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Laboratory Research

Influence of Conditioning Time of Universal Adhesives on Adhesive Properties and Enamel-Etching Pattern

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Clinical Relevance

The active and prolonged application of universal adhesives in the self-etch mode may be a viable alternative comparable to enamel etching with phosphoric acid.

SUMMARY

Objectives: To evaluate the effect of application protocol in resin–enamel microshear bond strength (μSBS), *in situ* degree of conversion, and etching pattern of three universal adhesive systems.

Methods and Materials: Sixty-three extracted third molars were sectioned in four parts (buccal, lingual, and proximals) and divided into nine groups, according to the combination of the main factors—Adhesive (Clearfil Universal, Kuraray Noritake Dental Inc, Tokyo, Japan; Futurabond U, VOCO, Cuxhaven, Germany; and Scotchbond Universal Adhesive, 3M ESPE, St Paul, MN, USA)—and enamel treatment/application time (etch-and-rinse mode [ER], self-etch [SE] application for 20 seconds [SE20], and SE application for 40 seconds [SE40]). Specimens were stored in water (37°C/24 h) and tested at 1.0 mm/min

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(μSBS). The degree of conversion of the adhesives at the resin–enamel interfaces was evaluated using micro-Raman spectroscopy. The enamel-etching pattern was evaluated under a scanning electron microscope. Data were analyzed with two-way analysis of variance and Tukey test (α=0.05).

Results: In general, the application of the universal adhesives in the SE40 produced μSBS and degree of conversion that were higher than in the SE20 ($p<0.01$) and similar to the ER mode. The deepest enamel-etching pattern was obtained in the ER mode, followed by the SE40.

Conclusions: The active and prolonged application of universal adhesives in the SE mode may be a viable alternative to increase the degree of conversion, etching pattern, and resin–enamel bond strength.

INTRODUCTION

The current adhesive systems can be classified based on the number of application steps or according to the adhesion strategies in etch-and-rinse (ER) and self-etch (SE).[1] Recently, a new group of dental adhesives has been introduced to the market as "universal" or "multimode" adhesives. They are essentially one-step SE adhesives that may be associated with phosphoric acid etching.[2,3] This versatile new adhesion philosophy advocates the use of the simplest option of each strategy, that is, one-step SE or two-step ER,[2-4] with the advantage that the dentist can decide which adhesive strategy to use for each specific clinical situation.

In spite of this, bonding to enamel is still a concern. Similar to what has been reported for one-step SE adhesive applied on enamel,[5,6] reduced bonding effectiveness is observed in enamel when universal adhesives are used in the SE mode.[7-9]

The most recognized technique for improving the bonding of one-step SE to enamel is to use selective enamel etching,[10] which led dental manufacturers and researchers to advocate selective enamel etching for one-step SE and for the majority of universal adhesives. A shortcoming of this technique is that accidental dentin etching may occur, particularly in small cavity preparations or when a low-viscosity etchant is used.[4,11]

Although prior phosphoric acid etching increases the immediate bond strength of universal adhesives to dentin when applied as ER for the majority of universal adhesives,[2-4,12,13] earlier signs of degradation are observed in the ER mode compared to the SE approach.[14,15] Additionally, some universal adhesives applied as SE showed stable bond strength and reduced the nanoleakage at the interface after six months of aging.[15]

To improve adhesion of one-step SE adhesives applied to enamel[16] without the use of phosphoric acid etching, some authors suggested increased application time or multiple adhesive layers. This approach increases the contact of acidic resin monomers with the enamel surface, creating a more retentive pattern. While some authors reported improved enamel bond strength using these aforementioned techniques, this is not consensual, probably because it seemed to be dependent on others factors, such as the pH of the adhesive tested as well as the application technique for each adhesive.[17-22]

Therefore, taking into consideration the recent launch of universal adhesives on the market, the aim of the present study was to compare the effect of different active application times on resin–enamel microshear bond strength (μSBS), *in situ* degree of conversion, and etching pattern of three universal adhesive systems. The following null hypotheses were tested: 1) the different active application times will not influence the μSBS of the different adhesives, 2) the different active application times will not influence the degree of conversion of the adhesives at the resin–enamel interfaces, and 3) the different active application times will not influence the enamel-etching pattern of the universal adhesives.

METHODS AND MATERIALS

Sixty-three extracted, caries-free human molars were used. The teeth were collected after obtaining the patients' informed consent under a protocol approved by the Ethics Committee Review Board of the local university. The teeth were disinfected in 0.5% chloramine, stored in distilled water, and used within six months of extraction.

The roots of all teeth were removed by sectioning at the enamel-cementum junction. Each dental crown was then sectioned in the diagonals across the long axis of the tooth to produce four enamel specimens (buccal, lingual, and proximals).[9] Two hundred and sixteen enamel specimens, which originated from 54 teeth, were ground wet with 180- and 600-grit SiC paper for 60 seconds each and used for evaluation of μSBS and *in situ* degree of conversion at the resin–enamel interfaces. The other 36 enamel specimens, which originated from

Table 1: *Adhesive System (Batch Number), Composition, and Application Mode of the Adhesive Systems According to the Manufacturers' Instructions*

Adhesive (Batch Number)	Composition[a]	Application Mode	
		Self-Etch[b]	Etch-and-Rinse
Clearfil Universal (01416)	1. Etchant: 35% phosphoric acid, colloidal silica, polyethyleneglycol, pigment, and water (K-ETCHANT) 2. Adhesive: HEMA, MDP, Bis-GMA, ethanol, camphorquinone, hydrophilic aliphatic dimethacrylate, silane coupling agent, colloidal silica, water, and accelerators	1. Apply bond and rub for 20 s[a] or 40 s 2. Dry by blowing mild air for 5 s 3. Light cure for 10 s at 1200 mW/cm^2	1. Apply etchant for 10 s 2. Rinse thoroughly 3. Dry 4. Apply adhesive as for the self-etch mode
Futurabond U (1346518)	1. Etchant: 34% phosphoric acid, water, synthetic amorphous silica, polyethylene glycol, aluminum oxide (Scotchbond Universal Etchant) 2. Adhesive: Liquid 1: Acidic adhesive monomer HEMA BISGMA, HEDMA, UDMA Catalyst Liquid 2: Ethanol initiator, catalyst	1. Apply the adhesive to the entire preparation with a microbrush and rub it for 20 s[a] or 40 s. If necessary, rewet the disposable applicator during treatment 2. Direct a gentle stream of air over the liquid for about 5 s until it no longer moves and the solvent is evaporated completely 3. Light cure for 10 s at 1200 mW/cm^2	1. Apply etchant for 15 s 2. Rinse for 10 s 3. Air dry 2 s 4. Apply adhesive as for the self-etch mode
Scotchbond Universal Adhesive (D-82229)	1. Etchant: 34% phosphoric acid, water, synthetic amorphous silica, polyethylene glycol, aluminum oxide (Scotchbond Universal Etchant) 2. Adhesive: MDP phosphate monomer, dimethacrylate resins, Bis-GMA, HEMA, methacrylate-modified polyalkenoic acid copolymer, camphorquinone, filler, ethanol, water, initiators, silane	1. Apply the adhesive to the entire preparation with a microbrush and rub it in for 20 s[a] or 40 s. If necessary, rewet the disposable applicator during treatment 2. Direct a gentle stream of air over the liquid for about 5 s until it no longer moves and the solvent is evaporated completely 3. Light cure for 10 s at 1200 mW/cm^2	1. Apply etchant for 15 s 2. Rinse for 10 s 3. Air dry 2 s 4. Apply adhesive as for the self-etch mode

Abbreviations: HEMA, 2-hydroxyethyl methacrylate; MDP, methacryloyloxydecyldihydrogen phosphate; Bis-GMA, bisphenolglycidyl methacrylate; UDMA, urethanedimethacrylate; HEDMA, hexamethylenedimethacrylate.
[a] *As per manufacturer's instructions.*
[b] *The intensity of the light-curing procedure was standardized for all materials.*

nine teeth, were not ground and used for the evaluation of the etching pattern produced on the enamel surface.

Experimental Design

The enamel specimens were randomly assigned into nine experimental conditions according to the combination of the independent-variable adhesive system—Clearfil Universal (Kuraray Noritake Dental Inc, Tokyo, Japan), Futurabond U (VOCO, Cuxhaven, Germany), and Scotchbond Universal Adhesive (3M ESPE, St Paul, MN, USA; also known as Single Bond Universal in some countries) (Table 1)—and enamel treatment/application time: ER mode, SE mode with application time of 20 seconds, and SE mode with application time of 40 seconds.

μSBS

Each enamel specimen was embedded in a polyvinyl chloride tube (10 mm high × 13 mm diameter) using a chemically cured acrylic resin (Jet Clássico, São Paulo, Brazil) in a way that the enamel surface was left exposed on the top of the cylinder. The delimitation of the bonding area was performed according to Shimaoka and others.[23] Six to eight perforations, with an internal diameter of 0.8 mm were made in an acid-resistant, double-faced adhesive tape (AdelbrasInd e Com Adesivos Ltda, São Paulo, Brazil) that was adapted to the enamel surface. This procedure was performed with the Hygienic Ainsworth–style rubber-dam punch (Coltene, Alstätten, Switzerland) and adapted to the enamel surface. The variation in the number of perforations for each enamel surface was dependent on the dimensions of the enamel specimens.

The randomization of the specimens for the μSBS was done using block randomization. A person not involved in the research protocol performed this procedure using computer-generated tables. The universal adhesives were applied to the enamel surface as described in Table 1. A single operator performed all bonding procedures according to the description below:

1) ER mode: The phosphoric acid gel of each adhesive system was applied and left undisturbed for the time recommended for each manufacturer. Then the surfaces were water rinsed with an air–water syringe for 10 seconds.
2) SE mode, 20-second application: Each adhesive was applied actively on the enamel for 20 seconds without phosphoric acid etching. The manual pressure exerted on the microbrush (Microbrush International, Grafton, WI, USA) during application was equivalent to 35 g.[24,25]
3) SE mode, 40-second application time: Each adhesive was applied actively on the enamel for 40 seconds without phosphoric acid etching. As reported earlier, the manual pressure exerted on the microbrush during application was equivalent to 35 g.

After the application of the adhesive system, polyethylene transparent Tygon tubes (Tygon Medical Tubing Formulations 54-HL, Saint Gobain Performance Plastics, Akron, OH, USA) with the same internal diameter of the perforations and a height of 0.5 mm were positioned on the perforations over the double-faced tape, ensuring that their lumen coincided with the circular areas exposed by the perforations. Resin composite (Filtek Z350, 3M ESPE) was carefully packed inside each tube, and a clear Mylar matrix strip was placed over the filled Tygon tube and pressed gently into place. The resin composite was light cured for 20 seconds using an LED light-curing unit set at 1200 mW/cm^2 (Radii-cal, SDI Limited, Bayswater, Victoria, Australia). A radiometer (Demetron L.E.D. Radiometer, Kerr Sybron Dental Specialties, Middleton, WI, USA) was used to check the light intensity every five specimens. These procedures were carried out under 10x magnifying loupes.

After storage of the specimens in distilled water for 24 hours at 37°C, the Tygon tubes and the double-faced adhesive tape were carefully removed with a blade, exposing the composite cylinders. Each specimen was examined under a stereomicroscope at 10× magnification. The bonded cylinder was discarded if there was evidence of porosities or gaps at the interface.[26]

The specimens were attached to a shear-testing fixture (Odeme Biotechnology, Joaçaba, SC, Brazil) and tested in a universal testing machine (Kratos IKCL 3-USB, Kratos Equipamentos Industriais Ltda, Cotia, São Paulo, Brazil). Each specimen was positioned onto the universal testing machine, and a thin wire (0.2-mm diameter) was looped around the base of each composite cylinder. The orthodontic wire contacted the composite resin cylinder in half of its circumference. The setup was kept aligned (resin–enamel interface, the wire loop, and the center of the load cell) to ensure the correct orientation of the shear forces.[27] The crosshead speed was set at 1 mm/min until failure.

The μSBS values (MPa) were calculated by dividing the load at failure by the surface area (mm^2). After testing, the specimens were examined in an optical microscope (SZH-131, Olympus Ltd, Tokyo, Japan) at 100× magnification to define the location of the bond failure. The type of failure was determined based on the percentage of substrate-free material as adhesive (A; failure at the resin–enamel interface), cohesive (C; failure exclusively within enamel or resin composite), and/or mixed (M; failure at the resin–enamel interface that included cohesive failure of the neighboring substrates).

In Situ Degree of Conversion

Four enamel specimens were randomly assigned for each group as described earlier for the μSBS. The adhesives were applied, and composite resin build-ups were constructed on the bonded enamel using the same materials and protocols described for the μSBS test. After storage of the restored teeth in distilled water at 37°C for 24 hours, the resin–enamel specimens were longitudinally sectioned across the bonded interface with a low-speed diamond saw (Isomet, Buehler Ltd, Lake Bluff, IL, USA) to obtain two resin–enamel slices.

The resin–enamel slices were wet polished with 1500-, 2000- and 2500-grit SiC paper for 15 seconds each. Then they were ultrasonically cleaned for 20 minutes in distilled water and stored in water for 24 hours at 37°C. The micro-Raman spectrometer (Bruker Optik GmbH, Ettlingen, Baden-Württemberg, Germany) was first calibrated for zero and then for coefficient values using the following micro-Raman parameters: 20-mW neon laser with 532-

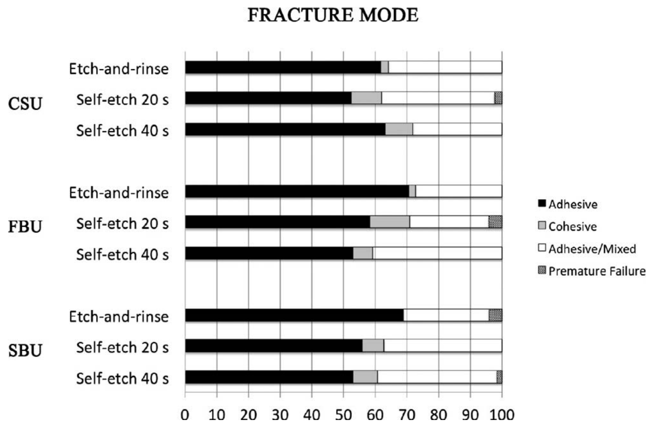

Figure 1. *Number of specimens (%) according to fracture mode for all experimental groups.*

nm wavelength, spatial resolution ≈3 μm, spectral resolution ≈5 cm^{-1}, accumulation time of 30 seconds with five coadditions, and magnification of 100× (Olympus UK, London, UK) using a ≈1-μm beam diameter.[28]

Spectra were taken at the resin–enamel adhesive interface at three different sites for each specimen. Spectra of uncured adhesives were taken as references. Post-processing of spectra was performed using the Opus Spectroscopy Software, version 6.5 (Bruker Optik). The ratio of double-bond content of monomer to polymer in the adhesive was calculated according to the following formula: Degree of conversion (%) = $(1 - R_{cured}/R_{uncured}) \times 100\%$, where R is the ratio of aliphatic and aromatic peak areas at 1639 cm^{-1} and 1609 cm^{-1} in cured and uncured adhesives, respectively.

Enamel-Etching Pattern

The enamel-etching pattern was evaluated on the enamel surface under a scanning electron microscope (JSM 5600LV, JEOL, Tokyo, Japan). For this purpose, the adhesives were applied in the SE strategy (20 and 40 seconds) according to the experimental conditions (Table 1), but the adhesives were not light cured. For the ER mode, the phosphoric acid gel was applied on enamel for 15 seconds, rinsed for 10 seconds, and air-dried according to each manufacturer's instructions (Table 1). Then the adhesives were applied without the light-curing step.

The enamel surfaces were then immediately stored in acetone for 24 hours to dissolve the resinous material from the enamel surface. Then the specimens were rinsed off in deionized water (5 minutes), 96% alcohol bath (5 minutes), and deionized water (5 minutes) in order to dissolve and remove the SE primer and the adhesive resins.[29]

All specimens were dried and dehydrated in a desiccator for 12 hours, and the conditioned enamel surfaces were sputter coated with gold/palladium in a vacuum evaporator (SCD 050, Balzers, Schaan, Liechtenstein). The entire surface of treated enamel was examined under a scanning electron microscope (JSM 5600LV, JEOL). Photomicrographs of representative surface areas were taken at 1000×, 1200×, and 5000× magnification.

Statistical Analysis

The resin–enamel μSBS values with adhesive/mixed failure mode and obtained from the same enamel specimen were averaged for statistical purposes. Similarly, the values of the degree of conversion from the same resin–enamel specimen were averaged so that the experimental unit in this experiment was the enamel specimen.

Specimens with cohesive and premature failures were not included in data analysis of the μSBS due to their low frequency in the experiment. Data from μSBS and degree of conversion were analyzed separately using two-way analysis of variance (adhesive vs. enamel treatment/application time) and Tukey *post hoc* test at a level of significance of 5%. The enamel-etching pattern was evaluated only qualitatively.

RESULTS

Microshear Bond Strength

The majority of the specimens (92.2% to 100%) showed adhesive/mixed failures (Figure 1). For all adhesives, the SE mode with an application time of 40 seconds yielded bond strength values that were statistically superior to that obtained in the SE mode for 20 seconds ($p<0.005$; Table 2). For Futurabond U and Scotchbond Universal, the SE modes for 40 seconds were statistically similar to the respective adhesives in the ER mode ($p>0.62$; Table 2). For Clearfil Universal, the application in the SE mode for 40 seconds produced adhesive interfaces with higher bond strength values than that obtained in the ER mode ($p<0.0002$; Table 2).

In Situ Degree of Conversion

For all adhesives, their application in the SE mode for 40 seconds produced a statistically higher degree of conversion than that obtained in the SE mode for 20 seconds ($p<0.003$; Table 3) and ER strategies ($p<0.00002$; Table 3). The lowest degree of conver-

Table 2: *Means and Standard Deviations of the Microshear Bond Strength (MPa) Values of the Different Experimental Groups*

Adhesives	Application Mode		
	Etch-and-Rinse	Self-Etch (20 s)	Self-Etch (40 s)
Clearfil Universal	19.1 ± 1.8 BC[a]	20.1 ± 1.2 B	23.1 ± 2.0 A
Futurabond Universal	17.1 ± 2.0 C	15.9 ± 1.3 D	18.2 ± 1.1 C
Scotchbond Universal	19.3 ± 1.7 BC	16.9 ± 1.1 D	20.4 ± 1.5 B

[a] *Similar letters indicate groups that are statistically similar (analysis of variance, Tukey test;* $p>0.05$*).*

Table 3: *Means and Standard Deviations of the Degree of Conversion (%) Values for Each Experimental Condition*

Adhesives	Application Mode		
	Etch-and-Rinse	Self-Etch (20 s)	Self-Etch (40 s)
Clearfil Universal	67.3 ± 2.6 D[a]	83.2 ± 3.8 B	92.3 ± 3.5 A
Futurabond Universal	60.2 ± 3.3 E	76.2 ± 2.7 C	93.5 ± 4.2 A
Scotchbond Universal	73.1 ± 4.3 C	76.0 ± 2.7 C	89.9 ± 4.6 A

[a] *Similar letters indicate groups that are statistically similar (analysis of variance, Tukey test;* $p>0.05$*).*

sion was obtained in the ER protocol, except for Scotchbond Universal, in which the degree of conversion in the SE mode for 20 seconds was statistically similar to the ER condition (p=0.45; Table 3). For the other two adhesives (Clearfil Universal and Futurabond U), the degree of conversion in the SE mode for 20 seconds was superior to the degree of conversion measured in the ER protocol (p<0.002; Table 3).

Enamel-Etching Pattern

The ER strategy resulted in the deepest and most pronounced etching pattern, mainly when compared with the SE mode applied for 20 seconds (Figure 2). In the SE mode applied for 20 seconds, all universal adhesives resulted in featureless morphology in which the superficial enamel layer was removed without exposure of the subsurface enamel. The application for 40 seconds in the SE mode improved the etching pattern, resulting in exposure of the periphery of the prisms, with signs of hydroxyapatite dissolution (Figure 2).

DISCUSSION

The results of this study confirmed previous findings[5,6,30,31] that adhesives applied in the SE mode for shorter application times resulted in reduced resin–enamel bond strength values compared to the ER protocol, which led us to reject the first null hypothesis. This means that even for universal adhesives, bonding to enamel is still a concern for clinicians when they are used in the SE mode.

Acidic monomers presented in the SE composition partially dissolve the smear layer and etch the enamel, and their effect is more pronounced when applied on ground enamel. But their acidity is not sufficient to produce retentive etching patterns equivalent to those produced by 35% phosphoric acid.[30-33] Several studies have shown a shallow intercrystallite infiltration of the monomers and a slight formation of interprismatic resin tags,[30,31] indicating a very superficial interaction of the SE adhesives, applied for shorter periods, with the enamel surface. This may be the reason why reduced resin–enamel μSBS was usually reported for SE adhesives.[32,34]

The universal adhesive systems evaluated in the present study have variable pH values around 2.5. The adhesives Clearfil Universal and Futurabond Universal have pH values of approximately 2.3, while Scotchbond Universal has a pH of 2.7.[4,9,35] Within this pH range, SE adhesives applied for only 20 seconds do not etch enamel as effectively as phosphoric acid.[6,9,30,31,36,37]

On the other side, Clearfil Universal and Futurabond U adhesives showed a higher degree of conversion when applied as SE for 20 seconds in comparison with ER, as was previously shown by Loguercio and others.[9] This indicates the rejection of the second null hypothesis.

Taking into consideration that, in the ER strategy, a double conditioning (phosphoric acid + acidic monomers from SE adhesives) occurred, these authors speculated that as Clearfil Universal and Futurabond U are more acidic when compared to Scotchbond Universal, the lower pH potentiates the demineralization mechanism and improves the superficial interaction.[6,32,36,38,39] Additionally, the uneven topography of etched enamel surfaces may increase the entrapment of solvents and air into the deeper prismatic and interprismatic portions of the etched enamel, leading to a lower *in situ* degree of conversion.[40]

The present study showed that the prolonged application of the adhesives in the SE mode might compensate for the higher pH of the universal adhesives. Improved enamel-etching pattern and

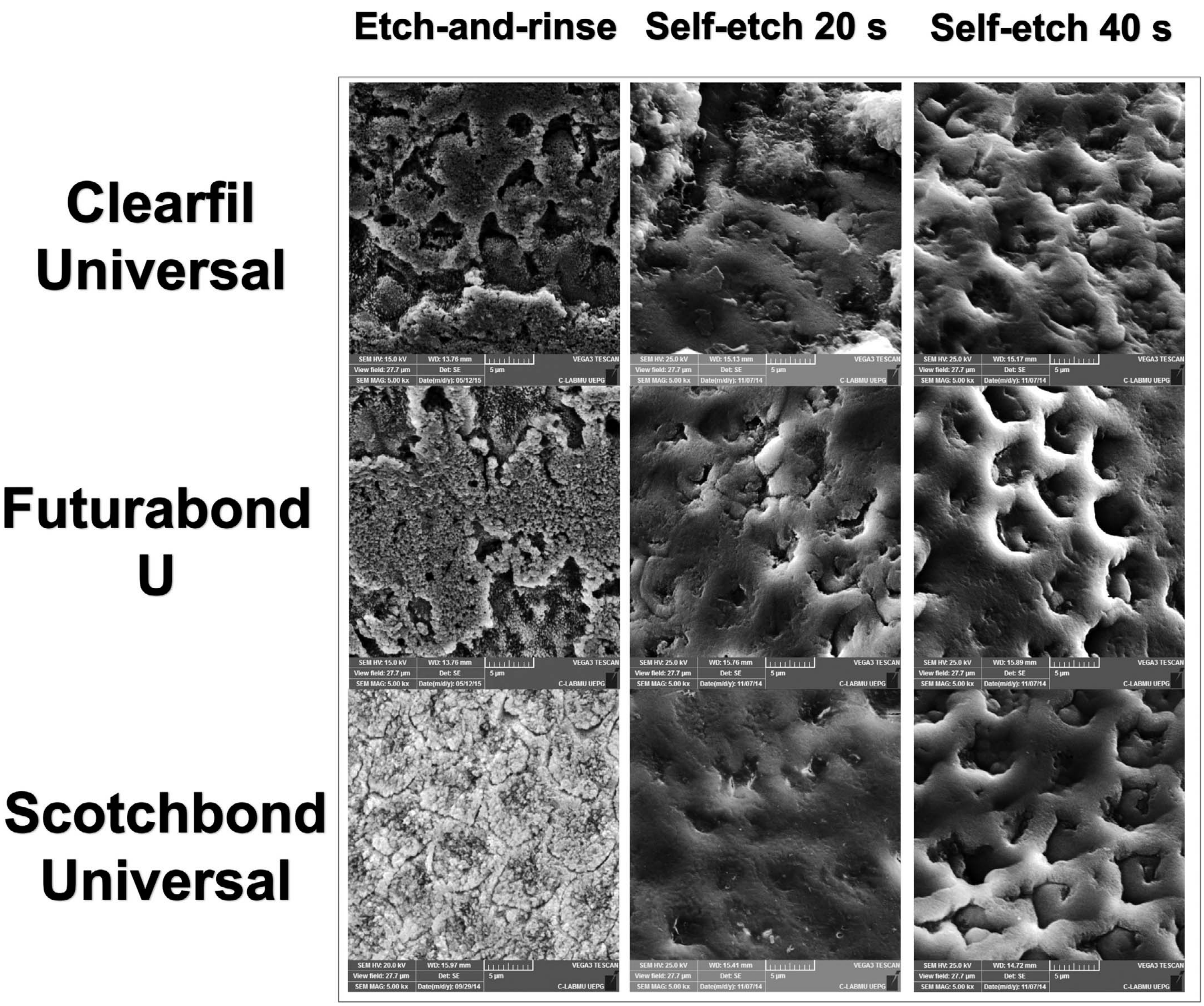

Figure 2. *Representative morphology of enamel treated with different applications protocol with the three universal adhesives used in this study. The etch-and-rinse group resulted in the best-defined etching pattern for all adhesives. Observe that all adhesive showed a more eroded pattern in comparison with Scotchbond Universal. A 20-second application showed signs of mild etching effect, with few islands without evidence of acid conditioning. A 40-second application of adhesives showed a better etching pattern with signs of interprismatic conditioning.*

increased resin–enamel bond strength values were observed when the universal adhesives were applied for 40 seconds instead of the conventional 20 seconds. This may be the result of improved diffusion and interaction of acidic resinous monomers with the underlying enamel, increasing not only the potential of etching but also resin impregnation into the underlying enamel.[17,22]

Although the etching pattern achieved with the SE mode for 40 seconds was still not as retentive as that produced in the ER mode, the ultramorphological images of the present investigation support the findings that a deeper demineralization occurred with the universal adhesives when they were used in the SE mode for 40 seconds, leading us to reject the third null hypothesis. It should be mentioned that a more retentive etching pattern could have been achieved had we removed the outer enamel layer before adhesive application.[30,31]

Apart from that, the literature findings are not consensual on whether increased application times produce higher resin–enamel bond strength values.[17-22] Several methodological differences could explain these differences. For example, in two studies where similar results were obtained, it is not clear whether the adhesives were applied passively or actively.[19,21]

In the present experimental study, the good results associated with the increased application time in the SE mode cannot be dissociated from the benefits of the active application method. In all groups, the adhesives were applied actively, as this method was shown to improve enamel demineralization and interaction of resin monomers with prismatic and interprismatic areas by carrying fresh resin monomers to the deeper enamel layers during active application.[9,18,37,41-43]

Additionally, active and prolonged application of universal adhesives in the SE mode may increase solvent diffusion outward, mainly for adhesives composed of solvents with low vapor pressure, such as water.[33] This solvent evaporation may allow room for changes in polymer topology by reducing in the intrinsic fraction of nanopores, allowing increased cross-linking and improved mechanical properties of the polymer inside the enamel hybrid layer.[44,45] This may be an explanation of why the highest degree of conversion was obtained when adhesives were applied in the SE mode for 40 seconds.

In short, the prolonged and active application of the universal adhesives in the SE mode improved the etching pattern of the enamel surface and produced a higher degree of conversion and higher resin–enamel bond strengths. Future clinical studies should be conducted to evaluate if this protocol may increase the retention rates and the quality of the margins of composite resin restorations placed in cervical lesions without the need of selective phosphoric acid etching of the enamel margins.

Due to the difficulty of SE adhesives bonding to enamel, several manufacturers have been recommending selective enamel etching, although no significant difference exists in the retention rate of composite resin restorations in non-carious cervical lesions when selective enamel etching is compared with SE adhesives without selective enamel etching.[46-50] The only benefit of selective enamel etching with SE adhesives is minor reduction of marginal discoloration at the restoration interface.[46,47]

On the other hand, at least one clinical study showed that higher retention rates after 18 months were associated with the prolonged time of active SE application in noncarious cervical lesions, but, in this specific study, the increase of application time was associated with more coats of the SE adhesive. In addition, the mentioned adhesive is no longer available for use.[51] Further clinical trials should be conducted to validate the results obtained with the prolonged SE application of universal adhesives in this laboratory study, mainly because the prolonged and active application of SE adhesives makes the application procedure easier when compared to selective enamel etching, mainly because the former omitted the use of phosphoric acid.[52]

CONCLUSIONS

In summary, the active and prolonged application of universal adhesives in the SE mode may be a viable alternative to increase the degree of conversion, etching pattern, and resin–enamel bond strength.

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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: COEP/UEPG. The approval code for this study is: 0123/2009.

Conflict of Interest

The authors 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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