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Durability of Enamel Bonding Using Two-step Self-etch Systems on Ground and Unground Enamel

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Clinical Relevance

The effect of surface preparation was adhesive-dependent. Improvements in resin-enamel bond strength after enamel preparation were observed only for AdheSE and Optibond Solo plus Self-Etch Primer. Among the self-etch systems, mild, self-etch Clearfil SE Bond showed the highest bond strength values. No degradation of resin-enamel bonds was observed after 12 months of water storage, regardless of the adhesive tested.

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SUMMARY

This study examined the early and long-term microtensile bond strengths (µTBS) and interfacial enamel gap formation (IGW) of two-step self-etch systems to unground and ground enamel. Resin composite (Filtek Z250) buildups were bonded to proximal enamel surfaces (unground, bur-cut or SiC-treated enamel) of third molars after the application of four self-etch adhesives: a mild (Clearfil SE Bond [SE]), two moderate (Optibond Solo Plus Self-Etch Primer [SO] and AdheSE [AD]) and a strong adhesive (Tyrian Self Priming Etchant + One Step Plus [TY]) and two etch-and-rinse adhesive systems (Single Bond [SB] and Scotchbond Multi-Purpose Plus [SBMP]). Ten tooth halves were assigned for each adhesive. After storage in water (24 hours/37°C), the bonded specimens were sectioned into beams (0.9 mm²) and subjected to µTBS (0.5 mm/minute) or interfacial gap width measurement (stereomicroscope at 400x) either immediately (IM) or after 12 months (12M) of water storage. The data were analyzed by three-way repeated measures

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ANOVA and Tukey's test (α=0.05). No gap formation was observed in any experimental condition. The μTBS in the Si-C paper and diamond bur groups were similar and greater than the unground group only for the moderate self-etch systems (SO and AD). No reductions in bond strength values were observed after 12 months of water storage, regardless of the adhesive evaluated.

INTRODUCTION

Enamel phosphoric acid etching associated with etch-and-rinse adhesives is considered the most durable and predictable method of bonding esthetic materials to tooth structure.[1] In an effort to simplify the dentin/enamel bonding systems, the self-etch bonding approach was proposed. These systems eliminated the separate step of acid etching, turning these materials user-friendly and popular among clinicians.[2]

However, as the etching pattern of self-etch adhesives is not as well defined as that provided by phosphoric acid,[3-5] several studies have attempted to make the enamel substrate more receptive to bonding via abrasion, similar to what is done during a bevel or cavity preparation. Promising results were reported by some authors,[3-4,6-7] while others did not reach the same conclusions.[8-9] Differences in the way the enamel was prepared and material-related factors, such acidity,[9-10] can explain the lack of consensus among investigators and deserves further investigation.

Although much information is available in the literature regarding long-term resin-dentin bonds,[11-13] few studies have evaluated the durability of resin-enamel interfaces bonded with etch-and-rinse and self-etch adhesives.[14] The high retention rates achieved by sealants over time, when applied over phosphoric acid-treated enamel[15-16] provide evidence of the long-term durability of the etch-and-rinse approach to enamel bonding. However, sealants are composed of solvent-free hydrophobic monomers, and they are not as hydrophilic as two-step etch-and-rinse adhesives and do not contain acidic monomers similar to the ones employed in self-etch systems. In addition, the high water/solvent concentration in these simplified adhesive systems precludes the formation of a high cross-linked polymer[17-19] within the enamel porosities created by phosphoric acid or acidic monomers, which makes these materials more sensitive to water sorption and compromises their bonding effectiveness over time.

Therefore, this study evaluated the early and long-term effectiveness of resin-enamel bonds by means of microtensile testing and gap width measurements for etch-and-rinse and self-etch adhesives on unground, bur and silicon carbide paper-treated enamel. This study tested three null hypotheses: 1) enamel surface pre-treatment does not affect the bonding effectiveness of etch-and-rinse and self-etch systems to enamel; 2) all the adhesive systems tested can achieve similar bond strengths to enamel, regardless of their bonding approach; 3) the storage period does not affect the bonding effectiveness of etch-and-rinse and self-etch adhesives to enamel.

METHODS AND MATERIALS

This study was approved by the Institutional Review Board, Dental School, Unoesc, Brazil under protocol #208/03. Ninety extracted third human molars were used in this study.

Teeth Preparation and Restorative Procedures

All the teeth were sectioned in the mesio-to-distal direction in order to obtain tooth halves. The buccal and lingual surfaces were cleaned with slurry of pumice and water and examined under a 40x stereomicroscope (HMV-2, Shimadzu, Tokyo, Japan) to ensure that they were free of surface cracks, decalcification or any sign of previous grinding. The enamel surface was then demarcated to outline the flattest area for bonding. The occlusal third of the buccal and lingual surfaces was usually outside the bonding area due to its inclination. The teeth were then randomly divided into three groups according to the type of enamel surface preparation (Figure 1):

Group 1: No enamel preparation was performed before adhesive application, except for teeth prophylaxis with pumice slurry.

Group 2: After teeth prophylaxis, a wheel medium-grit diamond bur (#4142, particle size ca 100 µm, KG Sorensen, Barueri, SP, Brazil) was applied to the surface using a high-speed handpiece with water coolant. This procedure created 0.5 mm deep grooves on the surface. The surface was then flattened with the tapered, rounded end of a fine-grit diamond bur (#4138, particle size ca 46 µm, KG Sorensen, Barueri, SP, Brazil).

Group 3: After teeth prophylaxis, the enamel surfaces were manually ground with 60-grit silicon carbide paper under water-cooling for 60 seconds in order to flatten the enamel area for bonding.

Each group was further subdivided into six subgroups according to the adhesive used (Figure 1). Four two-step self-etch adhesives were selected based on the acidity of their adhesive solutions: Clearfil SE Bond (Kuraray Medical Inc, Tokyo, Japan), as a mild self-etch adhesive (pH≅2); OptiBond Solo Plus Self-Etch Primer (Kerr Co, Orange, CA, USA) and AdheSE (Ivoclar Vivadent, Schaan, Liechtenstein, Germany) as two intermediate, strong self-etch adhesives (pH≅1.5) and Tyrian Self Priming Etchant (BISCO Inc, Schaumburg, IL, USA) as an acidic self-etch system (pH<1). Two etch-and-rinse adhesives were also evalu-

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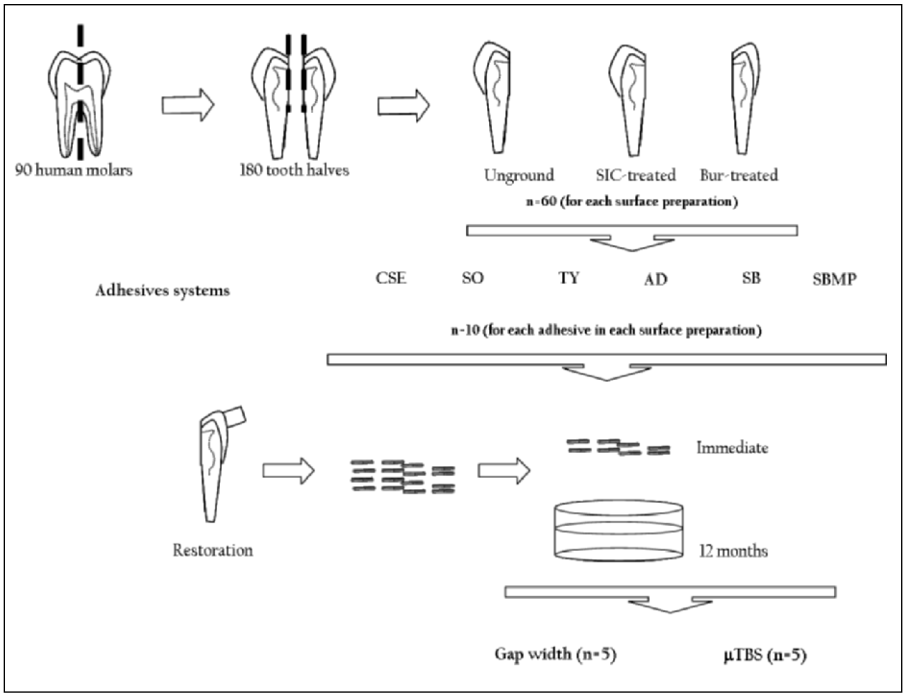

Figure 1. *Experimental design.*

ated: Single Bond and Scotchbond Multi-Purpose Plus (3M ESPE, St Paul, MN, USA) as two- and three-step adhesives, respectively. A single operator applied all the adhesives in a controlled environment (24°C/75% relative humidity) using the bonding protocols summarized in Table 1.

Special care was taken to ensure that the enamel surfaces were adequately covered by monomers after evaporation of the solvents. In the event that matte enamel was encountered, an additional coat of adhesive was applied to produce shiny surfaces prior to light curing with a VIP unit (600 mW/cm^2, BISCO Inc). Bonded buccal and lingual enamel surfaces were coupled with a hybrid composite (Filtek Z250, 3M ESPE) that was light activated in three 1-mm thick increments. Half of the sample were used for microtensile testing (n=5 tooth halves) and the other half (n=5 tooth halves) were used for gap width measurement.

Storage and Specimen Preparation

After storage in distilled water at 37°C for 24 hours, the tooth halves were sectioned perpendicular to the adhesive-tooth interface using a Labcut diamond saw (Extec, Enfield, CT, USA) to obtain rectangular beams (0.9 mm^2). The beams were prepared with the resin composite forming the upper half of the beam and the underlying enamel and dentin forming the lower half. The bonded beams that originated from the same teeth were randomly divided into two groups to be tested immediately [IM] or after 12 months [12M] of storage in distilled water containing 0.5 % chloramine[14] at 37°C (Figure 1). The storage solution was not changed over time.[20]

Microtensile Bond Strength

For microtensile bond strength testing, the number of prematurely debonded beams [PD], those that did not survive the slicing procedures, was recorded. Each beam was examined in a stereomicroscope (100x; HMV-2, Shimadzu, Tokyo, Japan) to check the inclination of the bonding interfaces on the four sides of each stick. Sticks with visually detected failures in the bonded interface ("visually" detected gaps or resin/enamel cracks) were not tested in tension. The cross-sectional area of each stick was measured with a digital caliper to the nearest 0.01 mm and recorded for calculation of the bond strength values (Absolute Digimatic, Mitutoyo, Tokyo, Japan).

The beams from each adhesive group were stressed to failure using a universal testing machine (Emic, São José dos Pinhais, PR, Brazil) at a crosshead speed of 0.5 mm/minute. The bond failure modes were evaluated at 400x under a light stereomicroscope (HMV-2, Shimadzu, Tokyo, Japan) and classified as cohesive (failure exclusively within enamel or resin composite) or adhesive/mixed (failure at resin/enamel interface or mixed with cohesive failure of the neighboring substrates).

Gap Width Measurement

The gap width was measured by means of a light microscope (Shimadzu HMV-2, Tokyo, Japan) at 400x magnification. The measurement of the mean gap width of each stick was performed in different rectangular sections having approximately similar geometric structure. The area of these sections was calculated based on their width and length.[21] The sum of all stick sections, divided by the total length of the interface, resulted in the mean stick gap width. The mean gap width of the individual tooth sections was than calculated.

Statistical Analysis

The bond strength and gap width values (at their respective storage periods) that originated from the same tooth were averaged for statistical purposes. For microtensile bond strength analysis, the premature debonded specimens were included in the tooth half mean. 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.[22] The microtensile bond strength and gap

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

Adhesive Systems	Composition	Application Mode	Batch #
Clearfil SE Bond (Kuraray)	1. Primer—water, MDP, HEMA, camphoroquinone, hydrophilic dimethacrylate 2. Adhesive—MDP, Bis-GMA, HEMA, camphoroquinone, hydrophobic dimethacrylate, N,N-diethanol p-toluidine bond, colloidal silica	1. Appy two coats of the primer with slight agitation (20 seconds) 2. Air-dry (10 seconds at 20 cm) 3. Appy one coat of the adhesive (15 seconds) 4. Air-dry (10 seconds at 20 cm) 5. Light-activate (10 seconds—600 mW/cm)	00176A 001185A
Optibond Solo Self-EtchPrimer and Optibond Solo Plus (Kerr)	1. Primer–alkyl dimethacrylate resins, barium aluminoborosilicate glass, fumed silica (silicon dioxide), sodium hexafluorosilicate and ethyl alcohol 2. Adhesive—alkyl dimethacrylate resins (25-28%), ethyl alcohol, water, stabilizers and activators	1. Apply one coat of the primer with slight agitation (15 seconds) 2. Air-dry (10 seconds at a distance of 20 cm) 3. Apply one coat of the adhesive (15 seconds with slight agitation) 4. Air-dry (10 seconds at 20 cm) 5. Apply one coat of the adhesive (15 seconds with slight agitation) 6. Air-dry (10 seconds at 20 cm) 7. Light-activate (20 seconds–600 mW/cm)	205187 203D20
AdheSE (Ivoclar Vivadent)	1. Primer–dimethacrylate, phosphonic acid acrylate, initiators, stabilizers, water 2. Adhesive–HEMA, BisGMA, GDMA, silicon dioxide, initiators, stabilizers	1. Apply one coat with slight agitation (15 seconds). The primer is then left undisturbed (for 15 seconds) 2. Air-dry (10 seconds at 20 cm) until the mobile liquid film disappears 3. Apply one coat of the adhesive without pooling 4. Light-activate (10 seconds–600 mW/cm)	G03221 G02780
Tyrian SPE and One Step Plus (BISCO)	3. Primer–2-Acrylamido-2-methyl propanesulfonic acid (2-15%); Bis-GMA; Ethanol (25-50%) 4. Adhesive—Bis-GMA, BPDM, HEMA, Glass Frit initiator and acetone (40%-70%)	1. Mix Tyrian SPE (A and B) and apply two coats with slight agitation (10 seconds) 2. Air-dry (10 seconds at 20 cm) 3. Apply two consecutive coats of the adhesive, brushing (for 10 seconds each) 4. Air-dry (10 seconds at 20 cm) 5. Light-activate (10 seconds–600 mW/cm)	200002694 200004295
Single Bond (3M ESPE)	1. 37% phosphoric acid 2. Adhesive–Bis-GMA, HEMA, dimethacrylates, polyalknoic acid copolymer, initiators, water and ethanol	1. Acid etch (15 seconds), rinse (15 seconds) and air-dry (10 seconds) kept dentin moist 2. Apply one coat of the adhesive (10 seconds with slight agitation) 3. Air-dry (10 seconds at 20 cm) 4. Apply one coat of the adhesive (10 seconds with slight agitation) 5. Air-dry (10 seconds at 20 cm) 6. Light-activate (10 seconds–600 mW/cm^2)	2GM
ScotchBond Multi-Purpose Plus (3M ESPE)	1. 37% phosphoric acid 2. Primer—aqueous solution of HEMA, polyalkenoic acid copolymer (Vitrebond) 3. Adhesive—Bis-GMA, HEMA, dimethacrylates and initiators	1. Acid etch (15 seconds), rinse (15 seconds) and air-dry (10 seconds) kept dentin moist 2. Apply two coats of the primer (10 seconds with slight agitation) 3. Air-dry (10 seconds at 20 cm) 4. Apply one coat of the adhesive (10 seconds with slight agitation) 5. Air-dry (10 seconds at 20 cm) 6. Light-activate (10 seconds–600 mW/cm^2)	3008 7543

Abbreviations: MDP (10-methacryloyloxydecyl dihydrogen phosphate); HEMA (2-hydroxyethyl methacrylate); Bis-GMA (bisphenol-glycidyl methacrylate); BPDM (biphenyl dimethacrylate); GDMA (glycidyl dimethacrylate).

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Table 2: *Overall resin-enamel bond strength means and standard deviations (MPa) from the different adhesive systems under the experimental conditions of the present investigation.*

Adhesive Systems	**Unground**		**SiC Paper**		**Diamond Bur**	
	IM	**12M**	**IM**	**12M**	**IM**	**12M**
Clearfil SE Bond	18.7 ± 4.6	17.2 ± 4.4	22.7 ± 1.8	18.3 ± 2.9	19.9 ± 4.1	18.2 ± 4.0
AdheSE	14.5 ± 2.1	9.5 ± 2.8	16.5 ± 2.0	15.3 ± 2.4	16.9 ± 5.3	13.1 ± 3.5
Optibond Solo Self-etch Primer and Optibond Solo Plus	8.2 ± 1.2	9.9 ± 2.6	12.7 ± 2.5	9.6 ± 2.1	13.3 ± 3.0	12.5 ± 2.9
Tyrian SPE and One Step Plus	8.9 ± 1.6	10.1 ± 1.9	12.8 ± 2.0	7.1 ± 1.0	9.9 ± 3.8	8.0 ± 2.3
Single Bond	23.4 ± 1.9	18.7 ± 2.4	24.3 ± 3.4	19.2 ± 2.8	25.3 ± 4.2	19.5 ± 4.9
ScotchBond Multi Purpose Plus	24.9 ± 4.5	24.8 ± 5.1	23.8 ± 3.7	19.9 ± 3.1	20.4 ± 4.9	17.7 ± 7.8

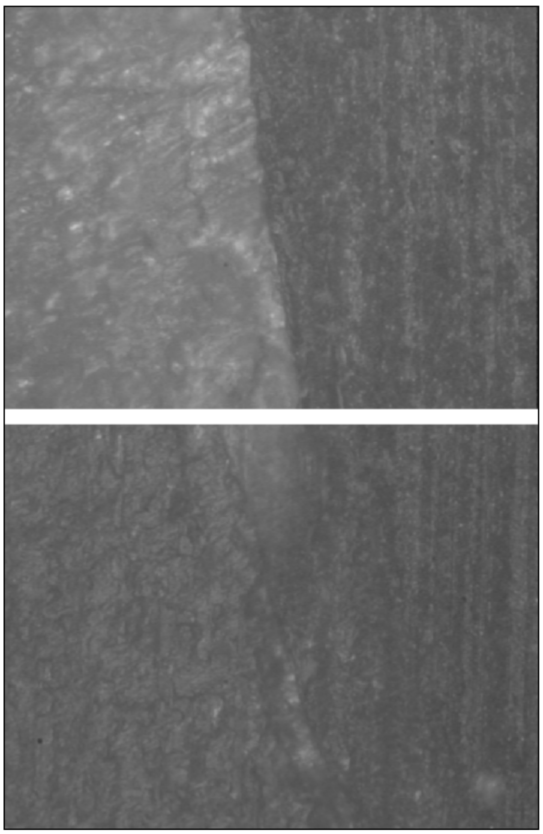

Figure 2. *Adhesive interface of Clearfil SE Bond and enamel after diamond bur preparation. The upper figure illustrates the bonded interface in the immediate group; the lower figure illustrates the bonded interface after 12 months. Note that there is no gap formation in the bonded interface. Original magnification: 400x.*

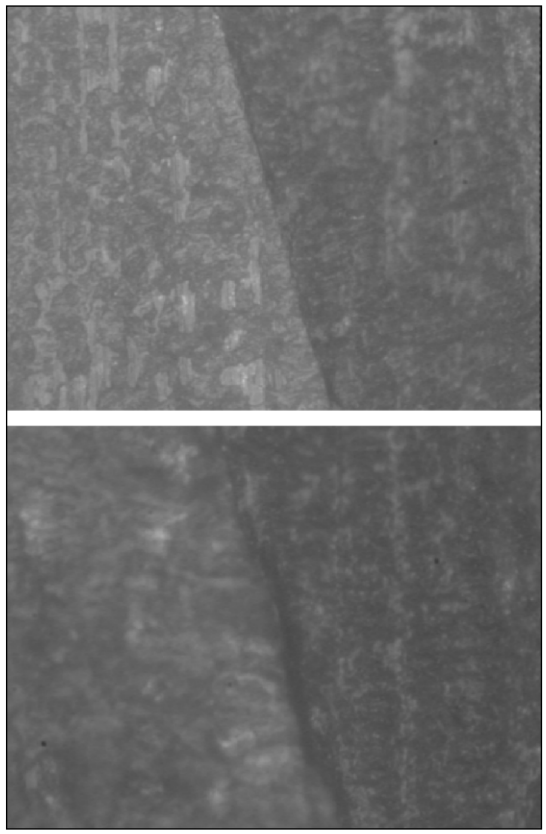

Figure 3. *Adhesive interface of Single Bond and enamel after SiC paper preparation. The upper figure illustrates the bonded interface in the immediate group, while the lower figure shows the bonded interface after 12 months. Note that there is no gap formation in the bonded interface. Original magnification: 400x.*

width means were subjected to a three-way repeated measures analysis of variance (Adhesive vs Substrate treatment vs Storage period) and Tukey's test for pairwise comparisons (α=0.05). The storage period was the repeated factor.

RESULTS

Microtensile Bond Strengths

The mean cross-sectional area was 0.73 ± 0.2 mm^2, with no difference detected among the treatment groups (p>0.05). The bond strength value assigned to premature debonded specimens was 3.5 MPa, which corresponded to half of the minimum bond strength value measured in this study.

The overall means and standard deviations (MPa) of the bond strength values are shown in Table 2. The number of beams tested and the number of premature debonded specimens is shown in Table 3. No cohesive failure in enamel or resin composite was observed in this study. The results from the three-way ANOVA revealed that the main factor Adhesive (p<0.00001), the interaction of Adhesive vs Storage period (p=0.0001) and the interaction of Adhesive vs Surface Preparation (p=0.037) were statistically significant.

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Table 3: *Number of Tested vs Premature Debonded Beams for Each Experimental Condition*

Adhesive Systems	Unground		SiC Paper		Diamond Bur	
	IM	12M	IM	12M	IM	12M
Clearfil SE Bond	14/05	15/04	13/00	12/02	11/02	14/02
AdheSE	12/03	16/02	13/04	13/01	11/03	15/04
Optibond Solo Self-etch Primer and Optibond Solo Plus	12/03	12/01	11/00	12/02	13/03	14/01
Tyrian SPE and One Step Plus	12/03	16/02	13/04	13/01	11/03	15/04
Single Bond	12/03	12/01	11/00	12/02	13/03	14/01
ScotchBond Multi Purpose Plus	14/04	11/04	12/02	09/02	12/04	16/01

Table 4: *Resin-enamel bond strength means, standard deviations (MPa) and statistical analysis for the interaction of Adhesive vs Surface Preparation.*

Adhesive Systems	Surface Preparation		
	Unground	SiC Paper	Diamond Bur
Clearfil SE Bond	20.1 ± 2.7[b]	20.6 ± 2.0[b]	20.0 ± 3.1[b]
AdheSE	11.2 ± 3.2[d]	16.1 ± 3.1[c]	14.7 ± 3.0[c]
Optibond Solo Self-etch Primer and Optibond Solo Plus	9.7 ± 2.3[d]	10.7 ± 2.7[d]	13.4 ± 2.4[c]
Tyrian SPE and One Step Plus	10.6 ± 1.6[d]	10.6 ± 1.8[d]	9.3 ± 2.1[d]
Single Bond	21.5 ± 1.8[b]	21.7 ± 2.3[b]	21.1 ± 2.6[b]
ScotchBond Multi Purpose Plus	24.3 ± 3.7[a,b]	22.5 ± 3.4[b]	20.0 ± 3.7[b]

Means with the same superscripted letters are statistically similar (p>0.05).

Table 5: *Resin-enamel bond strength means, standard deviations (MPa) and statistical analysis for the interaction Adhesive Systems vs Storage Period.*

Adhesive Systems	Storage Period		
	Immediate	6 Months	12 Months
Clearfil SE Bond	20.5 ± 3.6[b]	21.4 ± 3.4[b]	18.9 ± 2.7[b]
AdheSE	16.0 ± 3.1[c]	13.9 ± 5.0[c]	12.6 ± 2.9[c,d]
Optibond Solo Self-etch Primer and Optibond Solo Plus	11.4 ± 2.2[d]	11.7 ± 2.3[d]	10.7 ± 2.5[d]
Tyrian SPE and One Step Plus	10.5 ± 2.5[d]	11.6 ± 1.9[d]	8.4 ± 1.8[d,e]
Single Bond	24.3 ± 3.1[a,b]	20.8 ± 2.0[b]	19.1 ± 3.4[b]
ScotchBond Multi Purpose Plus	23.1 ± 4.4[a,b]	21.7 ± 1.8[b]	21.9 ± 3.8[b]

Means with the same superscripted letters are statistically similar (p>0.05).

Table 4 depicts the bond strength values for the interaction of Adhesive vs Surface Preparation. Significantly higher resin-enamel bond strength means were observed for the adhesives AdheSE and Optibond Solo Plus SE when the enamel was previously abraded. Only for AdheSE was the diamond-bur treatment effective, while both enamel pre-treatments (SiC paper and diamond bur) increased the bond strength of the Optibond Solo Plus SE system. All other adhesives showed similar resin-enamel bond strengths under the three conditions of enamel treatment.

Table 5 describes bond strength values for the interaction of Adhesive vs Storage Period. No significant difference was observed among the different storage periods for each adhesive system.

Gap Width Measurements

No gap formation was found in the early or long-term storage for any of the adhesives tested. Representative resin-enamel interfaces can be seen in Figures 2 and 3.

DISCUSSION

The etching pattern of self-etch adhesives is not as well defined as that of etch-and-rinse adhesives.[3-5] Some studies have demonstrated that abrading the enamel during a bevel or cavity preparation results in the substrate being more receptive to bonding with two-step self-etch systems.[3-4,6-7] However, this is not consensual in the literature, since other researchers have not detected any difference in the self-etch systems' performance when these materials were applied on ground and unground enamel.[8-9]

Improvements in resin-enamel bond strength after enamel preparation were only detected for two of the four self-etch adhesive systems tested. This led the

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authors of the current study to reject the first null hypothesis. This finding indicates that the benefits of enamel preparation are material-dependent, as demonstrated by Perdigão and Geraldelli[3] and Perdigão and others.[23]

Mild self-etch Clearfil SE Bond showed the highest bond strength values, while the most acidic Tyrian Self Priming Etchant + One Step Plus showed the lowest values, despite the deeper, more retentive etching pattern created by the latter.[5,24] This led the authors of the current study to reject the second null hypothesis. This finding is consistent with previous literature findings, since mild Clearfil SE Bond usually performs better than acidic Tyrian Self Priming Etchant + One Step Plus.[5,24]

One can speculate that the poorer performance of self-etch systems on enamel[6,24-25] cannot be solely attributed to the presence of a less reactive superficial enamel layer or the acidity of the self-etch solution. Other features, individual for each self-etch system, can be responsible for such differences. For instance, variations in adhesive viscosity, surface tension, chemical interaction of acidic monomers with enamel,[26] water concentration[24] and cohesive strength of the adhesives[10,13,28] are important features to be considered.

Most of the laboratory studies that evaluated strong acidic one-step self-etch systems under a microtensile bond strength approach demonstrated relatively lower bond strength values for these materials despite the more retentive etching pattern achieved by them.[5,25,29] Some hypotheses can explain the lower performance of acidic self-etch systems as follows: 1) the amount of solvent presented in the self-etch primer and 2) the hydrophilic nature of the adhesive resin placed over the self-etch primer.[13,30] In addition, Tyrian SPE + One Step Plus also lacks monomers, which are capable of promoting chemical adhesion to dental substrate. Clearfil SE Bond, a mild two-step self-etch system with successful *in vitro*[24-25,31] and *in vivo* investigations[32-33] has 10-methacryloxydecyl dihydrogen phosphate (10-MDP) as an acidic monomer, which has bonding potential to the calcium of residual hydroxyapatite.[34-35] This fact justifies the good performance of Clearfil SE Bond in the current and other *in vitro* studies[24,31] and turns this material into the "gold standard" against which other self-etch systems should be compared.[1]

AdheSE and OptiBond Solo Plus Self-Etch Primer cannot be classified as "mild" or "strong" two-step self-etch adhesives. Based on their interaction to dentin, these systems have been referred to as "intermediary strong" or moderate two-step self-etch adhesives.[1] The pH of the aforementioned materials is lower than that of Clearfil SE Bond and, therefore, these materials are capable of providing a more defined enamel etching pattern.[4-5] However, these materials do not contain monomers capable of chemically interacting with tooth substrates. The retention provided by these materials is solely based on micromechanical interlocking, which makes them more sensitive to enamel preparation.[23,29]

Previous studies demonstrated that, when the etching pattern of these two moderate two-step systems were compared, a deeper etching pattern was observed when these were applied to abraded enamel, suggesting that removal of the superficial layer of enamel is essential in improving their performance.[32-33] Further studies need to be conducted in order to investigate these assumptions.

Although significant differences were observed in microtensile bond strength values, no gap formation was found between the composite and enamel substrate, as already reported by other authors.[3] This is in agreement with previous literature findings, since no correlation between marginal sealing, micro- or nanoleakage with bond strength values measured immediately or over time has been established.[34,36]

Several studies have reported resin-dentin bond degradation after water storage.[11-13] Hydrolysis of the interface components (resin and/or collagen) is usually blamed for such degradation. Water can infiltrate into the resin matrix and, through swelling, can reduce frictional forces between the polymer chains in a process known as plasticization. This water-driven process can, therefore, decrease the mechanical properties of the polymer matrix,[37-38] and the breakdown of products from the aforementioned plasticization mechanisms can be eluted, weakening the bonded interface.[39] More recently, evidence has demonstrated that the breakdown of unprotected collagen fibrils[40-41] can also occur via the activation of host-derived matrix metalloproteinases.[42-43]

Contrary to long-term resin-dentin bond strength results, resin-enamel bonds were stable after 12 months of water storage, which led the authors of the current study to accept the third null hypothesis. The insignificant amount of organic matrix in the enamel structure makes this tissue completely different from dentin. Although copious amounts of protein are secreted during enamel matrix development[44-45] when the maturation stage is complete, enamel achieves its final hardened structure, where it contains less than 1% protein by weight.[46-47] This means that, compared to dentin, enamel does not contain suitable organic components to be degraded over time, or that the period of evaluation was too short to detect any significant difference in enamel adhesion.

Polymerization of adhesive monomers in the presence of water prevents the formation of a highly cross-linked polymer.[48] Thus, poor polymerization of the adhesive due to the wet nature of dentin is not expected in enamel. It is possible that a stronger polymer is formed when applied to enamel and, therefore, water absorption is

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much less than in dentin, making the resin-enamel interface less prone to the plasticizing effects of water over time.

It was also well documented that one-step self-etch systems behave as permeable membranes after polymerization.[49-50] This is probably caused by a lack of a hydrophobic layer placed over the self-etch primer.

In agreement with the current results are the studies which demonstrated that resin-dentin bonds can be stable over time, as long as the cavity margins are surrounded by enamel.[11] The stability of bond strength values in specimens with an enamel border can be attributed to the protective role of the surrounding resin-enamel bond against degradation,[11,38] avoiding inward water diffusion.

Although no significant reductions in resin-enamel bond strengths and gap formation were observed in this study after long-term water storage of the specimens, several clinical trials have reported marginal discoloration around enamel borders bonded with two-step self-etch systems.[32-33] Nonetheless, lower bond strength values, or the absence of enamel gaps, do not automatically mean worse retention rates. This explains why studies evaluating the retention of brackets to enamel bonded with etch-and-rinse and self-etch systems do not show higher retention rates for the former.[51-52] Further *in vivo* and *in vitro* studies should be conducted in order to elucidate the hypothesis raised in this study.

CONCLUSIONS

Based on the limitations of the current investigation, one can conclude that:

1. No interfacial enamel gap was observed between composite and enamel for all experimental conditions.
2. The effect of the enamel preparation was adhesive-dependent. The microtensile bond strength values for the SiC paper and diamond bur groups were similar and higher than the unground group only for the two moderate two-step adhesive systems tested.
3. No degradation of resin-enamel bonds was observed for any of the adhesives tested after 12 months of water storage.

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