

# Microtensile Bond Strength Test and Failure Analysis to Assess Bonding Characteristics of Different Adhesion Approaches to Ground *versus* Unground Enamel

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This study evaluated the bonding characteristics to ground and unground enamel obtained with different strategies. For this purpose, 24 sound third-molars were bisected mesiodistally to obtain tooth halves. A flat enamel area was delimited in the tooth sections, which were randomly distributed into 8 groups (n=6), according to the enamel condition (ground and unground) and adhesive system (Adper Single Bond 2 - SB2; Adper Prompt L-Pop - PLP; Adper Prompt - AD; Clearfil SE Bond - SE). Each system was applied according manufacturers' instructions and a 6-mm-high resin composite "crown" was incrementally built up on bonded surfaces. Hourglass-shaped specimens with 0.8 mm<sup>2</sup> cross-section were produced. Microtensile bond strength ( $\mu$ TBS) was recorded and the failure patterns were classified. Results were analyzed by two-way ANOVA and Tukey's test ( $\alpha=0.05$ ). There were no statistically significant differences among the  $\mu$ TBS values of SB2, PLP and AD ( $p>0.05$ ). SE values were significantly lower ( $p<0.05$ ) than those of SB2 and PLP, but not different from those of AD ( $p>0.05$ ). There was prevalence of cohesive failure within enamel, adhesive system and resin composite for SB2. The self-etch systems produced higher incidence of cohesive failures in the adhesive system. Enamel condition did not determine significant differences on bonding characteristics for the same bonding system. In conclusion, the bonding systems evaluated in this study resulted in specific  $\mu$ TBS and failure patterns due to the particular interaction with enamel.

Key Words: enamel, adhesive systems, bond strength, failure analysis, scanning electron microscopy.

## INTRODUCTION

Contemporary adhesive systems can hybridize dental hard tissues through the total-etch or self-etch approaches. The total-etch approach is an accepted and widely used technique to improve bonding of dental resins to enamel in both restorative and preventive dentistry. The enamel etched with phosphoric acid increase in wettability and surface contact area what turns it more favorable to monomer infiltration and form a stable micro-mechanical retention after the polymerization (1,2). In order to simplify the bonding

protocol, manufacturers have attempted to produce the self-etch adhesives systems that combine tooth surface etching and priming in one single procedure. The adhesives that use this adhesion approach are classified into two- or one-step systems according to the number of procedures required for bonding, as well as, ultra-mild, mild, intermediate strong and strong systems depending on the initial pH (3).

The elimination of separate etching and rinsing steps shorter the application time and reduced the technical sensibility, what has been responsible for the increased popularity in the clinical practice. However,

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the effectiveness of self-etch approach on enamel is still controversial. The two-step self-etch primers have shown much less conditioning ability than the phosphoric acid, especially on unground (UN) enamel, because of their high pH (4,5). This performance could partially explain the lower resin-enamel bond strength in comparison to the results on ground (GR) enamel, which was similar to the total-etch systems (5). In contrast, other results do not show any significant difference on bond strength when two-step and one-step self-etch adhesives were applied on GR and UN enamel (6). Pashley and Tay (7) reported that one-step self-etch adhesives have the potential to etch the UN enamel, producing an etching pattern comparable to that of phosphoric acid treatment. A similar tendency has been observed by other authors, who demonstrated a correlation between the pH of the adhesive systems and the level of morphological alterations of the enamel surface (8).

In spite of the bonding stability obtained on acid-etched enamel, marginal discoloration and recurrent caries are still frequent and have been claimed to be responsible for most replacements of resin composite restorations (9). Resin margins usually become stained when the restorative material is extended onto unetched enamel around the prepared cavity (10). Ideally, the restorations should not present any resin composite or adhesive excess beyond the margins. However, from a clinical perspective, it seems difficult to achieve such condition. According to Opdam et al. (11), most *in vivo* class II resin composite restorations have overextended margins. Thus, a good clinical performance of composite restorations depends on an effective bonding to UN enamel around the prepared cavity.

Due to the diversity of results, the combination of microtensile bond strength ( $\mu$ TBS) test and failure pattern analysis was purposed to assess bonding characteristics to GR *versus* UN enamel using the total-etch and the self-etch adhesion approaches. The hypotheses tested were: 1. there is no significant difference among adhesive systems regarding bond strength and failure pattern; 2. the enamel condition does not influence the performance of the adhesive systems.

## MATERIAL AND METHODS

Twenty-four caries-free human third molars extracted for orthodontic reasons were used in this study, according to the protocol approved by Research Ethics Committee of the Piracicaba Dental School, University

of Campinas, Brazil (073/2007). The donors of these teeth live in a geographic region where the water supply is fluoridated. The teeth were stored in 0.5 chloramine T at 4°C and were used within 2 months following extraction. They were decoronated with a diamond disk (KG Sorensen, Barueri, SP, Brazil) mounted in a slow-speed handpiece. The crown of each tooth was sectioned in a mesiodistal direction using a diamond-impregnated disk (Exttec; Enfield, CT, USA) under water lubrication in specific cutter machine (Isomet 1000; Buehler, Lake Bluff, IL, USA) to obtain tooth halves that were randomly distributed to each experimental condition. After that, the bonding area (nearly 9 mm<sup>2</sup>) was demarcated to outline the flattest surface. The occlusal third of the buccal and lingual surfaces were usually outside the bonding area due to their inclination. The demarcated surfaces were cleaned with pumice/water slurry, and examined under a stereomicroscope to ensure that they were free of decalcification, surface cracks or previous grinding. Half of the tooth halves were kept intact (UN specimens), while the other tooth halves had the enamel surface ground with 600-grit SiC paper under water cooling for 60 s to create a standardized smear layer prior the adhesive application (GR specimens).

### Bonding Procedure

The GR and UN enamel surfaces were randomly assigned to 8 groups of 6 specimens each, according to the combination of enamel surfaces (UN and GR) and adhesive systems (Adper Single Bond 2 - SB2; Adper Prompt L-Pop - PLP; Adper Prompt - AD; Clearfil SE Bond - SE). The adhesives systems, their compositions, pH and manufacturers are listed in Table 1. All adhesive systems were applied under controlled environmental conditions (24°C/60% relative humidity) by a single operator, according to manufactures' instructions. A "crown" of resin composite (FilteK Z-250; 3MESPE, St. Paul, MN, USA) was incrementally build up to a height of 6 mm on the UN and GR enamel bonded surfaces. Each increment (2 mm thick) was light-cured for 40 s with a quartz-tungsten-halogen light-curing unit (XL 3000; 3MESPE). The bonded surfaces were then stored in tap water at 37°C for 24 h.

### Microtensile Bond Strength Test and Failure Analysis

The GR and UN enamel bonded surfaces were serial sectioned in 1-mm-thick slabs. The slabs were

trimmed and shaped with a high-speed diamond bur (#1122 FF - KG Sorensen) in a high-speed handpiece under air/water spray coolant. Hourglass specimens with approximately 0.8 mm<sup>2</sup> cross-section area were produced. The specimens were fixed with cyanoacrylate adhesive (Zapit DVA; Corona, CA, USA) to the grips of a microtensile device. Maximal tensile bond strength measurement was performed in a universal testing machine (4411; Instron Co., Canton, MA, USA) using a load cell of 50 N at a cross-head speed of 0.5 mm/min

until failure. A schematic presentation of the preparation of specimens for the  $\mu$ TBS test is illustrated in Figure 1.

Following the  $\mu$ TBS test, failed specimens were carefully removed from the grips with a scalpel blade and the cross-sectional area at the site of fracture was measured to the nearest 0.01 mm with a digital caliper (Starret 727-6/150; Starret, Itu, SP, Brazil). Maximal tensile load was divided by specimen cross-sectional area to express results in MPa. Statistically significant differences between the mean bond strength of the 4

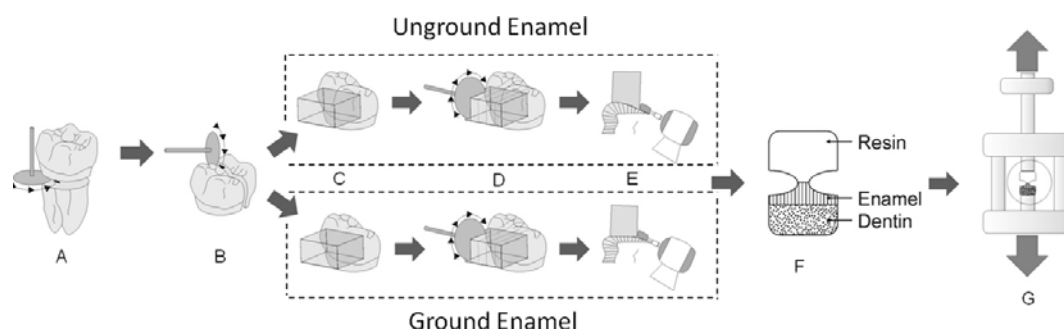


Figure 1. Schematic representation of specimen preparation for the  $\mu$ TBS test. Root removal (A); cutting of the tooth crown mesiodistally (B); incremental build up of a resin composite “crown” (C); serial sectioning (D) and trimming (E) of the enamel bonded surfaces to obtain hourglass-shaped specimens (F); microtensile device (G).

Table 1. Composition, pH and manufactures of the adhesive systems used in this study.

Adhesive system	Composition	pH	Manufacturer
Adper Single Bond 2	Water, ethanol, HEMA, Bis-GMA, dimethacrylates, initiators, methacrylate functional copolymer of polyacrylic and polyitaconic acids and silica nanofillers Etching: 35% phosphoric acid	0.6 <sup>a</sup>	3M ESPE, St. Paul, MN, USA
Clearfil SE Bond	<i>Primer:</i> water, MDP, HEMA, hydrophilic dimethacrylates, camphoroquinone, <i>Adhesive:</i> MDP, Bis-GMA, HEMA, camphoroquinone hydrophobic dimethacrylate, N/N-diethanol p-toluidine bond, colloidal silica	2	Kuraray Medical Inc., Kurashiki, Okayama, Japan
Adper Prompt L-Pop	Water, methacrylates phosphoric acid-HEMA esters, BAPO initiator stabilizer, fluoride complex parabenes	0.8	3M ESPE, St. Paul, MN, USA
Adper Prompt	<i>Prompt A:</i> Methacrylated phosphoric esters, Bis-GMA, initiators based on camphorquinone, stabilizers <i>Prompt B:</i> Water, HEMA, polyalkenoic acid, stabilizers	0.4	3M ESPE, St. Paul, MN, USA

HEMA=2-hydroxyethyl methacrylate; Bis-GMA=bisphenol-glycidil dimethacrylate; MDP= 10-10-methacryloyloxydecyl dihydrogen phosphate; BAPO = bis-acylphosphine oxide; <sup>a</sup> = phosphoric acid 35%.

adhesive systems and the 2 enamel conditions (GR or UN) were analyzed by two-way ANOVA and Tukey's test at 5% significance level.

Common procedures for preparation of the specimens for examination by scanning electron microscopy (SEM) for failure analysis were undertaken, including fixation with 2.5% glutaraldehyde and 2% paraformaldehyde in 0.1 M cacodylate buffer, pH 7.3 (Karnovsky's fixative), dehydration in ascending concentrations of ethanol (25, 50, 75, 95 and 100%) and immersion in hexamethyldisilazane (HMDS) for 10 min to chemical drying (12). After mounting on aluminum stubs, the specimens were sputtered coated with gold/palladium (SCD 050; Balzers, Schaan, Liechtenstein) and examined using a scanning electron microscope (JSM 5600LV; JEOL, Tokyo, Japan) operating at 15 kV. The failure patterns were classified in one of the three following categories: type I - cohesive failure in the enamel; type II - cohesive failure in the adhesive system and type III - cohesive failure in the enamel, adhesive system and resin composite. Representative SEM micrographs were taken at the enamel side of the fractured specimens with the following magnifications:  $\times 110$  to  $\times 150$  (general view) and  $\times 350$  to  $\times 1,500$  (marked sections).

## RESULTS

The means and standard deviations of the  $\mu$ TBS values are given in Table 2. Two-way ANOVA revealed no statistically significant difference ( $p > 0.05$ ) among the  $\mu$ TBS mean values of SB2, PLP and AD for both enamel conditions. SE showed significantly lower ( $p < 0.05$ ) bond strength than SB2 and PLP, but no significant difference ( $p > 0.05$ ) from AD in both enamel conditions. The GR and UN enamel conditions did not differ significantly ( $p > 0.05$ ) from each other for same adhesive system.

The failure pattern distribution (%) as analyzed by SEM can be observed in Figure 2. There was prevalence

of type III failure pattern for SB2 on both GR and UN enamel (Fig. 3A,B). The three self-etch adhesive systems showed a prevalence of type II failure pattern for both enamel conditions (Fig. 3C-H). A low percentage of the type I failure pattern was found for PLP in GR enamel, AD in GR enamel and in both enamel conditions for SB2.

## DISCUSSION

Several studies have employed traditional shear and tensile bond strength tests to evaluate the adhesion of adhesive systems to enamel and dentin. However, these tests frequently induces cohesive failure in the substrate at the fractured surface due to stress. Cohesive failure within enamel as substrate to adhesion is especially common because of its brittle properties. The  $\mu$ TBS test uses small cross-section areas, from 0.5 to 1.5 mm<sup>2</sup> depending on the technique, allowing a more uniform stress distribution due to the supposedly more homogeneous structure. As a result, the incidence of cohesive fracture at the substrate is reduced and a more realistic assessment of the bond strength can be performed. Moreover, the failure analysis at the fractured surface after  $\mu$ TBS test can be readily performed, providing extremely important information about the integrity of the bond structures and bonding mechanisms (13).

SB2 showed a prevalence of the type III failure pattern (Fig. 3A,B), for both enamel condition. Probably, the high incident of this failure pattern occurred due to the interaction form of the total-etch approach with enamel. The deep and complex inter-prismatic demineralization produced by the phosphoric acid is not completely filled out by the resin monomers, especially at the bottom of hybrid-like layer (14), creating a discrepant zone that is thought to be less resistant to the mechanical efforts. Thus, the authors suppose that there was a tendency to the failure initiated in the severely

demineralized enamel prisms at the discrepant zone during the  $\mu$ TBS test, propagating within the adhesive system and resin composite.

Self-etch adhesives vary in their acidity according to the composition and concentration of the polymerizable acids and/or acidic resin monomers.

Table 2. Tensile bond strength of the four adhesive systems tested. Mean (MPa)  $\pm$  SD.

Enamel	Adhesive system			
	Adper Single Bond 2	Adper Prompt L-Pop	Adper Prompt	Clearfil Se Bond
Unground	23.95 $\pm$ 6.05 <sup>a</sup>	23.74 $\pm$ 4.01 <sup>a</sup>	20.49 $\pm$ 5.72 <sup>a,b</sup>	17.19 $\pm$ 5.03 <sup>b</sup>
Ground	24.11 $\pm$ 5.22 <sup>a</sup>	23.51 $\pm$ 5.11 <sup>a</sup>	20.88 $\pm$ 5.54 <sup>a,b</sup>	17.92 $\pm$ 3.09 <sup>b</sup>

Different subscript letters in the rows indicate statistically significant difference ( $p < 0.05$ ).

The two-step self-etch primer SE contains the unsaturated methacrylated phosphate monoester 10-MDP (10-methacryloxydecyl dihydrogen phosphate), as the acidic resin monomer. It is considered a “ultra-mild” self-etch primer with a pH of 2, what results in a reduced potential to demineralize enamel (7,8). This characteristic may have a direct effect on the magnitude of bond strength and was considered as the

main reason for the lower bond strength in comparison to SB2. The larger percentage of the uniform type II failure pattern (Fig. 3C,D), in both enamel conditions, was probably determined by the self-etch approach. As demineralization and resin monomer infiltration occur almost simultaneously, the discrepant zone is minimized and no cohesive failure in the enamel was observed. In addition, hydroxyapatite crystals are not removed by the

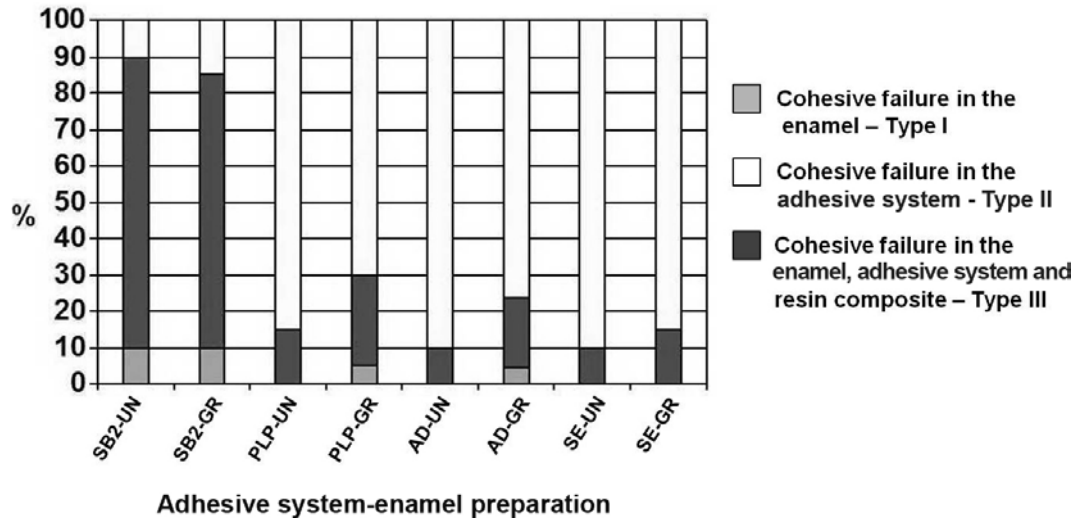


Figure 2. Distribution of the failure patterns (%) of the adhesive systems Adper Single Bond 2 (SB2), Adper Prompt L-Pop (PLP), Adper Prompt (AD) and Clearfil Se Bond (SE) on ground (GR) and unground (UN) enamel.

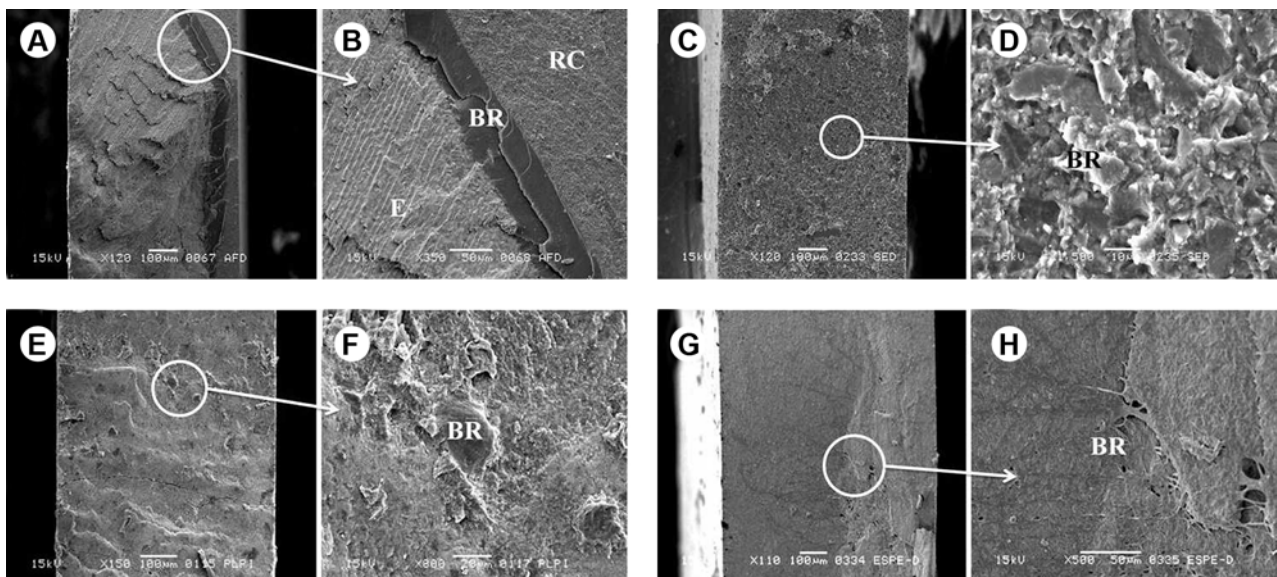


Figure 3. Representative SEM micrographs illustrating the fractured surfaces of the enamel side of the specimens. A general view of the fractured surfaces of SB2 bonded to ground enamel (A), SE to ground enamel (C), PLP to unground enamel (E) and AD to ground enamel (G) is shown. Higher magnification of the sections (white circle) shows type III failure pattern for Adper Single Bond 2 (B) and type II failure pattern for the self-etching systems (D, F and H). E = Enamel; BR = Bonding resin; RC = Resin composite.

water rinsing after demineralization with the SE primer and can act as a kind of inorganic reinforcement in the bonding region, making it physically resistant to tensile forces. According to this interpretation, the weakest link of the resin-enamel interface is the adhesive system, which was proven more susceptible to fracture during the  $\mu$ TBS test.

PLP and AD present methacrylated phosphoric mono and diesters combined in balanced proportions, in which monoesters are prevalent. Since these three self-etch systems are composed basically of acidic monoesters, it would be expected that they could produce equivalent dissolution of enamel. However, the concentration of acid esters in PLP and AD ( $\approx 80\%$ ) is higher than in SE ( $\approx 25\text{-}30\%$ ). Such difference explains why the two one-step self-etch have been described to produce a consistent demineralization of the enamel surfaces and are considered as strong adhesives, forming defined 1-2  $\mu\text{m}$  hybrid-like layers (7,8). This potential to achieve micromechanical interlocking similar to the total-etch approach was probably decisive for the bond strength in the same range that SB2. In spite of the demineralization comparable to phosphoric acid, a prevalence of type II failure pattern was observed (Fig. 3E,F for PLP; Fig. 3G,H for AD) on GR and UN enamel surfaces. This finding can be attributed to the self-etch interaction in a similar way to SE, where the discrepant zone is thought to be reduced. In general, the predominance of cohesive failure in the adhesive system can be considered a goal of the self-etch approach to enamel. When the bonding region was submitted to tensile strength until fracture, the enamel was preserved and remained protected by the hybrid-like layer and the adhesive system.

Despite the difference in aggressiveness, AD and SE produced similar bond strength. As already reported, this means that other factors, apart from the etching pattern, may have important role on the bond strength. For instance, the carboxylic acid-based monomer 10-MDP present in SE has a chemical bonding potential to the hydroxyapatite (15). This interaction could theoretically have contributed to the equivalent bond strength between these self-etch systems, despite the reduced potential for micromechanical interlocking observed with SE to enamel (7,8).

Another important point to be considered has been described by Pashley and Tay (7) about the acidic environment of one-step self-etch adhesives that can disturb the polymerization reaction and reduces its

mechanical properties. As the  $\mu$ TBS can be correlated with the ultimate bond strength of the self-etch adhesive systems (16), such characteristic could have decreased the efficiency of PLP and AD. However, it can be speculated that this interference on the process was not relevant on the bond strength to enamel. In addition to the reduced content of water in enamel, the surface is dried prior to adhesive application and afterwards to evaporate the solvent. This condition is unfavorable for the intensification of the acidic environment and might have minimized the interference in the adhesive system polymerization. Even without this immediate interference in the bond strength, methacrylates have low hydrolytic stability in acidic solutions. The ester portion of functional methacrylates, such as HEMA, commonly used for self-etch primers, undergoes rapid hydrolysis under acidic aqueous conditions. This commits the long-term performance of this category self-etch systems, which degraded up to 90% after 16 weeks (17).

Differences between GR and UN enamel have been discussed in the literature. Studies have shown the presence of the prismless layer on the UN enamel surface and its imperviousness to mild-acidic attack (4,8,18). UN enamel is high mineralized and can incorporate fluoride in the hydroxyapatite structure, what turns it more resistant to demineralization. Therefore, the resulting etching pattern of UN enamel is less homogeneous than that of GR enamel, especially with mild acids (4,5,7,8), which do not etch the underlying prismatic enamel (4,7). On the other hand, the presence of smear layer on GR enamel and its claimed capacity to buffer the effect of a number of acids (4,19) could hinder the etchant action. These compensatory factors may be one of the reasons why differences between enamel surface preparation was not found to significantly change the  $\mu$ TBS and failure pattern employing the ultra-mild self-etch adhesive SE, which is in accordance with other results (5,6). In relation to the total-etch system SB2, under SEM observation at high magnification, no micromorphological differences was observed when the GR and UN enamel surfaces were compared after conditioning with phosphoric acid. Complete dissolution of the aprismatic layer with exhibition of the prismatic enamel was observed (4,8). One-step self-etch adhesives have a similar performance due their low pH (7,8), suggesting that initial enamel condition was not decisive for the  $\mu$ TBS. In addition to these factors, the authors suggest that the eventual absence of the prismless layer on the UN enamel surface of some teeth might have contributed with the

statistically equal bond strength values for GR and UN enamel, considering the same adhesive system. The medium thickness of the prismless enamel layer is 30  $\mu\text{m}$ . However, this layer can become thinner or be completely removed due to the functional wear in the oral cavity (20). In face of this possibility, the bonding procedures to UN enamel could have been accomplished on a prismatic surface similar to the GR enamel.

According to the methodology employed and based on the obtained results, it may be concluded that the integrity of the bond surfaces varied according to the adhesion approach. Each one of the adhesive systems tested showed specific bond strength and failure pattern. The hypothesis 1 was rejected because the bond strength of SE was significantly lower than that of SB2, which showed different failure patterns from the other three self-etch systems evaluated. The hypothesis 2 was accepted as enamel condition did not influence the performance of the adhesive systems.

## RESUMO

Este estudo avaliou a união ao esmalte íntegro e desgastado obtida com diferentes estratégias. Para tanto, 24 terceiros molares hígidos foram seccionados ao meio. Um plano de esmalte foi delimitado nos fragmentos de dente, aleatoriamente distribuídos em 8 grupos ( $n=6$ ) conforme a condição do esmalte (íntegro ou desgastado) e o adesivo (Adper Single Bond 2: SB2; Adper Prompt L-Pop: PLP; Adper Prompt: AD; Clearfil SE Bond: SE). Estes foram aplicados seguindo recomendações dos fabricantes e uma “coroa” de compósito (altura - 6 mm) incrementalmente construída. Espécimes (ampulheta/secção transversal - 0,8 mm<sup>2</sup>) foram confeccionados. O ensaio de resistência da união à microtração (RU $\mu$ T) foi realizado e os padrões de fratura classificadas. Os resultados analisados pela ANOVA (dois fatores) e teste de Tukey ( $\alpha=0,05$ ). Os valores de RU $\mu$ T do SB2, PLP e AD não foram significativamente diferentes entre si ( $p>0,05$ ); SE foi inferior ( $p<0,05$ ) ao SB2 e PLP, mas não diferiu do AD ( $p>0,05$ ). Houve prevalência de fratura no esmalte, adesivo e compósito no SB2. Nos adesivos autocondicionantes, predominou a fratura no adesivo. A condição do esmalte não influenciou significativamente as características da união, considerando o mesmo adesivo. Em conclusão, os adesivos apresentaram RU $\mu$ T e padrões de fratura específicos, devido a forma de interação com o esmalte.

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