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Research Article

Dentin Bond Strength of Universal Adhesive Systems under Different Strategies: Self-Etch (SE) and Etch-and-Rinse (ER)

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Abstract

Objective: To evaluate the short-term µTBS (Micro-tensile bond strength) of universal adhesive systems under different strategies: self-etch (SE) and etch-and-rinse (ER).

Materials and Methods: Twelve sound third molars were divided into 6 groups (n = 2) according to the adhesive system: Scotchbond Universal - 3M [SBU], All Bond Universal - BISCO [ABU] and the adhesive strategy: ER and SE. The adhesives Clearfil SE Bond - Kuraray [CSE] and Adper Single Bond 2 - 3M [SB] were used as control for SE and ER, respectively. The restored teeth were placed in 37°C distilled water for 24 h. Twenty sticks (1 mm x 1 mm approximately) were obtained from each tooth and they were subjected to a tensile force at a crosshead speed of 0.5 mm / min in a universal testing machine (KRATOS K 2000) until failure occurred. The resultant µTBS values were evaluated with Kruskal-Wallis test ($p < 0.05$).

Results: Significant differences were detected in the mean µTBS in the ER group ($p = 0.012$) and SE group ($p < 0.001$). The mean µTBS of ABU/ER (34,59 MPa) and SB (39,86 MPa) did not differ statistically and they showed higher mean µTBS than that of SBU/ER (25,16 MPa). The mean µTBS of ABU/SE was the highest (45,47 MPa) followed by that of CSE (35,26 MPa) and SBU/SE (24,44 MPa).

Conclusion: The adhesive All Bond Universal showed better bonding performance in both adhesive strategies when compared to Scotchbond Universal.

Keywords: *Bond Strength; Adhesive; Self-Etch; Etch-and-Rinse*

Abbreviations

ER: Etch-and-Rinse; SE: Self-etch; SBU: Scotchbond Universal; ABU: All Bond Universal; CSE: Clearfil SE Bond; SB: Adper Single Bond 2; MDP: Methacryloyloxydecyl dihydrogen phosphate; MMP: Metalloprotreinases; µTBS: Micro-tensile bond strength

Introduction

The adhesive systems can be classified according to the way they act on the smear layer: etch-and-rinse (ER) and self-etch (SE). One difference between these two kinds of dentin adhesives is that the first one promotes the removal of the smear layer, amorphous layer of debris of enamel, dentin and odontoblast processes that is formed after the use of an instrument to remove dental tissue, and the second one promotes its incorporation into the adhesive process [1]. The ultimate goal, common to these adhesives, is the formation of a qualitatively good, compact and homogeneous hybrid layer that is a layer formed by dentin infiltrated by resin monomers. The adhesion achieved by these adhesive systems is based on micromechanical adhesion to dentin obtained by infiltration and subsequent polymerization of resin monomers [2] in the interfibrillar spaces of the exposed collagen network [3] and in the dentinal tubules [4].

During etch-and-rinse adhesive system application in three-steps or two-steps, the collagen fibrils are demineralized by acid etching. The action of these adhesive systems generates a limitation that is the poor solvent evaporation when this solvent is water. The 37% phosphoric acid promotes hydroxyapatite dissolution, leaving collagen fibers, not involved by hydroxyapatite, soaked in water, leading to swelling of the collagen fibers which, in turn, close the nanospaces created by the mineral dissolution. The non-evaporation of this water associated to poor infiltration of the adhesive resin monomers will contribute negatively to the formation of a good hybrid layer between the resin and the exposed collagen fibers [5]. The other limitation also related to the etching technique is the activation of metalloproteinases (MMP's) present in the dentin matrix. These proteins are secreted in inactive form, however, phosphoric acid promotes their activation, making them able to hydrolyze the collagen fibers present in the demineralized dentin, influencing negatively in the success of the restorations [6].

In order to improve not only the adhesive bond strength as well as the quality and longevity of adhesion, a monomer, 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP), has been developed, synthesized and incorporated into the primer and the adhesive resin [7]. This monomer is able to bond to the hydroxyapatite from the enamel and dentin, increasing the longevity of the connecting interface. Initially synthesized by Kurakay (Osaka, Japan), it is currently considered as the most promising functional monomer in the chemical adhesion to enamel and dentin [8].

The recently introduced class of multi-mode adhesive system or universal adhesive was designed to bond to dental structures through the etch-and-rinse or one bottle self-etch technique. In its composition it was incorporated the 10- MDP. These adhesives' manufacturers suggest that they may also be used for selective etching technique on enamel margins [9].

The purpose of the present study was to evaluate the short-term μTBS (Micro-tensile bond strength) of two universal adhesive systems under different strategies: self-etch (SE) and etch-and-rinse (ER). The null hypothesis of the study was that there is no difference on dentin bond strength between the two universal adhesives under both adhesive strategies.

Materials and Methods

Tooth selection and preparation

Twelve extracted sound human third molars were selected. The teeth were cleaned and disinfected in 0.1% thymol solution. A flat dentine surface was exposed after wet grinding the occlusal enamel on a #180 grit SiC paper. The exposed dentine surfaces were further polished on wet #600-grit silicon-carbide paper for 60s to standardize the smear layer.

Test Groups

The teeth were assigned randomly to 6 groups as follows:

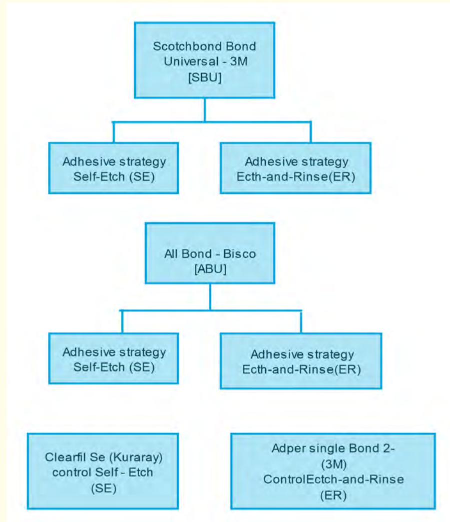

Figure 1: *Test Groups.*

Restorative Procedure

The adhesive systems were applied according to the manufacturers' instructions. After the bonding procedures, all teeth were restored with resin composite (Filtek Z350 XT, 3M ESPE) in 5 mm thick increments. Each incremental layer was light cured for 40 s using light curing unit (600 mW/cm^2, Optilight Plus, Gnatus, Ribeirão Preto, SP, Brazil). Then, the restored teeth were stored in distilled water at 37°C for 24 h. The application mode of each adhesive system can be seen in Table 1.

Adhesive system (Batch number)	Composition	Self-etch strategy	Etch-and-rinse strategy
Adper Single Bond 2 (3M) Batch number: N364098BR	1. Etchant: 35% phosphoric acid (Scotchbond Etchant) 2. Adhesive: bis-GMA, HEMA, dimethacrylates, ethanol, water, photoinitiator, methacrylate functional copolymer of polyacrylic and poly (itaconic) acids,10% by weight of 5 nm-diameter spherical silica particles	N.A.	1. Apply etchant for 15 s 2. Rinse for 10 s 3. Blot excess water 4. Apply 2–3 consecutive coats of adhesive for 15 s with gentle agitation 5. Gently air dry for 5 s 6. Light polymerize for 10 s at 1,200 mW/cm^2
Clearfil SE Bond (Kuraray) Batch number: 01714-A	1. Primer: water, MDP, HEMA, camphorquinone, hydrophilic dimethacrylate 2. Bonding: MDP, bis-GMA, HEMA, camphorquinone, hydrophobic dimethacrylate, N,N-diethanol p-toluidine bond, colloidal silica	1. Apply primer to tooth surface and leave in place for 20 s 2. Dry with air stream to evaporate the volatile ingredients 3. Apply bond to the tooth surface and then create a uniform film using a gentle air stream 4.Light polymerize for 10 s at 1,200 mW/cm^2	N. A.

All-Bond Universal (Bisco) Batch number: 1300001647	1. Etchant Uni-Etch: 32% phosphoric acid, benzalkonium Chloride 2. Adhesive: MDP, bis-GMA, HEMA, ethanol, water, initiators	1. Apply two separate coats of adhesive, scrubbing the preparation with a microbrush for 10–15 s per coat. Do not light polymerize between coats 2. Evaporate excess solvent by thoroughly air-drying with an air syringe for at least 10 s, there should be no visible movement of the material. The surface should have a uniform glossy appearance 3. Light polymerize for 10 s at 1,200 mW/cm^2	1. Apply etchant for 15 s 2. Rinse thoroughly 3. Remove excess water with absorbent pellet or high volume suction for 1–2 s 4. Apply adhesive as for the self-etch mode
Scotchbond Universal (3M) Batch number: (619545)	1. Etchant: 34% phosphoric acid, water, synthetic amorphous silica, polyethylene glycol, aluminium oxide. (Scotchbond Universal Etchant) 2. Adhesive: MDP phosphate monomer, dimethacrylate resins, HEMA, methacrylate-modified polyalkenoic acid copolymer, filler, ethanol, water, initiators, silane	1. Apply the adhesive to the entire preparation with a microbrush and rub it in for 20 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 has evaporated completely 3. Light polymerize for 10 s	1. Apply etchant for 15 s 2. Rinse for 10 s 3. Air dry for 2 s 4. Apply adhesive as for the self-etch mode

Table 1: *Adhesive system (batch number), composition and application mode according to the manufacturer's instructions.*

Micro-tensile bond strength (μTBS) test

Each specimen was longitudinally sectioned in the mesio-distal and buccal-lingual directions across the bonded interface, using a slow-speed diamond saw (Isomet,Buehler Ltd., Lake Bluff, IL, USA) to obtain 20 resin–bonded sticks (1 mm x 1 mm approximately).

Resin–bonded sticks were then fixed to a Geraldeli device with cyanoacrylate and subjected to a tensile force at a crosshead speed of 0.5 mm/min in a Universal Testing Machine (Kratos Dinamometros, Cotia, SP, Brazil) until failure occurred. The μTBS values were expressed in MPa dividing the applied force (N) at the time of fracture by the bonded area (mm^2).

Statistical Analysis

Data were expressed by mean ± standard deviation. The normality hypothesis was rejected by Shapiro-Wilk test. Kruskal-Wallis test with its multiple comparisons was used to compare bond strength of the groups. The significance level was 5%. (SPSS, V21, Chicago, USA).

Results

The mean μTBS and standard deviations are displayed in Table 2. Significant differences were detected in the mean μTBS in the ER group ($p = 0.012$) and SE group ($p < 0.001$). Comparing the ER groups, the mean μTBS of ABU/ER and SB (ER control) did not differ statistically and they showed higher mean μTBS than that of SBU/ER. Comparing the SE groups, the mean μTBS of ABU/SE was the highest, followed by that of CSE (SE control) and SBU/SE. Comparing the application mode for each experimental group, significant difference was detected in the mean μTBS of All Bond Universal ($p = 0.001$).

Adhesive System	Application Mode	μTBS (MPa)
Adper Single Bond 2	Etch-and-rinse	39,86 ± 9,64 A
Scotchbond Universal	Etch-and-rinse	25,16 ± 10,25 B
All Bond Universal	Etch-and-rinse	34,59 ± 13,18 A
Clearfil SE Bond	Self-etch	35,26 ± 9,34 a
Scotchbond Universal	Self-etch	24,44 ± 8,10 b
All Bond Universal	Self-etch	45,47 ± 9,26 c

Table 2: *Micro – tensile bond strength (μ TBS) values (means ± standard deviations) of the experimental groups. Similar capital (ER groups) and lower (SE groups) letter are not statistically significant ($p < 0.05$).*

Discussion

The null hypothesis that there was no difference between the two universal adhesives on dentin bond strength under different strategies (etch-and-rinse and self-etch) was rejected. ABU groups resulted in higher mean μTBS than those of SBU groups in both adhesive strategies. It can be explained by the fact that All Bond Universal has in its composition the monomer MDP whose function is to carry out chemical bonds to free Ca+ ions. A study [10] showed that an effective chemical interaction occurs between MDP and hydroxyapatite forming a stable nano-layer inducing the formation of a strong adhesive layer, thereby, increasing the mechanical strength. Moreover, the deposition of stable salt MDP-Ca, along with nano - layers, may explain the high bond strength. It is worth noting that ABU requires the application of two layers of adhesive, different from SBU whose manufacturer recommends applying one layer. This double layer increases the concentration of phosphate monomer (MDP) enhancing the chemical dentin bonding.

The application mode can be crucial to the adhesive quality of some systems, especially the two-step self-etch ones (self-etching primer). As they are less acidic, the adhesive quality can be improved if the primer is applied actively, it means, rubbing the applicator instrument on the surface for the time recommended by the manufacturer, dissolving, thus, the particles of the smear layer and facilitating the infiltration of acidic monomers in the underlying dentin [11,12].

Although the adhesive Scotchbond Universal has in its chemical composition the phosphate monomer MDP, which is important for chemical adhesion, an agent of "wetting" (HEMA), alcohol and water as solvent, similar to Clearfil SE Bond, and in addition the copolymer Vitrebond, with the potential to make chemical bonds to free Ca^{+} ions, the bonding performance of this system was not so good. These results can be explained by the need of removing the residual monomers and water. Water is an essential factor in the activation of the acidic monomers in the self-etching systems, however, its permanence after polymerization is undesirable and must be removed at the moment of solvent evaporation [5]. A study [11] has shown that only periods of adhesive drying longer than 12-20 s can ensure complete evaporation of the solvent. The adhesive system Clearfil SE Bond discloses advantage for having its application performed in two steps, facilitating the removal of the solvent (water). But even with such recommendation of gentle drying, this two-step system needs the application of a layer of hydrophobic resin (Bond) overlying the conditioning primer. This hydrophobic coverage improves the bond strength of the self-etch adhesives [13].

It should be taken into consideration that as Scotchbond Bond Universal is a one-step adhesive, it is generally more hydrophilic, containing higher amount of solvent and diluent than the 2-step self-etch primers [1]. The applied layers of SBU have a tendency to be thinner, especially after the evaporation of large amounts of solvents. Due to the fact that these systems do not require the application of a layer of adhesive (Bond) separately, one runs the risk that the layer is quite thin, allowing the oxygen in the air to diffuse, thereby compromising the polymerization and reducing the bond strength to the dentin substrate [14].

The µTBS difference between Scotchbond Bond Universal and Clearfil SE Bond can also be due to the presence of polyalkenoic acid copolymer in the first system. The copolymer can compete with the phosphate monomer MDP by bonding to the calcium of hydroxyapatite damaging the chemical bonding of MDP to dentin [10]. The Vitrebond copolymer could have prevented the approach of the monomers during polymerization due to its high molecular weight. Furthermore, the system Scotchbond Bond Universal is a one-step self-etch adhesive which leads to a lower concentration of monomer MDP compared to Clearfil SE Bond that has this monomer incorporated into the conditioning primer and in the adhesive resin (bond) [15].

During dentin etching, the Ca^+ ions are lost and the phosphate monomers cannot bond to them, thus affecting significantly the adhesive function of these monomers, hence, an unstable hybrid layer will be formed. Thus, the weakening of the adhesive interface of composite restorations, over time, can be associated with the presence of water inside the hybrid layer, leading to nanoleakage [3], hydrolytic degradation of the adhesive or the degradation of the demineralized collagen fibers not impregnated by the adhesive [16]. Another disadvantage of the etching agents action on dentin is the risk that, after applying the adhesive, it does not cover completely the collagen fibers that were exposed in the previous step. Such fibers become susceptible to the action of proteolytic enzymes, leading to the dissolution of the hybrid layer. These enzymes are known as endogenous metalloproteinases (MMP) and they are found in human dentin [6].

In order to obtain a more stable hybrid layer, the use of acid etching should be recommended only for enamel, since its morphological microstructure allows such adhesive treatment, thus, promoting a better marginal adhesion [13]. As dentin is a very complex substrate due to its organic part, these new materials show better performance when used in the self-etch strategy. It is important to understand that adhesive systems have inherent limitations related to the method of use, however, essential care must be taken to provide its maximum performance.

Conclusion

Within the limitations of this study, the authors conclude that All Bond Universal showed better bonding performance in both adhesive strategies when compared to Scotchbond Universal.

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N/A

Conflict of Interest

No financial interest or any conflict of interest exists.

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