

Microtensile bond strength of a universal adhesive to deep dentin

Resistência de união de um sistema adesivo universal aplicado em dentina profunda

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ABSTRACT

Objective: This study evaluated the microtensile bond strength of a universal adhesive system applied to deep dentin under different bonding strategies. **Material and Methods:** Fifteen human third molars had the coronal portion removed exposing deep dentin and were assigned into 3 groups according to the adhesive system: G1(control): 2-steps total-etch (Adper Single Bond 2, 3M ESPE); G2: 1-step self-etch universal adhesive (Scotchbond Universal, 3M ESPE); G3: 2 steps total-etch universal adhesive (Scotchbond Universal, 3M ESPE). Composite build-ups were performed on the dentin surfaces and after water storage for 24 h, teeth were sectioned to obtain 40 bonded beams per group with sectional area of 0.9 mm². The specimens were submitted to the microtensile bond strength (μ TBS) test until failure. Statistical analyses were computed using one-way ANOVA ($p = 0.05$). **Results:** The mean μ TBS (in MPa) were G1 = 22.27, G2 = 22.85 and G3 = 20.3. After statistical analysis, no significant differences were observed among the groups. **Conclusions:** Universal adhesive system performed similarly to the total-etch adhesive and was not affected by the adhesion strategy.

KEYWORDS

Microtensile; Adhesive system; Dentin.

RESUMO

Objetivo: O estudo avaliou a resistência de união à microtração de um sistema adesivo universal aplicado em dentina profunda sob diferentes estratégias adesivas. **Material e Métodos:** 15 terceiros molares tiveram a porção coronária removida, expondo dentina profunda, sendo então divididos em 3 grupos de acordo com o sistema adesivo: G1 (controle) sistema adesivo convencional de 2 passos e condicionamento ácido total (Adper Single Bond 2, 3M ESPE); G2: sistema adesivo universal, 1 passo, autocondicionante (Scotchbond Universal, 3M ESPE); G3: sistema adesivo universal, 2 passos e condicionamento ácido total (Scotchbond Universal, 3M ESPE). As porções coronárias foram reconstruídas em resina composta e após 24 h em água destilada os espécimes foram seccionados para obtenção de 40 filetes por grupo. Os filetes foram submetidos ao teste de microtração à velocidade de 0,5 mm/min e os dados analisados estatisticamente. **Results:** Não foram observadas diferenças estatísticas entre os grupos, sendo as médias de resistência de união (MPa): G1 = 22,27, G2 = 22,85 and G3 = 20,3. **Conclusão:** O sistema adesivo universal apresentou performance similar ao sistema adesivo convencional e seu desempenho não foi afetado pela estratégia adesiva utilizada.

PALAVRAS-CHAVE

Microtração; Sistemas adesivos; Dentina.

INTRODUCTION

The basic mechanism of bonding to enamel and dentin is an exchange process that involves the replacement of minerals, removed from enamel and dentin, by resin monomers that become micro-mechanically interlocked in the created porosities. [1] While bonding to enamel is a reliable technique, bonding to dentin still represents a greater challenge because of the complex composition of this tissue. [2].

Adhesive systems can be classified as total-etch or self-etch adhesives [3], in versions of three steps, two or just one step. [4]

In the total-etch adhesive systems, the first step involves the application of conditioners or acid etchants to enamel and dentin with the objective to completely remove the smear layer and simultaneously exposure of the collagen fibrils in dentin [4] and increase the surface energy in the enamel substrate. [5] The second step involves the application of primers, that are considered adhesion-promoting agents and contain monomers with hydrophobic properties for co-polymerization with the adhesive resin and hydrophilic properties that have an affinity for the exposed collagen fibril [4]. The main function of the primer is to transform the hydrophilic dentin surface into a hydrophobic surface, allowing the adhesive, to penetrate the network of collagen fibers in an efficient manner. [6] In the total-etch strategy, there is a risk of collapsing collagen fibers during drying, obstructing the interfibrillar spaces, which prevents infiltration of the adhesive. [7] Incomplete infiltration of the adhesive in the demineralized dentin can leave exposed collagen in the dentin-adhesive interface[8], which can be degraded by bacteria, compromising the integrity of the union.[9]

In order to simplify the dentin bonding, acidic monomers were combined with the bond component [10] and proved to be effective in the smear layer removing and to improve the

effectiveness of the adhesive thus, originating the self-etch adhesive systems. In these systems, the acidic part demineralizes dentin and simultaneously infiltrate it with monomers that can be polymerized *in situ* [2], therefore the whole extension of the demineralized dentin depth is likely to be impregnated by resin monomers.

Considering the differences in opinion regarding the adhesive strategy to be adopted and the number of steps, some manufactures have released most versatile adhesive systems that can be used on the self-etch strategy and also as a total-etch adhesive system. These latter materials are called “Universal”, “Multi-purpose” or “Multi-mode” adhesive systems [11]. The performance of one of these adhesive systems, ScotchBond Universal, is reported in some studies in the literature. [5,12-14]

Since most cavity preparations show not only areas of exposed superficial dentin but also of deep dentinal areas [15], several reports were performed indicating lower bond strength on deep dentin [16-19].

This laboratory study evaluated the influence of different adhesive systems on the immediate microtensile bond strength to deep dentin. The null hypothesis was that the adhesive system did not influence the bond strength.

MATERIAL AND METHODS

Once approval was obtained from the Ethical Committee of the Federal University of Santa Catarina (approval number 390.073), fifteen intact sound molars were collected and stored in 0.5% chloramine solution for up to 1 month.

The teeth were analyzed with a magnifying glass (Carl Zeiss Jena, Germany) to exclude teeth with some structural defect.

After cutting the root (slow-speed diamond saw, Buehler Wafering Blades, Buehler Ltd, Lake Bluff, IL, USA) perpendicular to the long axis, 4 mm below the enamel-dentin limit, in a cutting

machine (ISOMET 1000, Buehler Ltd, Lake Bluff, IL, USA), the pulp chamber of each tooth was cleaned and filled with resin composite, making it possible to obtain resin–dentin sticks of adequate length for the microtensile test in the region corresponding to the roof of the pulp chamber. Deep dentin (0.5-1mm over the highest pulp horn) was exposed by sectioning the crowns parallel to the occlusal surface in a slow-speed diamond saw (Buehler Wafering Blades, Buehler Ltd, Lake Bluff, IL, USA) under water-cooling.

Dentin was polished with wet 600-grit SiC abrasive paper for 60 seconds to create a standardized smear layer. Teeth were randomly assigned into three groups (n=5) according to the adhesive system and bonding strategies (Table 1).

G1 (control group): Adper Single Bond 2 (3M ESPE, St Paul, MN, USA) total-etch adhesive system. After acid etching (37% phosphoric acid, Power Etching, BM4 Materiais Odontológicos, Palhoça, SC, Brazil) for 15 seconds, the dentin surface was rinsed and dried with absorbent

paper disks. The adhesive system was applied in 2 consecutive coats for 15 seconds with gentle agitation and gently air dried for 5 seconds, followed by light-curing for 10 seconds at 750 mW/cm² (Translux Power Blue-Hareaus Kulzer GmbH-Hanau, Germany). Irradiance was monitored with a radiometer (RD-7, Ecel Ind. e Com. Ltda, Ribeirão Preto/São Paulo, Brazil).

G2: ScotchBond Universal(3M ESPE, St Paul,MN, USA) applied on dentin surface on the one step self-etch strategy with gentle agitation for 20 seconds. Then, the dentin surface was gently dried for 5 seconds, followed by light-curing for 10 seconds at 750 mW/cm².

G3: ScotchBond Universal applied on dentin surface on the total-etch strategy. After acid etching (37% phosphoric acid, Power Etching, BM4 Materiais Odontológicos, Palhoça, SC, Brazil) for 15 seconds, the dentin surface was rinsed and dried with absorbent paper disks and the adhesive system was applied as for the one step self-etch mode.

Table 1 - Materials composition and instructions for use.

Composite resins	Composition	Manufacturer's	
		Self-etch	Total-etch
Adper Single Bond 2	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.	X	<ol style="list-style-type: none"> 1. Apply etchant for 15 s. 2. Rinse for 15s. 3. Blot excess water. 4. Apply 2 consecutive coats of adhesive for 15 s with gentle agitation. 5. Gently air dry for 5 s. 6. Lightcure for 10 s.
SingleBond Universal	BisGMA, HEMA, water, ethanol, silane-treated silica, decamethylene-dimethacrylate (10MDP), 2-propenoic acid, 2-methyl, reaction products with 1,10 decanediol and phosphorous oxide (P ₂ O ₅), copolymer of acrylic and itaconic acid (Vitre-bond Copolymer), dimethylaminobenzoat (-4),CQ,(dimethylamino) ethylmethacrylate, methyl ethyl ketone, silane.	<ol style="list-style-type: none"> 1. Apply the adhesive to the entire preparation and rub it in for 20seconds. 2. Direct a gentle stream of air over the liquid for 5 seconds. 3. Light polymerize for 10 s. 	<ol style="list-style-type: none"> 1. Apply etchant for 15 s. 2. Rinse for 15 s. 3. Blot excess water. 3. Apply adhesive as for the self-etch mode.
Filtek Z-350	Bis-GMA,UDMA, TEGDMA, Bis-EMA, zirconium, silica.		
Gel etchant	37% H ₃ PO ₄ , water, fumed silica		

Abbreviations - BisGMA: bisphenol A diglycidyl methacrylate; Bis-EMA (Bisphenol A polyethylene glycol diether dimethacrylate); CQ: camphorquinone; HEMA; 2-hydroxyethyl methacrylate, 10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate.

After the bonding procedures, all teeth received a composite restoration (Filtek Z-350, 3M ESPE, St. Paul, MN, USA) in three increments of 2 mm each. Each increment was irradiated for 20 seconds at 750 mW/cm² (Translux Power Blue-Hareaus Kulzer GmbH-Hanau, Germany). When the build-up was completed, a 3 × 3 mm² square was painted in the central area of the composite occlusal surface with a colored permanent marker to allow for the selection of central bonded beams.

After the restored teeth had been stored in distilled water at 37°C for 24 hours, the specimens were sectioned longitudinally in the mesio-distal and buccal-lingual directions, using a slow-speed diamond saw (Buehler Wafering Blades, Buehler Ltd, IL, USA) to obtain beams with a cross sectional area of approximately 0.9 mm² measured with a digital caliper (KingTools, São Paulo-SP, Brazil).

Each beam was attached to a stainless steel notched Geraldeli's jig [20] using cyanoacrylate glue (Loctite, Henkel Ltd, São Paulo, SP, Brazil) and tested under tension using a universal testing machine (Instron 4444, Instron Corp., Canton, MA, USA) at 0.5 mm/minute crosshead speed until failure. The fracture load and the bonding area of the specimen were registered, and microtensile bond strength was calculated in MPa. The fractures were analyzed by two observers under a stereomicroscope (Olympus SZ40, Tokyo, Japan) at ×100 magnification. The mode of failure was classified as adhesive, mixed, and cohesive. Failures were considered adhesive when they occurred at the dentin-adhesive interface; they were of cohesive nature when the failure occurred in dentin; and of mixed nature when there was composite and dentin at the interface. [12] The results were analyzed by one-way ANOVA. The level of statistical significance was set at $p < 0.05$.

RESULTS

The analysis of variance test (ANOVA) accepted the hypothesis of equality between the groups ($p = 0.454$), ie, no statistical difference was found between groups.

Microtensile Bond Strength: The number of microtensile beams (N), specimens with premature failures (PF), and standard deviations (SD) are shown in Table 2. A small number of premature failures were observed in the present study. Specimens with premature failures (PF) were excluded from the statistical analysis.

Analysis of fracture mode: The evaluation under stereomicroscope showed that the majority of fractures, over 93%, were of adhesive nature, equally distributed among the three groups.

Table 2 - Number of beams (N), premature failures (PF), mean microtensile bond strength values and standard deviations (SD).

Groups	N	PF	Mean (MPa)	SD
G1	40	1	22.27	8.67
G2	40	2	22.85	9.73
G3	40	0	20.3	7.13

DISCUSSION

Universal adhesives represent the last generation of contemporary adhesive systems, created based on the "all-in-one" concept of one-step self-etch. This category of bonding agents allows the general practitioner to adapt them to the clinical situation by using it either as a 'etch-and-rinse' or 'self-etch' adhesive approach, depending merely on his/her interpretation of what seems most appropriate upon clinical case.

In the present study, the use of SBU following a self-etch or an etch-and-rinse protocol did not affect significantly the deep

dentin microtensile bond strength and its performance was similar to the control group.

Considering that the microtensile bond strength is related to the surface area, ie, the smaller the area the greater resistance [21], in this study no trimming specimens were tested [22], avoiding concentration of stress resulting from the preparation to obtain hourglass shape specimens. The greatest advantage of the microtensile method is that one can obtain exclusively adhesive bond failures of materials if the bonded surface area is about 1 mm². [23] In this study, the majority of the specimens showed adhesive failures. Another advantage of the microtensile method is that multiple specimens can be obtained from a single tooth. [23]

In the present study, the behavior and the composition of ScotchBond Universal suggest that it may possess an intrinsic ability to bond chemically to deep dentin.

ScotchBond Universal is considered a mild self-etch adhesive because its pH is relatively high (pH = 2.7), therefore, it demineralizes dentin only partially, leaving hydroxyapatite partially attached to collagen, enabling a chemical bond between the MDP and hydroxyapatite. [24]. Calcium ions released upon partial dissolution of hydroxyapatite diffuse within the hybrid layer and assemble MDP molecules into nano-layers. This chemical interaction between MDP and hydroxyapatite creates a stable nano-layer that could form a stronger phase at the adhesive interface, which increases the mechanical strength of the adhesive interface in the self-etch strategy. [25]

For the total-etch adhesive system Adper Single Bond 2, the major elements that contribute to bond strength are intratubular resin-tag formation and resin infiltration into demineralized intertubular dentine. [26] In

deep dentin it can be more difficult to happen because of the smaller amount of intertubular dentin to form the hybrid layer [17], therefore deep dentin is more porous and retains more water within its enlarged tubule openings, which may avoid appropriate lateral bonding of the resin tags. [19]

Both adhesive systems tested in this study contain the polyalkenoic acid copolymer, that bonds chemically to the calcium in hydroxyapatite. [27] Clinical studies have shown a good performance of copolymer containing self-etch and etch-and-rinse adhesives [28] that may be attribute to chemical bonding of these materials to hydroxyapatite.

In the present study, there was no difference between the adhesive systems tested or among the different adhesive strategies adopted for ScotchBond Universal, with the average bond strength 22.27 MPa, 22.85 MPa and 20.3 MPa for G1, G2 and G3 respectively. Such bond strength values were lower than those reported by Perdigão *et al.* [12] and Muñoz *et al.* [5]. The lowest result obtained in this study can be explained by the different dentin depths tested by the others authors, middle and superficial respectively. The lower content of calcium present in deep dentin [16] for chemical bond with MDP and with polyalkenoic acid copolymer and the increased permeability [18] found in deep dentin may explain the lower value of bond strength.

Considering the “universal application” concept, the application mode should not impair the ScotchBond Universal bond strength. When clinically tested on noncarious cervical lesions, Mena-Serrano *et al.* [13] and Perdigão *et al.* [14] also found no significant differences in the different adhesive strategies used for ScotchBond Universal. In the present study, the similar bonding performance of the ScotchBond Universal after the different adhesive strategy

suggests its reliability when applied under different clinical situations.

As the bond strength was measured upon 24-h water storage, it should be considered as 'immediate' bond strength test. Thermal fatigue was not performed in our study because the susceptibility of adhesives to this type of fatigue depends on the specific composition of each adhesive. [29] Future studies in our laboratory will evaluate the effect of long-term water storage on the *in vitro* performance of the universal adhesive system.

With regard to the degree of moisture of the dentin, in the present study the dentin was kept moist after acid etching, according to the manufacturer's instructions.

Both adhesive systems contain water in the composition, capable to re-expand the collagen network collapsed by the air-drying almost to the original level, allowing better penetration of resin monomers. [30]

In the present study, the similar bonding performance of the ScotchBond Universal after the different adhesives strategies indicates its reliability when working under different clinical situations. Moreover, this Universal adhesive system showed similar performance to the control – group, thus, the null hypothesis was accepted.

REFERENCES

- Nakabayashi N, Kojima K, Masuhara E. The promotion of adhesion by the infiltration of monomers into tooth substrates. *J Biomed Mater Res.* 1982;16 (3):265-73.
- Perdigão, J, Lopes M. Dentin bonding questions for the new millennium. *J Adh Dent.* 1999; 1(3):191-209.
- De Munck J, Van Landuyt K, Peumans M, Poitevin A, Lambrechts P, Braem M, Van Meerbeek B. A critical review of the durability of adhesion to tooth tissue: methods and results. *J Dent Res.* 2005; 84:118-132.
- Meerbeek B, Perdigão J, Lambrechts P, Vanherle G. The clinical performance of adhesives. *J of Dent.* 1998; 26(1): 1-20.
- Muñoz MA, Luque I, Hass V, Reis A, Loguercio AD, Bombarda NH. Immediate bonding properties of universal adhesives to dentine. *J Dent.* 2013; 41: 404-11.
- Nakabayashi N, Takarada K. Effect of HEMA on bonding to dentin. *Dent Mat.* 1992; 8:125-130.
- Tay FR, Gwinnett JA, Wey SH. Micromorphological spectrum from overdrying to overwetting acid-conditioned dentin in water-free acetone-based, single-bottle primer/adhesives. *Dent Mater.* 1996; 12: 236-44.
- Spencer P, Swafford J. Unprotected protein at the dentin-adhesive interface. *Quint Intern.* 1999; 30:501-7.
- Eick JD, Gwinnett AJ, Pashley DH, Robinson SJ. Current concepts on adhesion to dentin. *Crit Rev Oral Biol Med.* 1997; 3:306-35.
- Hasegawa T, Manabe A, Itoh K, Wakumoto S. Investigation of self-etching dentin primers. *Dent Mater.* 1989; 5(6): 408-10.
- Hanabusa M, Mine A, Kuboki T, Momoi Y, Van Ende A, Van Meerbeek B, De Munck J. Bonding effectiveness of a new 'multi- mode' adhesive to enamel and dentine. *J Dent.* 2012; 40: 475-84.
- Perdigão J, Sezinado A, Monteiro P. Laboratory bonding ability of a multi-purpose dentin adhesive. *Am J Dent.* 2012; 25(3):153-8.
- Mena-Serrano A, Kose C, De Paula E, Tay LY, et al. A new universal simplified adhesive – 6 month clinical evaluation. *J Esth Rest Dent.* 2013; 25(1):55-9.
- Perdigão J, Kose C, Mena-Serrano AP, De Paula EA, Tay LY, Reis A, Loguercio AD. A New Universal Simplified Adhesive: 18-Month Clinical Evaluation. *Op Dent.* 2013; 38(6):113-27.
- Kaaden C, Powers JM, Friedl KH, Schmalz G. Bond strength of self-etching adhesives to dental hard tissues. *Clin Oral Investig* 2002; 6(3):155-60.
- Causton BE. Improved bonding of composite restorative to dentine. *Br Dent J.* 1984; 156: 93-95.
- Suzuki T, Finger WJ. Dentin adhesives: site of dentin vs. bonding of composite resins. *Dent Mater.* 1988; 4(6): 379-83.
- Tagami J, Tao L, Pashley DH. Correlation among dentin depth, permeability, and bond strength of adhesive resins. *Dent Mater.* 1990; 6:45-50.
- Giannini, M, Carvalho RM, Martins LR, Dias CT, Pashley DH. The influence of tubule density and area of solid dentin on bond strength of two adhesive systems to dentin. *J Adh Dent.* 2001; 3(4): 315-24.
- Perdigão J, Geraldini S, Carmo AR, Dutra, HR. In vivo influence of residual moisture on microtensile bond strengths on one-bottle adhesives. *J Esthet Restor Dent.* 2002;14:31-8.
- Sano H, Shono T, Sonoda H, Takatsu T, Ciucchi B, Carvalho R, Pashley DH. Relationship between surface area for adhesion and tensile bond strength: evaluation of a micro-tensile bond test. *Dent Mater.* 1994; 10(4):236-40.
- Shono Y, Ogawa T, Terashita M, Carvalho RM, Pashley EL, Pashley DH. Regional measurement of resin-dentin bonding as an array. *J Dent Res.* 1999; 78(2):699-705.
- Pashley DH, Sano H, Ciucchi B, Yoshiyama M, Carvalho RM. Adhesion testing of dentin bonding agents: a review. *Dent Mat.* 1995; 11(2):117-25.
- Yoshida Y, Nagakane K, Fukuda R, Nakayama Y, Okazaki M, Shintani H, Inoue S, Tagawa Y, Suzuki K, De Munck J, Van Meerbeek B. Comparative study on adhesive performance of functional monomers. *J Dent Res.* 2004; 83: 454-8.

25. Yoshida Y, Yoshihara K, Nagaoka N, Hayakawa S, Torii Y, Ogawa T, Osaka A, Meerbeek BV. Self-assembled nano-layering at the adhesive interface. *J Dent Res.* 2012; 91: 376-81.
26. Pashley DH, Ciucchi B, Sano H, Carvalho RM, Russell CM. Bond strength versus dentine structure: a modelling approach. *Arch Oral Biol.* 1995; 40:1109-18.
27. Mitra SB, Lee CH, Bui HT, Tantbirojn D, Rusin RP. Long-term adhesion and mechanism of bonding of a paste-liquid resin-modified glass-ionomer. *Dent Mater* 2009; 25: 459-66.
28. Bittencourt DD. An 18-months evaluation of self-etch and etch & rinse adhesive in non-carious cervical lesions. *Acta Odontol Scand.* 2005; 63:173-8.
29. Perdigão J, Gomes G, Sezinando A Bonding ability of three ethanol-based adhesives after thermal fatigue. *American Journal of Dentistry.* 2011;24(3) 159-64.
30. Van Meerbeek B, Inokoshi S, Braem M, Lambrechts P, Vanherle G. Morphological aspects of the resin-dentin interdiffusion zone with different dentin adhesive systems. *J Dent Res.* 1992; 71:1530-40.

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