**Microtensile Bond Strength of a Universal Adhesive to Deep Dentin**

**Introduction**

The basic mecanism 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 porosities1. While bonding to enamel is a reliable technique, bonding to dentin still represents a greater challenge because of the complex composition of this tissue2.

Adhesive systems can be classified as total - etch or self - etch adhesive 3, in versions of three steps, two or just one step4.

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 substrate5. 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 fibril4. 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 manner6. In the total-etch strategy, there is a risk of collapsing collagen fibers during drying, obstructing the interfibrillar spaces, which prevents infiltration of the adhesive7.Incomplete infiltration of the adhesive in the demineralized dentin can leave exposed collagen in the dentin-adhesive interface8, which can be degraded by bacterias, compromising the integrity of the union9.

In order to simplify the dentin bonding, acidic monomers were combined with the bond component10 and proved to be effective in the smear layer removing and to improve the effectiveness of the adhesive thus, originating the self-etch adesive systems. In these systems, the acidic part demineralizes dentin and simultaneously infiltrate it with monomers that can be polimerized *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 system5. This latter materials are called “Universal’’, ‘‘Multi-purpose’’ or ‘‘Multi-mode’’ adhesive systems11. The performance of one of these adhesive systems, ScotchBond Universal, is reported in some studies in the literature5,12,13,14.

Since most cavity preparations show not only areas of exposed superficial dentin but also of deep dentinal areas15, several reports were performed indicating lower bond strenght on deep dentin16, 17,18,19.

This laboratory study evaluated the influence of different adhesive systems in the 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 excludeteeth 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 consecutives coats for 15 seconds with gentle agitation and gently air dried for 5 seconds, followed by light-curing for 10 seconds at 750mW/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 750mW/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.**

|  |  |  |  |
| --- | --- | --- | --- |
|  | Composition | Manufacter’s | instructions |
|  |  | Self-etch | Total- etch |
| Adper  Single Bond 2 | Bis-GMA; HEMA, dimethacrylates,  ethanol,water,pho-  toinitiator, metha-  crylate functional copolymer of  polyacrylic and poly(itaconic) acids,  10% by weight of  5 nm-diameter sphe-  rical silica particles. | X | 1. Apply etchant for 15 s .  2. Rinse for 15s .  3. Blot excess water.   4. Apply 2 con-secutive coats of adhesive for 15 s with gentle agitation.  5. Gently air dry for 5 s .  6.Light poly-merize for 10s . |
| SingleBond Universal | BisGMA,HEMA,water,  ethanol,silane-treated silica,decamethylene-dimethacrylate(10MDP),  2-propenoic acid, 2-me-thyl, reaction products with 1,10 decanediol and phosphorous oxide (P2O5), copolymer of acrylic and itaconic acid (Vitre-bond Copolymer),  dimethylaminobenzoat  (-4),CQ,(dimethyla-mino) ethylmetha-crylate, methyl ethyl ketone, silane. | 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. | 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% H3PO4, 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 750mW/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 370C 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 jig20 using cyanoacrylate glue (Loctite, Henkel Ltd, São Paulo, SP, Brazil) and tested under tension using an 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 strengths were 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 interface12. 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%, was 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**

The null hypothesis was acceptedas there were no statisticaldifferences in dentin microtensile bond strength among the different adhesion strategies for ScotchBond Universal and the control group.

Considering that the microtensile bond strength is related to the surface area, ie, the smaller the area the greater resistence21, in this study no trimming specimens were tested22, 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 1mm2 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 tooth23.

In the present study, the behavior and the composition of ScotchBond Universal suggests 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 hydroxyapatite24. 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 strategy25.

For the total-etch adhesive system Adper Single Bond 2, the major elements which contribute to bond strength are intratubular resin-tag formation and resin infiltration into demineralized intertubular dentine26. In deep dentin it can be more difficult to happen because of the smaller amount of intertubular dentin to form the hybrid layer17, therefore deep dentin is more porous and retains more water within its enlarged tubule openings, which may avoid appropriate lateral bonding of the resin tags19.

Both adhesive systems tested in this study contain the polyalkenoic acid copolymer, that bonds chemically to the calcium in hydroxyapatite27.Clinical studies have shown a good performance of copolymer containing self-etch and etch-and-rinse adhesives28 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 dentin16 for chemical bond with MDP and with polyalkenoic acid copolymer and the increased permeability18 found in deep dentin may explain the lower value of bond strength.

When clinicaly 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.

With regard to the degree of moisture of the dentin, in the present study the dentin was kept moist after acid etchig, according to the manufacter’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 monomers29.

In the present study the ScotchBond Universal adhesive system showed similar performance to the control – group, thus, the null hypothesis was accepted.

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