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Influence of a hydrophobic resin coating on the bonding efficacy of three universal adhesives

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ABSTRACT

Objectives: To evaluate the effect of an additional hydrophobic resin coating (HE) on the resin–dentine microtensile bond strengths (μTBS), nanoleakage (NL), and *in situ* degree of conversion (DC) of three universal adhesives used in the etch-and-rinse (ER) and the self-etch (SE) modes.

Methods: Sixty caries-free extracted third molars were divided into 12 groups according to the combination of the factors adhesive (All-Bond Universal [ABU]; G-Bond Plus [GBP] and Scotchbond Universal [SBU]), adhesive strategy (ER and SE), and the use of HE (Heliobond; yes or no). After restorations were constructed, specimens were stored in water (37 °C/24 h) and sectioned into resin–dentine beams (0.8 mm^2) to be tested under tension (0.5 mm/min). Selected beams from each tooth were used for DC quantification and for NL evaluation. Data from each adhesive were analyzed with two-way ANOVA and Tukey's test ($\alpha = 0.05$).

Results: ABU and GBP resulted in higher μTBS in the ER mode. The use of HE increased the μTBS of ABU and GBP only in the SE mode. Lower NL was observed for SBU and ABU in the ER mode + HE, and for GBP in the SE mode + HE. SBU and GBP showed higher DC when used in the ER mode, which was increased with HE application. The DC of ABU was similar in all conditions.

Conclusions: The conversion of 1-step SE to 2-step SE may increase the μTBS and DC of current universal adhesives. The reduction in the NL is more dependent on the adhesive composition than on the bonding strategy.

1. Introduction

For each of the two current adhesive strategies dental manufacturers have introduced simplified adhesive systems, known as 2-step etch-and-rinse [ER] and 1-step self-etch [SE] adhesives,[1,2] making them more attractive to the clinician and reducing the sensitivity of the application technique. This task of simplification was possible through the inclusion of hydrophilic monomers and the increase in the amount of

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solvents, to make adhesives compatible with the inherent wet dentine substrate.[3,4] Recently, newly universal or multi-mode adhesive systems were introduced with manufacturers' claims that one monomer solution can be used for either adhesive strategy[5,6] without compromising the bonding effectiveness,[7] therefore being able to replace existing simplified adhesives.

The increased amount of solvents and hydrophilic monomers in the adhesive formulations lead to greater amount of residual solvents entrapped in the adhesive layer.[4] The accumulation of hydrophilic monomers and especially residual solvents may hinder the formation of a high cross-linking polymer,[8–10] decreasing the degree conversion (DC)[11] which may reduce resin–dentine bond strengths,[12–15] and increase the permeability of the adhesive layer after polymerization.[16,17] Consequently, the resulting polymers will be more susceptible to degradation over time.[2,18]

One of the methods used to circumvent these drawbacks includes the application of an additional layer of a hydrophobic resin coating over the polymerized simplified adhesive.[19] This extra resin coat aims at increasing the thickness and uniformity of the adhesive layer, as well as to reduce the fluid flow across the adhesive interface.[19,20] Excellent *in vitro* and clinical results have been reported after placement of an hydrophobic resin coating over 1-step SE adhesives[21,22]; however this technique has not been tested with new universal or multi-mode adhesives. Thus, the aim of this study was to compare the immediate resin–dentine microtensile bond strengths, nanoleakage and *in situ* degree of conversion of three universal adhesives with or without an additional hydrophobic resin coating. We tested the null hypothesis that the application of a hydrophobic resin coat on the cured adhesives will not influence the selected properties of the universal adhesives systems used in both adhesive strategies.

2. Materials and methods

Sixty caries-free extracted human third molars were disinfected in 0.5% chloramine, stored in distilled water and used within six months after extraction. The teeth were collected after obtaining the patients' informed consent under a protocol approved by the local Ethics Committee Review Board under the protocol number 17878/11.

A flat occlusal dentine surface was exposed in all teeth after wet-grinding the occlusal enamel with # 180 grit SiC paper. The exposed dentine surfaces were further polished with wet # 600-grit silicon-carbide paper for 60 s to standardize the smear layer.[23]

2.1. Experimental design and specimen preparation

The specimens were randomly assigned to twelve experimental conditions ($n = 5$) resulting from the combination of the factors "adhesive system" (Scotchbond Universal Adhesive [SBU, 3M ESPE, St. Paul, MN, USA, also known as Single Bond Universal in some countries], All-Bond Universal [ABU, Bisco Inc., Schaumburg, IL, USA] and G-Bond Plus [GBP, GC Corporation Tokyo, Japan – also known as G-ænial Bond]) (Table 1); "adhesive strategy" (etch-and-rinse [ER] or self-etch [SE]); and hydrophobic resin coating (yes or no; HE, Heliobond, Ivoclar Vivadent, Schaan, Liechtenstein).

The adhesive systems were applied according to the respective manufacturers' instructions (Table 1), except for G-Bond Plus, for which the manufacturer does not recommend dentine etching with phosphoric acid (Table 1). Composite resin crowns were built with a nanofilled composite (Filtek Z350 XT, 3M ESPE, St. Paul, MN, USA; also named Filtek Supreme XTE or Filtek Supreme Ultra in some countries) in two increments of 2 mm each. Each increment was light-cured for 40 s using a LED light-curing unit set at 1200 mW/cm^2 (Radii-cal, SDI Limited, Bayswater, Victoria, Australia).

After storage in distilled water for 24 h at 37 °C, the specimens were longitudinally sectioned in mesio-distal and buccal–lingual directions across the bonded interface with a slow-speed diamond saw (Isomet Ltd., Buehler Ltd., Lake Bluff, IL, USA) to obtain resin–dentine beams with a cross sectional area of approximately 0.8 mm^2 measured with a digital calliper (Digimatic Caliper, Mitutoyo, Tokyo, Japan). All specimens from each tooth were used for the μTBS test, except four that were randomly selected for measurement of nanoleakage (NL) and *in situ* DC.

2.2. Microtensile bond strength (μTBS)

The resin–dentine bonded beams were attached to a Geraldeli jig[24] (Odeme Biotechnology, Joaçaba, SC, Brazil) with cyanoacrylate adhesive and tested under tension (Model 5565, Instron Co., Canton, MA, USA) at 0.5 mm/min until failure. The μTBS values were calculated by dividing the load at failure by the cross-sectional bonding area.

The failure mode was classified as cohesive [C] failure (exclusively within dentine or resin composite), adhesive [A] failure (at the resin/dentine interface), or mixed [M] failure (at the resin/dentine interface that included cohesive failure of the neighbouring substrates). The failure mode analysis was performed under a stereomicroscope at 100× magnification (Olympus SZ40, Tokyo, Japan). Specimens with premature failures (PF) were included in the tooth mean as zero MPa and those with cohesive failures were excluded.

2.3. Nanoleakage (NL)

Two resin-bonded beams from each tooth were used for NL evaluation. Ammoniacal silver nitrate was prepared according to the protocol previously described by Tay et al.[25] The beams were placed in the ammoniacal silver nitrate solution in darkness for 24 h, rinsed thoroughly in distilled water, and immersed in photo developing solution for 8 h under a fluorescent light to reduce silver ions into metallic silver grains. Specimens were polished with wet 600-, 1000-, 1200-, 1500-, 2000- and 2500-grit SiC paper and 0.25 μm diamond paste (Buehler Ltd., Lake Bluff, IL, USA) using a polishing cloth. Specimens were then ultrasonically cleaned, air dried, mounted on Al stubs, and coated with carbon-gold (MED 010, Balzers Union, Balzers, Liechtenstein). Resin–dentine interfaces were analyzed in a field-emission scanning electron microscope operated in the backscattered mode (LEO 435 VP, LEO Electron Microscopy Ltd., Cambridge, UK).