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# Effects of solvent evaporation time on immediate adhesive properties of universal adhesives to dentin

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## ABSTRACT

**Objective.** To evaluate the microtensile bond strengths ( $\mu$ TBS) and nanoleakage (NL) of three universal or multi-mode adhesives, applied with increasing solvent evaporation times.

**Methods.** One-hundred and forty caries-free extracted third molars were divided into 20 groups for bond strength testing, according to three factors: (1) Adhesive – All-Bond Universal (ABU, Bisco, Inc.), Prime&Bond Elect (PBE, Dentsply), and Scotchbond Universal Adhesive (SBU, 3M ESPE); (2) Bonding strategy – self-etch (SE) or etch-and-rinse (ER); and (3) Adhesive solvent evaporation time – 5 s, 15 s, and 25 s. Two extra groups were prepared with ABU because the respective manufacturer recommends a solvent evaporation time of 10 s. After restorations were constructed, specimens were stored in water (37 °C/24 h). Resin–dentin beams (0.8 mm<sup>2</sup>) were tested at 0.5 mm/min ( $\mu$ TBS). For NL, forty extracted molars were randomly assigned to each of the 20 groups. Dentin disks were restored, immersed in ammoniacal silver nitrate, sectioned and processed for evaluation under a FESEM in backscattered mode. Data from  $\mu$ TBS were analyzed using two-way ANOVA (adhesive vs. drying time) for each strategy, and Tukey's test ( $\alpha = 0.05$ ). NL data were computed with non-parametric tests (Kruskal–Wallis and Mann–Whitney tests,  $\alpha = 0.05$ ).

**Results.** Increasing solvent evaporation time from 5 s to 25 s resulted in statistically higher mean  $\mu$ TBS for all adhesives when used in ER mode. Regarding NL, ER resulted in greater NL than SE for each of the evaporation times regardless of the adhesive used. A solvent evaporation time of 25 s resulted in the lowest NL for SBU-ER.

**Significance.** Residual water and/or solvent may compromise the performance of universal adhesives, which may be improved with extended evaporation times.

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## 1. Introduction

New multi-mode or universal one-bottle adhesives have been recently introduced for use as either self-etch or as etch-and-rinse adhesives [1]. The respective manufacturers suggest that multi-mode adhesives may also be used with separate etching of enamel margins, or selective enamel etching.

Due to the intrinsic wetness of the dentin substrate, hydrophilic monomers have been used in the composition of dentin bonding systems for years [2,3]. Hydrophilic resins result in high dentin bond strengths. However, several studies have demonstrated that degradation of the resin–dentin interface occurs over time [4,5]. It has been questioned whether current monomers have become too hydrophilic [6]. In fact, all self-etch adhesives, including the newest universal adhesives, contain water, which is required for ionization of the hydrophilic acidic monomers [7].

Their hydrophilicity makes one-step self-etch adhesives behave as semi-permeable membranes, allowing fluid transudation across the resin–dentin interface [8]. Some etch-and-rinse adhesives also contain water and hydrophilic monomers, which makes them behave as permeable membranes as well, allowing exudation of dentin fluid [9]. The presence of residual water may accelerate the degradation of the bonding interface [3,10].

Commercial dental adhesives include organic solvents, such as ethanol or acetone, to facilitate monomer infiltration into the humid dentin substrate. Although water and organic solvents are essential components of one-step adhesives, solvents should be completely removed during clinical application of the adhesive. If solvents are not evaporated, residual water and organic solvents may inhibit the polymerization of monomers in current dentin adhesives [11].

Solvent evaporation is usually accomplished by agitating the adhesive on dentin/enamel surfaces followed by solvent evaporation with compressed air [12]. An extended solvent evaporation time has been used to successfully improve the degree of conversion and mechanical properties of 1-step self-etch and 2-step etch-and-rinse adhesives [13,14]. There is no consensus, however, regarding the proper solvent evaporation time for 1-step self-etch [13,15,16], or for 2-step etch-and-rinse adhesives [17–19].

Taking into account that new universal adhesives contain both water and, at least, one organic solvent (ethanol or acetone), the aim of this study was to compare the immediate microtensile bond strengths ( $\mu$ TBS) and nanoleakage (NL) of three universal or multi-mode adhesives, applied with increasing solvent evaporation times. The null hypotheses tested were that extended solvent evaporation time would not improve: (1) the immediate bond strengths of universal adhesives and; (2) the sealing ability of resin–dentin interfaces formed with universal adhesives.

## 2. Material and methods

### 2.1. Tooth selection and preparation

One hundred and forty extracted, caries-free human third molars were used. The teeth were collected after obtaining

the patient's informed consent under a protocol approved by the local Ethics Committee Review Board. The teeth were disinfected in 0.5% chloramine, stored in distilled water and used within six months after extraction.

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

### 2.2. Experimental design, restorative procedure and specimen preparation

Teeth were randomly assigned into 20 groups ( $n=7$ ) according to the adhesive strategy and different solvent evaporation times of three universal adhesive systems: All-Bond Universal (ABU – Bisco Inc., Schaumburg, IL, USA); Prime&Bond Elect (PBE – Dentsply Caulk, Milford, DE, USA); and Scotchbond Universal Adhesive (SBU – 3M ESPE, St. Paul, MN, USA).

Each adhesive was applied (1) as etch-and-rinse (ER) adhesive or as self-etch (SE) adhesive; and (2) with three adhesive solvent evaporation times (5 s, 15 s, and 25 s). Two extra groups were tested to include the recommended manufacturer's solvent evaporation time of 10s for ABU in both adhesive strategies. All details regarding the adhesive composition are displayed in Table 1.

Solvent evaporation was accomplished with an oil-free air-water syringe. The air pressure was adjusted to 1 bar using a pressure regulator, and the air nozzle was held at 45° to the dentin surface at a distance of 1.5 cm. The adhesive systems were applied as per the respective manufacturer's instructions, except for the different experimental solvent evaporation times. Please refer to Table 1 for more details.

After the bonding procedures, a nanofilled composite restoration (Filtek Z350, 3M ESPE, St. Paul, MN, USA) was built in two increments of 2 mm. Each increment was light polymerized for 40 s using a LED light-curing unit set at 1200 mW/cm<sup>2</sup> (Radii-cal, SDI Limited, Bayswater, Victoria, Australia).

### 2.3. Microtensile bond strength ( $\mu$ TBS)

After storage in distilled water for 24 h at 37°C, one-hundred restored teeth ( $n=5$  for each experimental group) were sectioned longitudinally in a mesio-distal and buccal-lingual directions across the bonded interface with a low-speed diamond saw (Isomet, Buehler Ltd, Lake Bluff, IL, USA) with water irrigation to obtain resin–dentin beams with a cross sectional area of approximately 0.8 mm<sup>2</sup> measured with a digital caliper (Digimatic Caliper, Mitutoyo, Tokyo, Japan).

Resin–dentin bonded beams were attached to a Geraldini jig [20] (Odeme Biotechnology, Joaçaba, SC, Brazil) with cyanoacrylate adhesive and tested under tension (Model 5565, Instron, Norwood, MA, USA) at 0.5 mm/min until failure. The  $\mu$ TBS values (MPa) 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 dentin or resin composite), adhesive ([A] failure at the resin/dentin interface), or mixed ([M] failure at the resin/dentin interface that included cohesive failure of the neighboring substrates). The failure mode analysis was

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