

LONG-TERM BOND STRENGTH OF A MULTIMODE ADHESIVE TO DENTIN

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To the Editor,

The long-term survival of composite resin restorations depends on the bonding efficiency and the ability to achieve a satisfactory marginal seal at the cavosurface (1). The durability of the adhesive layer is therefore of considerable concern. The challenges of achieving predictable adhesion to dentin differ greatly from the challenges of achieving adhesion to enamel due to the histologic differences between the two surfaces (2-4). Manufacturers have recently introduced universal adhesives (multimode one-bottle adhesives), which have gained popularity due to their versatility in usage strategies/modes. The most studied adhesive, Scotchbond™ Universal adhesive (SU; 3M ESPE, St. Paul, MN, USA), penetrates the demineralized dentin more deeply in the etch-and-rinse (ER) mode than in the self-etch (SE) mode (5). The quality of SU is partly due to 10-methacryloyloxydecyl-dihydrogen-phosphate (10-MDP), a phosphate monomer that provides acidity to the adhesive, enables it to etch, and bonds ionically to dentin more effectively than other functional monomers (6). Conflicting reports are found in the literature regarding the immediate (3-4, 7) and long-term performance of these multimode adhesives (3-4). These, together with limited clinical

data, suggest that the performance of universal adhesives has not yet been fully validated. A systematic review revealed a large number of papers related to the static bond strength of these systems to enamel, and only few studies have examined the bond strength to dentin. However, the most important questions of long-term performance and possible deterioration were not addressed (6, 8-9).

The aim of the current study was to evaluate the shear bond strength (SBS) of SU to dentin, using either SE or ER modes, after short- and long-term incubation periods, relative to a two-step ER adhesive used as a control.

MATERIALS AND METHODS

Tooth preparation and study materials

Forty-eight caries-free, freshly extracted third molars from healthy, non-smoking patients were selected. The study protocols were approved by the Tel Aviv University Ethics Committee (# 160514), and all patients signed an informed consent form. The teeth were cleaned with hand cures to eliminate soft tissue remnants and/or calculus and then stored in tap water containing 0.01% thymol (Sigma-Aldrich, Rehovot, Israel) at 5°C until the experiment was performed. The teeth were embedded

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parallel to the long axis using a custom-designed alignment apparatus. Each tooth was suspended in the middle of an aluminum ring (25 mm × 20 mm) and mounted 2 mm apical to the cement enamel junction (CEJ) in poly methyl methacrylate (PMMA) resin (Quick Resin, Ivoclar, Schaan, Liechtenstein) (Fig. 1A).

The lingual and buccal superficial enamel was removed using a rigidly secured high-speed hand piece equipped with a diamond bur under air-water irrigation (F1-Strauss, Ra'anana, Israel) to expose a flat dentin surface. The high-speed hand piece was mounted on a custom-designed surveyor-like apparatus (6.5° taper) (Fig. 1, B and C). A new bur was used for each tooth. SU multimode adhesive (3M ESPE, St. Paul, MN, USA) was used in both the ER and SE modes. The control adhesive was the two-step ER adhesive Adper Single Bond 2 (SB; 3M ESPE, St. Paul, MN, USA).

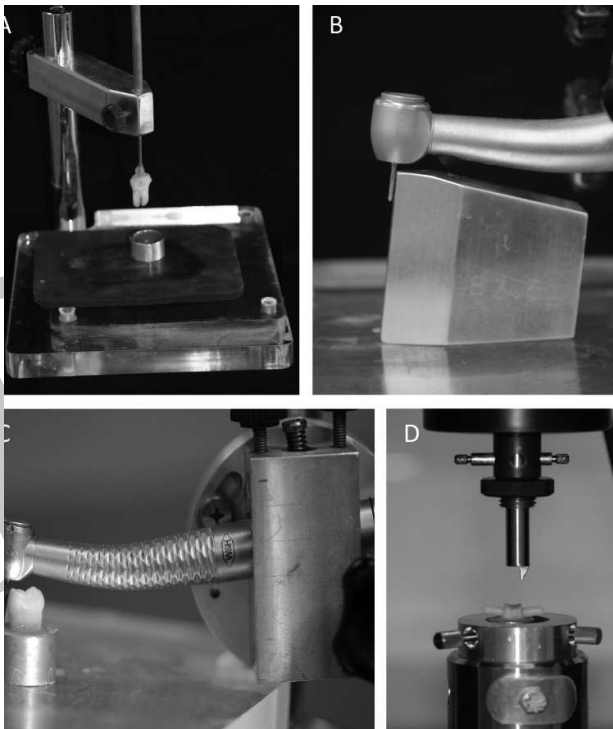


Fig. 1. Sample preparation. *A)* Teeth were suspended in the middle of an aluminum ring and mounted 2 mm apical to the CEJ in a PMMA resin. *B)* A high-speed hand piece was mounted on a custom-designed surveyor-like apparatus with a jig to ensure a 6.5° taper. *C)* The lingual and buccal superficial enamel was removed, exposing a flat dentin surface. *D)* The specimen was loaded into a universal loading machine.

Experimental design

The 48 prepared teeth (96 surfaces) were randomly assigned to 2 experimental and 1 control groups according to the adhesive used and its mode of application. Adhesive systems were applied to the dentin surfaces in accordance with the manufacturer's instructions (Table I) and light-cured using a LED light-curing unit (Elipar FreeLight 2, 3M ESPE, St. Paul, MN, USA) with a light intensity of 600 mW/cm². The light intensity was monitored with a curing radiometer (model 100, Demetron, Kerr, Danbury, CT, USA). Gelatin capsules with an inner diameter of 6 mm (10 mm long) (Torpac, Inc. NJ, USA) were incrementally filled with resin composite (Filtek Supreme XT, 3M ESPE, St. Paul, MN, USA) to 1 mm short of their margins and light-cured for 40 s from each side. The remaining 1 mm of the capsule was filled with the resin composite and was approximated to the adhesive-treated dentin surface. The specimens were clamped in a bonding jig to ensure that the capsule was oriented at 90° to the dentin surfaces. Excess material was removed, and the final curing was conducted for 20 s from 3 different directions. All specimens were incubated in 37°C distilled water. Half of the specimens in each group (8 specimens, 16 surfaces) were tested after one week, while the other half was tested after 6 months. The latter group was subjected to thermocycling for 10,000 cycles between 5 and 55°C with a 10 s dwell time (TC-2000 automatic device, Y Manes, Tel Aviv, Israel).

Shear bond strength testing

SBS testing was performed according to ISO 29022 (10) (using the notched-head SBS test). The specimens were loaded in a universal loading machine (Instron, Model 4502, Buckinghamshire, England) with a crosshead speed of 1.0 mm/min until failure (Fig. 1D). The SBS (in MPa) was calculated as the force exerted $(MPa) = \frac{FB + FL}{2\pi R^2}$ by the surface area: F

Failure analysis

After testing, the debonded dentin surfaces and resin composite cylinders were observed under an optical microscope at 18× magnification (M8 stereo microscope, Wild, Heerbugg, Switzerland). The modes of failure were classified as follows: cohesive failure (either within the composite resin or within the adhesive layer), adhesive

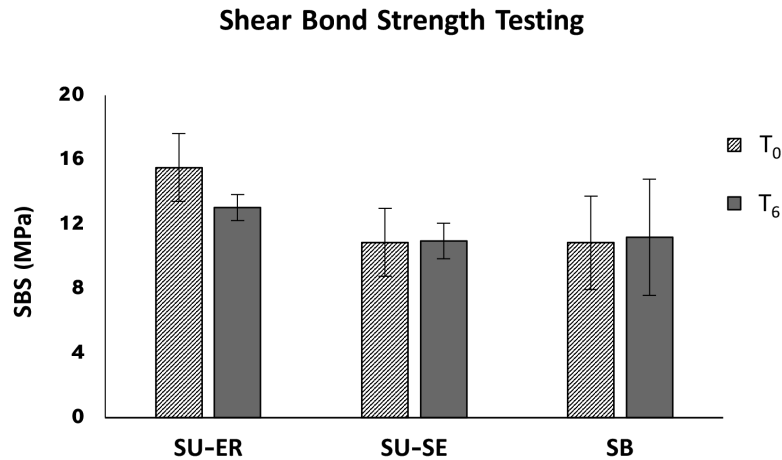


Fig. 2. Shear bond strength (MPa) after one week (T₀) and 6 months (T₆). SU: Scotchbond™ Universal; ER: etch-and-rinse; SE: self-etch; SB: Adper Single Bond 2.

failure (either between the adhesive and dentin or between the adhesive and composite) or mixed failure. For each category, the number of surfaces was summed, and the results are presented as a percentage of all surfaces exposed to the specific treatment (11).

Selected representative specimens were examined by scanning electron microscopy (SEM, JSM 840A, JEOL) using a Scandium SEM imaging platform (Olympus). The samples were fixed in 2.5% glutaraldehyde in PBS, washed, and dehydrated by ethanol treatment. After reaching the critical drying point, the samples were mounted on aluminum stubs and sputter-coated (E5100, Polaron) with gold. Images were captured at an operating voltage of 10 kV and a working distance of 8 mm.

Statistical analysis

Statistical analyses were performed using SPSS. A paired *t*-test showed no statistically significant difference between the SBS of the buccal and lingual surfaces ($p=0.365$). Accordingly, the average SBS of the two surfaces of each tooth was recorded as the representative value.

Two-way ANOVA was conducted. The dependent variable was the SBS, and the independent variables were the adhesive and time. Tukey's post hoc test was performed to analyze subgroup differences. The level of significance was set at 0.05.

RESULTS

Shear bond strength

The SBS results are shown in Fig. 2. Two-way ANOVA revealed a significant difference between the adhesive systems ($p=0.001$) and a nonsignificant difference between the incubation times ($p=0.305$). The interaction of time X material was also nonsignificant ($p=0.178$). The highest SBS value at T₀ was obtained with SU-ER (15.5±2.1 MPa) and was significantly higher than the values obtained with SU-SE (10.87±2.1 MPa) and SB (10.85±2.92 MPa). No significant difference was observed between the latter two methods. A similar pattern was obtained at T₆. SU-ER yielded the highest SBS value (13.02±0.83 MPa), which was significantly higher than the values obtained with the SU-SE and SB adhesive strategy (10.94±1.09 and 11.17±3.59 MPa, respectively). No significant difference was observed between the latter two methods.

Mode of failure analysis

The frequencies of the failure modes for the different adhesive systems at T₀ and T₆ are presented in Fig. 3A. For SU-ER, mixed failures were dominant, and very few pure adhesive failures

Table I. Application protocol for the tested adhesives.

Adhesive system	Application protocol
SB	The etchant was applied to the dentin for 15 s, followed by 10 s of water spray and medium air spray. Two coats of adhesive were then applied to the etched dentin with a rubbing action for 15 s without light curing between coats, followed by a gentle air stream (3 bar) for 5 s. Light irradiation was applied for 10 s.
SU-SE	The adhesive was applied to the air-dried dentin surface with a rubbing action for 20 s, followed by gentle air pressure (3 bar) for 5 s. Light irradiation was then applied for 10 s.
SU-ER	The etchant was applied to the dentin for 15 s, followed by water spray for 10 s and medium air spray for 2 s. Adhesive was then applied to the dentin surface with a rubbing action for 20 s, followed by medium air pressure for 5 s. Light irradiation was then applied for 10 s.

SB: Adper Single Bond 2; SU-SE: Scotchbond™ Universal/self-etch mode; SU-ER: Scotchbond™ Universal/etch-and-rinse mode

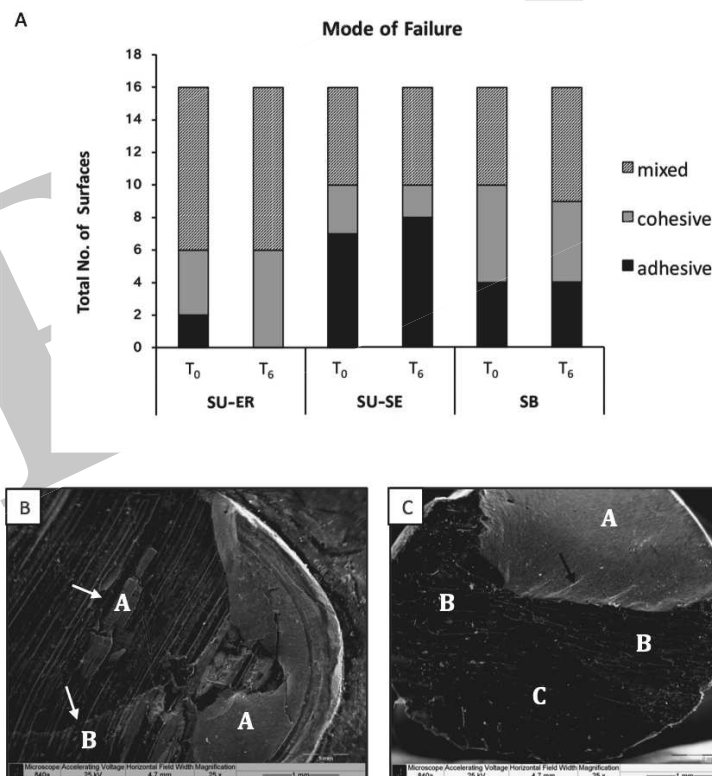


Fig. 3. Frequencies of failure modes for the different adhesive systems determined by light microscopy at one week (T₀) or 6 months (T₆). SU: Scotchbond™ Universal; ER: etch-and-rinse; SE=self-etch; SB=Adper Single Bond 2 (A). Representative SEM micrographs of resin-dentin interfaces treated with SU-ER at T₀ indicating a mixed mode of failure, determined at a magnification of 25× (B). Dentin surface. A (and arrow) clearly denote parts of the composite. B indicates a region partially coated by SU, and the remaining area is clear dentin with intense polishing tracks. The dentin polishing tracks are continuous, thus reducing the possibility of cohesive dentin failure. C). Composite surface of the gelatin capsule. Three regions can be identified: A: Cohesive composite failure. Note the material striations (black arrow). Two levels seem to be present: an inclined level and a more horizontal level. B: Cohesive failure of SU. C: Exposed composite due to adhesive debonding from the SU.

occurred (2/32 surfaces). When the same adhesive was used in the self-etch mode (SU-SE), adhesive failure was dominant (47% of surfaces), followed by mixed failure (38%). The SBS failure rates were approximately 40%, 35% and 25% for mixed, cohesive and adhesive failures, respectively. The incubation time had a minor effect on the mode of failure; for all adhesives, the trends at T0 and T6 were similar.

Mixed failure, which was more frequent in the SU-ER and SB groups, is illustrated in an SEM image at a magnification of 25× in Fig. 3, B and C.

DISCUSSION

We found, in the current study, that the SBS to dentin for SU applied in the ER mode was significantly higher (~30%) than the values obtained for the SE mode and for the control, SB. The higher SBS values in the ER mode were accompanied by only a few pure adhesive failures, and the majority of the failures were of mixed mode, whereas in the SE mode and the SB condition, adhesive and mixed modes of failure occurred with similar frequencies. We also found that the bond strength did not deteriorate in either mode after 6 months aging. Our findings regarding the superiority of the ER mode are consistent with those of Vermelho et al. (4). A recent finding showed that pre-etching the dentin before the application of SE adhesives increased the bond strength and did not increase the nanoscale leakage. This result was emphasized by the fact that the universal adhesives penetrated both demineralized and non-demineralized dentin when used in the ER mode, leaving behind no detectable freezable water (5). Lack of deterioration after aging was also shown by Manfroi et al. (3) and was supported clinically over a period of 24 months for both the ER and SE modes (2). The durability of the bond strength might be explained by the bonding of 10-MDP to calcium, which creates a hydrolytically stable salt (MDP-Ca) that protects against hydrolysis (12). The chemical bonding between the carboxylic groups of the methacrylate-modified polyalkenoic acid copolymer and the calcium ions within the hydroxyapatite also contributes to the stability of the adhesive layer (3).

The results of the current study suggest that etching did not disrupt the bonding strength of SU to dentin, either at baseline or after six months of aging. Further clinical studies are required to validate the results of this *in vitro* study.

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