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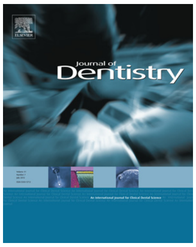

Q1 Bonding performance of universal adhesives in different etching modes

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ABSTRACT

Objectives: The aim of this study was to compare the microtensile bond strength (μTBS) and resin penetration into dentine of three universal adhesives (UAs) applied in two different Q3 etching modes (i.e. self-etch or etch-and-rinse). The effect of thermocycling on the μTBS was also evaluated.

Methods: The occlusal third of sound human molars was removed and the exposed surfaces were treated with three UAs (Futurabond Universal, Scotchbond Universal Adhesive and All-Bond Universal) in self-etch or etch-and-rinse mode. Two one-step self-etch adhesives (Futurabond DC and Futurabond M) were applied on additional teeth as reference. After composite build up, the specimens were stored for 24 h in distilled water at 37 °C or thermocycled for 5000 cycles. Composite/dentine beams were prepared (1 mm^2) and μTBS test was performed. Data was analyzed using three-way ANOVA and Tukey's test ($\alpha = 0.05$). One additional tooth was prepared for each group for evaluation of infiltration ability into dentine by dyeing the adhesives with a fluorochrome (Rhodamine B). After longitudinal sectioning, the generated interfaces were examined under confocal light scanning microscopy.

Results: The addition of an etching step did not significantly affect the μTBS of none of the UAs, when compared to their self-etch application mode. All pre-etched specimens showed considerably longer resin tags and thicker hybrid layers. Thermocycling had no significant effect on the μTBS of the UAs.

Conclusions: Application of an etching step prior to UAs improves their dentine penetration, but does not affect their bond strength to dentine after 24 h or after thermocycling for 5000 cycles.

Clinical significance: Similar bond strength values were observed for the UAs regardless of Q4 application mode, which makes them reliable for working under different clinical conditions.

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1. Introduction

Current dental adhesive systems and adhesive approaches seek to provide long-term bonding, while ensuring simplification of the technique. Self-etch adhesives (SEAs) were introduced with the goal of eliminating the highly sensitive technique step of acid etching, as their acidic monomers simultaneously etch and infiltrate the dental substrate,[1] thereby minimizing the discrepancies between hybridized and etched zones in the substrate.[2] Among them, "all-in-one" or "one step self-etch adhesives" (1-SEAs) go even further, as they intend to combine all-steps in only one application. While their bonding ability to dentine has been progressively improved with respect to the first 1-SEAs by means of better Q6 chemical interaction,[3] adhesion to enamel still remains unsatisfactory. Therefore, application of selective acid etching on enamel before SEA application has been recommended, especially when the use of mild-pH SEAs is intended.[4] However, inadvertent pre-etching of dentine is a clinical risk, which can negatively affect bonding efficacy.[5,6] Aiming to eliminate complications and providing a single product for all Q7 situations, "universal adhesives" (UAs) that might be indistinctly applied, either in self-etch (SE) or etch-and-rinse (E&R) mode,[7,8] have been recently introduced.

One of the keys of success with self-etching adhesives is the chemical bonding capability of their functional monomers to hydroxyapatite (HAp),[1] as described by the "adhesion/decalcification concept".[9,10] Among the currently used functional monomers, 10-methacryloyloxydecyl dihydrogenphosphate (MDP) has demonstrated a very effective and durable bond to dentine,[11,12] due to the low solubility of the calcium salt that forms on the hydroxyapatite surface.[12] On the other hand, micromechanical interlocking by means of good dentine hybridization (i.e. resin tags and hybrid layer), has been proposed to improve the bond strength of SEAs.[13] Phosphoric acid etching of dentine prior to application of SEAs significantly improves the interface infiltration morphology, by generating thicker hybrid layers[14,15] and longer resin tags.[16] Removal of the smear layer and smear plugs by this pretreatment[17] facilitates the adhesive penetration, especially in mild SEAs. Nevertheless, a clear correlation to higher bond strengths by these enhanced interfaces has not been established.[18]

According to their ability to demineralize dentine, SEAs have been classified into strong (pH $\leq$1), intermediately strong (pH between 1 and 2), mild (pH $\approx$ 2) and ultra-mild (pH >2.5).[1] This "etching aggressiveness" is strongly related with their interaction depth in dentine,[19] which varies from few nanometers in ultra-mild SEAs[20] to several micrometres, in Q8 the strong SEAs.[1] Thus, hybrid layers of mild SEAs are much thinner than those generated by stronger SEAs or etch-and-rinse adhesives, although hybrid layer thickness may not be of major importance to bonding efficacy.[2] By demineralizing dentine only incompletely, mild SEAs leave HAp partially attached to collagen, so it is available for chemical interaction[12] and protective nanolayering.[21]

At present, there is only sparse literature reporting on the efficacy of UAs. Similar adhesive performance has been observed for these adhesives regardless of their application mode,[7,22] although bond strength degradation has been observed after ageing for pre-etched samples.[23] The aim of Q9 the present study was to compare the bond strength and resin penetration pattern into dentine of three commercial UAs applied in two different etching modes (i.e. SE or ER). Two 1-SEAs were compared as reference. The effect of thermocycling on µTBS was also evaluated. The null hypotheses were that (i) the application mode of the UAs did not affect their µTBS, (ii) nor their resin penetration pattern into dentine; and (iii) that the bond strength is not affected by thermocycling.

2. Materials and methods

2.1. Bonding procedures, specimen fabrication and µTBS testing

Sound human third molars were stored in 0.5% chloramine solution and used within 3 months of extraction. The occlusal third was removed using a low-speed diamond saw (IsoMet; Buehler Ltd., USA) under water irrigation and flat surfaces were prepared in mid-coronal dentine with a remaining thickness in the range of 2.5 ± 0.2 mm. The dentine surface was ground with a 600-grit SiC paper for 60 s in order to Q10 produce a clinical relevant smear layer. Twelve teeth were assigned randomly to each of the three experimental groups and the UAs Futurabond U (FbU) (Voco, Cuxhaven, Germany), Scotchbond Universal Adhesive (SbU) (3M ESPE, Seefeld, Germany) and All-Bond Universal (AbU) (Bisco, Schaumburg, USA) were applied either in self-etch (SE) or total etch (E&R) Q11 mode. Two self-etching adhesives, Futurabond M (FbM) and Futurabond DC (FbDC) (Voco, Cuxhaven, Germany), were used for comparison and only applied in the SE mode (each on six teeth). Composition and manufacturer's instructions are described in Table 1.

For the SE mode, the dentine surface was left slightly wet and the adhesives were applied following manufacturer's instructions and light-cured for 20 s under a halogen light-curing unit (EliparTrilight, 3M ESPE, USA) with an output intensity of 750 mW/cm^2. For the E&R mode, before adhesive application, Scotchbond Etchant (35% H_3PO_4, 3M ESPE, USA) was applied on the dentine surface for 15 s, rinsed for 30 s and left slightly wet.

Resin composite crowns (GrandioSO; Voco, Germany) were incrementally build-up in 1 mm increments up to 5 mm under the same curing conditions as described above. Three teeth in each group were stored in distilled water for 24 h at 37 °C, whereas the other three were thermo-cycled (Willytec V2.8; Willytec/SD Mechatronik, Germany) for 5000 cycles (cyclic immersion for 30 s at 5.5 °C/55 °C each, 7 s dwelling time).

All specimens were then longitudinally sectioned in both 'x' and 'y' directions across the bonded interface with a low-speed diamond saw under sustained water-cooling (IsoMet low speed saw with a Buehler 10.2 cm × 0.3 mm diamond wafering blade, No. 11-4244), following a non-trimming Q12 microtensile technique.[24] Each beam dimensions were controlled with a digital calliper to ensure a cross-sectional area of 1 ± 0.05 mm^2. Only beams from the central region of each Q13 tooth were used. After sectioning, the specimens were fixed to

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