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Resin–dentin bonds to EDTA-treated vs. acid-etched dentin using ethanol wet-bonding. Part II: Effects of mechanical cycling load on microtensile bond strengths

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ARTICLE INFO

Article history:

Received 25 July 2010

Received in revised form

22 November 2010

Accepted 22 February 2011

Keywords:

Microtensile bond strength
 Scanning electron microscopy
 Ethanol-saturated dentin
 Hydrophobic hybrid layer
 Cycling loading

ABSTRACT

Objective. To compare microtensile bond strengths (MTBS) subsequent to load cycling of resin bonded acid-etched or EDTA-treated dentin using a modified ethanol wet-bonding technique.

Methods. Flat dentin surfaces were obtained from extracted human molars and conditioned using 37% H₃PO₄ (PA) (15 s) or 0.1 M EDTA (60 s). Five experimental adhesives and one commercial bonding agent were applied to the dentin and light-cured. Solvated experimental resins (50% ethanol/50% comonomers) were used as primers and their respective neat resins were used as the adhesives. The resin-bonded teeth were stored in distilled water (24 h) or submitted to 5000 loading cycles of 90 N. The bonded teeth were then sectioned in beams for MTBS. Modes of failure were examined by scanning electron microscopy.

Results. The most hydrophobic resin 1 gave the lowest bond strength values to both acid and EDTA-treated dentin. The hydrophobic resin 2 applied to EDTA-treated dentin showed lower bond strengths after cycling load but this did not occur when it was bonded to PA-etched dentin. Resins 3 and 4, which contained hydrophilic monomers, gave higher bond strengths to both EDTA-treated or acid-etched dentin and showed no significant difference after load cycling. The most hydrophilic resin 5 showed no significant difference in bond strengths after cycling loading when bonded to EDTA or phosphoric acid treated dentin but exhibited low bond strengths.

Significance. The presence of different functional monomers influences the MTBS of the adhesive systems when submitted to cyclic loads. Adhesives containing hydrophilic comonomers are not affected by cycling load challenge especially when applied on EDTA-treated dentin followed by ethanol wet bonding.

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doi:10.1016/j.dental.2011.02.010

1. Introduction

Resin bonding agents with high hydrophilicity are characterized by substantial water sorption and plasticization [1–3]. Adhesive bond strengths to water-saturated, acid-etched dentin matrices are directly related to the hydrophilicity of the resin bonding agents [4]. Dentin can be bonded with hydrophobic resins to decrease water sorption and increase the longevity of the resin–dentin interfaces using ethanol wet-bonding [5,6]. Indeed, it is possible to coax hydrophobic monomers into the hydrophilic dentin collagen matrix by using absolute ethanol [6–8] without any sign of phase change and/or microporosity at the resin–dentin interface [10]. This technique leads to better infiltration of hydrophilic and/or hydrophobic dimethacrylates into ethanol–dentin matrices also when applied on 37% phosphoric acid-demineralized or EDTA-treated dentin for only 1 min [11].

Moreover, the use of EDTA-conditioning, combined with ethanol wet-bonding seems allow better penetration of adhesive monomers into a thinner layer of demineralized dentin and to offer high and more durable microtensile bond strength (μ TBS) [11–13].

However, restored teeth are constantly subjected to cyclic stresses during physiologic chewing and swallowing. This occlusal stress may cause mechanical degradation and accelerate chemical degradation within resin–dentin interfaces [14,15].

No information is available on the effect of mechanical load cycling on the resin–dentin bonds to EDTA-treated vs. acid-etched dentin using ethanol wet-bonding. Thus, the purpose of this study was to compare resin–dentin bond strengths before and after mechanical load cycling of resin bonding agents applied to acid-etched or EDTA-treated dentin created with a simplified (1 min) ethanol wet-bonding technique. The test null hypotheses were that there is no difference in bond strength following cyclic loading of EDTA vs. phosphoric acid pre-treatment using hydrophobic vs. hydrophilic resins.

2. Materials and methods

2.1. Specimen preparation

The materials and methods sessions of this paper are similar to those of Sauro and collaborators [11] as it reports the second part of the experiments performed on the resin–dentin bonds to EDTA-treated vs. acid-etched dentin.

In brief, human molars extracted for surgical reasons were stored at 4 °C in 0.5% chloramine T for up to 1 month before use. The specimens were sectioned below the dentin–enamel junction using a water-cooled diamond saw (330-CA RS-70300, Struers, Copenhagen, Denmark). The occlusal surfaces were ground flat (LaboPol-4, Struers, Copenhagen, Denmark) using 500 grit SiC abrasive paper under constant water irrigation to provide standardized smear layer-covered dentin surfaces. Two principal groups were created according to the dentin conditioning treatments (i.e. EDTA or H_3PO_4). The teeth were subsequently divided into seven subgroups according to the resin adhesives used in this study. A further sub-division of

each subgroup was performed according to whether or not the teeth were cyclically loaded. All bonded teeth were stored in water for 24 h prior to cyclic loading or water storage for the same time.

2.2. Experimental resins

The five experimental comonomer blends used in this study as dentin bonding agents (DBAs) were formulated based on known concentrations of all ingredients, including 50 wt.% ethanol-solvated resin mixtures used as primers (Table 1) [11]. All experimental neat resins contained 0.25 wt.% camphorquinone and 1.0 wt.% ethyl-dimethyl-4-aminobenzoate. Resins 1 and 2 were hydrophobic resins similar to those used in pit-and-fissure sealants. Resin 3 represented the formulation of typical two-step, etch-and-rinse adhesives, while resins 4 and 5 had a hydrophilicity similar to one-step, self-etching adhesives, containing carboxylic- or phosphate-substituted methacrylates, respectively [11].

2.3. Bonding procedures

Dentin surfaces were acid-etched for 15 s with 37% phosphoric acid (PA) or treated with 0.5 M EDTA (pH 7.8) for 60 s and copiously rinsed with deionized water. The dentin surface was covered with absolute ethyl alcohol for 1 min and kept visibly moist with ethanol prior to the application of the resin blends [11].

Two consecutive coats of the five experimental primers (50% ethanol/50% resin) were applied to the conditioned dentin. Gentle air-drying was performed for 3 s to evaporate the excess solvent from the primed dentin. A layer of each respective neat comonomer adhesive was spread thin with a microbrush and light-cured for 15 s (Translux EC halogen light-curing unit, Kulzer GmbH, Bereich Dental, Werheim, Germany). A light output intensity of 600 mW/cm² intensity was employed for the experiments (Demetron Radiometer Model 100, Demetron Research, Danbury, CT, USA). A commercial adhesive, Scotchbond Multi-Purpose (SBMP) (3M ESPE, St. Paul, MN, USA) was also applied with the ethanol wet-bonding either as per manufactures' instructions (i.e. application of the primer and adhesive layers) or the adhesive was diluted in 50% ethanol and applied as a primer in two consecutive coats, followed by one layer of the neat Scotchbond MP adhesive. Five mm high composite build-ups were constructed for each specimen with a light-cured flowable resin composite, Tetric EvoFlow® (Ivoclar, Vivadent, Schaan, Liechtenstein – batch number: L26398) in 1-mm-thick increments. The resin-bonded specimens were stored in de-ionized water for 24 h at 37 °C.

2.4. Mechanical cycling load

The resin-bonded teeth of each subgroup that were created for the mechanical cycling load test were mounted in plastic rings with dental stone for placement in the load cycling machine using load control (5000 cycles, 12 Hz, 90 N). This compressive load was applied to the flat resin composite build-ups using a 5-mm diameter spherical stainless steel plunger, attached to a cyclic loading machine (model S-MMT-250NB; Shimadzu, Tokyo, Japan) while immersed in deionized water [15].

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