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Polymerization contraction stress in resin–tooth bonds under hydrated and dehydrated conditions

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ABSTRACT

Objective. This study hypothesizes that, with enamel or dentin as a bonding substrate, intrinsic water affects the development of polymerization contraction stress in the bonds of self-etching adhesives during bonding.

Materials and methods. The influence of the water content in dentin and enamel (wetness with water as control and acetone-dried specimens) on the stress development in self-etching adhesives was determined with a tensilometer. Thin layers of self-etching primer and/or adhesive resins were created between a glass plate and a flat enamel or dentin surface.

Results. After an initial maximum shortly after light curing for 30 min, the contraction stress was decreased in the dentin (30–70%) and enamel (approximately 20%). In the acetone-dried specimens, the stress was continuously increased for 20–50%.

Significance. The intrinsic water content of tooth tissue influences the initial polymerization of polymers. This effect is favorable for stress relief in resin restoration but causes unwanted nanoleakage channel formation in resin–tooth bonds.

1. Introduction

It has been shown that presently used adhesive resins contain relatively high concentrations of water or solvent which may induce water sorption into the resin from the tooth tissue which in turn results in accelerating hydrolysis of bonds [1–8]. Fluid movement study [9] has shown displacement of water in resin–dentin bonds during bonding procedures such as air-blasting and light irradiation. Further, a recent study showed that water sorption of bonding resins results in softening of the resin matrix and polymerization contraction stress relief [10]. This effect occurs in resins bonded to dentin as well as to enamel [11]. A TEM study [12], using silver nitrate as a tracer, has shown that transparent carious dentin containing mineral dentinal tubules prevented water movement from the dentinal tubules to the adhesive interface. Therefore, the presence of water during polymerization may create an expanded polymer network that offers greater swelling potential, and lead to un-desirable long-term effects [1–8]. However, it is not clear at what level of intrinsic water content the characteristics of adhesives is significantly affected and whether the risk is dependent on the type of hard tooth tissue (enamel or dentin) and its condition (wet or dry).

The first aim of this study was to evaluate the effect of the water content of the bonding substrate (hydrated vs. dehydrated) on the development of contraction stress during initial

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Table 1 – Two self-etching adhesive system investigated.

Material (manufacturer)	Primer	Adhesive
Two-step self-etching adhesive Imperva FL-Bond (Shofu Inc., Kyoto, Japan)	4-AET, HEMA, ethanol, water	4-AET, UDMA, HEMA, filler
All-in-one self-etching adhesive Xeno III (Dentsply deTrey, Konstanz, Germany)	–	Pyro-EMA-SK,PEM-F, UDMA, BHT, EDAB, HEMA, ethanol, water

Abbreviations: 4-AET, 4-aeryloxythyltrimellitic; UDMA, urethane dimethacrylate; HEMA, 2-hydroxylethyl methacrylate; Pyro-EMA-SK, tetramethacryl-ethyl-cyclo-phosphazen-monofluride; BHT, 2,6-di-tert-butyl-*p*-cresol; EDAB, ethyl 4-dimethylaminobenzoate and pyrophosphate.

bonding. The second aim was to evaluate the effect of the pH of self-etching primers on the dynamic stress development within resin–tooth bonds. To monitor the effect on the stress development when the bonding substrates are dried, specimens were divided into two groups, control and acetone-dried (24 h of acetone storage) enamel or dentin. The null hypothesis tested is that there are no significant differences in the stress development for hydrated and dehydrated substrates.

2. Materials and methods

2.1. *Adhesives resin systems and pH measurements*

Two commercially available self-etching primer systems (Xeno III, Dentsply de-Trey, Konstanz, Germany and Imperva FL-Bond, Shofu Inc., Kyoto, Japan) were used in this study (Table 1). The pH values of the adhesive resin systems were measured with a pH meter (Delta 350, Metter-Toledo, Tiel, The Netherlands, pH surface electrode: Orion Ross model 8135, Thermo Electron Corporation, Breda, The Netherlands). The measurements were performed at 23 °C in a dark room with special red light on approximately 10 drops of each liquid and values were read for 15 s when the pH was stable ($n = 5$ for each group).

2.2. *Preparation of enamel and dentin specimens*

Eighty cylindrical cores of dentin, 7.0 mm in diameter were cut from the roots of central bovine incisors, normal to the flat ground mesial or distal root surface, using a hollow diamond drill with copious water cooling (Diamant Boart Nederland B.V., Vianen, The Netherlands). Enamel cores were made from the crowns of central bovine incisors, using the same diamond drill. Each enamel or dentin core was mounted at the free end of a rotatable rod with the central axes aligned. A 1.0 mm length of the free end of each dentin core was trimmed to a diameter of 6 mm using a diamond bur in an air-rotor with water spray (Fig. 1). The flat end of each core was finally wet ground with SiC-paper up to 600 grit at the bonding site.

2.3. *Test groups*

The specimens were divided into two groups; wet (control group) and dry (experimental group). For the dry group, the enamel or dentin cores were stored in 100% acetone solution for 24 h at room temperature to remove water. Subsequently, the cores were air-blasted and then left in air for 2 h. For the wet groups, the cores were stored in water and air-blasted prior to bonding.

2.4. *Surface treatment prior to bonding*

For Xeno III (all-in-one self-etching adhesive), the enamel or dentin surfaces were covered with the self-etching adhesives (mixture of adhesive A and B), left undisturbed for 20 s and then air-dried for 3 s to remove the volatile solvents. For FL-Bond (two-step self-etching adhesive), the enamel or dentin surfaces were covered with the self-etching primers (mixture of primer A and B), left undisturbed for 20 s, and then air-dried for 3 s. The bonding resin was then applied at the primed-dentin surface.

2.5. *Contraction stress measurements*

The polymerization contraction stress was determined in a universal testing machine (ACTA Intense, ACTA, Amsterdam, The Netherlands) [10,11]. The cores were fixed in a specially machined steel cylindrical specimen holder, from which 0.5 mm of the 6 mm diameter part of the cores could protrude, and connected to the crosshead with the load-cell of the universal testing machine. The specimens with adhesives-applied were lowered toward the glass plate and adjusted to a position to form an adhesive layer of approximately 15 μm. Adhesion to the glass plate was ensured by sandblasting and silanizing. The adhesive layer was then light cured from underneath the glass plate for 20 s (600 mW/cm^2) using a light-curing unit (Astralis 10, Ivoclar-vivadent). The contraction stress development was recorded continuously from the start of light curing up to 30 min. The relative polymerization contraction stress was calculated by the following equation: (stress value after 30 min/stress value after 1 min) × 100 (%). One-way ANOVA and Tukey's post hoc tests were used to analyze differences ($p < 0.05$) in the ratio of polymerization contraction stress. Differences between the maximum and 30 min values within each group were analyzed with paired *t*-tests ($p < 0.05$). The number of experiments was $n = 10$ for each group.

2.6. *Tensile bond test*

After 30 min of contraction stress measurements, a tensile load was immediately applied with a universal testing