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Permeation of intrinsic water into ethanol- and water-saturated, monomer-infiltrated dentin bond interfaces

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ABSTRACT

Objectives. The aim of this study was to evaluate the formation of dentin bonding interfaces using the water-wet and the ethanol-wet techniques under simulated pulpal pressure, and to assess the effect of adhesive solvent and thermomechanical loading.

Methods. Flat dentin surfaces were restored under 20 mm-simulated pulpal pressure following two bonding approaches (water-wet and ethanol-wet bonding) in combination with dental adhesives containing ethanol (Single Bond Plus and Scotchbond Multi-Purpose) or acetone (One-Step Plus and All-Bond 2) as solvent. Half of the restorations of each subgroup were subjected to thermocycling followed by cyclic loading (three teeth per group). Bond strength was measured using the microtensile bond strength test and fitted to a Weibull distribution ($\alpha = 0.05$). Ultrastructural analyses of the interface and leakage/nanoleakage evaluation were performed using confocal scanning microscopy (CLSM) and transmission electron microscopy (TEM).

Results. Water permeation through dentin tubules during adhesive application prevented adequate penetration of adhesive monomers into the demineralized collagen matrix in both bonding techniques, but more severely for water-wet bonding. Acetone-solvated adhesives showed worse bonding performance and hybridization than ethanol-based systems when applied in the ethanol-wet mode, both before and after thermomechanical challenge.

Significance. The ethanol-wet bonding technique helps to compensate for water permeation from dentin tubules during the bonding procedures to form more stable dentin bonds, especially when used in conjunction to ethanol-solvated systems.

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1. Introduction

Water is a polar solvent that plays a polar role in dentin bonding. Water is needed to prevent the collapse of the etched, hydroxyapatite-depleted collagen network, serving as a medium for subsequent infiltration of solvated hydrophilic monomer mixtures [1–3]. This approach has shaped the concept of dentin adhesion and seems to persevere as a successful clinical technique [4–6]. Water therefore helps to achieve high quality bonds to dentin – immediate quality. Long-term degradation of dentin bonds, on the other hand, is generally associated to processes linked to the presence of water [7–9]. To name some, water promotes the phase separation of hydrophobic monomers and creates a comonomer gradient within the hybrid layer [10–12], where the low-molecular weight, more hydrophilic monomers occupy its deepest regions retaining humidity, and soaking further water from the substrate. Water during application interferes with monomer polymerization, and trapped and absorbed water later flow through uncured routes within the polymer to hydrolyze and plasticize the resin [13,14]; chemical degradation proceeds invariably to mechanical degradation of the interface. Parallel to that, interfibrillar water mediates the activity of endogenous enzymes (endopeptidases) set loose during etching, triggering the proteolytic degradation of unenveloped collagen fibers in the hybrid layer, reducing its long-term structural integrity [15,16].

To circumvent these issues some current strategies aimed to prolong the integrity of the dentin bonding interface bet on the complete replacement of water from the etched surface with ethanol before infiltration with resin [17,18]. Saturation with absolute ethanol promotes the full dehydration and shrinkage of collagen fibers, and in principle grants the use of more hydrophobic monomer solutions that are less prone to water uptake and hydrolysis. So far this rationale was proven right *in vitro*, in that hydrophobic bonding to dentin following the so-called *ethanol-wet bonding* technique has shown longstanding ultrastructural integrity and bond strength in comparison to its hydrophilic *water-wet technique* counterpart [19–21]. Accountable for these improvements are, essentially, the deeper penetration of hydrophobic monomers that increase the mechanical quality of the hybrid layer in its entire depth [22], the minimal permeation from substrate moisture into the interface [22,23] and the removal of unbound water from interfibrillar spaces prior to application of adhesive monomers [24,25]. However, bound water in dentin matrices cannot be evaporated or removed by organic solvents such as ethanol [26].

Yet in vital teeth dentin permeability increases considerably after etching, and pulpal fluid is continuously pumped toward the interface during adhesive application and after polymerization. Pulpal pressure places water at the interface unless dentin tubules are occluded, potentially undermining the aforementioned benefits of a hydrophobic seal. Also playing against the clinical feasibility of the ethanol-wet technique is the technical sensitivity of the dehydration procedure [27], the prolonged clinical time for proper dehydration to take place, and the immense variability in composition of available commercial adhesive systems.

Motivated by these open issues the present study was set to evaluate the role of intrinsic water permeability in the formation of dentin bonds following the ethanol and water-wet techniques using commercial adhesives having different compositions and solvents. Our null hypotheses were: (1) that bond integrity in terms of interface morphology and bond strength are comparable for bonds produced by the ethanol-wet and water-wet techniques; (2) that adhesive system and solvent (ethanol or acetone) have no significant effect on bond integrity assessed by microscopy, and; (3) that thermo-mechanical loading results in no bond degradation irrespective of adhesive system or technique employed.

2. Materials and methods

One hundred and twelve intact, freshly extracted, non-carious human third molars were collected after the donors' informed consent and under a protocol reviewed and approved by an Ethical Committee. The teeth were scaled, cleaned, stored in 0.5% chloramine solution at 4 °C to prevent bacteria growth and used within 3 months after extraction. The teeth were sectioned perpendicular to the longitudinal axis of the tooth 3.5 mm below the cement-enamel junction (CEJ), using a diamond saw under water-cooling. A second parallel cut removed the occlusal enamel and superficial dentin creating a flat dentin surface of middle dentin A caliper was used to control the remaining dentin thickness to 2.5 ± 0.1 mm from the deepest pulpal horn. The pulpal tissue was gently removed from the exposed pulp chamber without damaging the pre-dentin. The occlusal surfaces of crown segments were polished using a 600-grit silicon carbide paper under running water for 60 s to create a standard smear layer. The crown segments were attached to perforated plexiglass disks and connected to polyethylene 18-gauge tubings to simulate pulpal pressure. Twenty centimeters of water pressure was delivered to the prepared dentin surface during the adhesive procedures and restoration build-up.

The teeth were divided into two main groups according to the bonding technique: water-wet (W) or ethanol-wet (E) bonding. Adhesive systems were selected (see Table 1) based on their solvent (ethanol or acetone) and number of application steps (two-step or three-step etch-and-rinse systems). The exposed occlusal dentin surface was etched with 35% phosphoric acid gel (Scotchbond Etchant, 3M ESPE) for 15 s and rinsed with distilled water for 15 s. The excess of water was blotted with a tissue but left visibly moist to prevent collapse of the collagen matrix. In the water-wet bonding group the primers/adhesives were applied to the moist dentin surface, while in the ethanol-wet bonding groups the surface was saturated with 1 ml of 100% ethanol for 30 s before primer/adhesive application.

The adhesive systems were applied to the etched dentin strictly following the manufacturers' instructions in regard to application layers and time (see Table 1). Light-curing was performed using a halogen unit (Elipar 2500, 3M ESPE) with 600 mW/cm^2 light output. Resin composite restorations made with a micro-hybrid composite resin (Filtek Z250, shade A2, 3M ESPE) were built on top of the bonded surfaces in five successive increments of 1 mm, each light-cured for 20 s. From

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