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# Evaluation of the micro-mechanical strength of resin bonded–dentin interfaces submitted to short-term degradation strategies

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**ABSTRACT**

The aim of this study was to evaluate the microtensile bond strength ( $\mu$ BTBS) and confocal micropermeability of resin bonded–dentin specimens created using two representative two-step/self-etch adhesives submitted to short-term period degradation strategies such as simulated pulpal pressure, thermo- or mechanical-cycling challenges. Clearfil SE Bond (CSE) and Silorane adhesive (SIL) were bonded to flat deep dentin from seventy extracted human molars and light-cured for 10 s. Composite build-ups were constructed using with Filtek Z350 XT and Filtek P90 respectively. The specimens of each adhesive group were subjected to three different accelerated aging methods: (1) thermo-cycling challenge (5000 cycles); (2) mechanical-cycling load (200,000 cycles); (3) experiment and (4) conventional method for simulated pulpal pressure (20 cm H<sub>2</sub>O). Control resin-bonded specimens were stored in distilled water for 24 h.  $\mu$ BTBS and confocal microscopy (CLSM) micropermeability evaluation were performed and the results were analyzed using Two-way ANOVA and Tukey's tests ( $\alpha=0.05$ ). The CLSM evaluation revealed micro-cracks within the Silorane-bonded dentin subsequent to mechanical-cycling load, whereas, the simulated pulpal pressure induced evident micropermeability in both bonding agents. Mechanical loading provides discernible bonding degradation in a short-term period in resin-bonded dentin created using two-step/self-etch adhesives. However, simulated pulpal pressure may reduce the sealing ability of self-etch adhesives causing greater water uptake within the resin–dentin interface.

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## 1. Introduction

Direct and indirect adhesive restorations are commonly realized in practice using resin-based materials (Ferracane, 2011). Self-etch adhesives have become increasingly popular due to more user-friendly and less technique-sensitive characteristics (Van Meerbeek et al., 2003). They do not require a separate etching step because they contain acidic monomers that simultaneously demineralize and infiltrate the dentin to create a chemical/micro-mechanical bond; thus, no discrepancy could be expected between the demineralization depth and the resin infiltration depth (Perdigão et al., 2006). Nevertheless, a decrease in bond strength has also been reported for this type of dental adhesive subsequent to challenge conditions (Cadenaro et al., 2005; De Munck et al., 2003; Osorio et al., 2008).

Novel dentin bonding agents (DBA) are being continuously developed and launched in the market without full knowledge of their bonding efficacy and durability (Amaral et al., 2007; Feitosa et al., 2010) due to a lack of long-term *in vitro* investigations and/or *in vivo* clinical trials. Simple water immersion over “few” weeks may not provide sufficient degradation to challenge the bonding performance of these bonding agents bonded to dentin and enamel. Therefore, alternative *in vitro* short-term period degradation strategies to reveal valuable clinical information are needed (Hashimoto et al., 2004).

It has been suggested that a reliable method to challenge the bonding durability should employ accelerated degradation strategies (Amaral et al., 2007), such as chemical challenge (Sauro et al., 2009) thermo- (Bedran-de-Castro et al., 2004; Feitosa et al., 2010; Ulker et al., 2010), and mechanical-cycling (Bedran-de-Castro et al., 2004; Feitosa et al., 2010; Osorio et al., 2008). Mechanical-cycling load and thermo-cycling testing performed in water at temperatures between

5 and 55 °C which subject the adhesive interface to water infiltration, mechanical and expansion/contraction strain (Ferracane et al., 1998; Gale and Darvell, 1999) may be considered suitable aging methods for dental adhesive materials.

A further suitable strategy to challenge the resin bonded-dentin is based on the use of a simulated pulpal pressure (20 cm H<sub>2</sub>O) in order to induce water seepage, polymer degradation and droplets formation within the resin–dentin interface jeopardizing the μTBS (Bakry et al., 2009; Hosaka et al., 2007; Sauro et al., 2007; Sauro et al., 2007).

The purpose of this study was to evaluate the microtensile bond strength (μTBS) and confocal micropermeability of resin bonded–dentin created with two representative “gold-standard” self-etch adhesive when submitted to short-term period degradation strategies such as simulated pulpal pressure, thermo- or mechanical-cycling challenges. SEM ultra-morphological analysis of the fractured specimens subsequent to challenge strategies used in this study was also performed. The null hypotheses to be tested is the strategies used in this study to accelerate degradation in a short-term period have no effect on the micro-mechanical adhesive properties (μTBS) or on the ultra-morphology/micropermeability of the resin–dentin interfaces created using self-etch adhesives.

## 2. Materials and methods

### 2.1. Sample preparation

Seventy human third molars extracted under the approved protocol of the appropriate institutional Research Ethics Committee (protocol 167/2009) were used in this study. The teeth were stored in 0.5% chloramine water solution for a period not exceeding 4 months at a temperature of 4 °C.

Deep dentin specimens with a remaining dentin thickness (RDT) of approximately 0.9 mm were obtained by removing

**Table 1 – Materials, compositions, application procedures and batches.**

Materials	Composition	Application procedure	Batch no.
P90 System adhesive	<i>Self-etch primer:</i> Vitrebond copolymer, phosphorylated methacrylates, BisGMA, HEMA, water, ethanol, silica filler, stabilizers, photoinitiator	Apply one coat of the self-etch primer for 15 s with gentle agitation. Gently air dry. Light curing for 10 s.	9BN
	<i>Bond:</i> Hydrophobic methacrylates, phosphorylated methacrylates, TEGDMA, silica filler, initiators, stabilizers	Apply the Bond. Gently air thin until the Bond is spread to an even film and does not move any longer. Light curing for 10 s.	9BK
Clearfil SE Bond	<i>-Primer:</i> MDP, HEMA, water, photoinitiator	Apply primer for 20 s, gently air-dry; apply bond. Light cure for 10 s	896A
	<i>-Bond:</i> MDP, BisGMA, HEMA, hydrophobic dimethacrylates, photoinitiator		1321A
Filtek P90 Shade A3	<i>Matrix:</i> TEGDMA, ECHCPMS <i>Filler:</i> Quartz filler	Apply in 1–2 mm increments and light cure for 40 s	N110333
Filtek Z350 XT Shade A3	<i>Matrix:</i> BisGMA, BisEMA, TEGDMA, UDMA. <i>Filler:</i> Silica and zirconia nanofiller	Apply in 1–2 mm increments and light-cure for 40 s	9XN

BiSGMA: bisphenol A diglycidylmethacrylate; HEMA: hydroxyethylmethacrylate; MDP: 10-methacryloyloxi decyl phosphate; TEGDMA: triethylene glycol dimethacrylate; ECHCPMS: 3,4-epoxycyclohexylcyclopolymethylsiloxane; BisEMA: ethoxylated bisphenol A diglycidylmethacrylate.

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