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ORIGINAL ARTICLE

Effect of six month storage on microtensile bond strength of new elective etching adhesive system on dentin in self-etching or etch-and-rinse approach

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Abstract The aim of this study was to evaluate the microtensile bond strength (μTBS) to dentin of an elective etching adhesive system applied in etch-and-rinse (ER) or self-etching (SE) mode after 6-months of storage in water. Thirty-six caries-free, human third molars were collected and stored in a 0.1% thymol solution. Dentin surfaces were exposed by 600-grit silicon carbide paper and teeth were divided into six groups ($n = 6$), according to the adhesive systems: a 2-step SE system, Clearfil SE Bond (CSE); a 1-step SE adhesive Adper Prompt L-Pop (LPOP); Scotchbond Universal applied as a 1-step SE adhesive (SBU-SE) and applied as a 2-step ER adhesive (SBU-ER); and two 2-step ER adhesives: Adper Single Bond Plus (SBP) and Optibond Solo Plus (OSP). Composite build-ups were constructed with TPH3 and cured in three increments of 2 mm each. Specimens were sectioned with a slow-speed diamond saw under water in *X* and *Y* directions to obtain bonded beams that were tested to failure in tension at a crosshead speed of 1.0 mm/min after one week or 6 months of storage in water. Statistical analyses were computed using Repeated-Measures ANOVA and Fisher's LSD Tests ($\alpha = 0.05$). There were no significant differences between 1-week and 6-months. SBU-ER and SBUSE showed the highest μTBS values and statistically differed from LPOP (Fisher's LSD). The SBP, OSP, and CSE groups showed intermediary μTBS and did not differ statistically from SBU-ER, SBUSE or LPOP, which presented the lowest μTBS values. The use

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of elective etching adhesive system in dentin with the etch-and-rinse or self-etching approach did not compromise the bond strength and showed stable bonds after six months of storage in water.

1. Introduction

Bonding to dental enamel is considered effective and usually presents high bond strength values.[20] Resin-dentin bonds are more difficult to achieve than resin-enamel bonds, because dentin bonding relies on organic components[12] However, adhesive systems should present similar performance on enamel and dentin to increase the clinical performance. Resin-dentin bonds are normally created by infiltration of hydrophilic resin monomers into a previously demineralized dentin matrix.[1,21,22] An essential condition for the formation of the hybrid layer is the maintenance of the dentin organic matrix moist after demineralization, which supports the expansion of the collagen fibrils and preserves the integrity of the interfibrillar spaces. This disposition allows an appropriate infiltration of the resin monomers dissolved in non-aqueous organic solvents or an aqueous solution of hydrophilic primers.[16]

Alternatively, the self-etch bonding technique uses acidic monomers that combine tooth surface etching and priming in a single procedure, reducing the risk of technical deficiencies from etch-and-rinse systems.[25] The advantage of the self-etching adhesives (SEA) technique is the simultaneous demineralization and resin infiltration, which should lead to a thin and an optimally infiltrated hybrid layer.[19,21,24] Also, SEAs are less aggressive than phosphoric acid used in the etch-and-rinse technique.[21] However, bonding to enamel is not as effective as it is in dentin, and it is generally not indicated for use on enamel surfaces without a prior phosphoric acid etching step for direct or indirect restorative procedures, especially in unground enamel.[8,10] The selective enamel etching combined with a 'mild' pH SEA can therefore today be recommended to achieve effective and durable bonding to enamel and dentin.[24,27]

However, clinicians should not inadvertently etch dentin walls in the selective etching process. If it occurs, the adhesive may not be able to properly infiltrate etched dentin to produce a good-quality hybrid layer. SEAs may have reduced performance when bonding to etched dentin surfaces.[11,19,23]

In an attempt to overcome this problem, manufacturers have developed the so-called "Universal" adhesives, that can be applied in both self-etching or etch-and-rinse modes. However, few studies describe the use and the performance of such adhesive systems.

The aim of this study was to evaluate the microtensile bond strength (μTBS) of an elective etching adhesive system applied in the etch-and-rinse and self-etching modes and the stability after six months of water-storage. The null hypothesis of this study is that the bond strength values produced by the elective etching adhesive system do not differ from strictly etch-and-rinse or the self-etching adhesives after seven days or six months of storage in water.

2. Methods and materials

2.1. Experimental design

The factor under study was "adhesive" system in six levels ($n = 6$), analyzed by repeated measurements at seven days and six months in water storage. The experimental adhesive groups were: SBU-SE- Scotchbond Universal (3M ESPE, St. Paul, MN, USA) applied as a 1-step self-etch adhesive; SBU-ER- Scotchbond Universal (3M ESPE, St. Paul, MN, USA) applied as a 2-step etch-and-rinse adhesive, the etch-and-rinse control groups were SBP- Adper Single Bond Plus (3M ESPE, St. Paul, MN, USA), OSP- Optibond Solo Plus (Kerr, Orange, CA, USA), and the control self-etching groups were CSE- Clearfil SE Bond (Kuraray, Kurashiki, Japan), and LPOP- Adper Prompt L-Pop (3M ESPE, St. Paul, MN, USA) (Table 1). The dependent variable was microtensile bond strength (μTBS) in MPa.

2.2. Specimens' preparation

Thirty-six caries-free, human third molars were collected according to the local Institutional Review Board (# 19/2009), with the informed consent of the donors. They were stored in a 0.1% thymol solution at 4 °C and used within one month following extraction.

Prior to bonding procedures, the roots and occlusal enamel were removed and the exposed middle dentin surfaces were wet-polished with 600-grit silicon carbide paper under running water to create a standard smear layer.[5]

2.3. Bonding procedures

For groups SBU-ER, SBP and OSP the 35% phosphoric acid gel (ScotchBond™ Etchant, 3M ESPE, St. Paul, MN, USA) was used to etch dentin surfaces for 15 s. Dentin was thoroughly rinsed with water and excess water was removed prior to adhesive application. Adhesive was applied following manufacturer directions (Table 1) and light-cured using an LED (Radii Plus – SDI, Victoria, Australia) with a power output of 2000 mW/cm^2. Subsequently, composite build-ups were made incrementally (TPH3 Dentsply Caulk, Milford, DE, USA) on each bonded surface. Three 2 mm increments were individually placed and light-cured for 20 s.

2.4. Microtensile bond strength test

Restored teeth were stored in distilled water at 37 °C for 7 days. Afterward, they were serially sectioned in both "x" and "y" directions using a diamond saw (IsoMet 1000; Buehler Ltd, Lake Bluff, IL, USA) under water cooling, resulting in

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