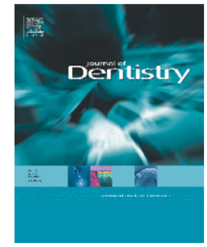
journal of dentistry 39 (2011) 578–587

available at www.sciencedirect.com

ScienceDirect

journal homepage: www.intl.elsevierhealth.com/journals/jden



Effect of 3-year water storage on the performance of one-step self-etch adhesives applied actively on dentine

Alessandro Dourado Loguercio *, *Rodrigo Stanislawczuk*, *Alexandra Mena-Serrano*, *Alessandra Reis*

School of Dentistry, State University of Ponta Grossa, Paraná, Brazil

ARTICLE INFO

Article history:
Received 1 April 2011
Received in revised form
15 June 2011
Accepted 16 June 2011

Keywords:
Adhesives systems
Active application
Durability
Long-term
Nanoleakage
Microtensile bond strength

ABSTRACT

Objectives: To evaluate the effect of the application method on the immediate and 3-year resin–dentine bond strength (μTBS) and nanoleakage (NL) for 3 one-step self-etch adhesives (Adper Prompt L-Pop; Clearfil S^3 Bond and Xeno III).

Methods: The occlusal enamel of 42 human molars were removed to expose a flat dentine surface. The adhesives were applied under inactive or active application. After light-curing (600 mW/cm^2 for 10 s), composite buildups were constructed incrementally and sectioned to obtain bonded sticks (0.8 mm^2) to be tested in tension immediately or after 3 years of water storage of water storage. For NL, 2 bonded sticks from each tooth at each time were coated with nail varnish, placed in silver nitrate and polished down with SiC paper. The μTBS data was submitted to a two-way repetead mesures ANOVA and Tukey's test for each adhesive ($\alpha = 0.05$).

Results: The active application showed higher immediate and 3-year μTBS than the inactive application ($p < 0.05$). An increase in the silver nitrate deposition was seen for all conditions after 3 years; however this was more pronounced for all materials under inactive application ($p < 0.05$).

Conclusions: The active application improves the immediate bonding performance of the adhesive systems. Reductions of the bond strength were observed for all materials after 3 years, however reduced degradation rate was observed when the materials are applied actively in dentine.

1. Introduction

Success in adhesive dentistry involves long lasting restorations; however the resin–dentine interface degradation appears as the biggest obstacle to achieve this goal. Independent of the adhesive strategy used for bonding to tooth substrates (etch-and-rinse or the newest self-etch adhesive systems) degradation of the hybrid layer can be observed.[1]

The etch-and-rinse or the self-etch approach are the available adhesive strategies employed nowadays for dentine bonding.[2–4] The advantage of the self-etch approach is that these materials do not require etching and rinsing. The first non-rinsing self-etch adhesives were composed of two solutions, an acidic primer and a bonding resin. More recently, this trend has shifted to a more simpler procedure, which led to the development of one-step self-etch adhesives, in which all components were combined into one single solution.

* *Corresponding author at*: Rua 7 de setembro 125 – apto 41 – Centro, Ponta Grossa, Paraná 84010-350, Brazil. Tel.: +55 42 3027 7898; fax: +55 42 3220 3741.
E-mail address: aloguercio@hotmail.com (A.D. Loguercio).

0300-5712/$ – see front matter
doi:10.1016/j.jdent.2011.06.005

The self-etch adhesives vary in their acidity by virtue of the composition and concentration of polymerizable acids.[5] Usually, the self-etch systems can be classified according to the pH of the adhesive solutions in strong ($pH < 1.0$), intermediately strong or moderate (pH between 1.0 and 2.0) and mild ($pH > 2.0$),[2–4] and this seems to have an influence in their ability to interact with the underlying dentine.

Several studies have demonstrated that the smear layer thickness may jeopardize the bonding effectiveness of self-etch systems and therefore the use of fine-grit diamond or carbide burs were suggested for cavity preparation in order to improve the resin penetration into dentine.[6–9] This is an important concern regarding self-etch adhesives, as they may fail to penetrate beyond the smear layer and reach the mineralized dentine to form a hybridized complex[10] compromising the strength of the bond.

In an attempt to improve the bonding effectiveness of these materials, Miyazaki et al.[11] was the first to suggested that the active primer application may be helpful to remove the smear layer in order to achieve micromechanical as well as chemical interaction with underlying dentine regardless the adhesive acidity and different research centres also indicating several advantages for this technique.[12–14]

Even though these adhesives are very user-friendly and less technique sensitive, they exhibit lower immediate bond-strength values[2,15,16] and produce a less stable interface under ageing when compared with the other adhesives categories.[3,17] This improvement of the dissolution of smear layers produced by active application could also have an impact on the immediate and short term longevity.[11–14]

To the extent of the author's knowledge no *in vitro* study has so far evaluated the effect of application technique on the long-term durability of resin–dentine bonds produced by one-step self-etch systems. Therefore, the purpose of this study was to evaluate the early and 3-year resin–dentine bond strength and silver nitrate deposition of one-step self-etch adhesive under active and inactive application. The following null hypotheses were tested in this study: (1) the application method does not affect the early and 3-year resin–dentine bond strength for one-step self-etch system and (2) the application method does not affect the early and 3-year silver nitrate deposition for one-step self-etch systems.

2. Methods and materials

2.1. *Teeth selection and preparation*

Forty-two extracted, caries-free human third molars were used. The teeth were collected after the patient's informed consent. The Ethics Committee from the State University of Ponta Grossa approved this study. Teeth were disinfected in 1% thymol, stored in distilled water and used within 6 months after extraction. A flat and superficial dentine surface was exposed on each tooth after wet grinding the occlusal enamel on # 180-grit silicon carbide paper. The enamel-free, exposed dentine surfaces were further polished on wet # 600-grit silicon-carbide paper for 60 s to standardize the smear layer. One schematic diagram of tooth preparation, restoration specimen sectioning and testing can be seen in Fig. 1.

Three one-step self-etch adhesive systems were selected according to their aggressiveness potential reported by their manufacturers: Adper Prompt L-Pop ([AD] – 3MESPE, St. Paul, MN, USA) – aggressive ($pH < 1$); Xeno III ([XE] – Dentsply Caulk, Milford, DE, USA) – moderate ($1 < pH < 2$) and Clearfil S^3 Bond ([S3] – Kuraray Medical Inc., Osaka, Japan) – mild ($pH > 2$).[2–4] Their composition, application mode and batch number are described in Table 1.

A single operator applied all adhesives on the dentine as follows:

1) *Inactive application*: In this group, the adhesive was only spread, over the entire surface and left undisturbed for 15–20 s according to the manufacturer's recommendation depicted in Table 1. In the case of Adper Prompt L-Pop, a second coat is indicated to be applied. Then, an air stream was applied for 10 s at a distance of 20 cm (Table 1).
2) *Active application*: The adhesive was actively applied on the entire dentine surface for approximately 15–20 s according to manufacturer's recommendation (Table 1). The microbrush was scrubbed on the dentine surface under manual pressure (equivalent to approximately 34.5 ± 6.9 g).[18,19] In the case of Adper Prompt L-Pop, a second coat is indicated to be applied. An air stream was applied for 10 s at a distance of 20 cm (Table 1).

A VIP light-curing unit set with a light intensity of 600 mW/cm^2 (Bisco, Schaumburg, IL, USA) was used throughout the restorative procedure. Following the adhesive application, resin composite build-up "crowns" (Opallis, FGM, Joinville, SC, Brazil) were constructed in 1.5 mm increments and light cured for 40 s each. Seven teeth were used for each experimental group.

After storage of the bonded teeth in distilled water at 37 °C for 24 h, they were longitudinally sectioned in both "x" and "y" directions across the bonded interface with a diamond saw in a Labcut 1010 machine (Extec Corp., Enfield, CT, USA), under water cooling at 300 rpm to obtain bonded sticks with a cross-sectional area of approximately 0.8 mm^2. All sticks from each tooth were divided for silver nitrate uptake (SNU) evaluation under Scanning Electron Microscopy (SEM) and microtensile bond strength test (μTBS). Usually, 4 sticks was used for SNU, two in each storage time and the remaining sticks was used for μTBS, half in the immediate time and half after 3-year storage time.

2.2. *Microtensile bond strength test (μTBS)*

The number of premature failures (PF) per tooth during specimen preparation was recorded. The cross-sectional area of each stick was measured with the digital calliper to the nearest 0.01 mm and recorded for subsequent calculation of the microtensile bond strength (Absolute Digimatic, Mitutoyo, Tokyo, Japan). The bonded sticks originated from the same teeth were randomly divided and assigned to be tested immediately or after 3 years of storage in distilled water at 37 °C. The storage solution was not changed and its pH was monitored monthly.

Each bonded stick was attached to a modified device (Odeme Prod. Med. Odont., Joaçaba, SC, Brazil) for μTBS with cyanoacrylate resin (Zapit, Dental Ventures of North America, Corona, CA, USA) and subjected to a tensile force in a universal