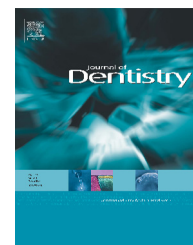


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Immediate bonding properties of universal adhesives to dentine

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ARTICLE INFO

Article history:

Received 1 November 2012

Received in revised form

27 February 2013

Accepted 1 March 2013

Keywords:

Microtensile bond strength

Nanoleakage

Degree of conversion

Etch-and-rinse

Self-etch

Universal simplified adhesive systems

ABSTRACT

Objectives: To evaluate the dentine microtensile bond strength (μ TBS), nanoleakage (NL), degree of conversion (DC) within the hybrid layer for etch-and-rinse and self-etch strategies of universal simplified adhesive systems.

Methods: forty caries free extracted third molars were divided into 8 groups for μ TBS ($n = 5$), according to the adhesive and etching strategy: Clearfil SE Bond [CSE] and Adper Single Bond 2 [SB], as controls; Peak Universal Adhesive System, self-etch [PkSe] and etch-and-rinse [PkEr]; Scotchbond Universal Adhesive, self-etch [ScSe] and etch-and-rinse [ScEr]; All Bond Universal, self-etch [AlSe] and etch-and-rinse [AlEr]. After restorations were constructed, specimens were stored in water (37 °C/24 h) and then resin–dentine sticks were prepared (0.8 mm²). The sticks were tested under tension at 0.5 mm/min. Some sticks from each tooth group were used for DC determination by micro-Raman spectroscopy or nanoleakage evaluation (NL). The pH for each solution was evaluated using a pH metre. Data were analyzed with one-way ANOVA and Tukey's test ($\alpha = 0.05$).

Results: For μ TBS, only PkSe and PkEr were similar to the respective control groups ($p > 0.05$). AlSe showed the lowest μ TBS mean ($p < 0.05$). For NL, ScEr, ScSe, AlSe, and AlEr showed the lowest NL similar to control groups ($p < 0.05$). For DC, only ScSe showed lower DC than the other materials ($p < 0.05$).

Conclusions: Performance of universal adhesives was shown to be material-dependent. The results indicate that this new category of universal adhesives used on dentine as either etch-and-rinse or self-etch strategies were inferior as regards at least one of the properties evaluated (μ TBS, NL and DC) in comparison with the control adhesives (CSE for self-etch and SB for etch-and-rinse).

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1. Introduction

The bonding mechanism of adhesive systems basically involves the replacement of minerals removed from the

hard dental tissue by resin monomers, in such a way that a polymer becomes micro-mechanically interlocked to the dental substrate.¹ However, the adhesive systems available on the market can be classified into two categories: etch-and-rinse (Er) and those applied using self-etch strategies

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Abbreviations: Co, composite; De, dentine; HL, hybrid layer; AL, adhesive layer.

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<http://dx.doi.org/10.1016/j.jdent.2013.03.001>

(Se), in versions of three (only Er), two or one application step.^{2,3}

When using the Er strategy, the first step involves the application of a phosphoric acid gel to both dental substrates, which allows removal of the smear layer, exposure of the collagen fibrils in dentine, and increase in surface area and surface energy in the enamel substrate. The primer is then applied (second step) followed by the bond (third step) resin separately or in a single solution.^{2–4} Irrespective of the number of steps, the main disadvantage of the Er system, mainly two-step versions, is that there is risk of collagen fibre collapse during the process of demineralized dentine drying, which leads to a decrease in bond strength.^{5,6} The collagen collapse is prevented by keeping demineralized dentine moist, which is a difficult task to perform clinically. In fact, adequate moisture depends on both the solvent used in the material⁷ and on the clinician's interpretation of the manufacturer's directions.

The incomplete impregnation of collagen fibers⁸ and the need to protect them against the degrading mechanisms present in the oral cavity environment,^{9,10} led to the development of the second category, an adhesive using the self-etch strategy.

In the Se strategy (one-step or two-step), there is no need to apply a preliminary phosphoric acid gel on dental substrates as dentine demineralization and priming occur simultaneously.^{3,11} The dissolved hydroxyapatite crystals and residual smear layer are incorporated in the hybridized complex.^{3,12} Except for very acidic Se systems,^{13,14} the whole extension of the demineralized dentine depth is impregnated by resin monomers, which may be the reason why Se systems are not associated with the technique sensitivity characteristic of bonding to moist etched dentine.^{7,15,16} This advantage makes Se materials suitable for areas where adequate control of moisture is rather difficult, such as in posterior restorations.

A clear disadvantage of the Se protocol is the reduction in enamel bonding effectiveness.^{17,18} The increase in surface area in intact and ground enamel obtained with Se adhesives is lower than that achieved with phosphoric acid, and it depends on the pH of the Se adhesive.¹⁸ The performance of Se adhesives has improved when these systems were applied to phosphoric acid-treated enamel.^{12,19,20} However, this procedure has been shown to be unsuitable for use on the dentine substrate,^{21–23} because accidental dentine etching may occur during the enamel-etching process, particularly when a low-viscosity etchant is used. The effect of intentionally etching dentine with phosphoric acid prior to the application of self-etch adhesives has been studied.^{21,23–26} The results are controversial and material-dependent.

Considering the differences in professional judgement regarding the selection of the adhesive strategy and number of steps, some manufacturers have released more versatile adhesive systems that include etch-and-rinse (two step) and self-etch (one or two step) options. These new materials are called “Universal”, “Multi-purpose” or “Multi-mode” adhesives.^{23,27} There is little information in the literature about the performance of this new class of universal adhesives.^{23,27} Thus, this study compared the immediate microtensile bond strengths (μ TBS), nanoleakage (NL), *in situ* degree of conversion (DC) of three universal adhesives applied to dentine according to the etch-and-rinse and the self-etch strategies.

The two-step etch-and-rinse, Adper Single Bond 2 (SB, 3M ESPE, St. Paul, MN, USA), and two-step self-etch, Clearfil SE Bond (CSE, Kuraray, Okayama, Japan) were also evaluated as control groups. The following null hypotheses were tested in this study: (1) universal adhesives applied to dentine according to the Er and the Se strategies when compared to their respective control groups do not affect the immediate resin-dentine bond strength; (2) universal adhesives applied to dentine according to the Er and the Se strategies when compared to their respective control groups do not affect the immediate silver nitrate deposition and (3) universal adhesives applied to dentine according to the Er and the Se strategies when compared to their respective control groups do not affect the degree of conversion of the adhesives.

2. Materials and methods

2.1. Tooth selection and preparation

Forty extracted, caries-free human third molars were used. The teeth were collected after obtaining the respective patients' informed consent under a protocol approved by the local Ethics Committee Review Board. The teeth were disinfected in 0.5% chloramine, stored in distilled water and used within six months after extraction. A flat dentine surface was exposed after wet grinding the occlusal enamel on a #180 grit SiC paper. The exposed dentine surfaces were further polished on wet #600-grit silicon-carbide paper for 60 s to standardize the smear layer.

2.2. Experimental design

The teeth were randomly assigned into eight groups ($n = 5$) according to the different bonding strategies of the selected adhesive system. As control materials, the 2-step etch-and-rinse (Er), Adper Single Bond 2 (SB, 3M ESPE, St. Paul, MN, USA); and the 2-step self-etch (Se), Clearfil SE Bond (CSE, Kuraray, Okayama, Japan) were used. The following three universal adhesive systems were tested: Peak Universal Adhesive System (Peak LC Bond and Peak SE Primer, Ultradent Products Inc., South Jordan, UT, USA), applied as a 2-step Er (PkEr) and 2-step Se (PkSe); Scotchbond Universal Adhesive (3M ESPE, St. Paul, MN, USA), applied as a 2-step Er (ScEr) and 1-step Se (ScSe); and All Bond Universal (Bisco Inc., Schaumburg, IL, USA) applied as a 2-step Er (AlEr) and 1-step Se (AlSe).

2.3. Restorative procedure and specimen preparation

The adhesive systems were applied strictly in accordance with the respective manufacturer's instructions, described in Table 1. After the bonding procedures, all teeth received a micro-hybrid composite restoration (Opallis, FGM Produtos Odontológicos, Joinville, SC, Brazil) in two increments of 2 mm. Each increment was light polymerized for 40 s using a LED light curing unit set at 1200 mW/cm² (Radium-cal, SDI Limited, Bayswater, Victoria, Australia).

After the restored teeth had been stored in distilled water at 37 °C for 24 h, the specimens were sectioned longitudinally in the mesio-distal and buccal-lingual directions across the

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