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Effect of smear layer thickness and acidity of self-etching solutions on early and long-term bond strength to dentin

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Summary *Objectives*. To evaluate the effect of smear layer thickness (SL) on early and 6-month bond strength (BS) of self-etching adhesives to dentin and to measure the ultimate microtensile strength (UTS) of the adhesives.

Methods. Clearfil SE Bond; Optibond Solo Plus Self-Etch Primer; Tyrian Self Priming Etchant (TY) and as controls, Single Bond (SB) and Scotchbond Multi-Purpose Plus (SBMP) were applied on flat superficial dentin surface with thick and thin SL thicknesses. After adhesive's application ($n=6$) a resin build-up was made. After 24 h, resin–dentin beams (0.8 mm^2) were prepared to be tested immediately and after 6-month (6M) at 0.5 mm/min. For the UTS measurement, hour-glass specimens were prepared with the bonding resin alone or after mixing (1:1). BS values were analyzed by three-way repeated measures ANOVA and Tukey's multiple comparison tests. Two-way ANOVA (bonding resin and bonding resin + self-etching primer) and Tukey's test were used for the UTS values. The bonding resins were re-evaluated separately by a one-way ANOVA and Tukey's test, since Single Bond is a one-bottle adhesive ($\alpha=0.05$).

Results. The SL thickness was not significant ($p=0.64$). BS values were reduced after 6M, except for the SBMP. TY provided the lowest BS mean while SB and SBMP the highest BS. The UTS of the SBMP was the highest. TY yielded the lowest UTS. Regression analysis revealed a linear and significant relationship between the UTS of self-etch systems and the mean BS ($R=0.95$, $p=0.02$).

Conclusions. The performance of a self-etching system does not seem to be dependent on the SL thickness. The total-etch, three-step system provided the highest BS to dentin and maintained the BS stable over 6 months. The performance of the self-etching systems can be envisaged by their UTS.

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Introduction

Total-etch systems require a conditioning, a rinsing and a priming step in order to allow encapsulation of collagen fibers by the resin monomers and the formation of the so-called 'hybrid layer'.[1] Demineralization of intertubular dentin and maintenance of interfibrillar porosity is required for monomer penetration into dentin. Thus, wet bonding technique has been designated as essential for the current adhesive systems in order to prevent the known collapse of collagen fibrils in air-dried conditions.[2,3] The proper degree of moisture is individual for each solvent-based adhesive system;[4,5] however, even under ideal condition, an actual discrepancy between the extent of the demineralization and monomers infiltration is likely to occur.[6–8] The development of self-etch systems avoided the occurrence of the above disadvantages from total-etch systems. This bonding approach has reintroduced the concept of employing the smear layer as a bonding substrate, but with novel insights and improved formulas that can etch beyond the smear layer into the underlying dentin. Simultaneous infiltration of the demineralized dentin matrix during acid etching is possible, as the demineralizing component of the primer is also the infiltrating resin.[9]

The hybridized complex of self-etch systems is comprised of a surface zone of hybridized smear layer and a subsurface zone of hybridized intertubular dentin.[10–12] To achieve this goal, the self-etching primer should penetrate beyond the smear layer into the intact, mineralized dentin. Under clinical circumstances, the thickness, coarseness, and roughness of smear layers may vary according to the rotary instruments used for cavity preparation.[13,14] Thick smear layers might affect the ability of self-etching systems to penetrate through intact, mineralized dentin, since early neutralization of the adhesive by the dentin buffering components presented in the smear layer[15] might hamper superficial demineralization of solid dentin, which is required for collagen exposure. Studies addressing this matter have not reached a conclusion about the performance of self-etching systems applied to varied smear layer thickness. Some studies reported low resin–dentin bond strengths over thick dentin smear layers,[16–18] while others reported no influence of smear layer thickness on resin–dentin bond strengths.[11,13]

To the authors' knowledge, there is no information as to whether the thicknesses of smear layers incorporated in the hybridized complex affect the long-term resin–dentin bond strength. Other important issue that has been disregarded in most studies and may play a role on the adhesive performance is the ultimate strength of the adhesive systems employed. Therefore, the objective of this study was to determine the effects of varied dentin smear layer thicknesses on the early and six-month bond strength of three two-step self-etching systems possessing different levels of acidity, and to measure the ultimate microtensile strength of the bonding resin and the self-etching primer. The null hypothesis to be tested was: (1) there will be no influence of smear layer thicknesses on early and 6-month bond strengths to dentin; (2) the bond strengths over thick and thin smear layer covered dentin will not be dependent on the acidity of self-etch systems and; (3) the ultimate strength of the adhesives will not be different among each other.

Materials and methods

Experimental design and teeth preparation

This study had the approval of the Ethical Committee of the University of São Paulo, School of Dentistry. Thirty non-carious, human third molars were used. The teeth were disinfected in 0.5% chloramine and used within 6 months following extraction. The occlusal enamel was wet ground using a 180-grit SiC paper, in order to expose a superficial flat dentin surface. The teeth were longitudinally sectioned in a buccal to lingual direction (Labcut 1010, Extec Corp., Enfield, CT, USA) into two halves (Fig. 1). The occlusal surface of one half was further polished with wet 600-grit SiC paper for 60 s to create thin smear layer and the other half was treated alike with a 60-grit SiC (thick smear layer). Then, six teeth were randomly assigned into five experimental groups (Fig. 1).

Three self-etching adhesive systems were selected according to their acidity, as provided by the manufacturers: Clearfil SE Bond (SE—Kuraray Medical, Inc., Osaka, Japan)—mild ($pH > 2$), Optibond Solo Plus Self-Etch Primer + Optibond Solo Plus (SO—Kerr; Orange, CA, USA)—moderate ($1 < pH < 2$) and Tyrian Self Priming Etchant (SPE) + One-Step Plus (TY—Bisco, Schaumburg, IL, USA)—aggressive ($pH < 1$). Other two total-etch adhesive systems were used as controls: Single Bond (SB—3M ESPE, St Paul, MN, USA), a two-step adhesive system, and Scotchbond Multi Purpose Plus (SBMP—3M ESPE), a three-step adhesive system. Their composition, application mode, and batch number are described in Table 1.