### Effect of Surface Treatment on Bond Strength of Two Self-Etching Adhesives to Astringent Exposed Dentin

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## Dedication

I wish to dedicate this work to

my Great Parents,

my supporting brothers,

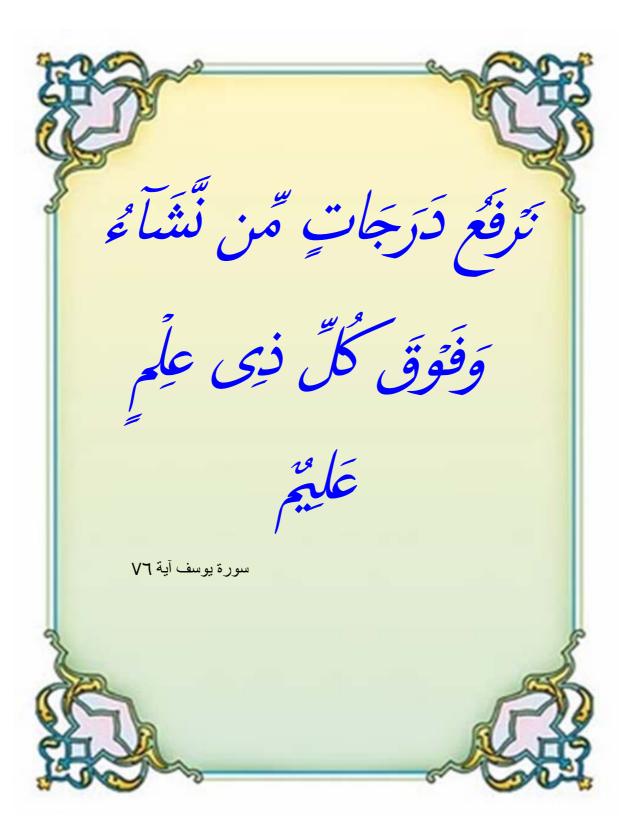
and my Lovely Wife.

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Dentin continues to represent a challenge in bonding with resin-based adhesives because of its complexity and dynamism. Quality bonding of composite resins to dentin depends on appropriate incorporation of the resin into the demineralized dentinal matrix formed through etching. It is possible that clinical contaminants such as saliva, blood, or handpiece oil prevent the composite resin from wetting and penetrating into the acid conditioned dentin surface. Contaminants may serve to obstruct the flow of resin monomers into the dentinal tubules. Small contaminant particles may penetrate the dentinal tubules and ultimately affect the development of the hybrid Layer (1-4).

While preparing for and placing restorations below the gingival margins, hemostasis becomes of utmost importance in maintaining the ideal, contaminant-free working field. If bleeding does occur, one approach to accomplish a successful bonding procedure is to use a hemostatic agent. Examples of these materials are aluminum chloride, aluminum sulfate and ferric sulfate <sup>(5-7)</sup>.

Previous studies have demonstrated that these hemostatic agents or astringents are highly acidic and their pH varies from 0.7-3.0 and that hemostatic agents interfere with bonding of composite resins. Contamination with astringents or hemostatic agents can have a detrimental effect on the bond strength of self-etching adhesive systems to enamel and dentin. Total-etch adhesives may have the benefit of separate etching step which removes the smear layer with all contaminants incorporated, therefore less pronounced effect of the contamination on the bond strength to dentin. However, self-etching adhesives appear to be more susceptible to reduction in bond strength than total-etch adhesives because of the absence of pre-conditioning of the dentin that removes the smear layer with its contaminants which is incorporated within the hybrid layer; therefore

contamination can affect the quality of bond to dentin and has more pronounced effect on the bond strength of self-etching adhesives (8-11).

A recently introduced astringent based on aluminum chloride has been advocated for use with resin composite because it doesn't result in discoloration of the operative site on the contrary to ferric sulphate astringents which cause discoloration due to the residues left. This is attributed to the mechanism of action of aluminum chloride which depends on shrinkage of blood vessels to achieve hemostasis. As opposed to ferric sulphate that depends on blood coagulation with resultant residues (11).

Many decontamination procedures were suggested in literature prior to the application of the adhesive (glycolic acid, chlorhexedine or doubling the application time of the self-etching primer) (7,11). However, there is little information about the effect of pre-conditioning of dentin with phosphoric acid (for 5 seconds) after exposure to astringent in the literature. From all the mentioned above, it was found beneficial to investigate the effect of aluminum chloride astringent on the shear bond strength of one-step and two-step self-etching adhesives and the effect of different surface treatments protocols.

#### **I-Self-etching adhesives:**

Probably, in regard to user-friendliness and technique sensitivity, clinically, the most promising approach is self-etch. It no longer needs an "etch & rinse" phase, which not only lessens clinical application time, but also significantly reduces technique-sensitivity or the risk of making errors during application and manipulation. Another important advantage of the self-etch approach is that infiltration of resin occurs simultaneously with the self-etching process, by which the risk of discrepancy between both processes is low or non-existent <sup>(12)</sup>.

Tay and Pashley, 2001 (13), examined with the use of transmission electron microscopy (TEM), the aggressiveness of three self-etching adhesive systems in penetrating dentin smear layers of different thickness. Dentin disks were produced from extracted human third molars. For the control group, the middle dentin surface was cryofractured to create a bonding surface that was devoid of a smear layer. The experimental teeth were polished with wet 600 or 60 grit SiC paper to produce bonding surfaces with thin and thick smear layers. They were bonded using one of the three self-etching systems: Clearfil Mega Bond (Kuraray), Non-Rinse Conditioner and Prime&Bond NT (Dentsply DeTrey) and Prompt L-Pop (ESPE). Bonded specimens were then demineralized and embedded in epoxy resin for TEM examination. It was found that for Mega Bond, thin authentic hybrid layers between 0.4-0.5 µm were found. Smear layer and smear plugs were retained as part of the hybridized complex. For Non-Rinse Conditioner/Prime&Bond NT, the authentic hybrid layers were between 1.2-2.2 µm thick. Smear layer and smear plugs were completely dissolved in dentin with thin smear layers, but were partially retained as part of the hybridized complex in those with thick smear layers. For Prompt L-Pop, authentic hybrid layers were 2.5-5 µm thick and smear layer and smear plugs were completely dissolved even in dentin with thick smear layers. It was concluded that contemporary self-etching systems may be classified as mild, moderate and aggressive based on their ability to penetrate dentin smear layers and their depth of demineralization into the subsurface dentin. The more aggressive system completely solubilized the smear layer and smear plugs and formed hybrid layers with a thickness approaching those of phosphoric acid conditioned dentin.

Moll et al, 2002 (14), compared the bonding potential to human dentin of adhesive/composite combinations including five 2-step and two 3-step total-etch (TE) bonding systems, two systems with self-conditioning (SC) primers, and one SC all-in-one adhesive by use of the microtensile bond test and the debonded surfaces were then examined under the SEM for mode of failure. The results showed that the mean bond strengths of the simplified (2-step) TE systems were not significantly lower than that of the traditional 3-step TE systems, and not related to phosphoric acid concentration. Dentin treatment with SC primers was as effective as etching with phosphoric acid. The SC all-in-one adhesive produced significantly lower bond strength than all other systems evaluated. It was suggested that the use of adhesive/composite combinations including simplified bonding systems does not necessarily result in reduced bond strength to dentin. This study showed that SC primers offer a promising alternative to phosphoric acid etching as far as bonding to dentin is concerned. In contrast, the SC all-in-one adhesive evaluated needs to be improved.

Armstrong et al, 2003 <sup>(15)</sup>, evaluated dentin bond durability using dentin adhesive resin bonding approaches over a 15-month period of water storage. Forty four extracted human molars were polished with 600 grit SiC papers exposing occlusal dentin, and randomly distributed into four adhesive groups: total-etch 3-

step (TE3), total-etch 2-step (TE2), self-etch 2-step (SE2), and a self-etch 1-step (SE1). A resin composite crown was incrementally formed and light cured. Microtensile specimens were fabricated and stored in distilled water containing 0.5% chloramine T and tensile tested at 1 mm/min after 1, 6, and 15 months. The debond pathway was recorded as either involving the substrate or joint using scanning electron microscopy. The results showed that the TE2 was significantly weaker than TE3 and SE2 after 1 and 6 months of storage, but all three systems were equivalent after 15 months of storage. The SE1 system could not be tested due to 58 of 65 specimens failing during specimen preparation. Failure modes were observed to be dependent upon adhesive system, with only the total-etch 2-step system demonstrating an increasing involvement in the adhesive joint over time. This study revealed that although differences in bond strength were observed across adhesive systems up to 6 months of storage, no differences were noted at 15 months. This may represent common degradative mechanisms.

*Mallmann et al, 2003* <sup>(16)</sup>, compared the bond strength of a self-etching (Clearfil SE Bond) and a single-bottle (Excite) adhesive system using two cavity configurations (C-factors 5 and 1). Class I cavities were prepared in 28 extracted human molars using diamond burs under water cooling. Teeth were divided into 4 groups: G1: Excite, C-factor 5; G2: Excite, C-factor 1; G3: Clearfil, C-factor 5; G4: Clearfil, C-factor 1. To determine C-factor 5, systems were applied to all cavity walls according to the manufacturers' instructions (5 bonded, 1 unbonded). For C-factor 1, lateral walls were isolated using nail varnish, and adhesive systems were only applied to the pulpal floor (5 unbonded, 1 bonded). Cavities were restored using Tetric Ceram composite resin, and bulk light cured. Teeth were stored in distilled water for 24 h at 37°C, and then sectioned using a diamond disk, yielding stick-shaped specimens. Specimens were submitted to the microtensile

bond test at a rate of 1 mm/min speed in a universal testing machine. The results revealed that there was no statistically significant difference between adhesive systems for C-factor 1. For C-factor 5, Clearfil SE Bond produced higher values when compared to the other group. It was found that changes in C-factor only affected the total-etch adhesive system tested. This may be a result of the different filler volume in the self-etching system, and not of the bonding technique itself.

Ernest et al, 2004 (17), determined the shear bond strength (SBS) of different established (Resulcin Aqua Prime & Monobond N: RA, Prompt L-Pop III: PLP) and experimental (AC-Bond: AC, AC-Bond + Desensitizer: ACD) selfetching adhesives in comparison to fourth FL: FL) (Optibond and fifth generation (Excite: EX, Gluma Comfort Bond: CB) adhesives. All adhesives were applied on flat enamel and dentin surfaces and light cured following manufacturers' directions. Composite cylinders were sheared off after thermocycling. FL resulted in significantly higher SBS in enamel and dentin than RA, AC, ACD, and PLP, and in higher SBS to dentin than CB. In enamel and dentin, RA performed significantly superior to PLP, but was not different from AC and ACD. EX and CB were both on the same level of significance as AC and RA, but showed superior results to ACD and PLP (enamel and dentin). PLP resulted in significantly lower SBS values in enamel and dentin than all the other materials investigated, except ACD in dentin, to which it was equivalent. It was found that Resulcin Aqua Prime & Monobond N and AC-Bond were not significantly different than established 5th generation products. AC-Bond + Desensitizer and significantly different **Prompt** L-Pop have SBS from established 4th and 5th generation products.