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Effect of the Number of Applications of Acetone-based Adhesives on Microtensile Bond Strength and the Hybrid Layer

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Clinical Relevance

The application technique of total- and self-etching bonding resins affects their bonding strength to dentin.

SUMMARY

Purpose: The current study was carried out to evaluate the effect of doubling the adhesive layers of three acetone-based adhesives on the microtensile bond strength and ultra morphological characterization of the resin dentin interface using SEM.

Materials and Methods: A total of 27 caries-free human molars were used. Superficial flat dentin surfaces were obtained by wet grinding the buccal surfaces. Three adhesive systems Prime & Bond NT (G1), XENO IV (GII) and G BOND (GIII) were used according to three different protocols: (A) according to the manufacturer's instructions, (B) doubling the adhesive layers and light curing and (C): doubling the adhesive layers with intermediate curing between each layer. Resin composite buildups were made using TPH Spectrum resin composite on the bonded surfaces in 1 mm light cured increments for 40 seconds each. The bonded teeth were sectioned to obtain sticks for microtensile testing. The testing was conducted using a universal testing machine at a crosshead speed of 1 mm/minute. The microtensile bond strength means and standard deviations were calculated and the data were statistically analyzed using Two-way Analysis of Variance (ANOVA) and Tukey's post-hoc tests ($p \leq 0.05$).

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DOI: 10.2341/08-089-L

Ultra morphological characterization of the resin-dentin interface and representative fractured dentin specimens were examined using SEM.

Results: When applied according to the manufacturer's instructions, Prime & Bond NT (total etch adhesive) showed significantly high microtensile bond strength with a mean value ± SD of 35 ± 12.7 MPa followed by XENO IV (21.2 ± 9.4 MPa), while G BOND presented a significantly lower mean value (10.9 ± 2.9MPa). Doubling the adhesive layers significantly reduced the mean strength of the total etch adhesive system; in contrast, it significantly increased the bond strength of both self-etch adhesives. The relatively thicker adhesive layer was seen with the total-etch adhesive when the application was doubled, while the hybrid layer appeared thicker with self-etch adhesives.

Conclusions: Doubling the adhesive layer applications significantly improved the bond strength of the two self-etch adhesives (XENO IV and G BOND); however, it had a negative effect on the bond strength of the total-etch adhesive (Prime & Bond NT).

INTRODUCTION

One of the challenges in restorative dentistry research is developing adhesive restorative materials that provide an effective bond to dental tissues and, consequently, offering successful restorative treatment.[1-2] Bonding to enamel is now considered a durable and predictable clinical procedure, while bonding to dentin has been inconsistent.[3-5] The dynamic nature, structure and chemistry of this anisotropic biologic composite affects the bonding mechanism.[6-8]

The basic mechanism of bonding to enamel and dentin is already well established and based on an exchange process between the inorganic tooth material and resin. This process involves two phases: the first phase consists of removing calcium phosphates from enamel and dentin with the subsequent formation of microporosities in both. The second phase involves infiltration and subsequent *in situ* polymerization of resin within the created microporosities forming the so-called hybrid layer. The resulting micromechanical interlocking is based on a diffusion mechanism that was first described by Nakabayashi and others in 1982 and is commonly referred to as "hybridization."[9-11] Based on the above adhesion strategy, the currently available adhesive systems involve two approaches: an etch and rinse approach and a self-etch approach.[12]

In the etch and rinse approach, the etching process is carried out using 37% phosphoric acid, resulting in removing the smear layer, opening the dentinal tubules and selectively demineralizing the intertubular dentin, thereby exposing a scafollad collagen network. The interfibrillar microporosities allow resin monomer to infiltrate the collagen layer, forming the resin-dentin interdiffusion zone.[13] Several authors have addressed the sensitivity associated with the above approach, especially when water miscible organic solvents are incorporated into such adhesives.[14-15]

Trends toward simplification of bonding procedures and overcoming the aforementioned problem have led to the introduction of self-etch adhesives. In this approach, the rinsing phase is eliminated, which does not only reduce clinical application time, but also significantly decreases technique sensitivity or the risk of making errors during application.[16-18] Unfortunately, it seems that this simplicity relates to the extent of efficacy.[19] Several authors have reported that self-etch adhesive systems did not improve bonding effectiveness to dentin in spite of their purported reduction in technique sensitivity.[20-21] Furthermore, their increased hydrophilicity compromised bonding durability, as it was found that one-step self-etch adhesives result in an overall poorer performance compared with total-etch alternatives.[22]

To offset the limitations of self-etching adhesives, altered bonding protocols that increase resin-dentin bond quality were suggested.[23-24] Among the alternative bonding strategies are the multiple application of additional coats of adhesive[23-25] or increased substrate contact time of the acidic primers.[24]

Bond efficacy has been investigated in the literature using several *in vitro* testing methods, including shear tensile and microtensile bond strength testing procedures. The microtensile bond testing technique enables the investigation of interfacial bond with the reduced probability of pulling out dentin from a flat surface, similar to that often reported when testing in shear or conventional tensile mode. It allows the highest stress to be focused on the bonded interface.[26-27] The original version of this test, which involved the use of a dumbbell-shaped specimen design, was not suitable for the evaluation of materials with relatively low bond strength to dentin (5-7 MPa), as premature bond failure often occurred during "free-hand" bur trimming of the specimens.[28] A recent non-trimming version of the microtensile bond test replaced the use of dumbbell-shaped test specimens with beams of a uniform cross-sectional area that were sectioned from the bonded restorations.[29] The interfacial analysis used by SEM was found to be an ideal complement to the μTBS method, which allows for looking into the clues of micro- and ultra-morphological appearances of the different attempts for successful bonding to human dentin.[30]

The objectives of this study were designed to evaluate the effect of doubling the adhesive layers of three ace-

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tone-based adhesives, one total-etch and two self-etch adhesives on microtensile bond strength and to study the ultra morphological characterization of the resin dentin interface of such a bond using SEM.

METHODS AND MATERIALS

Selection of Teeth and Teeth Preparation

Twenty-seven caries-free human molars were used in the current study. The teeth were stored in physiologic saline at 4°C for no more than two weeks. Superficial flat dentin surfaces were produced by wet grinding the buccal surfaces on a polishing machine (TF 250, Jeanwirtz, Germany) with 180 grit silicon carbide paper to produce a polished surface with a clinically relevant smear layer.[31]

Bonding Procedure

The teeth were divided into three equal groups and assigned to the three adhesives. The teeth from each group were further subdivided into three equal subgroups. Three adhesive systems were used in the current study (material composition and manufacturer are presented in Table 1): one total-etch adhesive (Prime and Bond NT) and two one-step self-etch adhesives (XENO IV and G BOND). The adhesives were applied using three different bonding protocols corresponding to the three subgroups: (A) applied following the manufacturers' instructions, (B) doubling the number of coats and light curing and (C) doubling the number of coats with light curing carried out between coats, as shown in the experimental design (Figure 1).

For each tooth composite, build-up was made in three increments of 1 mm each using TPH Spectrum resin composite (Dentsply Caulk, Milford, DE, USA). Each increment was light cured for 40 seconds using Elipar (ESPE light curing unit). The bonded teeth were then stored in deionized water at 37°C for one week.

Microtensile Testing

The bonded teeth were sectioned to obtain rectangular sticks with a cross sectional area of 0.8 x 0.8 mm^2 ± 0.1 mm^2 and 6 mm long. A low-speed diamond saw (Isomet 1000, Buehler Ltd, Lake Bluff, IL, USA) was used under copious water to section the bonded teeth serially perpendicular to the bonded surfaces. Only the six central sticks were selected from each tooth for each subgroup (n=18). The bonded surface area at the adhesive interface was calculated before testing by measuring the width and thickness of each specimen using the Nikon Measure scope UM-2 (Nikon Corporation, Kanagawa, Japan). Each stick was then mounted on a Bencor Multi-T device (Danville Engineering, Danville, CA, USA) with cyanoacrylate glue (Loctite Super Glue, Henkel Consumer Adhesives Inc, Avon, OH, USA) and loaded to failure in tension using a universal testing machine (Sintech Renew 1123, MTS Headquarters, Eden Prairie, MN, USA) using 25 N load cell traveling at a crosshead speed of 1 mm/minute. The microtensile bond strength was then calculated and expressed in MPa.

Statistical Analysis

Two-way Analysis of Variance (ANOVA) was carried out using SPSS 14.0 and Tukey's post-hoc test at $p \leq 0.05$.

Scanning Electron Microscopy (SEM)

A–Ultra morphological examination of the resin-dentin interface:

Additional bonded specimens from each subgroup were examined using SEM. The specimens were ground with 600 grit abrasive papers. The polished surfaces were immersed in 6 mol/L HCL for 30 seconds to remove the smear layer. They were then immersed in 1% NaOCl for 10 minutes and ultrasonically cleaned in

Table 1: *Materials' Composition, Manufacturers and Manufacturers' Instructions for Use*

Material	Composition	Manufacturers' Instruction	Manufacturer
Prime and Bond NT (P&B NT)	35% phosphoric acid UDMA, R5-62-1 resin-T resin, D resin butylated hydroxyl toluene, functionalized silica, PENTA, camphorquinone, stabilizers, cetylamine hydrofluoride and acetone. The bonding system is reinforced by nanofillers.	Apply using saturated brush and leave undisturbed for 20 seconds. The surfaces are then gently air dried for 1-3 seconds and cured for 20 seconds.	Dentsply Caulk, Milford, DE, USA
XENO IV (X IV)	PENTA, UDMA, mono-, Di- and Trimethacrylates, Photoinitiators, stabilizers, cetylamine hydrofluoride, acetone, water.	Apply two consecutive coats, with each coat actively scrubbed for 20 seconds. Then, gently air-dry the surfaces to obtain a smooth, glossy surface. Light cure for 10 seconds.	Dentsply Caulk, Milford, DE, USA
G BOND (G B)	4-MET, UDMA, TEGDMA, acetone, water, fumed silica fillers, photoinitiator.	Apply a single coat, leave undisturbed for 5-10 seconds, dry thoroughly under maximum air pressure for 5 seconds. Light cure for 10 seconds.	GC Corporation, Tokyo, Japan

PENTA: Dipentaerythritol pentaacrylate
4-MET: 4-Methacryloxyethyltrimellitate
TEGDMA: Triethyleneglycol dimethacrylate
UDMA: Urethane dimethacrylate

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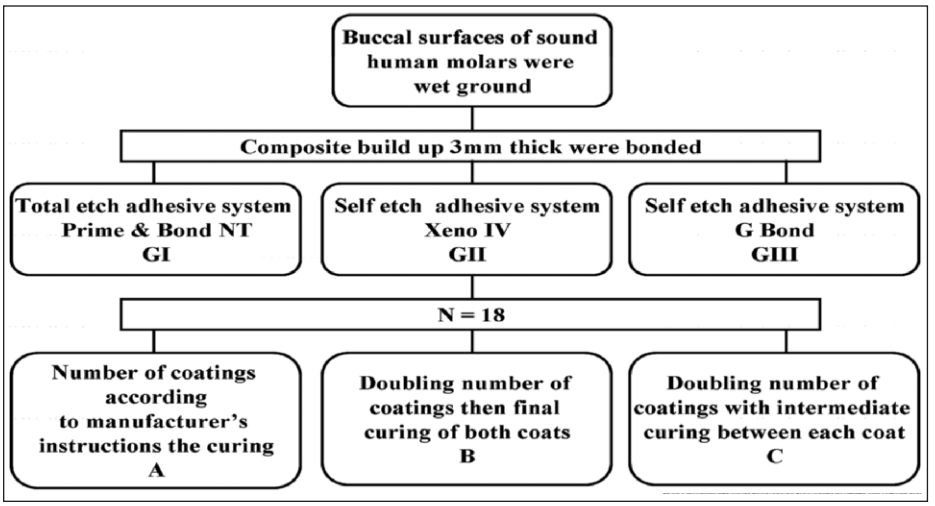

Figure 1. *Experimental design.*

distilled water for five minutes. The specimens were dehydrated using 99% ethyl alcohol for 30 seconds, then left to dry.

B–Ultra morphological examination of the fractured sticks:

Four representative fractured sticks from each subgroup, with a bond strength value close to the mean bond strength of the group, were selected for fractographic analysis. The dentin sides of the fractured specimens were air-dried.

All the specimens were gold sputtered under vacuum (Ladd Sputter Coater, Ladd Research, Williston, VT, USA) and examined using a scanning electron microscope (JSM 5310LV, JEOL Inc, Tokyo, Japan) at 20 kV accelerating voltage. Images of the resin-dentin interface were viewed at 1500x magnification, while images for fractographic analysis were examined at 1000x magnification.

RESULTS

Microtensile Bond Strength

The means and standard deviations of microtensile bond strength for all subgroups are shown in Table 2 and Figure 2. When applied according to the manufacturer's instructions, Prime & Bond NT showed the highest statistically significant bond strength compared to the two self-etch adhesives. However, doubling the number of adhesive layers significantly decreased the bond strength of Prime & Bond NT, while it significantly increased the values for the two self-etch adhesives. Furthermore, single curing of the doubled adhesive layers had a significant effect on G BOND by increasing its mean bond strength by more than twofold; it also increased the mean bond strength of XENO IV albeit, not significantly. Two-way Analysis of Variance (ANOVA) revealed that the adhesive systems factor significantly affected the microtensile bond strength ($p<0.036$); however, the technique of adhesive application had no significant effect ($p=0.196$). The interaction between both factors—adhesive type and application technique—was statistically significant ($p=0.003$). The mean microtensile bond strength statistical ranking is GIA>GIIIB=GIIB=GIIC=GIB>GIIA=GIC>GIIIC>GIIA.

SEM

Figure 3 shows SEM image of the resin-dentin interfaces treated with Prime & Bond NT. The hybrid layer can be seen with long and numerous resin tags extending down from the resin-impregnated dentin layer in all subgroups (A, B and C). Doubling the number of adhesive layer applications leads to increased thickness of the adhesive layer only (Figures 3B and 3C); however, this was more pronounced in the subgroup with intermediate curing.

Figure 4 shows SEM images of the fractured dentin specimen's interfaces treated with Prime & Bond NT applied according to the manufacturer's instructions (A), doubling the number of adhesive layers followed by curing (B) and doubling the number of layers with intermediate curing (C). In Figure 4A, the failure was mostly at the top of the hybrid layer (THL), with the

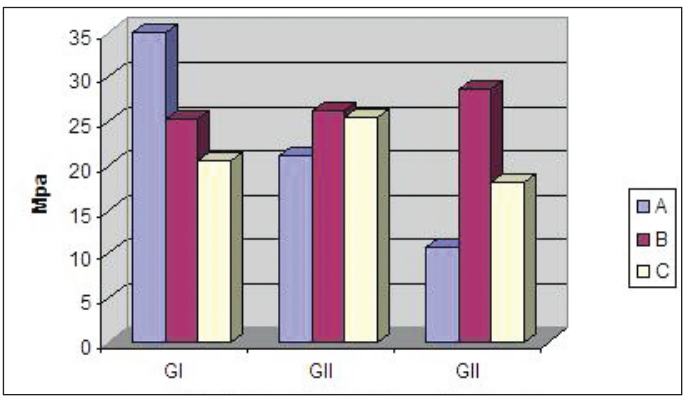

Figure 2. *Means of microtensile bond strength.*

Table 2: *Means (SD) of Microtensile Bond Strength Values of All Groups (MPa)*

	GI	GII	GIII
A	35 (12.7)[a]	21.2 (9.4)[c]	10.9 (2.9)[e]
B	25.3 (9.5)[b]	26.2 (7.8)[b]	28.6 (11.6)[b]
C	20.5 (8.1)[c]	25.5 (5.9)[b]	18.1 (7.3)[d]

Means with the same letters are not significantly different at p≤0.05.

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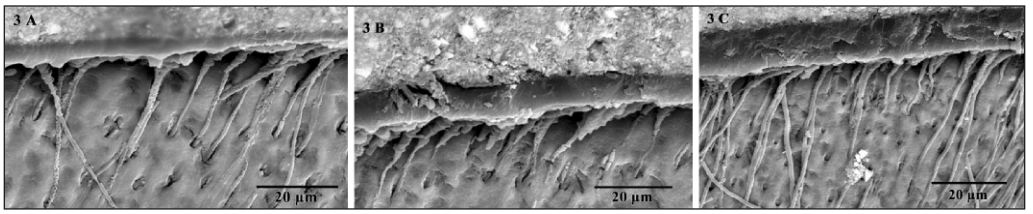

Figure 3: *SEM of the resin dentin interfaces treated with Prime & Bond NT adhesive system. Figure 3A applied according to the manufacturer's instructions, Figure 3B doubling the number of adhesive layers followed by curing, Figure 3C doubling the number of layers with intermediate curing.*

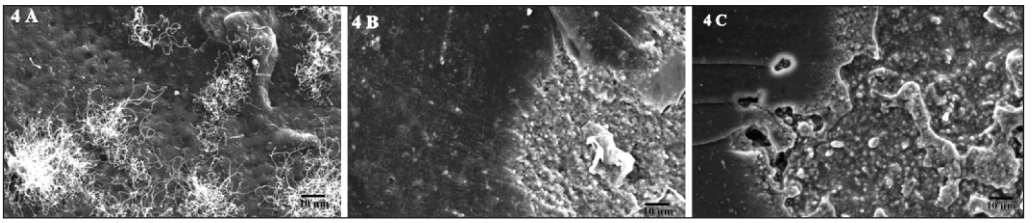

Figure 4: *SEM of fracture dentin surfaces treated with Prime & Bond NT adhesive system. Figure 4A applied according to the manufacturer's instructions, Figure 4B doubling the number of adhesive layers followed by curing, Figure 4C doubling the number of layers with intermediate curing.*

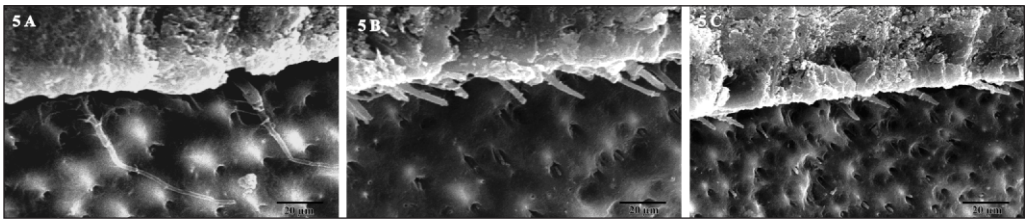

Figure 5: *SEM of the resin dentin interfaces treated with the XENO IV adhesive system. Figure 5A applied according to the manufacturer's instructions, Figure 5B doubling the number of adhesive layers followed by curing, Figure 5C doubling the number of layers with intermediate curing.*

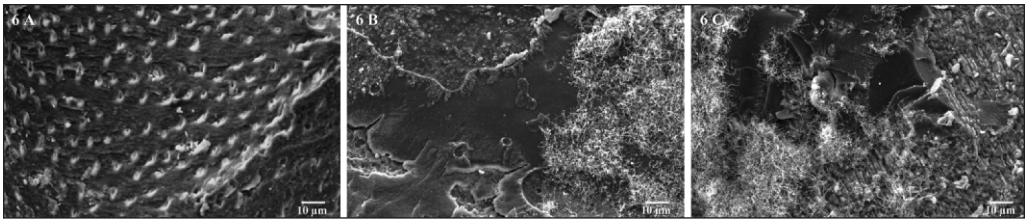

Figure 6: *SEM of fracture dentin surfaces treated with the XENO IV adhesive system. Figure 6A applied according to the manufacturer's instructions, Figure 6B doubling the number of adhesive layers followed by curing and Figure 6C doubling the number of layers with intermediate curing.*

presence of irregular collagen fibrils, while in Figures 4B and 4C, fractured surfaces did not show exposed collagen fibrils, indicating that the failure was transferred to the junction between THL and the adhesive layer.

Figure 5A shows a SEM image of the resin dentin interface treated with the XENO IV subgroup when the adhesive was applied according to the manufacturer's instructions. This figure revealed the relatively thin hybrid layer with scarce resin tags. While doubling the number of adhesive layers, followed by curing or with intermediate curing, Figures 5B and 5C show the relative increase in thickness of the hybrid layer and the increase in the number of resin tags compared to Figure 5A; however, these resin tags appear short.

SEM of the fractured dentin surfaces bonded with XENO IV applied according to the manufacturer's instructions (Figure 6A) showed failure partly at THL and partly at the base of the hybrid layer (BHL). By doubling the number of adhesive layers followed by curing (Figures 6B and 6C), the same failure occurred with relatively open dentinal tubules, where numerous irregular collagen fibrils on the THL are visible.

Figures 7A, B and C show SEM images of the resin dentin interfaces treated with G BOND. An ultra-thin hybrid layer formed a nano-interaction zone, with the absence of resin tags being seen when the adhesive was applied according to the manufacturer's instructions (Figure 7A). Figure 7B shows a relative increase in thickness of the hybrid layer with an increase in the number of resin tags when the number of adhesive layers was doubled followed by curing. However, when intermediate curing was carried out between the adhesive layers (Figure 7C), there was an increase in the thickness of the adhesive layer only without resin tags.

Figures 8A, B and C show SEM images of the fracture dentin surfaces of the above group. In Figure 8A, a mixed type of failure can be seen at THL and BHL, with numerous air bubbles present. With doubling the number of adhesive layers followed by curing, the failure was mainly at THL, with the presence of numerous air bubbles and fractured resin tags occluding dentinal tubules orifices. Figure 8C shows a mixed mode of failure, with part of the failure at THL and the other at BHL.

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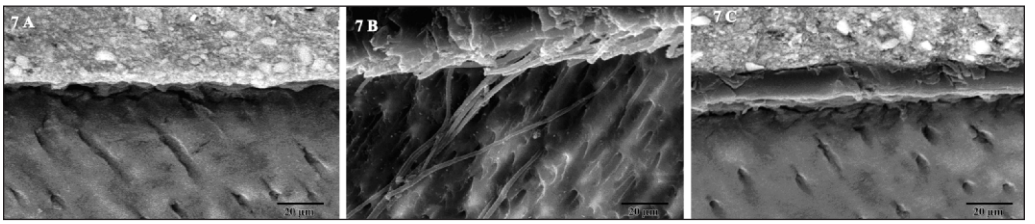

Figure 7: *SEM of the resin dentin interfaces treated with the G BOND adhesive system. Figure 7A applied according to the manufacturer's instructions, Figure 7B doubling the number of adhesive layers followed by curing, Figure 7C doubling the number of layers with intermediate curing.*

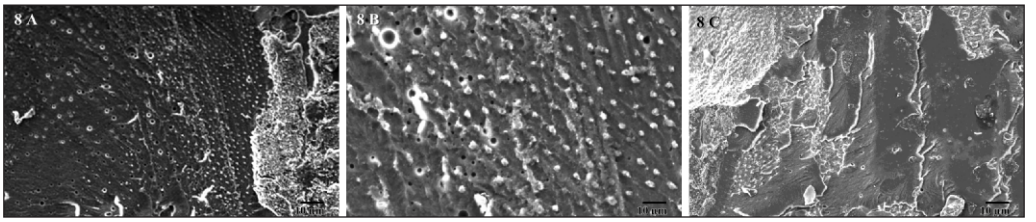

Figure 8: *SEM of fracture dentin surfaces treated with the G BOND adhesive system. Figure 8A applied according to the manufacturer's instructions, Figure 8B doubling the number of adhesive layers followed by curing, Figure 8C doubling the number of layers with intermediate curing.*

DISCUSSION

Prime & Bond NT showed the highest statistically-significant mean microtensile bond strength when applied following the manufacturer's instructions. This reinforces the idea that etch-and-rinse adhesives are still the benchmark for other adhesives when it comes to *in vitro* performance.[10-32-34] These findings were also confirmed by the SEM photomicrograph in Figure 3A, where the hybrid layer had long and numerous resin tags, explaining the high bond exhibited by this group. This could be attributed to the relative compatibility between the depth of demineralized dentin created by acid etching and resin monomer infiltration. The fracture mode of this group also confirmed that failure was mainly noted at the top of the hybrid layer (THL), indicating the existence of a hybrid layer with sufficient load-bearing capacity. On the other hand, doubling the adhesive coats in this group led to an increase in the thickness of the adhesive layer without affecting the hybrid layer thickness. This thick adhesive acted as a weak point, since it transferred the point of failure from THL to the junction between THL and the adhesive resin (Figures 4B and 4C). This may have, in turn, resulted in the lower bond strength exhibited by this group.

The low bond strength exhibited by the self-etch adhesives in the current study could be attributed to the fact that, attempting to incorporate all the necessary qualities of effective dentin adhesive, such as wettability, acidity, capability of penetration and cohesive strength, into one chemical composition, may have compromised the overall performance.[34] This is in agreement with previous research that demonstrated that self-etch adhesives do not improve bonding effectiveness to dentin in spite of their purported reduction of technique sensitivity.[20-21] According to the findings of the current study, the absence of resin tags in the self-etch adhesive subgroups when adhesives were applied according to the manufacturer's instructions, Figures 5A and 7A, compared to the total etch subgroup Figure 3A, is obviously the contributing factor for low bond strength as suggested by the theoretical model proposed by Pashley and others.[35] One study suggested that contribution of the resin tags to bond strength in superficial dentin ranges from 9% to 15%.[36]

The two self-etch adhesives used in the current study are considered mild HEMA-free adhesives. The rationale behind the introduction of these HEMA-free adhesives is that HEMA has been recently recognized as promoting water to be bonded in unstable soft Hydrogels,[22,38] which tend to cluster together before polymerization and create hydrophilic domains and microscopic-filled channels called "water trees." It was found that these water trees permit movement of water from the underlying dentin through hybrid and adhesive layers to the adhesive-composite interface, where water sorption plasticizes polymers and lowers their mechanical properties,[39-41] leaving them prone to hydrolytic degradation.[42] The omission of HEMA from the adhesive blends has been considered advantageous in removing those water prone hydrophilic domains.[43] However, in the current study, when the self-etch HEMA-free adhesives were applied according to the manufacturer's instructions, they statistically showed the lowest microtensile bond strength values compared to the other tested groups, which is in agreement with Perdigão and others.[33] Although all tested adhesives were acetone-based, G BOND contained 40% acetone. Such a high content of acetone, together with the manufacturer's instructions to perform strong air drying after adhesive application, could have increased the convective and evaporative water fluxes from the underlying dentin, resulting in a low water/solvent evaporation ratio. This may lead to residual "free" water, which did not completely evaporate and was entrapped at the interfacial level, leading to phase separation of its components.[16,37-38] The above explanation could be the reason for the blis-

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ter formation noted in the fracture surface of G BOND Figures 8A and 8B. These blisters may have compromised the mechanical and chemical stability of the resultant interface,[44] increasing fracture stress, thus lowering the bond strength values.[45]

Doubling the number of adhesive coats significantly increased the bond strength values of the two self-etch adhesives. This is in agreement with Pashley and others,[23] Toledano and others[24] and Ito and others.[25] It is likely that the increased bond strength seen in these adhesives when multiple coats are applied is due to several mechanisms operating simultaneously. As the first layer of adhesive begins to etch dentin, it is probably rapidly buffered, so that the additional layer of unpolymerized co-monomers may improve the etching ability of adhesives. The solvent also evaporates between coats, thus the concentration of co-monomers that exist after each coat can be increased. Therefore, doubling the layer would facilitate co-monomer infiltration with a further increase in the hybrid layer thickness.[25] These explanations are in line with the SEM findings, where doubling the number of adhesive layers has transferred the hybrid layer from a nanointeraction level (Figure 7A) to a microinteraction level (Figures 7B and 7C) thickness approaching that observed with total-etch adhesives. Furthermore, dentinal tubular orifices were also widened to the extent that funnel-shaped resin tags were identified (Figures 5B and 7B). The presence of these resin tags may have played a role in increasing the bond strength. This was also observed in the fractured dentin sticks where dentinal tubules appeared occluded with fractured resin tags (Figure 8B).

The manufacturer's instructions for G BOND are the application of a single layer for 10 seconds, followed by thorough air drying, which may result in a layer that is too thin for successful photopolymerization. It is known that vinyl resin monomers that polymerize via a free radical addition polymerization mechanism may be inhibited by oxygen, which is an effective free radical scavenger. As oxygen quenches the excited triplet state of camphorquinone and also reacts with the free amine radicals and monomer propagation radicals, it produces peroxy radicals that result in premature chain termination.[23] Thus, placement of a second adhesive layer over an air-inhibited layer may seal the uncured resins from a continuous supply of atmospheric air and prevents oxygen inhibition of its entire thickness.[23,25] This explains the increased microtensile bond strength by more than twofold as a result of double application of the adhesive coats followed by light curing. This technique has indirectly doubled the application time for the first layer of the applied adhesive, which may have led to two actions: first, it allowed sufficient time for the water to be removed from the first layer and second, it has possibly allowed sufficient time for a chemical reaction to take place, as this HEMA-free adhesive system contains 4-MET (4-methacryloxyethyl trimellitic acid). This functional monomer (4-MET) is speculated to have a chemical interaction with hydroxyapaptite crystals.[46] This led to speculation that the short application time recommended by the manufacturer may not be sufficient to allow the chemical bonding mechanism to take place as also reported by Yoshida and others.[47] On the other hand, with the XENO IV adhesive and due to a lack of chemical bonding capacity, there was no statistically significant difference when the curing was carried out between each adhesive layer or when curing the doubled layers was carried out once, since it only relies on the micromechanical interlocking in its bonding mechanism to the underlying dentin surface.

The presence of irregular collagen fibrils on the THL of the fractured specimens of XENO IV with double application of the adhesive layer (Figures 6B and 6C) and Prime and Bond NT, when applied in a single layer (Figure 4A), may be attributed to the fact that the collagen fibrils within the hybrid layer may have been deformed and over-stretched during debonding, demonstrating evidence of irreversible plastic deformation.[48-49] This deformation might have occurred due to weak or a lack of interaction between the surface of the collagen fibrils and the resin, which allowed the collagen fibrils to be pulled out of the resin upon the application of tensile stress. Nakabayashi and Pashley[50] postulated three models (interface, interphase and denatured interphase) to describe the potential interactions between demineralized collagen and polymerized resin within the hybrid layer. In the interface model, the resin envelops the fibril like a sheath without any micromechanical or chemical interaction. As collagen fibrils have a modulus of elasticity of 5 to 7 MPa while they are enveloped by resins that have a moduli of 2,000 to 4,000 MPa, the collagen fibrils will elongate when the bonded interface undergoes stress-induced strain. The resin lattice will absorb most of the load failing at lower stresses than the collagen. Such a scenario may explain the fractured surface appearance of Prime & Bond NT and XENO IV. With G BOND, no collagen fibrils were observed on the fracture dentin sticks, which may indicate that interphase and denatured interphase models may have occurred due to its chemical bonding capacity.

CONCLUSIONS

Under the conditions of this *in vitro* study, it can be concluded that:

1. Prime and Bond NT, when used according to the manufacturer's instructions, significantly had the highest bond strength.

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2. Doubling the number of adhesive layer applications significantly improved the bond strength of self-etch adhesives, in particular G BOND; however, it had a negative effect on Prime & Bond NT.
3. SEM analysis indicated that Prime & Bond NT had the highest distribution of long resin tags.

(Received 17 November 2008)

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