

Influence of resin-tags on shear-bond strength of butanol-based adhesives

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Abstract: Objectives: The aim of this in vitro study was to assess micro-shear bond strength (μ SBS) of tertiary-butanol-based adhesive under moist and dry conditions and correlate the results to resin-tags surface-area. **Methods:** Thirty-extracted human molars were used. Flat dentin surfaces were prepared on buccal and occlusal surfaces ready for bonding. Specimens were randomly divided into three-groups; G1: Prime&BondNT, applied to moist dentin (control), G2: XPBond, applied to moist dentin, and G3: XPBond, applied to dry dentin. Etch&Rinse technique was used for both adhesives as per manufacturer's instructions. For G3, dentin was air-dried for 10s before XPBond application. Three-microcylinders of composite-resin (TPH A2 shade, Dentsply) were bonded to buccal dentin of each specimen for μ SBS testing, while 2mm composite-resin was bonded to occlusal dentin for tags surface-area analysis. Curing was performed for 40s (LED, Bluephase, Ivoclar/Vivadent). All specimens were stored in distilled water at 37°C for 24h. μ SBS testing was performed using testing machine (Model LRX-plus; Lloyd-Instruments Ltd., Fareham, UK) and data were recorded using software (Nexygen-MT Lloyd Instruments). Each specimen was then sectioned mesio-distally to expose resin-dentin interface, examined at 1500X using Environmental-Scanning-microscope, and tags surface-area were calculated. Data were analyzed by Pair-wise Newman-Keuls multiple comparison and regression-analysis ($P < 0.05$). **Results:** G2 (29.06MPa) showed insignificantly higher μ SBS than G1 (25.45MPa), while G3 (17.3MPa) showed significantly the lowest μ SBS. G3 produced significantly highest tags surface-area (200.4 μ m²) compared to G1 (149.4 μ m²) and G2 (94.54 μ m²). **Conclusion:** - Butanol-based adhesive bonded to moist dentin, produced high μ SBS and hybrid layer with short resin-tags that showed a perfectly infiltrated and sealed dentin-resin interface, - bonding to dry dentin showed lower μ SBS, - there was significant correlation between tags surface-area and μ SBS for G1&G2, - no correlation was found for G3. **Acknowledgement:** Dentsply/Caulk. The purpose of this in vitro study was to compare micro-shear bond strength (μ SBS) of a tertiary-butanol-based adhesive to a 2-step etch and rinse one, under moist and dry conditions and correlate the results to resin-tags surface-area.

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Keywords: resin-tag; shear-bond; butanol-based adhesives; micro-shear bond strength (μ SBS)

Introduction

The type of solvent used in dentin adhesives strongly influences their clinical application protocol. While acetone-based systems only work well on a moist dentin surface, acid-etched dentin with excess water shows detrimental effects, referred to as the "over-wet phenomenon"¹. On the other hand, water-based systems are not so sensitive with regard to dentin moisture content, as they have inherent rewetting properties, but require a longer evaporation time. If the solvent is not completely evaporated before light-curing the adhesive, flaws can weaken

the hybrid layer probably causing premature restoration failure². A new type of solvent for adhesives, namely tertiary-butanol was claimed to be less sensitive to residual dentin moisture and allow full resin penetration under a wide range of dentin conditions. Manufacturers claim that these adhesives contain phosphate esters that may chemically bond with the mineral apatite component of dentin and enamel³. Together with the formation of a hybrid layer and chemical bonding to dentin substrate, resin tags may become a key factor in the bonding of tertiary-butanol adhesives. Therefore, the purpose of this in vitro study was to compare micro-shear bond strength

(μ SBS) of a tert-butanol-based adhesive to a 2-step etch and rinse one, under moist and dry conditions and correlate the results to resin-tags surface-area.

Materials and Methods:

Flat bonding sites were prepared on the buccal and occlusal surfaces of thirty extracted

human molars. Teeth were sectioned using a water cooled diamond disc and rotary instrument (Diamond instruments, DiaTessin, Switzerland), to expose the superficial buccal and occlusal dentin. Specimens were randomly divided into three-groups according to the adhesives and conditions of use (Table 1, 2).

Table 1: Materials used in this study

Material	Type	Composition	Composite	Manufacturer
Prime& Bond NT	2-step Etch& rinse, Acetone solvent	PENTA UDMA, resin5-62-1, resin-T, resin-D, bis-phenol A dimethacrylate, acetone, nano-scale filler cetylaminehydrofluoride	TPH resin composite (Microhybrid)	DentsplyDetrey, Konstanz, Germany
XPBond	2-step Etch& rinse, t-butanol solvent	PENTA, TCB resin, UDMA, TEGDMA, HEMA, nanofiller, camphorquinone, DMABE, butylatedbenenediol, tertiary butanol	TPH resin composite (Microhybrid)	DentsplyDetrey, Konstanz, Germany

Table 2: Grouping, conditions of use, and material application

Grouping	Adhesive used and substrate condition	Application and Curing
G1 (control)	Prime&BondNT applied to moist dentin	Conditioner gel 36% is applied for 15 s, rinsed for 15 s and blot-dried with cotton. Application of P&B for 20 s, leave surface undisturbed for 20 s, gently air-dried for 5 s, and light cured for 10 s (LED, Bluephase, Ivoclar/Vivadent).
G2	XPBond, applied to moist dentin	Conditioner gel 36% applied for 15 s, rinsed for 15 s and blot-dried with cotton. Application of XP for 20 s, leave surface undisturbed for 20 s, gently air-dried for 5 s, and light cured for 10 s
G3	XPBond, applied to 10 s air-dried dentin	Conditioner gel 36% applied for 15 s, rinsed for 15 s and - dried for 10 s (to simulate over-drying). Application of XPBond for 20 s, leave undisturbed for 20 s, gently air-dried for 5 s, and light cured for 10 s

After application of the adhesive systems, three-microcylinders of composite resin were bonded to the buccal dentin bonding sites of each ground tooth for μ SBS testing. Each tooth also, had a 2mm composite resin block bonded to the exposed bonding site on the occlusal dentin for resin tag surface-area evaluation. All specimens

were stored in distilled water at 37°C for 24h. Micro-shear bond testing was performed first using testing machine (Model LRX-plus; Lloyd-Instruments Ltd., Fareham, UK). Data were recorded using software (Nexygen-MT Lloyd Instruments). Each specimen was then sectioned mesio-distally to expose resin–dentin interface, soaked in 0.5 N HCl for 20 s followed by 5% NaOCl for 2 min to reveal the hybrid layer, then examined at 1500X using Environmental Scanning Electron Microscope (ESEM, Quanta200). Resin tags' surface-area was calculated using XT Document software (Netherland). Data were analyzed by Pair-wise Newman-Keuls multiple comparison and regression-analysis ($P < 0.05$).

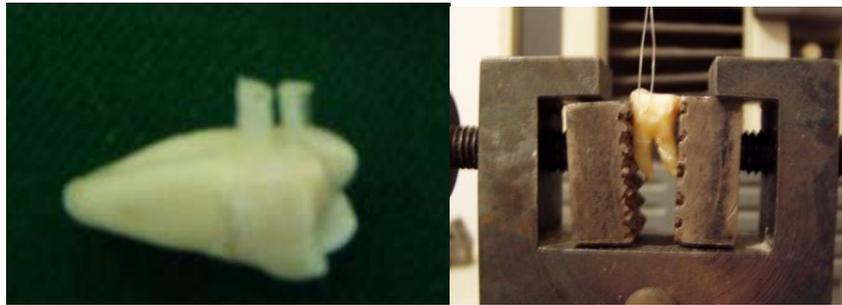


Figure 1: a, specimen with micro-cylinders bonded to buccal surface; b, μ SBS compartments

Results:

1. μ SBS results

XP group with moist dentin attained statistically significant highest bond strength (29.06 MPa). Intermediate bond strength was obtained with P&B group (25.45 MPa), while XP group with dry dentin showed the statistically significant lowest bond strength (17.3 MPa (table 3).

Table (3) Descriptive statistics of μ SBS in MPa for all groups ($p < 0.05$).

	P&B (G1)	XP moist dentin (G2)	XP dry dentin (G3)
Mean	25.45 ^a	29.06 ^a	17.31 ^b
Std. Deviation	3.531	4.039	3.311

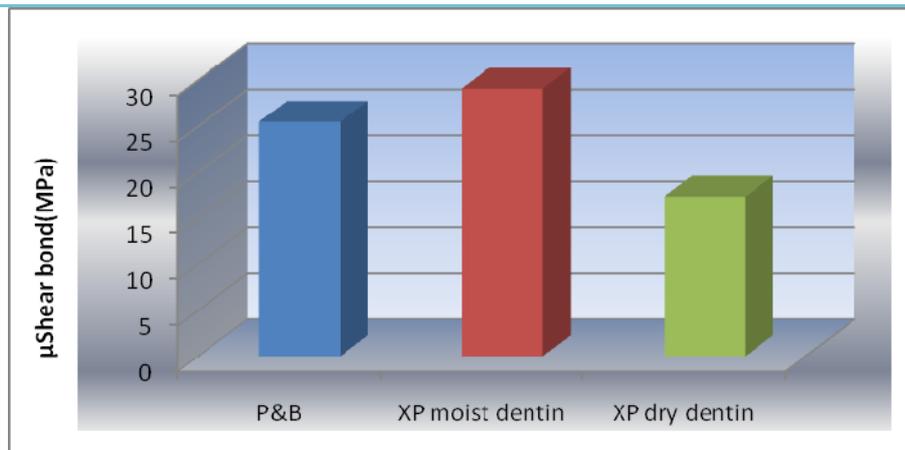


Figure (2) Column chart of μ SBS mean values (MPa) for all groups

2. Resin tag surface area Results

XP group with dry dentin attained statistically significant highest tags area ($200.4 \mu\text{m}^2$). Intermediate tags area was obtained with P&B group ($149.4 \mu\text{m}^2$), while XP group with moist dentin showed the statistically significant lowest tags area ($94.54 \mu\text{m}^2$).

Table (4) Descriptive statistics of tags area (μm^2) for all groups

	P&B (G1)	XP moist dentin (G2)	XP dry dentin (G3)
Mean	149.4 ^b	94.54 ^c	200.4 ^a
Std. Deviation	26.09	20.06	38.00

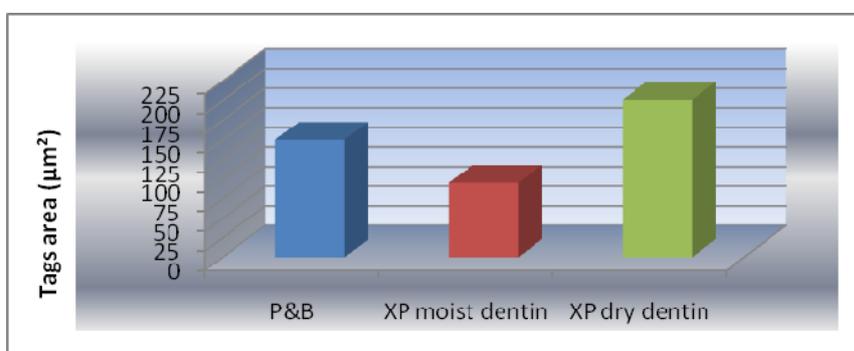


Figure (3) Column chart of tags area (μm^2) mean value for all groups

3. Correlation results

A statistically significant correlation was found between tags area and bond strength for Prime & Bond and XP-Bond with moist dentin as revealed by regression statistics. There was no correlation between tags area and bond strength for XP-Bond with dry dentin as revealed by regression statistics.

Table (5) Correlation of tags area and bond strength for all groups

<i>Regression Statistics</i>	Correlation coefficient R	R Square	P-value
Prime & Bond	0.8585	0.7370	0.0286*
XP moist dentin	0.9653	0.9318	0.0018*
XP dry dentin	0.62480	0.3904	0.185 ns

*; significant ($p < 0.05$)

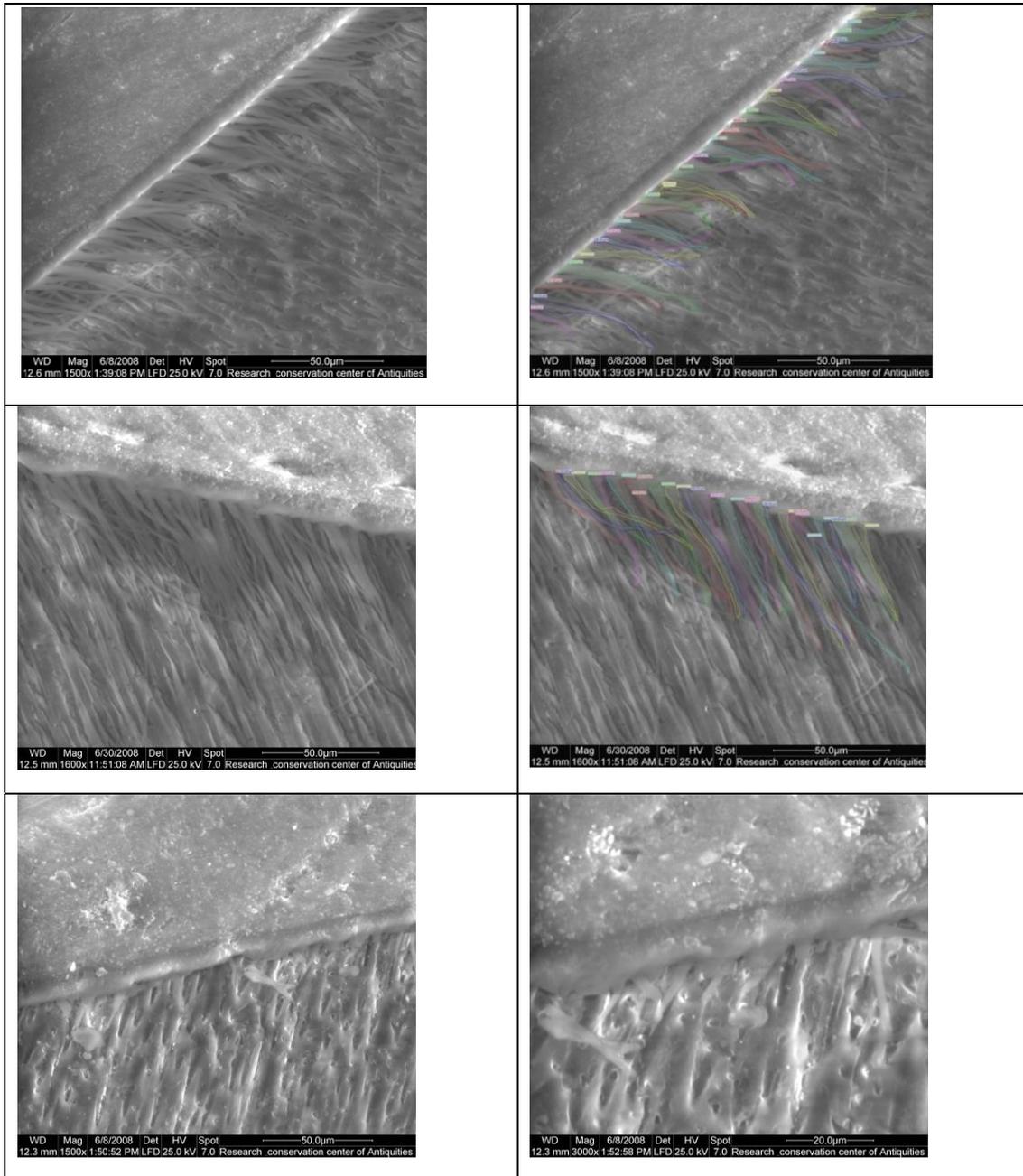
4. ESEM evaluation

Figure 4: Prime&Bond NT with moist dentin (G1) a-e: ESEM micrographs showed a distinct adhesive layer, well and homogenously infiltrated hybrid layer (Magnification X1500). F: Dense, sealed hybrid layer (Magnification X3000).

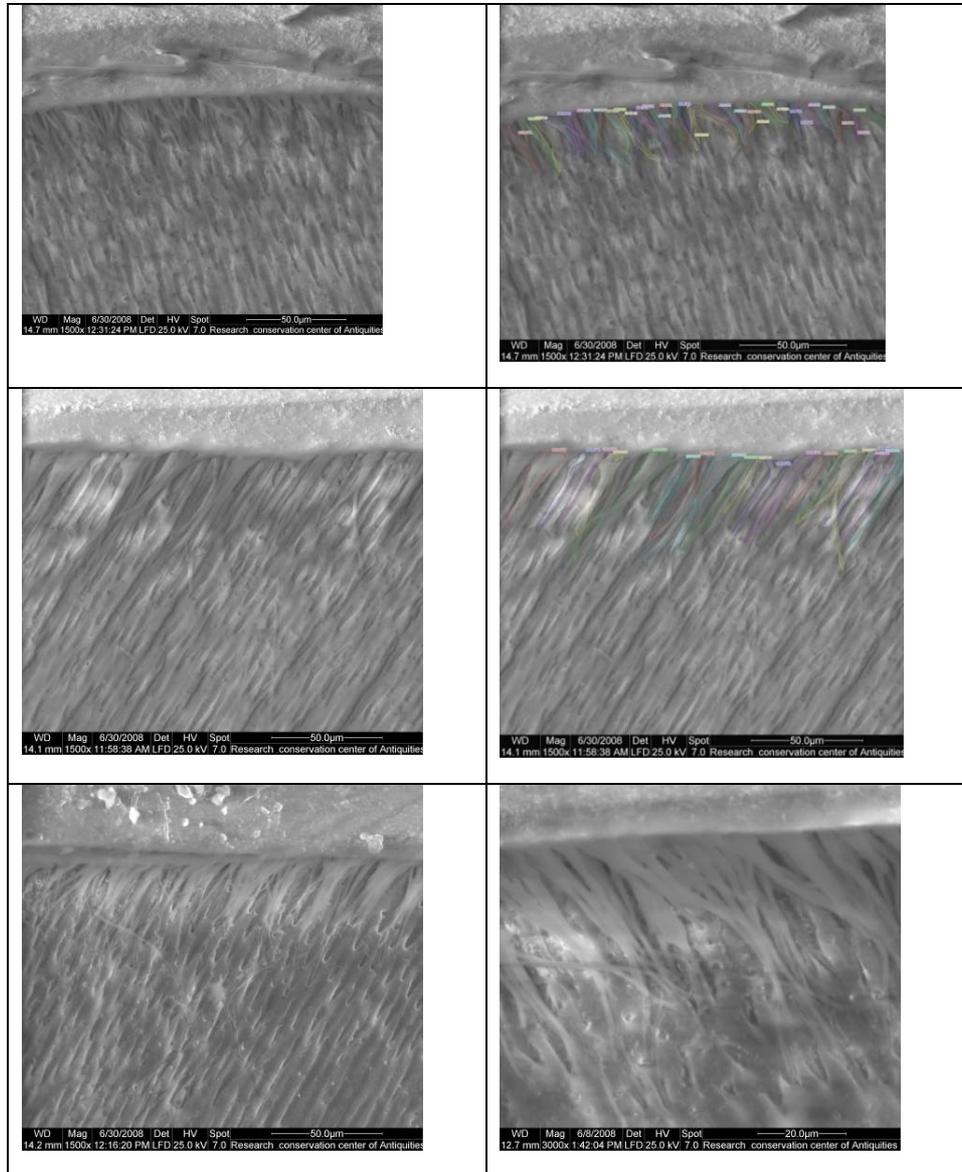


Figure 5: XP bond with moist dentin (G2): a-e: ESEM micrographs showed a distinct adhesive layer, a well and homogenously infiltrated hybrid layer with less resin tag area (Magnification X1500). f: Dense sealed hybrid layer (Magnification X3000).

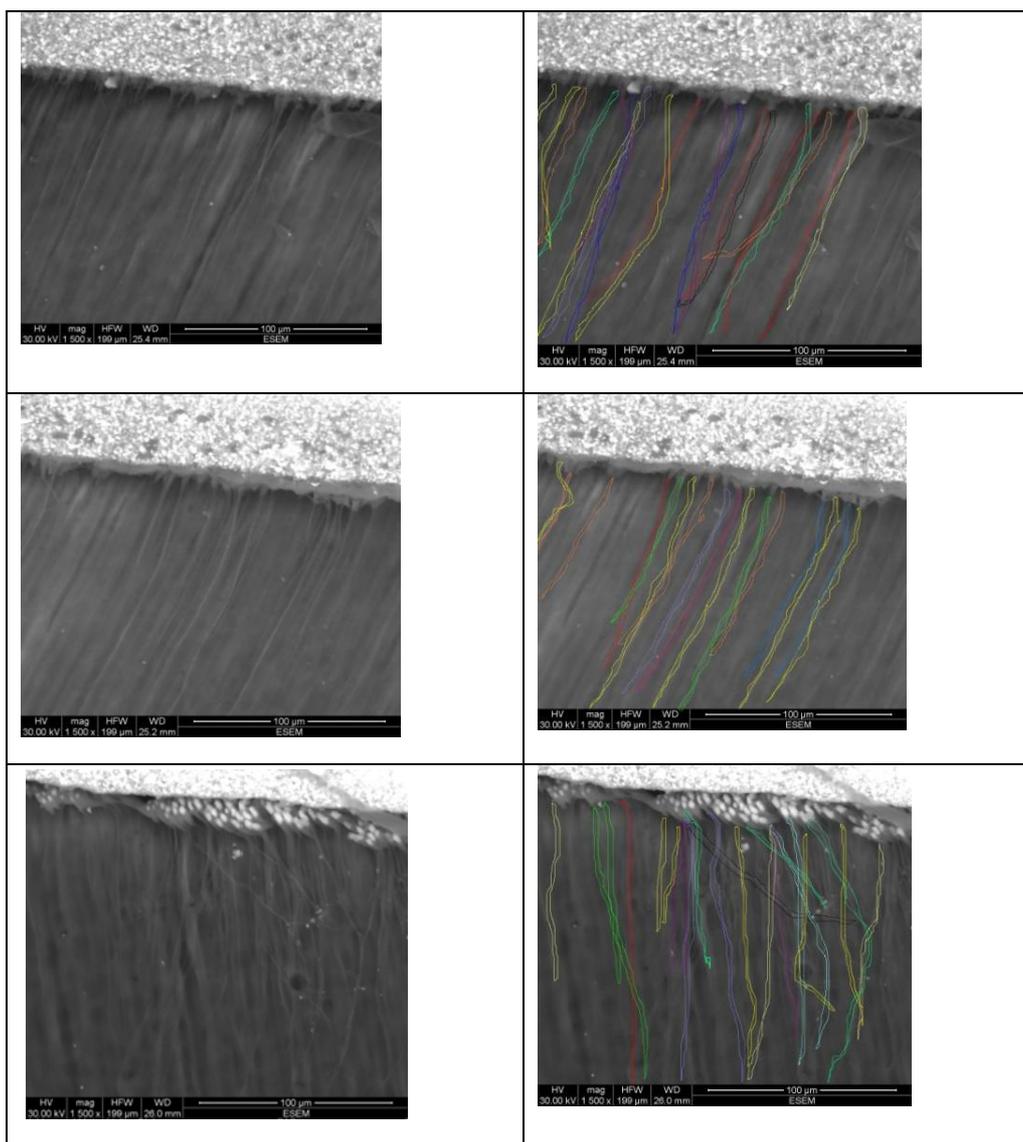


Figure 6: XP with dry dentin (G3) a-f: ESEM micrographs showed a distinct adhesive layer, infiltrated hybrid layer with areas of detachment from the underlying dentin. Long resin tags were observed. Hybrid layer seal was imperfect (Magnification X1500).

Discussion

Micro-shear bond strength testing (μ SBS) allows the measurements on small areas, making it possible to assess the adhesion strength of the resin composite to the clinically relevant dentin. The technique eliminates most of the cohesive resin or dentin fracture seen in more traditional shear strength test procedures that are due to nonuniform stress distributions.⁴ Monomers of adhesive systems are carried by a solvent which is usually water, ethanol, acetone, or a combination of those⁵.

Compared to Prime&Bond NT, in XP BOND acetone is replaced by tert-butanol. This solvent has a higher boiling point than acetone. Hence, tert-butanol is advantageous in daily practice by allowing the use of a dappen dish and the increase of the resin content. Moreover, it is totally miscible with both water and with the polymerisable resins³. It therefore helps the resin-containing adhesive to wet a moist tooth surface and produce dense, sealed hybrid layer that was recognizable in ESEM micrographs. Although Acetone has high vapor pressure, which is about four times as high as that of ethanol, it is highly

volatile which reduce its shelf life. Tert-butanol has similar vapor pressure as ethanol, but better stability towards chemical reaction with monomers². This may explain the high micro-shear bond strength of XP wet-bonded specimens. Tert-butanol is claimed to be less technique sensitive due to an improved ability to diffuse through partially collapsed demineralized dentin⁶. This could be attributed to the H-bonding capacity of a solvent which has been shown to be important to re-expand the shrunken demineralized collagen network after dehydration². Numerous publications have shown that collapsed air-dried dentin matrices do not always expand when bonding agents are applied^{7, 8, and 9}. Most monomers used in adhesive dentistry have h-values below those of dried dentin¹⁰. Thus, such resins cannot expand dried, acid-etched dentin. This is why dry bonding to acid-etched dentin seldom gave shear bond strengths over 5 MPa¹¹. In this study, shear bond strength of dry bonded dentin was significantly lower than wet bonded dentin specimens; however, micro-shear bond strength values were 17MPs. This could be credited to H-bonding capacity of tert-butanol solvent which breaks stabilizing H-bonds and other forces that keep the collagen in shrunken state. As seen in SEM micrographs of dry bonded dentin, the adhesive layer showed areas of detachment from underlying dentin which indicates incomplete resin infiltration or retention related to these areas. This may explain the lower micro-shear bond strength values. This was in contradiction to a previous investigation which stated that "The morphology of the hybrid layer when XP BOND was applied on dried dentin was not very distinct from the morphology corresponding to the application of the same adhesive on moist dentin."¹² The micro-shear bond strength of conventional etch and rinse systems has been theoretically modeled by Pashley et al as the sum of strength contributed by the resin tags, the hybrid layer and surface adhesion¹³. For etch and rinse adhesives, resin tag formation contribute quantitatively up to one-third of the total shear bond strength¹⁴. Even though significantly lower tag area was reported with both adhesives in moist condition, there was significant correlation between resin tag area and micro-shear bond strength. This could be related to the quality of the hybrid layer as shown in SEM micrographs and not the quantity of resin tags. Although resin tag area was significantly highest with XP dry bonding, no correlation was found between resin tag area and shear bond strength which may confirm the same finding.

Conclusion:

1. The type of solvent strongly influences the clinical application protocol of etch-and-rinse adhesive systems.
2. Butanol-based adhesive bonded to moist dentin, produced high μ SBS that was not considerably different from that of the P&BNT, with hybrid layer that showed a perfectly infiltrated, sealed dentin-resin interface with relatively short resin-tags.
3. Butanol-based adhesive bonded to dried dentin was able to infiltrate the demineralized collagen layer and produce long resin tags, however, hybrid layer was not perfectly sealed which may explain the lower bond strength values.
4. There was significant correlation between resin-tags surface-area and μ SBS for moist substrate bonding for the two tested adhesives. However, no correlation was found for dry substrate bonded to tert-butanol based adhesive.

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