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Effect of Adhesives and Thermocycling on the Shear Bond Strength of a Nano-Composite to Coronal and Root Dentin

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Clinical Relevance

The performance of an adhesive system may differ according to the dentin substrate and thermocyling.

SUMMARY

Objectives: To evaluate the effects of "etch & rinse" vs "self-etch" adhesives and thermocycling on the shear bond strength (SBS) of a nano-composite to coronal and root dentin. Materials and Methods: Fifty-six extracted human molars were mounted and ground to expose coronal and root dentin surfaces and were randomly divided into two groups according to adhesive system: SE—a two-step self-etch adhesive (Adper SE Plus, 3M ESPE) and ER—an etch & rinse adhesive (Adper Single Bond 2, 3M ESPE). The adhesives were applied to the coronal and root dentin of the mounted teeth. A nano-composite (Filtek Supreme XT, 3M ESPE) was applied to both dentin surfaces. The bonded specimens were stored in distilled water at 37°C for 24 hours. Half of the bonded specimens were tested for SBS in a universal testing machine without thermocycling. The remaining specimens were thermocycled (500 cycles between 5°C and 55°C) prior to SBS testing. Two specimens from each group were kept for Scanning Electron Microscope evaluations of the adhesive interfaces. Failure modes were determined under a stereomicroscope. The mean SBS value of each group was calculated, and the results were subjected to ANOVA, Duncan test and Paired samples *t*-test (p=0.05).

Results: Thermocycling did not affect the SBS of coronal dentin for either adhesive. However, it affected the SBS of SE in root dentin (p<0.05). The

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two different dentin substrates did not exhibit a significant difference except for higher values in root dentin with the SE group without thermocycling. The greatest number of cohesive failures was observed in root dentin with SE adhesive; however, the failure modes were mainly adhesive for the other groups.

Conclusion: The SE adhesive exhibited higher SBS values than the ER adhesive in root dentin. Thermocycling did not affect the SBS in coronal dentin for either adhesive but it decreased the SBS of SE in root dentin.

INTRODUCTION

The clinical use of resin composites has expanded considerably over the past few years due to increased esthetic demands by patients, new improved formulations and simplification of bonding procedures. Novel resin composites benefit from improved filler technology and modifications of the organic matrix for a greater degree of polymerization. These advances contribute to the improved mechanical and physical properties of contemporary restorative composites.[1-2] The longevity of a composite restoration is directly related to the effectiveness of the adhesive system used, as the lack of bonding and inadequate marginal sealing may lead to restoration failure.[3] Current adhesive systems interact with the enamel/dentin substrate, using two different strategies: either removing the smear layer prior to bonding (etch & rinse technique) or maintaining the smear layer as the substrate and allowing its modification by a low pH primer-adhesive (self-etch technique).[4] Reports on the effectiveness of self-etch adhesive systems, when compared to etch & rinse adhesives, are contradictory.[5-6] Some reports show a similarity between these systems,[6-7] while others suggest the superiority of etch & rinse adhesives.[5,8]

An effective bond between a composite material and tooth substrate must be of adequate strength to compensate for the shrinking and polymerization stress of the resin composite.[9] Although high early bond strengths of current adhesive systems to dentin have been reported,[10] the durability of the adhesive bond is still one of the areas of concern in adhesive dentistry. Several reports have evaluated long-term adhesive-bond durability and have shown that dentin bond strength decreased in long-term water storage.[11-12] Slow water absorption by bonded constituents can lead to the degradation of bond strengths, because of hydrolysis of the adhesive resin[13-14] and possibly the plasticizing effects of water on resin and collagen.[12]

Thermocycling is the *in vitro* process of subjecting a restoration and tooth to temperature limits similar to those experienced in the oral cavity, which can produce potential negative effects due to dissimilar coefficients of thermal expansion between the tooth and the restorative material.[15] Thermocyling stresses the bond between resin and the tooth, and it may affect bond strength, depending on the adhesive system.[15-17]

The bonding of resin-based restorative materials to dentin has always been more challenging, compared with enamel bonding. Dentin bonding is believed to be more difficult and less predictable over time, because dentin is a vital tissue with a high water organic content and its microstructure is dominated by tubules.[18] In selecting an adhesive system for clinical use, it is very important to evaluate the dentin's bond strength and sealing ability. It is increasingly important that clinicians recognize differences in dentin composition before planning restorations that depend on dentin bonding. Coronal dentin has been routinely used in *in vitro* studies,[15,19] since it is far more convenient than the smaller surfaces of root dentin. The root regions are not generally used as bonding substrates because of technical difficulties; therefore, little work has been published on the adhesive properties of resin composites to human root dentin.

In light of the importance of bonding to root dentin, this *in vitro* study was designed to compare the effects of etch & rinse vs self-etch adhesive systems and thermocycling on the shear bond strength (SBS) of a nanocomposite to coronal and root dentin.

METHODS AND MATERIALS

Table 1 shows the materials used in the current study. Fifty-six recently extracted, unerupted human third molars were collected and stored in distilled water for up to one month. The teeth were rinsed with distilled water for 30 minutes, then embedded in auto-polymerizing acrylic resin (Simplex Rapid, Kemmdent, Associated Dental Products Ltd, Wiltshire, UK) with the buccal surfaces positioned for surface treatment and composite bonding. After polymerization of the embedding resin, the buccal surfaces were abraded, then polished in a polishing machine (Mecapol P230, Presi Tavernoles 38, Brieet Angonnes, France) using 400 grit and 600 grit silicon carbide paper, sequentially, until a uniform layer of dentin was observed. The exposed dentin surfaces were inspected with a stereomicroscope (16x) (Leica MS5, Wetzlar, Germany) to ensure that no enamel or cementum was left and no pulpal exposure had occurred. All samples were randomly divided into two groups according to the adhesive system used:

Group 1: The two-step self-etch adhesive (Adper SE Plus, 3M ESPE, St Paul, MN, USA) was applied by first treating the dentin with Liquid A so that a continuous red-colored layer appeared on the surface. Liquid B was then applied and scrubbed into the surface of the bonding area for 20 seconds. The red color

Table 1: *Materials Tested in This Study*

Brand Name	Manufacturer	Composition
Adper SE Plus Two-step self-etch adhesive system	3M ESPE, St Paul, MN, USA	**Liquid A**: Water, HEMA, Surfactant, Pink colorant **Liquid B**: UDMA, TEGDMA, TMPTMA (hydrophobic trimethacrylate), HEMA phosphates, MHP (methacrylated phosphates), Bonded zirconia nanofiller, Initiator system based on camphorquinone
Adper Single Bond 2 Etch&rinse adhesive system	3M ESPE, St Paul, MN, USA	BisGMA, HEMA, dimethacrylates, ethanol, water, a novel photoinitiator system and a methacrylate functional copolymer of polyacrylic and polyitaconic acids, silica nanofiller
Filtek Supreme XT Nanofil resin composite	3M ESPE, St Paul, MN, USA	BIS-GMA, BIS-EMA, UDMA, TEGDMA non-agglomerated/non-aggregated silica nanofiller, agglomerate silica nanocluster consisting of agglomerates of primary silica nanoparticles

HEMA: 2-hydroxyethyl methacrylate
UDMA: urethane dimethacrylate
TEGDMA: triethyleneglycol dimethacrylate
Bis-GMA: bis-phenol A diglycidylmethacrylate
Bis-EMA: bis-phenol A polyethoxylated dimethacrylate

disappeared quickly, indicating that the etching components had been activated. After the treated dentin surface was air-dried thoroughly for 10 seconds to evaporate water, a second coat of Liquid B was applied and lightly air-thinned, then light-cured for 10 seconds (Elipar Free Light 2, 3M ESPE) light output: 1000 mw/cm^2).

Group 2: Dentin surfaces were etched with 35% phosphoric acid gel (Scotchbond Etching Gel, 3M ESPE) for 15 seconds, rinsed for five seconds and gently air-dried with a cotton pellet to remove excess water and keep the tooth surface moist. Two consecutive coats of Adper Single Bond 2 were then applied to the etched surfaces, left for 10 seconds and gently thinned with air and light-cured for 20 seconds.

Both adhesive systems were applied to coronal and root dentin surfaces according to the manufacturer's instructions. Light-curing was performed with the same LED light-curing unit. After adhesive applications, the specimens were clamped in the Ultradent Bonding Jig (Ultradent Products Inc, South Jordan, UT, USA), and a bonding template with an inner cylindrical opening of 2.3 mm in diameter and 3-mm deep was attached to the dentin surfaces. A nanofill resin composite (Filtek Supreme XT, 3M ESPE), with a shade of A2, was applied in two increments and each increment was light-cured for 40 seconds with the same curing light. Test specimens were prepared at room temperature (23°C ± 2°C) and stored in distilled water at 37°C ± 2°C for 24 hours. The specimens were subsequently tested in shear mode with a knife-edge shear blade in a universal testing machine (LR50K, Lloyd Instruments Ltd, Fareham, Hants, UK) at a crosshead speed of 1 mm/minute. Half of the bonded specimens in each group were first subjected to thermocycling between 5°C and 55°C for 500 cycles, then tested with the same protocol in the universal testing machine. The dwell time in each bath was 20 seconds and the transfer time between the two baths was 5-10 seconds. Shear bond strength values were calculated as the ratio of fracture load to bonding area and expressed in megapascals (MPa). After shear bond testing, the fractured test specimens were examined under a stereomicroscope (16x) (Leica MS5) and the type of bond failure (adhesive, cohesive or mixed) was recorded.[8] Bond failure was characterized according to the area of resin remaining on the dentin surface. Adhesive failures were characterized as having less than 25% resin composite remaining at the interfacial bond area. Cohesive failures had greater than or equal to 75% resin remaining at the interfacial bond area and mixed adhesive/cohesive failures had 25% to 75% resin composite remaining at the interfacial bond area. Cross sections of the exposed dentin-material interfaces of two unseparated specimens from each group were sequentially polished with a series of silicone carbide abrasive papers (600, 800, 1200, 1500 and 2000 grit) using running tap water as a lubricant to smooth the surfaces; they were then brought into relief by etching with 10% phosphoric acid for 10 seconds, followed by deproteinization in 5% sodium hypochlorite for five minutes. After rinsing with distilled water, each sample was mounted on metal stubs, sputter coated with gold and examined under a Scanning Electron Microscopy (SEM) (JEOL 6400, JEOL, Tokyo, Japan).

Mean shear bond strength values and standard deviations of each group were calculated and the results were subjected to two-way ANOVA and the Duncan test. A paired samples *t*-test was then used to evaluate the differences in SBS for each adhesive material between the coronal and root dentin in the same tooth. The level of statistical significance was set at 0.05.

RESULTS

Table 2 shows the mean SBS and standard deviations for the groups tested.

In coronal dentin, the etch & rinse adhesive system exhibited higher mean bond strength values than the

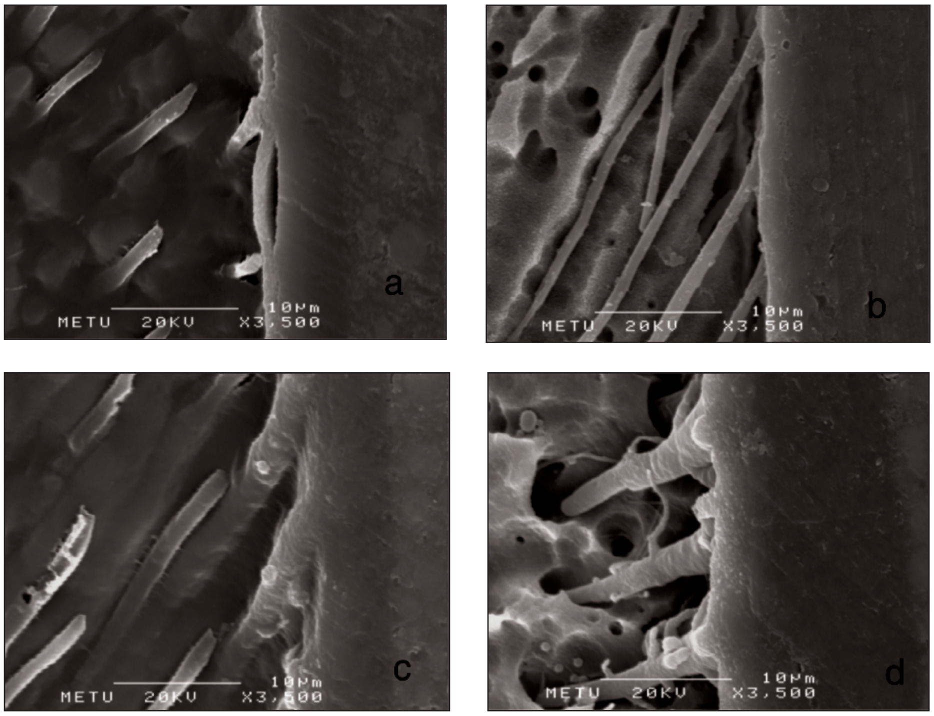

Figure 1. *SEM photomicrographs of Filtek Supreme XT/coronal dentin interfaces; a) used with Adper SE Plus without thermocycling, b) used with Adper SE Plus with thermocycling, c) Adper Single Bond 2 without thermocycling and d) Adper Single Bond 2 with thermocyling (Original magnification 3500x). Note resin tag formation with both adhesives.*

Table 2: *Mean Shear Bond Strength (MPa) ± Standard Deviations of the Adhesive Systems in Two Different Dentin Regions*

Groups	N	Coronal	Root
Adper SE Plus without thermocycling	12	$11.08 \pm 4.97^{a,A}$	$17.39 \pm 4.23^{a,B}$
Adper SE Plus with thermocycling	12	$11.01 \pm 4.56^{a,C}$	$10.89 \pm 1.73^{b,C}$
Adper Single Bond 2 without thermocycling	12	$14.80 \pm 5.95^{b,D}$	$10.93 \pm 2.49^{b,D}$
Adper Single Bond 2 with thermocycling	12	$15.36 \pm 6.00^{b,F}$	$14.35 \pm 5.70^{a,F}$

Homogenous statistical groups (p>0.05) within each column have the same lowercase superscript letter and homogenous statistical groups (p>0.05) within each row have the same uppercase superscript letter.

Table 3: *Failure Modes Observed in Dentin-composite Specimens*

Groups	Coronal			Root		
	Adhesive	Cohesive	Mix	Adhesive	Cohesive	Mix
Adper SE Plus without thermocycling	6	2	4	1	10	1
Adper SE Plus with thermocycling	9	0	3	3	5	4
Adper Single Bond 2 without thermocycling	8	0	4	9	1	2
Adper Single Bond 2 with thermocycling	9	0	3	4	0	8

self-etch adhesive system either with or without thermocycling ($p<0.05$). However, in root dentin, the self-etch adhesive system showed higher SBS than the etch & rinse adhesive system ($p<0.05$). The etch & rinse adhesive system specimens with thermocycling exhibited a higher mean bond strength value than the self-etch adhesive system in both coronal and root dentin specimens ($p<0.05$).

Thermocycling did not affect the SBS of coronal dentin of either adhesive system; however, in root dentin, thermocyling reduced the bond strength of the self-etch adhesive and increased the bond strength of the etch & rinse adhesive ($p<0.05$).

When the bond strengths of the coronal dentin and root dentin surfaces were compared, there was no significant difference ($p>0.05$), except for the self-etch adhesive, which showed higher bond strength values in root dentin when compared to coronal dentin without thermocycling ($p<0.05$).

Failure modes for each group are presented in Table 3. With bonding to coronal dentin regardless of thermocycling, the failure modes of the two adhesive systems were mainly adhesive. The highest number of cohesive failures were observed in root dentin with Adper SE Plus without thermocycling.

SEM observations of nano-composites bonded to coronal and root dentin are shown in Figures 1 and 2. The representative photomicrographs show some differences in resin tag length and possible adhesive infiltration of the dentin.

DISCUSSION

The bonding of resin composite to enamel and dentin is obtained

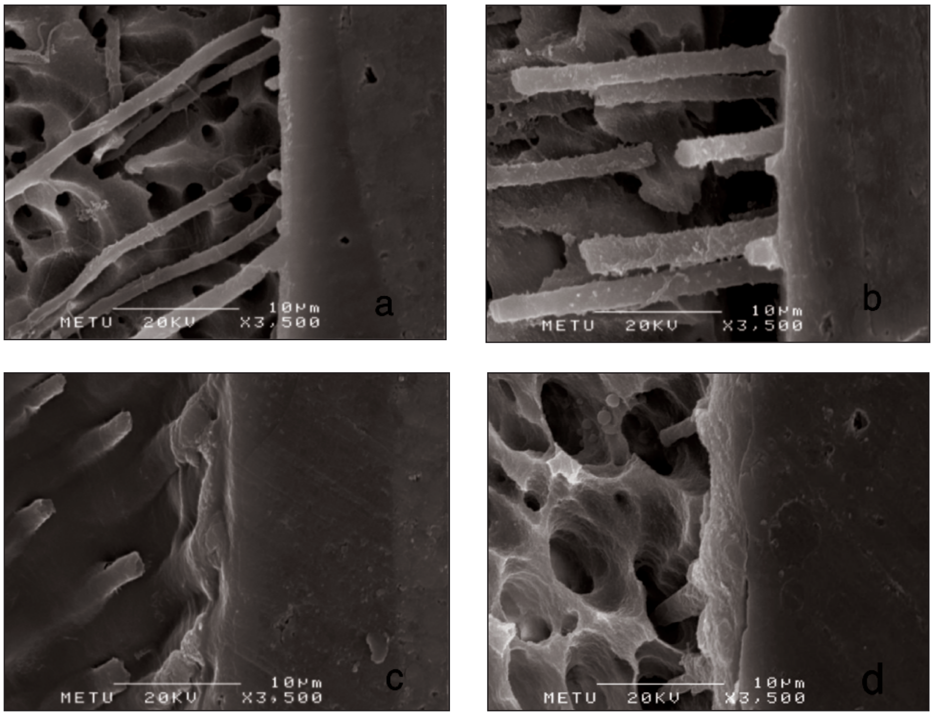

Figure 2. *SEM photomicrographs of Filtek Supreme XT /root dentin interfaces: a) used with Adper SE Plus without thermocycling, b) used with Adper SE Plus with thermocycling, c) Adper Single Bond 2 without thermocycling and d) Adper Single Bond 2 with thermocyling (Orginal magnification 3500x). Note resin tag formation with both adhesives.*

through the use of resin adhesive systems. *In vitro* and *in vivo* studies have found the efficacy of adhesive systems to differ greatly among the various brands and types of systems.[20-21] The efficacy has been shown to depend on numerous factors, such as infiltration of the adhesive into the demineralized tissue,[22] degree of conversion and strength of the adhesive,[23] mechanical properties of the resin composite[24] and compatibility between the resin composite and the adhesive-treated dentin surface, mostly with regard to the sensitivity of the resin composite initiator system to an acidic environment and surface energy parameters.[26]

Due to its heterogeneity, dentin can be described as a "dynamic" substrate and therefore represents a challenging substrate for bonding.[27] Teeth in elderly patients generally have some gingival recession, which puts the teeth at risk for root caries, abrasion, erosion and abfraction. Therefore, due to the increase in prevalence of root caries in the elderly, the bonding of restorations to root dentin becomes important.[28] Sidhu reported that the composition of dentin substrate may affect the performance of bonding agents.[29] It was shown that, as there were fewer dentinal tubules in root dentin, the permeability of root dentin was much lower than that of coronal dentin.[30] This might reduce the hydrophilic resin infiltration capacity of root dentin and result in lower bond strength values than that of coronal dentin.[30] Adhesive systems that use a separate acid-etching step are apparently more sensitive to the variation in morphological characteristics of dentin as a function of depth (tubule density and area of solid dentin) than are self-etch adhesives.[21] Another substrate variable that reduces bond strength is excessive sclerosis or hypermineralization of the exposed root surface dentin, characterized by a deeper yellow color and glossy surface.[21] In the current study, unerupted human third molars were used to standardize the tooth specimens. For this reason, the root dentin surfaces were not considered sclerotic, as is commonly observed in clinically exposed roots, which limits the observations to "healthy" roots made by the authors of the current study. The results showed that there was no significant difference in bonding to coronal and root dentin surfaces except for Adper SE Plus that exhibited higher bond strength values in root dentin before thermocycling.

By virtue of the clinical functions and characteristics of dental materials, they are difficult to evaluate under *in vivo* conditions. In part, this is due to the considerable time and resources required for controlled, randomized clinical trials. Therefore, *in vitro* studies that can simulate the intraoral environment are used as a predictor of the possible clinical performance of a material.[31]

Thermocycling is one widely used artificial aging methodology. It has been stated that a thermocycling regimen comprising a minimum of 500 cycles in water between 5°C and 55°C is an appropriate artificial aging test.[32] Some groups have reported simulation of thermal stress for shear bond strength evaluation. Titley and others[17] showed that the shear bond strength of Single Bond was not significantly affected by thermocycling. Davidson and others[33] examined the durability of the shear bond strength of adhesive systems to human dentin by thermocycling the specimens up to 300 cycles. The latter authors observed a significant decrease in bond strength after thermocycling, depending on the adhesive system tested. Price and others[34] also reported that thermocycling up to 5000 cycles had a significant negative effect on the bond strength of human dentin when a high C factor testing design was used. During thermocycling, the specimens are subjected to thermal changes and also additional exposure to water. Thermal stresses generate mechanical stresses by differences in the coefficients of thermal expansion and can result in bond failure at the tooth-restorative interface.[35] The main cause for the reduction in bond strengths is believed to be the possible effect of hydrolysis at the interfaces of the bonding resin and hybrid layer. Burrow and others[36] demonstrated that bonding resin absorbs a significant amount of water, which may adversely affect the longevity of restorations. Tay and others[37] showed that the cured single-step adhesives may act as semi-permeable membranes, allowing water diffusion from the bonded hydrated dentin to the intermixed zone between the adhesive and composite.

The permeability of single-step adhesives to water may hasten the rate of water sorption and leaching of resin components, challenging the durability of resin-dentin bonds produced by these adhesives.[38] This explains why the bond strength to dentin decreased after aging and using self-etch adhesives. Other studies have reported the bond strengths to dentin decreased dramatically[39] and nanoleakage gradually increased at the dentin interfaces.[40] On the other hand, an increase in shear bond strength was observed after thermocycling in enamel/material[41] and material/material.[42] Considering the data of all studies cited and the current study, the authors of the current study suggest that the influence of thermocycling was dependent on the number of cycles, restorative materials and dental substrates. In the current study, thermocyling did not affect bonding in coronal dentin for either adhesive system; however, it increased the shear bond strength in root dentin of one of the adhesive systems. The reason for this is that the resin used in the "etch and rinse" system is further polymerized by the repeated exposure to the 55°C environment (hot bath) in the thermocycling process.

The results of several studies have indicated that, when it comes to laboratory performance, etch and rinse adhesives remain the benchmark for other adhesives.[18,43-44] In the current study, for both the coronal and root dentin groups, Adper Single Bond 2 showed higher SBS values than Adper SE Plus after thermocyling.

Thermocyling may also have influenced the fracture modes. A decrease in the percentage of cohesive fracture and an increase in the percentage of mixed fracture was evaluated. The cohesive failures of adhesive materials are often associated with high bond strength values, indicating effective bonding.[45]

SEM examination (3500x) of the etch&rinse adhesive system revealed a thick, well defined hybrid layer. On the other hand, the self-etch system presented a thin hybrid layer, as observed by Senawonge and others.[6] Adper SE Plus appeared to have longer resin tags when compared to Adper Single Bond 2 for both dentin interfaces.

Manufacturers recommend the use of dentin adhesives and composite from the same manufacturer to achieve the maximum effect of dentin bonding, because the differences in chemical compositions might lead to unexpected chemical reactions that can affect the bonding performance.[46] In the current study, the nanofill composite and the adhesive systems were from the same manufacturer.

Future studies are necessary to further evaluate the effectiveness and durability of adhesive systems to root dentin with different aging protocols.

CONCLUSIONS

Within the limitations of the current study, it may be concluded that:

1. Shear bond strength values for the two adhesive systems tested were not significantly affected by thermocycling in coronal dentin. In root dentin, thermocyling adversely affected the bond strength of the etch & rinse adhesive.
2. The two-step self-etch adhesive system produced significantly higher bond strength values than the etch & rinse adhesive system in non-thermocycled root dentin.
3. The etch & rinse adhesive system exhibited better bond strength than the self-etch adhesive in coronal dentin.

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