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The Effect of Thermocycling on the Adhesion of Self-etching Adhesives on Dental Enamel and Dentin

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Abstract

Aim: The aim of the present study was to investigate the effectiveness of one total-etch self-priming adhesive and two one-step self-etching adhesive systems on the adhesion of a resin composite to both dentin and enamel. The effect of thermocycling on the adhesion was also investigated. The null hypothesis tested was thermocycling would not affect bond strengths to enamel and dentin treated with self-etching adhesives or a total-etch adhesive.

Methods and Materials: Two single-step self-etching adhesives [Xeno III (XE3) and Prompt L-Pop (PP)] and one two-step total-etch adhesive system (Prime & Bond NT) (P&B NT) were used in this study. Thirty caries-free unrestored human third molars were used to make specimens of enamel and dentin. Different adhesives were applied on enamel and dentin surfaces according to the manufacturer's instructions then hybrid composite restorative material was condensed on the surface using a mold. The bonded specimens were stored in distilled water at 37ºC for 24 hours before being tested. Half of the bonded specimens were tested for shear bond strength without thermocycling. The other half of the test specimens were thermocycled using a thermocycling apparatus in water baths held at 5ºC and 55ºC with a dwell time of one minute each for 10,000 cycles prior to shear testing. The mean shear bond strength before and after thermocycling was calculated, and the results were subjected to two-way analysis of variance (ANOVA) and repeated measure design to show the interaction between different materials and different times.

Results: The results showed shear bond strength on both enamel and dentin of the total-etch adhesive and the self-etching adhesives decreased after the specimens were subjected to thermocycling.

Conclusions: The null hypothesis tested “thermocycling would not affect bond strengths treated with self-etching adhesives” was rejected. Furthermore, the study revealed the following:

1. The shear bond strength to both enamel and dentin of the total-etch adhesive and the self-etching adhesives decreased after the specimens were subjected to thermocycling.
2. XE3 achieved the highest bond strength to both enamel and dentin (26.994±1.17 and 25.22±1.26, respectively).
3. XE3 showed even better bonding after thermocycling to enamel and dentin than the total-etching system or PP.
4. Although PP bonded to enamel showed lower shear bond strength value than XE3, it has durable bond strength even after thermocycling.

Keywords: Dental adhesives, thermocycling, shear bond strength

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Introduction

Several dentin bonding adhesives have been developed for the restoration of lost tooth structure.[1] The major goals of using dentin bonding agents are to enhance the bonding strength between the resin and the tooth structure, increase the retention of the restoration, reduce the microleakage across dentin-resin interface, and scatter the occlusal stress.[2]

Bonding to enamel has been successful since Buonocore introduced the acid-etch technique in 1955.[3] Bonding to dentin has been less predictable because of the wet tubular ultrastructure and organic composition of the dentin substrate.[4]

The introduction of the total-etch technique[5] and recent developments in the chemistry of dentin adhesives have made resin-based composite restorative materials nearly free of microleakage, with bond strengths approaching those of enamel bonding.[6-8] Improvements seen in the laboratory were confirmed in the clinical setting with the recent generation of adhesive systems.[9,10] Bonding to etched enamel and dentin, while relying on the entanglement of resin monomers with dental substrates or hybridization, is now considered the fundamental mechanism for retention of resin-based composite restorations.[11,12]

Current dentin adhesives employ two different means to achieve the goal of micromechanical retention between resin and dentin. The first method removes the smear layer completely and demineralizes the subsurface intact dentin via etching with acids. Following rinsing, a multi-step application of a primer and an adhesive, or a simplified self-priming adhesive is applied to the conditioned substrate to complete the bonding protocol.

The second method uses the smear layer as a bonding substrate. There are two types of simplified adhesives that are applied to the smear layer. One is a self-etching primer that includes two steps: the primer is applied without rinsing then a layer of adhesive resin is applied. The other type is more simplified, one-step self-etching adhesive that includes a single application to the tooth.

A self-etching adhesive system is another approach to prevent the risk of defective hybridization. With these systems, etching and priming of the dentin occurs simultaneously by infiltrating the smear-covered dentin with acidic resins. Thus, critical procedures like rinsing of the etchant and priming of the hydrated collagen fibers are eliminated. They are generally considered to be less technique sensitive compared to systems utilizing separate acid-conditioning and rinsing steps. However, it is still unclear whether these materials can produce strong, durable bonds.[13] Moreover, even if the decalcified zone is fully impregnated with resins, the long-term stability of this interdiffusion zone or hybrid layer is unknown. Inadequate polymerization or defects within the zone could occur due to diffusion gradients created by dentinal tissues, moisture content, residual solvents, or phase separation of monomers.[14,15]

Although high early bond strengths of current adhesive systems to dentin have been reported[16], the durability of adhesive bond is still one of the areas of current interest in adhesive dentistry.

Thermal cycling simulates the introduction of hot and cold extremes in the oral cavity and shows the relationship of the linear coefficient of thermal expansion between tooth and restorative material. Thermal cycling stresses the bond between resin and the tooth and, depending on the adhesive system, may affect bond strength.[17-20] It still has value in assessing the results of thermal stresses and prolonged water exposure.

Several studies have been done on the effectiveness of self-etching materials on the adhesion of composite to both dentin and enamel. However, controversial results have been reported about the bonding performance of the adhesives.[21-31] The aim of the present study was to investigate the effectiveness of one total-etch self-priming adhesive and two one-step self-etching adhesive systems on the adhesion of a resin composite to both dentin and enamel. The effect of thermocycling on the adhesion was also investigated. The null hypothesis tested was thermocycling would not affect bond strengths to enamel and dentin treated with self-etching adhesives or a total-etch adhesive.

Methods and Materials

Thirty caries-free unrestored human third molars, which had been stored in distilled water at 37ºC immediately after extraction, were selected for the study. The teeth were cleaned of debris using a rubber cup, pumice, and a low speed handpiece. The roots were cut off and the crowns were sectioned mesiodistally with a water-cooled diamond disc. The sectioned crowns were placed in a silicon mold and embedded in a self-curing acrylic resin with the buccal or lingual surfaces positioned for surface treatment and composite bonding. The crown surfaces were ground flat with 600-grit silicon carbide paper, under running tap water, to obtain flat enamel (n=30) or dentin (n=30) surfaces. For each surface type, the specimens were randomly divided into six adhesive treatment groups of ten specimens each. Table 1 shows the experimental groups with their respective treatment modalities. The adhesive systems employed in the study are shown in Table 2.

The adhesives were applied to enamel or dentin surfaces according to the manufacturer's instructions. The surface of dentin had to remain moist prior to the application of any adhesive system tested in this study. After the application of the bonding system, a cylindrical Teflon mold was placed on each sample (internal diameter = 3 mm; height = 5 mm). Hybrid composite restorative material was condensed into the mold and then light cured for 40 seconds. The bonded

Table 1. Experimental groups and treatment modalities.

Group	Treatment
1	Enamel + P&B NT + composite
2	Enamel + XE3 + composite
3	Enamel + PP + composite
4	Dentin + P&B NT + composite
5	Dentin + XE3 + composite
6	Dentin + PP + composite

Table 2. Adhesive systems tested in this study.

Code	Adhesive System		Manufacturer
P&B NT	Prime & Bond NT	Aceton-based total-etching adhesive	Dentsply/Caulk., Milford, DE, USA
XE3	Xeno III	One-step self-etching adhesive	Dentsply, Konstanz, Germany
PP	Prompt L-Pop	One-step self-etching adhesive	3M ESPE, St Paul, MN USA

Table 3. Mean shear bond strength to enamel in MPa (mean ± SD).

Groups	Before Thermocycling		After Thermocycling	
	Mean ± SD	Fracture mode	Mean ± SD	Fracture mode
P&B NT	23.039 ± 2.71^a	7 A+ 3 C	14.59 ± 1.66^d	10A
XE3	26.994 ± 1.17^b	7 A + 3C	24.27 ± 1.83^f	8 A + 2 C
PP	19.784 ± 1.73^c	9 A + 1C	18.14 ± 1.61^c	10 A

X = Number of samples; C = Cohesive failure; A = Adhesive failure.
Identical letter exponents identify groups that were not statistically significant different (P< 0.05).

specimens were stored in distilled water at 37ºC for 24 hours before being tested. Half of the bonded specimens were tested for shear bond strength without thermocycling. The other half of the test specimens were thermocycled using a thermocycling apparatus in water baths held at 5ºC and 55ºC with a dwell time of one minute each for 10,000 cycles prior to shear testing.[32]

The specimen cylinders were loaded by a chisel like metal rod parallel to the bonding interface in a shear mode until rupture occurred. The shear bond strengths were determined by means of a mechanical testing machine (Instron testing machine, Instron Inc., Norwood, MA, USA) at a cross head speed of 1 mm/min.[33] Shear bond strength was calculated as the ratio of fracture load and the cross-sectional area of the bonded composite cylinder. For each group, ten specimens were used to calculate the mean and standard deviation of shear bond strength to enamel and dentin.

Results

The shear bond strength data from enamel and dentin surfaces before and after thermocycling were subjected separately to two-way analysis of variance (ANOVA) and repeated measure design to show the interaction between different materials and different times.

Enamel-Adhesive Shear Bond Strength

The mean value of shear bond strength to enamel is presented in Table 3 and Figure 1. There was a significant difference in mean shear bond strength values of different groups. The Post Hoc Tukey test showed Group 1 (P&B NT) and Group 3 (PP) had the lower shear bond strength values to enamel, and they were not significantly different from each other. Xeno III (XE3) exhibited significantly higher shear bond strength value (26.99±1.17) to enamel than the other groups (P<0.0001).

Since the interaction effects between time and different groups were significantly different, the paired '*t*' test was employed for each group before and after thermocycling. The mean shear bond strength values before thermocycling for Group 1 (P&B NT) and Group 2 (XE3) were significantly higher after thermocycling. However, there was no significant difference in the mean shear bond strength values for Group 3 (PP) before and after thermocycling (P=0.065).

Dentin-Adhesive Shear Bond Strength

The mean value of shear bond strength to enamel is presented in Table 4 and Figure 2. Group 5 (XE3) showed significantly higher shear bond strength value to dentin before thermocycling (25.226±1.26) than the other two adhesives. The ANOVA revealed a significant difference at (P<0.0001).

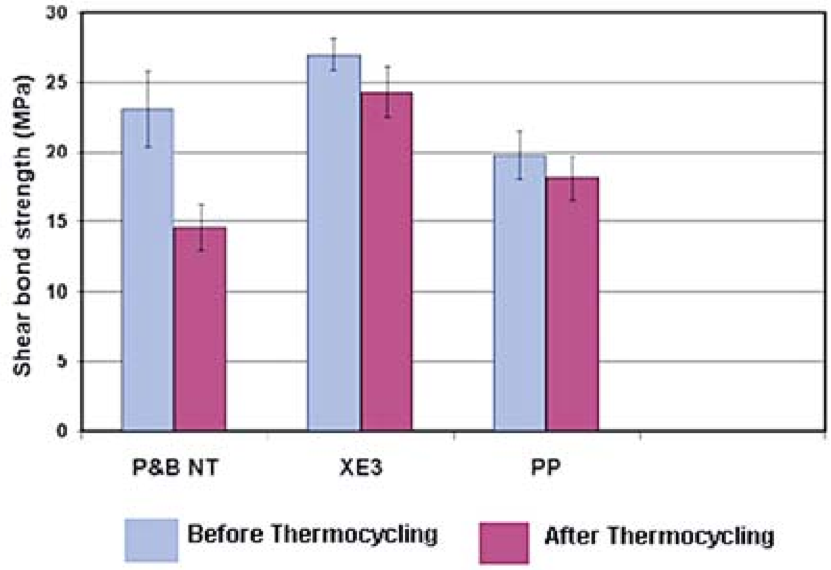


Figure 1. Mean shear bond strengths to enamel surfaces before and after thermocycling.

Table 4. Mean shear bond strength to dentin in MPa (mean ± SD).

Groups	Before Thermocycling		After Thermocycling	
	Mean ± SD	Fracture mode	Mean ± SD	Fracture mode
P&B NT	20.74 ± 1.24	8 A+ 1 C	12.14 ±1.08	10A
XE3	25.22 ± 1.26	7 A + 3C	23.24 ± 0.85	9 A + 1 C
PP	15.48 ± 0.58	9 A + 1C	11.19 ±1.67	10 A

X = Number of samples; C = Cohesive failure; A = Adhesive failure.

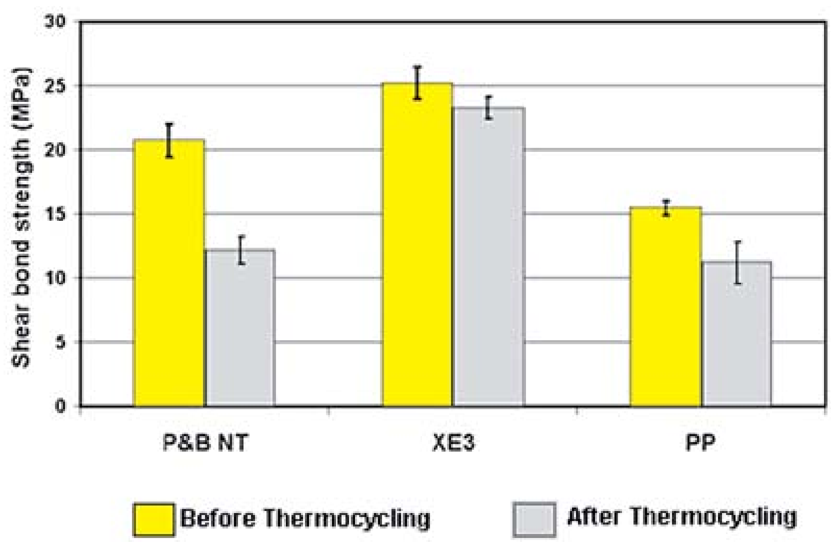


Figure 2. Mean shear bond strengths to dentin surfaces before and after thermocycling.

The Post Hoc Tukey test ranked these differences in the three subsets at a significant level of 5%. Group 6 (PP) resulted in the lowest shear bond strength. Group 4 was ranked in the intermediary subset, while group 5 (XE3) resulted in the highest shear bond strength. Paired '*t*' test was employed for each group showed there was a significant difference in shear bond strength before and after thermocycling.

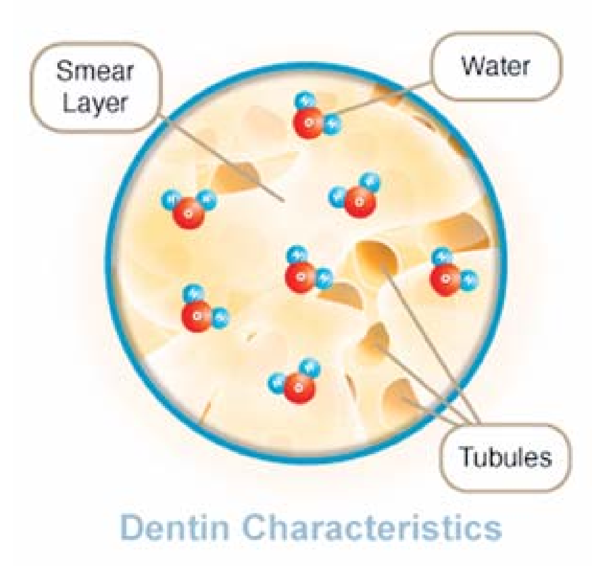
Discussion

The adhesion mechanisms of bonding to enamel using the acid conditioning technique are widely acknowledged and their efficiency is well established.[11,12,34,35] However, it is well known adhesion to dentin is more complex than adhesion to enamel due to the dentin tubular pattern, high water content, presence of smear layer, and the occurrence of pathophysiological alterations, such as sclerotic dentin and hypermineralization associated with erosion or abrasion lesions.[36,37]

Meerbeeck[38] described the concept of utilizing the smear layer as a bonding substrate but with improved formulations that can etch beyond the smear layer into the underlying dentin matrix a hybrid approach has gained interest. This approach to adhesion in dentin involves the use of primers, which combines the acid-etching step with the priming procedure. Therefore, the self-etching primers are generally less technique-sensitive since the acidic monomer demineralizes the dentin surface superficially by dissolving partially the mineral crystals around the collagen fibrils.[39,40]

Although the bonding qualities can be very appealing to clinicians, care should be taken to evaluate how these self-etching adhesives interact with the dentin surface. Since the current self-etching materials have higher pH values than the acids used with total-etch adhesive systems, and the self-etching materials are not rinsed away, the smear layer or its components are incorporated into the bonded layers.[41]

The behavior of self-etching adhesives on enamel and dentin has been a controversial subject. Some studies[21-23,27,30,42,43] have shown self-etching adhesives perform well on enamel and dentin *in vitro* whereas others reported insufficient bonding results.[26-28]



Several factors such as the origin of the tissue (human versus bovine), surface preparation method, smear layer thickness, test method, cross head speed, bonded surface area, and operator-related factors influence the bond strength of adhesives to dental tissues.[41,44,45]

Most often, bonding performances of dental adhesives are tested on bovine enamel and dentin. In an effort to simulate clinical conditions and to enhance human applicability in the present study, human molars were used and the shear bond strength was measured before and after thermocycling. Some of the specimens were subjected to thermocycling in water ranging from 5ºC to 55ºC before measurement of the bond strength.[32] This investigation revealed the potential of self-etching adhesives in creating durable resin-tooth structure bonding.

It has been postulated a minimum bond strength of 17 to 20 MPa to enamel and dentin is needed to resist contraction stresses of resin composite materials.[46] The shear bond strength of total-etching adhesive to enamel ranged between 26.9±1.17 and 19.784±1.73 MPa before thermocycling, and clinical experiences confirm this bond strength is sufficient for successful retention of resin restorations.[47-50]

All the adhesive systems used in this study achieved optimal bond strength values for enamel before thermocycling. XE3 (one-step self-etching adhesive) showed even better bonding after thermocycling to enamel and dentin than the total-etching system or PP. XE3 achieved the highest bond strength to both enamel and dentin (26.994±1.17 and 25.22±1.26, respectively). These values were significantly different among the total-etch adhesive system (P&B NT) and the other self-etching adhesive systems (PP). This means the self-etching adhesives used in this study behave differently. XE3 contains mono- and bi-functional monomers HEMA and UDMA. The first is proven to be an excellent priming molecule, and the latter is contributing to cohesive strength. Beside these two well-known monomers, XE3 contains two new monomers patented by Dentsply: Pyro-EMA and PEM-F. Both monomers contribute to the etching and adhesive function, respectively. This new formulation may contribute to the bonding stability to dentin over time. The acidity of XE3 is comparable to phosphoric acid gels. Its pH is less than one, which may be the reason for high bond strength to enamel achieved with this adhesive.[51]

Many studies have been carried out on the effectiveness of bonding of self-etching adhesives to enamel and dentin.[25-30] Bouillaguet et al.[29] reported PP had the lowest bond strength to dentin, whereas P&B NT had a higher bond strength to dentin. These results are in accordance with the results of the present study. On the other hand, Kiremitçi et al.[52] found PP exhibited significantly higher bond strength to enamel and dentin than P&B NT. These results were not in accordance with the results of the present study.

Helvatjoglu et al.[44] and Fritz et al.[27] reported no significant differences among the total-etch and self-etching adhesives. In another study, Rosa and Perdigao[25] reported acid etching produced higher shear bond strength to enamel than self-etching adhesives. These results are not in agreement with the results of this study. In another study[26], the authors reported the microtensile bond strength of PP to dentin was not statistically different when applied in multiple coats from those of total-etch adhesive P&B NT. The results showed the importance of the application method on bond strength.

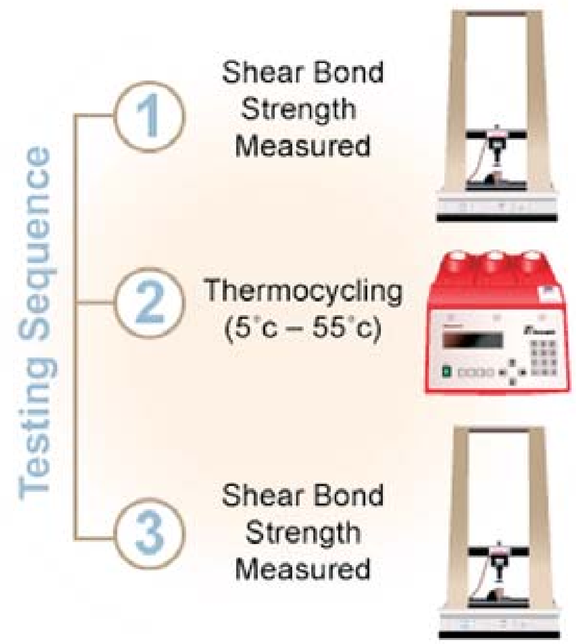
There is inconsistency in dentin and enamel bond strength results reported by various studies. The differences in testing conditions, operational factors, and the variable nature of dentin as a bonding substrate are possible explanations for the differences in the reported bond strength values.

The results obtained in the present study indicated the shear bond strength on both enamel and dentin of the total-etch adhesive (P&B NT) and the self-etching adhesives (XE3 and PP) decreased after the specimens were subjected to thermocycling. Group 3 (PP bonded to enamel) showed no significant difference in shear bond strength even after thermocycling. PP contained polycarboxylic acid, which may contribute to bonding stability to enamel. The Ca-polycarboxylic acid complex in the presence of water formed a polycarboxylate salt at the bonded interface with stress-relaxation capacity due to renewing the bond between calcium and carboxial groups.[53,54]

XE3 bonded to dentin showed the lowest fall in shear bond strength after thermocycling (7.8%). P&B NT bonded to dentin showed the greatest reduction in bond strength value (41.46%), whereas PP showed a 27.7% reduction in bond strength. The fall in bond strength of the tested adhesive systems was not directly related to the method of dentin pretreatment in terms of removing or keeping the smear layer intact.

Titley et al.[20] reported the shear bond strength of Single Bond was not significantly affected by thermocycling. The authors stated the effects of thermocycling proved to be variable and dependent upon the adhesive and the number of thermal cycles. Davidson et al.[18] examined the durability of the shear bond strength of adhesive systems to human dentin by thermocycling the specimens up to 300 cycles. They observed a significant decrease in bond strength after thermal cycling depending on the adhesive system tested. Price et al. also reported thermal cycling up to 5000 cycles had a very significant negative effect on bond strength in human dentin when a high C factor testing design was used.[55] During thermal cycling the specimens are subjected to thermal changes and also to additional exposure to water. Thermal stresses generate mechanical stresses by differences in the coefficient of thermal expansion and can result in bond failure at the tooth-restorative interface.[56] 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 et al.[57] reported bonding resin absorbs a significant amount of water which may adversely affect longevity of restorations. Tay et al.[58] reported 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 the composite. Permeability of single-step adhesives to water may hasten the rate of water sorption and leaching of resin components[59], challenging the durability of resin-dentin bonds produced by these adhesives. This explains why bond strength to dentin decreased on aging of self-etching adhesives. Other researchers have reported the bond strengths in dentin dramatically decreased[60] and nanoleakage was gradually increased at the dentin interfaces.[61]

The effects of thermal cycling on the bond strength of adhesive systems found in the present study reflect the findings of previous studies that examined the stability of dentin bonds after long-term water storage.[62-65]



Conclusions

The null hypothesis tested "thermocycling would not affect bond strengths treated with self-etching adhesives" was rejected.

1. The shear bond strength to both enamel and dentin of the total-etch adhesive and the self-etching adhesives decreased after the specimens were subjected to thermocycling.
2. XE3 achieved the highest bond strength to both enamel and dentin (26.994±1.17 and 25.22±1.26, respectively).
3. XE3 showed even better bonding after thermocycling to enamel and dentin than the total-etching system or PP.
4. Although PP bonded to enamel showed lower shear bond strength value than XE3, it has durable bond strength even after thermocycling.

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