

Effect of thermal cycling on microtensile bond strengths of various adhesives to dentin

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ABSTRACT

Objectives: The objective of this study was to evaluate the effect of thermal cycling on microtensile bond strengths (MTBS) of various adhesives to dentin. **Methods:** Three adhesive systems including a 1-step self-etch adhesive system (G-Bond), a 2-step self-etch adhesive system (AdheSE), and an etch&rinse adhesive system (Prime & Bond NT) were evaluated. Twenty-four extracted molars were used. After grinding the coronal enamel surface, the teeth were randomly divided into 6 groups ($n=4$) (G-Bond, G-Bond+thermal cycling, AdheSE, AdheSE+thermal cycling, Prime & Bond NT, and Prime & Bond NT+thermal cycling). Adhesives were applied according to each manufacturer's instructions followed by resin composite polymerization. Groups without thermal cycling were stored in distilled water at 37 °C for 24 hours and used for immediate testing of the MTBS. Groups with thermal cycling were subjected to thermocycling (10.000 cycles between 5 °C and 55 °C, for a dwell time of 30 seconds). For MTBS test, teeth were sectioned occluso-gingivally into a serial slabs and further sectioned into composite-dentin sticks. Testing was performed on a universal testing machine at a crosshead speed of 1.0 mm/min. One-way ANOVA and post hoc Tukey-Kramer multiple comparisons tests ($\alpha=0.05$) were performed on all data. **Results:** The results of the MTBS test showed that the highest bond strengths were observed in etch&rinse adhesive groups (3 and 3T). A statistically significant difference in MTBS was found between group 1 and group 1T ($p<0.05$). Moreover, there was no statistical difference in MTBS between group 1 and group 2 ($p=1.000$). **Conclusions:** Contrary to result with 1-step self-etch adhesive, thermal cycling was found ineffective on the MTBS of the 2-step self-etch and etch&rinse adhesive systems.

Keywords: Adhesive, thermal cycling, microtensile bond strength.

INTRODUCTION

Adhesion of resin composite to the tooth substance is required to provide retention, reduce microleakage and improve marginal adaptation.¹ Compared to the clinical success of enamel bonding, achievement of a predictable and clinically durable dentin bonding system has been more of a challenge.² The characteristics of the dentine substrate, including high organic content, tubular structure variations

the presence of outward fluid movement, dentine depth, sclerosis, caries,³ and the presence of a smear layer are responsible less reliable bonding to dentine.^{1,2}

Current adhesive systems utilize one of two approaches to interact with dental substrate- the 'etch&rinse technique' or the 'self-etch technique'.⁴ While etch&rinse adhesives include %35-40 phosphoric acid¹ that demineralizes dentin and enamel simultaneously, self-etch adhesives do not require a separate acid-etch step.⁴ Self-etch adhesives are composed of aqueous mixtures of acidic functional monomers, generally phosphoric acid esters or carboxylates, with a pH higher than that of phosphoric acid gels. Water is a very

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important component of self-etch adhesives, is needed for acidic monomers to ionize and trigger the demineralization of hard dental tissues.⁴ The self-etching products may have the priming and bonding steps combined (1-step systems) or they may require an additional step (2-step adhesives).¹ In respect of user-friendliness and technique sensitivity, the self-etch adhesive system is the most promising technique.⁵ Self-etch adhesives offer some advantages over etch&rinse adhesives, such as ease of use and faster manipulation, reduced technique sensitivity, and limited postoperative sensitivity. However, their etching potential is not as aggressive as that produced by phosphoric acid.⁶ It was reported that self-etch adhesives bond well to normal dentin and ground enamel *in vitro*.^{7,8} Conversely, self-etch adhesives may not bond as well to sclerotic dentin or intact enamel.^{4,9}

The evaluation of bonding durability is important, as the bond between the restorative material and the tooth substrate has a significant impact on the clinical success of a restoration. The thermal cycling test involves subjecting specimens to extreme temperatures that simulate intra-oral conditions.^{10,11} Thermal cycling also stresses the bond between resin and the tooth and, depending on the dentin bonding systems, may affect bond strength. However, only 19% of dentin bonding studies thermal cycle the specimens before testing. This may be because the value of *in vitro* thermal cycling of specimens has been questioned.¹² The purpose of this study was to determine the effect of thermal cycling on microtensile bond strengths of various adhesives to dentin. The hypothesis tested was that thermal cycling is ineffective on the bond strength of the adhesive systems.

MATERIALS AND METHODS

Twenty-four extracted; caries, crack, fracture and restoration free, human

maxillary third molar teeth were selected. After extraction, the teeth were cleaned of surface debris and stored in 0.5% chloramine T at 4 °C for less than one month. Each tooth was mounted in cold-curing acrylic resin (Meliodent, Bayer Dental Ltd., Newbury, UK). They were then submerged in tap water to reduce any temperature rise caused by the exothermic polymerization reaction of the acrylic resin. A flat dentin surface was created perpendicular to the teeth's longitudinal axis using a slow-speed diamond saw (Isomet, Buehler Ltd., Lake Bluff, IL, USA) to remove occlusal enamel and superficial dentin under water lubrication. The surface was ground with 600-grit silicon carbide paper (Carbimet Buehler, Buehler Ltd., Lake Bluff, IL, USA) under running water for 30 s to create a smear layer of clinically relevant thickness. The teeth were then randomly divided into 6 main groups of 4 teeth each and restored with investigated adhesive systems and corresponding resin composites.

Group 1: 1-step self-etch adhesive system, G-Bond (GC Corporation, Tokyo, Japan) + composite resin, Venus (Heraeus Kulzer, Armonk, NY, USA).

Group 1T: Group 1 + thermal cycling.

Group 2: 2-step self-etch adhesive system AdheSE (Ivoclar Vivadent, Schaan, Liechtenstein) + Venus.

Group 2T: Group 2 + thermal cycling.

Group 3: Etch&rinse adhesive system Prime & Bond NT (Dentsply Caulk, Milford, DE, USA) + Venus.

Group 3T: Group 3 + thermal cycling.

Adhesive systems were applied according to the manufacturer's instructions (Table 1). Composite buildups 6 mm high were constructed in three increments on the bonded surfaces. Each of the two resin composite increments were light cured with a halogen curing light (Hilux; Benlioglu Dental, Turkey) for 40 seconds. The restored teeth of groups 1, 2, and 3 were stored in distilled water at 37 °C for 24 hours. These groups were used

for immediate testing of the bond strength. The remaining groups (1T, 2T, and 3T) were subjected to thermocycling (10.000 cycles between 5 °C and 55 °C) (Nova, Nova Ticaret, Konya, Turkey). The dwell time in the water bath was 30 seconds and the transfer time was five seconds.

Under water cooling, teeth were sectioned occluso-gingivally into a serial slabs and further sectioned into 0.7 mm × 0.7 mm composite-dentin sticks. Approximately six sticks were obtained from each tooth using an Isomet diamond saw. Each stick was then attached to a custom jig of a universal testing machine (Lloyd LF Plus; Ametek Inc, Lloyd Instruments, Leicester, UK) and subjected to a tensile force at a crosshead speed of 1 mm/min until failure occurred. The fractured sticks were removed from the testing apparatus and the cross-sectional area at the site of failure was measured to the nearest 0.01 mm with digital calipers (Altas 905; Gedore-Altas, Istanbul, Turkey). The maximum force (in N) to produce fracture was recorded and the

bond strength (S) values (expressed in MPa) were calculated using the formula:

$$S = \frac{F}{A}$$

where F is the force (in N) and A is the bonded area (in mm²). Data were evaluated with 1-way ANOVA and post hoc Tukey-Kramer multiple comparisons tests ($\alpha=0.05$).

The fractured specimens were examined under a stereomicroscope (SMZ 800, Nikon, Tokyo, Japan) at 40X magnification to evaluate the fracture pattern. Failure modes were classified into one of three categories: adhesive failure if debonding occurred between resin and dentin; mixed failure if it exhibited partially adhesive, partially cohesive failure in bonding resin or in hybrid layer; or cohesive failure in resin or in dentin. All observations were conducted by one person.

Table 1. Materials used in the present study and their application methods.

Materials	Application protocol		Type
G-Bond	Apply one coat of adhesive, leave undisturbed for 10 s. Strong air-dry for 5 s. and light-cure for 10 s.		1-step self-etching (no rinse) adhesive
AdheSE	Apply primer, and when thoroughly coated, brush into for 15 s. (total reaction time 30 s.). Disperse excess amounts with a strong stream of air.	Apply bond beginning at dentin. Disperse with a very weak stream of air. Light cure.	2-step self-etching (no rinse) adhesive
Prime & Bond NT	Etchant 35% phosphoric acid 15 s, rinse and blot dry, adhesive application, gentle air stream, light polymerize 10 s.		etch&rinse adhesive
Venus			Resin composite

4-MET; 4-methacryloyxyethyl trimellitate anhydride, UDMA; urethane dimethacrylate, PENTA; dipentaerythritol pentaacrylate monophosphate.

RESULTS

1-way ANOVA tests results for MTBS measurements of the groups are summarized in Table 2. The highest mean force value was observed in group 3 specimens, and this was followed by group 3T specimens. There was no statistical difference in MTBS between groups 3 and group 3T ($p=0.988$). A statistically significant difference in MTBS was found between groups 1 and 1T ($p<0.05$). However, no significant differences were

found in MTBS between groups 2 and 2T ($p=0.962$). A statistically significant difference in MTBS was found between groups 1 and 3 ($p<0.05$), and groups 2 and 3 ($p<0.05$). However, there was no statistical difference in MTBS between group 1 and group 2 ($p=1.000$).

Modes of failure are presented in Table 3. Recorded failures were mainly adhesive (70–100%) in all experimental groups. In addition, there was an increase in adhesive failures after thermal cycling.

Table 2. Mean microtensile bond strength and SD of each group.

Groups	Mean (MPa)	SD
Group 1	29.59 ^a	4.21
Group 2	29.35 ^a	1.76
Group 3	34.47 ^b	4.15
Group 1T	25.91 ^c	2.58
Group 2T	30.26 ^a	4.19
Group 3T	33.77 ^b	3.13

$n=20$, $df=5$, $MS=198.828$, $F=16.492$ and groups with same superscripted letters not significantly different ($p>0.05$).

Table 3. Modes of failure in each group of specimens.

Groups	n	Adhesive failure	Cohesive failure	Mixed failure
Group 1	20	17	3	—
Group 2	20	14	4	2
Group 3	20	14	5	1
Group 1T	20	19	1	—
Group 2T	20	20	—	—
Group 3T	20	18	2	—

DISCUSSION

The results of the present study require partially rejection of the null hypothesis. Because, contrary to results with 2-step self-etch and etch&rinse adhesive systems, thermal cycling was found effective on the MTBS of the 1-step adhesive system. This result is not in accordance with the study of Asaka et al.¹⁰ who researched influence of different storage conditions on dentin bond strengths of 1-step self-etch adhesive systems and concluded that no changes in bond strength were found in each storage condition for G-Bond. Moreover, contrary to results with the present study, Asaka et al.¹¹ found that no significant differences between different storage conditions for 1-step self-etch adhesive systems. In addition, Miyazaki et al.¹³ advocated that dentin bond strength decreased as the number of thermal cycles increased in 2-step self-etch adhesive systems. Sadek et al.⁶ found that compared to 2-step self-etch adhesive systems, 1-step self-etch adhesive systems showed lower bond strengths and concluded that etch&rinse adhesive systems exhibited the highest MTBS. According to Knobloch et al.,¹⁴ no significant difference was noted between the mean MTBS values of the 1- and 2-step self-etch adhesives, except G-Bond, which was significantly lower than etch&rinse adhesive Prime & Bond NT. In the present study, the highest MTBS was found in etch&rinse adhesive systems and 1- and 2-step self-etch adhesives showed similar bond strengths. Results of the etch&rinse adhesive systems were consistent with those of Sadek et al.⁶ and Knobloch et al.¹⁴ However, results of self-etch adhesives contradicted to those of Sadek et al.⁶ and Knobloch et al.¹⁴ Generally, current interest in dentine bonding research is focused on reducing the number of application steps in the bonding procedure and reducing the technique sensitivity as well as operator variability. The self-etch adhesives have reduced the number of steps involved.¹ 1-

step self-etch adhesive systems exhibited similar bond strengths to the 2-step self-etch adhesives. Clinically, 1-step systems are generally less technique-sensitive than 2-step adhesives; however, 1-step systems tend to be more hydrophilic due to a higher concentration of acidic monomers to properly etch the enamel surface.

On the other hand, in the present study, modes of failure were also determined and all groups predominated in adhesive failure. These result was in agreement with the previous studies.^{10,11,14,15}

The use of thermal cycling in dental restorations is frequently seen in laboratory studies in order to simulate changing intraoral temperature conditions. Thermo-cycling is a widely used artificial aging methodology. A recent literature review concluded that 10.000 cycles corresponds approximately to 1 year of in vivo functioning.¹⁶ During thermal cycling, the specimens subjected to stresses that were generated by differential thermal conductivity. The temperature changes that occurred inside a specimen as a result of the temperature of the surrounding water are expected to have a significant impact.^{10,11,17} During the thermal cycling test, hot water might accelerate hydrolysis of the resin and extract poorly polymerized resin oligomers.¹⁸

CONCLUSIONS

Within the limitations of the present study, 1-step self-etch adhesive systems exhibited similar bond strengths to the 2-step self-etch adhesives. Moreover, etch&rinse adhesive systems exhibited the highest bond strength. Contrary to results of 1-step self-etch adhesive, thermal cycling was found ineffective on the microtensile bond strength of the 2-step self-etch and etch&rinse adhesive systems.

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