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Effect of thermal cycling on micro-tensile bond strength of composite restorations bonded with multimode adhesive

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Objective: The purpose of this *in vitro* study was to evaluate the effect of thermal cycling on the micro-tensile bond strength (Mtbs) of multimode adhesive agents. *Materials and methods*: Eight freshly extracted caries-free human third molars were used. The flat dentin surfaces were prepared and polished with 600-grit SiC abrasive paper for standard smear layer formation. The teeth were restored using Single Bond Universal Adhesive [(total etch (G1, G2)/self etch (G3, G4)] + Filtek Z550 and All-Bond Universal Adhesive [(total etch (G5, G6)/self etch (G7, G8)] + Aelite all-purpose. The specimens in groups G1, G3, G5, and G7 were subjected to thermal cycling (1000 cycles at 5–55 °C, for a 30 s dwell time), while the specimens in other groups were not exposed to an aging procedure. The Mtbs test was determined in all procedures. Data were submitted to three-way ANOVA and *post hoc* tests. The significance level was set at = 0.05. *Results*: Group five was highly affected by the thermal cycling following the total etch procedure, while group one was not significantly affected. Group seven was highly affected by thermal cycling, while group three was not significantly affected after the self etch procedure. Group eight exhibited a higher mean Mtbs value after the thermal cycling procedure. *Conclusion*: The bond strength of multimode (universal) adhesives was found to be material dependent. The total etch procedure showed a higher Mtbs value than the self etch procedure.

Keywords: micro-tensile bond strength; multimode adhesives; thermal cycling; universal adhesive

1. Introduction

In recent decades, adhesive dentistry has developed various approaches for bonding to hard dental tissues. Research regarding the mechanisms of adhesion to enamel and dentin has lead to more satisfactory clinical results, and it has enhanced the durability of preventive and restorative procedures.

Conventionally, adhesion to dental substrates has been accomplished through the use of bonding agents with advanced micro-mechanical interlocking to both the enamel and the dentin.[1,2] Approaches with different numbers of steps and degrees of sensitivity have been used to bond resin-based materials to enamel and dentin.[3,4] Early clinical studies reported the dentin bonding performances of different one- or two-step self etching adhesives.[5] Adhesive systems progress rapidly. Due to differences in

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professional decisions regarding the selection of adhesive strategies and the number of steps involved in the self etching process, some manufacturers have released all-purpose adhesive systems that include etch and rinse (two-step) and self etch (one- or two-step) options. These new materials are called universal, multipurpose, or multimode adhesives.[6,7] There is little information in the literature about the performance of this new class of universal adhesives.[6,7]

Restorative materials are used to seal dental cavities. Pain, marginal staining, and caries often occur after the use of restorative materials. These conditions may be associated with an insufficient cavity seal,[8,9] which is thought to be aggravated by the effects of thermal changes.[10]

Thermal cycling is a method used often in dental investigation, particularly when testing the performance of adhesive materials. This method aims to thermally stress the adhesive joint at the tooth restoration interface by subjecting the restored teeth to extreme temperatures concordant with temperatures encountered intraorally.[11]

Intraoral temperature variations may be induced by routines of eating,[12] drinking [10,13], and breathing.[14] Thermal stresses can be pathogenic in two ways: mechanical stresses induced through differences in bonded interfaces[10] and changing interval sizes associated with interval volume changes, which pump pathogenic oral fluids in and out of the intervals.[10] These stresses may negatively affect the resin–dentin bond. In this context, the use of thermal cycling *in vitro* has been considered a useful method for simulating *in vivo* challenges.

A new type of bonding system, called a universal (multipurpose or multimode) adhesive, was recently introduced for restorative procedures.[6,7] The purpose of this study is to evaluate the effect of thermal cycling on the micro-tensile bond strength (Mtbs) of composite restorations that have been bonded with multimode adhesive agents.

The null hypothesis of this study is that thermal cycling does not have an effect on the bond strength of the multimode adhesive systems applied with total etch or self etch techniques.

2. Materials and methods

2.1. *Teeth and surface preparation*

The research protocol of this study was designed according to the guidelines of the Ethics Committee of Bezmialem Vakif University (22 April 2013, 37/6).

Eight noncarious, nonrestored, intact, human mandibular third molar teeth were extracted and used in this study. After extraction, the teeth were scaled of their surface debris and stored in distilled water at room temperature for one week. Each tooth was mounted in cold curing acrylic resin (Meliodent, Bayer Dental Ltd, Newbury, UK). The occlusal surface of each tooth was reduced to expose the dentin with a slow-speed diamond saw under water lubrication (Isomet, Buehler Ltd, Lake Bluff, IL, USA). The surface of each tooth was ground with 600-grit SiC paper under running water for 30 s to create a smear layer of clinically relevant thickness.

2.2. *Experimental design*

The teeth were randomly divided into eight groups of one tooth each and were restored with investigated adhesive systems and their corresponding resin composites. The application protocols suggested by each manufacturer are listed in Table 1.

Two-millimeter high composite resin core buildups were created with the help of a matrix (Super Mat Adapt Supercap Matrix, Kerr, Switzerland). Incremental technique was used for this purpose, and each increment (2 mm) was cured for 40 s using an LED light-curing unit set at 1200 mW/cm^2 (Optima-10, BA International, Kingsthorpe, Northampton). The groups are shown in Table 2.

The specimens from groups 2, 4, 6, and 8 were stored in distilled water at 37 °C for 24 h after placement without thermal cycling, and those from groups 1, 3, 5, and 7 were subjected to 1000 thermal cycles between 5 and 55 °C. The dwelling time in the water was 30 s, and the transfer time was 10 s (SD Mechatronic Thermocycler, Germany). After treatment, all teeth were sectioned with a slow-speed saw (Isomet, Buehler Ltd, Lake Bluff, IL, USA) under water cooling into multiple 0.9 × 0.9 mm beams, with the 'non-trimming' version [15] of the micro-tensile test. The obtained composite resin–dentin sticks ($n = 15$) were performed in tension using a universal testing machine (SD Mechatronic MTD 500, Germany) at a crosshead speed of 0.5 mm/min until failure. The cross-sectional area at the site of failure was measured to the nearest 0.01 mm with a digital caliper (Model CD-6BS; Mitutoyo, Tokyo, Japan), from which the Mtbs was calculated and expressed in MPa. The data were analyzed using a three-way ANOVA to measure the effects of the bonding agent, thermal cycling, and different procedures. A one-way ANOVA and a *post hoc* test were then run at a 0.05 level of significance.

After the Mtbs testing was completed, the specimens were examined at 30× magnification with a stereomicroscope (Nikon SMZ 1000, Japan). The specimens were classified as either adhesive, cohesive within the resin composite or dentin, or mixed failures.

3. Results

The results of this study are shown in Table 3. Following the total etch procedures, significant differences were detected in the mean Mtbs between groups 5 and 6 ($p < 0.05$). The highest Mtbs value was observed in group 6, followed by group 2, which was not subjected to thermal cycling. There were no statistical differences in Mtbs values between these groups ($p > 0.05$). Group 5, which was subjected to thermal cycling, exhibited lower mean Mtbs values than groups 2 and 6.

When both adhesives were applied with self etch procedures, there were no statistical differences between groups 3–7 and 4–8 ($p > 0.05$). Group 3 revealed a significantly lower mean Mtbs value compared to group 2. In addition, there were statistically significant differences between groups 1–3 and 5–6.

The modes of failure are presented in Table 4. Recorded failures were mainly adhesive in all experimental groups, except group 2. A higher mix failure was observed in group 2 than in the other groups.

4. Discussion

The assessment of bonding durability is an important research topic, as the bond between restorative materials and tooth substrates has a major impact on the clinical success of a restoration. The thermal cycling test involves subjecting specimens to extreme temperatures that simulate intraoral conditions.[16,17] Thermal cycling also stresses the bond between the resin and the tooth. Depending on the dentin bonding systems, thermal cycling may also affect bond strength.

Table 1. Adhesive system (manufacturer's, lot number), composition, and 'application mode' of the adhesive systems used according to the manufacturer's instructions.

Adhesive system (lot number)	Composition	Self etch strategy	Total etch strategy
Single Bond Universal Adhesive (3MDeutschland GmbH, Neuss, Germany) Lot no: 490282	(1) MDP phosphate monomer (2) Dimethacrylate resins (3) Vitrebond™ copolymer (4) Filler (5) Ethanol (6) Water (7) Initiators (8) Silane	(1) Apply the adhesive to the entire preparation with a micro-brush and rub it in for 20 s. If necessary, rewet the disposable applicator during treatment (2) Direct a gentle stream of air over the liquid for about 5 s until it no longer moves and the solvent has evaporated completely (3) Light polymerize for 10 s	(1) Apply etchant phosphoric acid etching gel (35%) tooth structure and for 15 s (2) Rinse for 10 s (3) Air dry 2 s do not overdry (4) Apply adhesive as for the self etch mode
All-Bond Universal Adhesive (Bisco, Inc. Schaumburg, IL USA) Lot no: 1200011536	(1) **Etchant** Uni-etch: 32% phosphoric acid, Benzalkonium Chloride (2) **Adhesive** MDP, 5–15 wt.%, bis-GMA, 30–60 wt.%, HEMA, 5–15 wt.%, ethanol, water, 10–40 wt.%, photoinitiator	(1) Apply two coats and agitate adhesives for 10–15 s, gently air dry, light cure 10 s. (2) The surface should have a uniform glossy appearance (3) Light polymerize for 10 s at 1200 mW/cm^2	(1) Apply etchant for 15 s (2) Rinse thoroughly (3) Remove excess water with absorbent pellet or high volume suction for 1–2 s (4) Apply adhesive as for the self etch mode

Composite resin	Composition	Application
3 M Filtek Z550, shade A3 (3 M Espe, St.Paul, MN,USA) Lot no: 340139	**The filler system** (1) Surface-modified zirconia/silica (<3μ) (2) Nonagglomerated/nonaggregated 20 nm surface-modified silica particles (3) The filler loading is 82% by weight (68% by volume) **The resin system** BIS-GMA, UDMA, BIS-EMA, PEGDMA, and TEGDMA	After the bonding procedures, the teeth received a nanohybrid composite restoration in two increments of 2 mm. Each increment was light polymerized for 40 s using a LED light-curing unit set at 1200 mW/cm^2
Aelite All-Purpose Body, shade A3 (Bisco, Schaumburg, IL, USA) Lot no: 060005269	Ethoxylated bisphenol A dimethacrylate Triethyleneglycol dimethacrylate, Glass filler Amorphous silica filler weight (%) 73	After the bonding procedures, the teeth received a micro-hybrid composite restoration in two increments of 2 mm. Each increment was light polymerized for 40 s using a LED light-curing unit set at 1200 mW/cm^2

Table 2. Study groups.

Adhesive system	Application mode	Thermal cycling (TC)	Groups
Single Bond Universal Adhesive	Total etch	With TC	1
	Total etch	Without TC	2
	Self etch	With TC	3
	Self etch	Without TC	4
All-Bond Universal Adhesive	Total etch	With TC	5
	Total etch	Without TC	6
	Self etch	With TC	7
	Self etch	Without TC	8

Table 3. Mean Mtbs and SD values of the different experimental groups. Groups with different letters show statistically significant differences.

Adhesive system	Application mode	Thermal cycling (TC)	Mtbs & SD
Single Bond Universal Adhesive	Total etch	With TC	35.6 ± 10.3CDE
	Total etch	Without TC	38 ± 8.3 DE
	Self etch	With TC	26.5 ± 5.3 AB
	Self etch	Without TC	29.5 ± 9.9 ABCD
All-Bond Universal Adhesive	Total etch	With TC	27 ± 8.6 ABC
	Total etch	Without TC	39.3 ± 7.7 E
	Self etch	With TC	31.5 ± 6.8 BCDE
	Self etch	Without TC	21.3 ± 5.2 A

Table 4. Number of specimens according to the fracture mode of all experimental group.

Adhesive system	Application mode	Thermal cycling (TC)	Adhesive	Mix	Cohesive
Single Bond Universal Adhesive	Total etch	With TC	9	5	1
	Total etch	Without TC	1	9	5
	Self etch	With TC	9	6	–
	Self etch	Without TC	10	4	1
All-Bond Universal Adhesive	Total etch	With TC	15	–	–
	Total etch	Without TC	10	4	1
	Self etch	With TC	8	5	2
	Self etch	Without TC	12	1	2

Our aim in this study was to investigate age-induced changes in the bonding of multimode adhesives to human teeth. The specimens were subjected to 1000 cycles of thermal aging. According to the ISO TR 11405,[18] conducting 500 thermal cycles in water temperatures between 5 and 55 °C is an appropriate test for aging dental materials. In addition, 10,000 thermal cycles were suggested to correspond to approximately one year of *in vivo* functioning.[18,19] Some researchers question this method, since the temperatures used may not be the real temperatures of hot and cold beverages consumed by patients.[20]

The results of this study require partial rejection of the null hypothesis. Compared to the All-Bond Universal Adhesive system, the Single Bond Universal Adhesive system was not affected by thermal cycling when used with the total etch and self etch approaches. The differences in the compositions of these two products seem to be key reasons for the different performance of these materials.

When the Single Bond Universal Adhesive system was bonded with dentine, the bond strengths for both the total etch and the self etch procedures were not affected by the thermal cycling. These results are in accordance with the study of Asaka et al. [17] who reported that after 10,000 thermal cycles, the mean shear bonding strength of the one step self etch dentin bonding system remained unchanged. The Mtbs value of the total etch approach was higher than the self etch approach of the Single Bond Universal Adhesive. The phosphoric acid etching of dentin prior to the application of the total etch procedure increased the bond strength of dentin. Phosphoric acid can decalcify human dentin more deeply than a self etch adhesive is designed to infiltrate. Kenshima et al. [21] reported that the smear layer creates the true physical barrier and makes it extremely difficult for the bonding and hybrid layer formation to become fully integrated with the dentin. After beginning the etching with phosphoric acid, the smear layer is removed and the superficial dentin is demineralized. This promotes impregnation by the adhesive, allowing for the creation of a well-impregnated hybrid layer. This is done without modifying the potential for interfacial nanoleakage, as has been shown by several authors when a one-step self etch adhesive was applied on phosphoric acid etched dentin.[6,22]

Bond strength to dentin generally decreased with thermal cycles. In this study, the All-Bond Universal Adhesive had a higher Mtbs value in the self etch procedure after thermal cycling. In contrast, the total etch procedure was found to decrease the Mtbs value.

The specimens were subjected to stresses during thermal cycling that were produced by differential thermal conductivity. The temperature changes that occurred inside a specimen as a result of the thermal cycling were expected to have a major impact.[23] Collagen that is exposed to the acidic attack of a self etch adhesive can become sufficiently impregnated with resin components. When this happens, hydrolysis might occur, leading to bonding failure [24] during the thermal cycling test. Hot water might accelerate the hydrolysis of the resin and extract poorly polymerized resin oligomers.[25] However, this study found no effect of thermal cycling on the bond strength of All-Bond Universal Adhesives when applying the self etch strategy. The different values of bond strength between the self etch and the total etch strategies on dentin may be explained by the fact that 10-methacryloyloxydecyl dihydrogen phosphate (MDP), containing universal adhesives, benefits from the presence of residual apatite on the collagen fibrils. This results in additional chemical bonding, and it may produce more stability after thermal cycling. Yoshida et al. [26] suggested using self etching adhesives that utilize MDP to form self-assembled nanolayers at the tooth-bond interface. This could be the reason for their higher bond strengths to teeth. Despite these similarities, these universal adhesives are not equivalent to each other. The differences in terms of the Mtbs between self etch procedures in the two different systems may be due to the presence of polyalkenoic acid copolymer in the composition of the Single Bond Universal Adhesive system.

Munoz et al. [27] evaluated Mtbs values, nanoleakage, and degree of conversion within the hybrid layer for total etch and self etch strategies of universal simplified adhesive systems. They used Peak Universal Adhesive (Ultradent, USA), Scotchbond Universal Adhesive (3 M ESPE, USA), and the All-Bond Universal Adhesive system (Bisco, USA). They used the Clearfil SE Bond Adhesive system (Kuraray, Tokyo, Japan) as a control for the self etch strategy and the Adper Single Bond Adhesive system (3 M ESPE, USA) as a control for the total etch strategy. They reported the lowest Mtbs value in the All-Bond Universal Adhesive when it was applied with the self etch procedure according to the other universal adhesives (Scotchbond and Peak Universal adhesives). These results were compatible with our study; however, the groups in this study were not subjected to thermal cycling. The low Mtbs value of All-Bond Universal Adhesive may be attributed to its application mode. All-Bond Universal Adhesive was not applied actively on dentine, as the materials were applied in accordance with the respective manufacturer's directions for the self etching application. Previous literature demonstrates that active application improves the bonding performance of self etch adhesive systems to dentin.[28,29]

Modes of failure were also determined in this study. These were identified as mainly adhesive failures, with the exception of total etch failure in the Single Bond Universal Adhesive system. There were mainly mix failures in this group. These results matched those of the previous studies.[17,28]

5. Conclusion

The multimode adhesive systems tested in this study exhibited highest bond strength when they were applied with the total etch approach and were not subjected to thermal cycling. When the tested universal adhesive systems were subjected to thermal cycling, the All-Bond Universal Adhesive was highly affected, while the Single Bond Universal Adhesive was not. Further studies are needed to assess the long-term clinical performance and physical quality of new multimode (universal) simplified adhesives.

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Conflict of interest

This study has been presented as a poster at '18. Department of Restorative Dentistry Meeting "Restorative Current Approaches to Treatment" Symposium', Kayseri-Turkey.

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