Original Article

Effects of Artificial Aging on the Bond Strengths of Universal Dental Adhesives

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ABSTRACT

Aims: The effect of mechanical loading on the microtensile bond strength (μTBS) of universal adhesives to dentin was evaluated in this study. **Methods and Materials:** Human molar teeth had the occlusal dentin surfaces exposed and were allocated into ten groups (five experimental groups and five control groups) that used the following universal adhesive systems in self-etch mode: All-Bond Universal (ABU), Single Bond Universal (SBU), Gluma Bond Universal (GBU), Tetric N-Bond Universal (TBU), and Clearfil Universal Bond (CUB). Following the bonding procedures and build-ups, the specimens were either stored in water at 37°C for 24 h or were mechanically loaded (50 N for 60,000 cycles) prior to the μTBS test. Data were analyzed using the one-way analysis of variance (ANOVA) and Tukey's posthoc test ($P = 0.05$). **Results:** Both the adhesive type and mechanical loading had significant effects on the μTBS ($P < 0.05$). The μTBS values of SBU and ABU were significantly higher than the values of the other adhesives ($P < 0.05$). However, the μTBS values of ABU decreased significantly after mechanical loading ($P < 0.05$). **Conclusions:** With the exception of ABU, mechanical loading had no deleterious effects on the μTBS of the universal adhesive systems examined in this study.

Keywords: *Mechanical loading, self-etch, universal adhesives, μTBS*

Introduction

The techniques used in adhesive dentistry have been developed rapidly since Buonocore introduced the acid etching of dental tissues. Dental adhesives attach to dental hard tissues using micromechanical adhesion, and consist of an acid etchant, and primer and resin monomer agents.[1] The monomers penetrate the enamel/dentin tissues and form a strong link among the enamel, dentin, and restorative material after polymerization.[2] Currently, there are two types of adhesive systems according to the clinical application: total-etch and self-etch.[3] Total-etch adhesive systems involve washing the enamel and dentin hard tissue after the acid is applied and completely removing the smear layer.[4,5] In self-etch adhesive systems, etching and priming are carried out simultaneously because acidic monomers have been added to the primer. In this way, the acidification and the washing steps are eliminated.[1] Self-etch adhesives contain conventional components, such as fillers, polymerization initiators, and inhibitors, and various monomers. The monomers can be divided into three groups: self-etching (acidic) monomers, cross-linking monomers, and monofunctional co-monomers.[6] Self-etching adhesive monomers contain phosphoric acid groups, such as 10-methacryloxydecyl dihydrogen phosphate (10-MDP), or carboxylic acid groups, such as 4-methacryloxyethyl trimellitic (4-MET) acid, dipentaerythritol pentaacrylate phosphoric acid ester (PENTA), or di-2-hydroxyethyl methacryl hydrogen phosphate (di-HEMA-P).[7,8] Because the acids used in self-etch adhesive systems are not as strong as phosphoric acid used in the total-etch system, the adhesive forms a weaker bond with the enamel, which can lead to long-term discoloration of the enamel edges

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and the restorations.[9] In order to prevent this situation, it is recommended that only the enamel margin of the cavity be pre-etched.[10] However, in practice, during acid etching of the enamel, the dentin may be exposed to the acid.

New adhesive systems have been developed to eliminate the disadvantages of self-etch adhesive systems.[11] These new products are known as universal adhesives because they can be used as self-etch adhesives, total-etch adhesives, or total-etch adhesives on enamel and self-etch adhesives on dentin (i.e. selective enamel etching).[12] Universal adhesives have a composition similar to conventional single-stage self-etch adhesives, although most also contain specific carboxylate and/or phosphate monomers that ionically bond to calcium in hydroxyapatite.[13] Universal adhesive formulations may include functional monomers and silanes, such as 10-MDP, PENTA, polyalkenoic acid copolymer (Vitrebond copolymer), and phosphorous-containing monomers.[14] For example, Tetric N-Bond Universal (TBU) contains decandiol dimethacrylate (D3MA) and methacrylated carboxylic acid polymer (MCAP).[15] The 10-MDP monomer has been incorporated into the composition of many universal adhesives; it forms ionic bonds with dentin and a nanolayer of hydrolytically stable calcium salts on hydroxyapatite.[9] Many research papers have evaluated the chemical interactions of 10-MDP with the structures of teeth.[9,16] A 13-year study of a 2-step self-etch adhesive (Clearfil SE Bond) showed that the stability of the 10-MDP-mediated chemical bond resulted in excellent clinical performance.[17] Other studies have shown that adhesives containing 10-MDP and PENTA performed well in long-term clinical trials.[9,18] Both 10-MDP and PENTA have been included in the composition of different adhesives for decades to increase adhesion to tooth structure.

The most accurate way for the performance evaluations of restorative materials can be performed in clinical evaluations. However, the combination of different stresses and forces within the mouth can make it difficult to determine the cause of failure in the restorative materials. In addition, clinical tests are long-term and there is a lack of a standard testing protocol.[19] Different methods have been developed to mimic the stresses and forces occurring in the mouth and the effect they have on the behavior and life of the restorative material. One method that is used for artificially aging restorations involves chewing simulators; the bond strength of the restoration is assessed after the aging process.[20] Different results have been reported in the literature regarding the effect of mechanical loading on bond strength because the process can cause a decrease in the bond strength of the adhesive.[14,20] And there is insufficient information about the post-mechanical bonding of many of the newly developed adhesive systems.

Tensile bond strength testing is frequently used to evaluate the bonded interface of the adhesive. Sano *et al*. developed the microtensile bond strength (μTBS) test, which is widely used and has many advantages.[21] In addition to the small surface area (about 1 mm^2) that is required for the μTBS test, other advantages are that many samples can be taken from a tooth, the test allows for the determination of regional bonding differences, and the distribution of stress at the interface is more homogeneous.[21]

The aim of this study was to compare the μTBS values of five universal adhesive systems in self-etch mode on the surface of dentin tissue before and after mechanical loading (chewing simulation). The null hypotheses of this study were that mechanical loading does not decrease the μTBS values of the dentin tissue surface and that there is no difference between the five universal adhesives on the dentin μTBS values.

Materials and Methods

This study used 40 freshly extracted noncarious human third molars. Following a approval by the Dental Faculty Ethics Committee of Selçuk University, Konya, Turkey (no: 2006/02-04), the extracted teeth were thoroughly cleaned to remove hard and soft deposits and stored at 4°C in saline containing 0.01% thymol. The occlusal dentin surfaces of the teeth were exposed by removing the occlusal enamel and superficial dentin with a water-cooled, slow-speed diamond saw (IsoMet 1000 Precision Cutter, Buehler, Lake Bluff, IL, USA). The teeth were randomly assigned to five experimental groups and five control groups that used the following universal adhesive systems in self-etch mode: Clearfil Universal Bond (CUB) (Kuraray Medical Inc., Osaka, Japan); Gluma Bond Universal (GBU) (Kulzer, Hanau, Germany); Single Bond Universal (SBU) (3M ESPE, Neuss, Germany); All-Bond Universal (ABU) (Bisco Inc., Schaumburg, IL, USA); and Tetric N-Bond Universal (TBU) (Ivoclar Vivadent, Schaan Liechtenstein).

A smear layer was applied to the dentin surface by polishing the occlusal surface with 600-grit silicon carbide sandpaper. The teeth in each group were then embedded in acrylic resin. The bonded interface was prepared according to the adhesive group and applied in accordance with the manufacturers' instructions [Table 1]. After the adhesive was applied, the dentin surfaces were restored using composite resins obtained from the respective producer of each adhesive: Clearfil Photo Posterior (Kuraray Medical Inc., Osaka, Japan), Charisma Classic Syringe Refill (Kulzer, Hanau, Germany), Filtek Z550 Nano Hybrid (3M ESPE, St. Paul, MN, USA), Aelite All-Purpose Body (Bisco Inc., Schaumburg, IL, USA), and Tetric N-Ceram Bulk Fill (Ivoclar Vivadent, Schaan, Liechtenstein). Direct composite resins were built-up incrementally on the bonded surface to a height of 4 mm. Each 2-mm increment of resin composite was cured for 20 s under a VALO broadband LED curing light (Ultradent Products, Inc., South Jordan, UT, USA) at a standard curing distance of 0.5 mm and a light intensity of 1600 mW/cm^2, which was constantly monitored with a radiometer. After bonding, each group was divided into 2 subgroups (n = 4). One subgroup was stored in water for 24 h before undergoing the μTBS test. The other subgroup was tested after mechanical loading.

Mechanical load cycling test

The root surfaces of the teeth were covered with a 1-mm layer of polyether impression material (Impregum Penta DuoSoft; 3M ESPE, Seefeld, Germany) using a dispenser gun (3M ESPE). A scalpel blade was used to remove the excess silicone material to provide a flat surface 2 mm below the facial cementoenamel junction of each tooth. The teeth were then embedded in acrylic resin (Pattern Resin LS; GC America Inc., Alsip, IL, USA). The thin layer of silicone material simulated the periodontal ligament on the root surfaces of the teeth. Occlusal contact loading was simulated in an artificial oral environment sliding wear testing apparatus (chewing simulator; Selçuk University, Research Laboratory Center, Konya, Turkey).[22] Specimens were stored in distilled water in a simulator chamber at 37°C in occlusal contact to simulate normal physiological conditions [Figure 1]. Four specimens of each material were tested using a pin-on-block design; a stainless steel antagonist tip was placed in contact with a composite specimen and delivered a vertical load of 50 N for 60,000 cycles at a frequency of 1.2 Hz within an eccentric sliding radius of 0.3–0.8 mm.

Microtensile bond test procedures

After the mechanical load cycling test (artificial aging), the specimens were retrieved from the storage medium to determine the μTBS. The nontrimming technique, which was first described by Sano *et al*., was used for the μTBS test.[21] Four teeth were used for each bonding system. Each tooth was sectioned using a water-cooled slow-speed saw (IsoMet 1000 Precision Cutter; Buehler, Lake Bluff, IL, USA) into multiple 1.00 ± 0.03 mm × 1.00 ± 0.03 mm beams. Five standard beams were obtained for each tooth from the mechanically loaded teeth and the control teeth.

The specimens were then attached to a modified microtensile testing apparatus with a cyanoacrylate adhesive (Zapit; Dental Ventures of America, Inc., Corona, CA, USA) and subjected to tensile forces in a microtensile testing machine (Micro Tensile Tester; Bisco Inc., Schaumburg, IL, USA) at a crosshead speed of 1 mm/min [Figure 2]. The cross-sectional area at the site of failure was measured to the nearest 0.01 mm using digital calipers (model CD-6BS; Mitutoyo, Tokyo, Japan). This area was used to calculate the μTBS value in MPa: μTBS (MPa) = F (N)/bond area (mm^2).

Failure mode analysis

After the μTBS testing, the fractured surfaces of all the specimens were examined using a stereo microscope (LGP52; Olympus, Tokyo, Japan) to determine the mode of failure at 50 × magnification. Failures were classified as adhesive (interfacial failure), cohesive in dentin, cohesive in resin, which included failures in the resin composite or in the adhesive layer, or mixed.

Statistical analysis

Data were analyzed using the one-way analysis of variance (ANOVA) to evaluate the effects of the type of adhesive and the mechanical loading on the μTBS values. Tukey's honestly significant difference (HSD) test was used to compare the adhesives at a significance level of 0.05. Statistical analyses were performed using SPSS software (version 22.0; IBM Corp., Armonk, NY, USA).

Results

The means and standard deviations of the μTBS for each group are summarized in Table 2. Statistically significant differences were found among the μTBS values of the materials (P < 0.05) [Figure 3].

In the control group, SBU showed the greatest bond

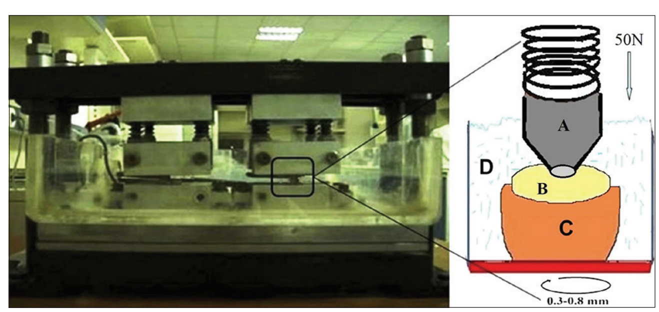

Figure 1: The chewing simulator employed in the study. The insert at the right schematically illustrates the alignment of one specimen in one chamber of the chewing simulator. (A) Antagonist stainless steel tip; (B) composite specimen; (C) tooth specimen; (D) medium

Table 1: Composition and application procedures of adhesives used in the study

	Composition	Self-Etch Mode
Gluma Bond Universal (GBU)	4-MET acid, methacrylate monomer, acetone, 10-MDP, and water	1. Adhesive is applied to the prepared tooth for 20 s. 2. The adhesive layer is dried until it is immobilized. 3. Light cure for 10 s.
Clearfil Universal Bond (CUB)	Bis-GMA, HEMA, ethanol, 10-MDP, hydrophilic aliphatic dimethacrylate, colloidal silica, DL-camphorcinone, silane, accelerators, initiators, and water	1. Adhesive is applied to the prepared tooth for 10 s. 2. The adhesive layer is dried for 10 s. 3. Light cure for 10 s.
Tetric N-Bond Universal (TBU)	10-MDP, MCAP, HEMA, Bis-GMA, D3MA, ethanol, highly dispersed silicon dioxide, water, initiators, and stabilizers	1. Adhesive is applied to the prepared tooth for 20 s. 2. The adhesive layer is dried until it is immobilized. 3. Light cure for 10 s.
All-Bond Universal (ABU)	Bis-GMA, 10-MDP, HEMA, ethanol, initiators, and water	1. Adhesive is applied to prepared tooth for 10–15 s for 2 layers without irradiation between the layers. 2. The adhesive layer is dried for 10 s. 3. Light cure for 10 s.
Single Bond Universal (SBU)	10-MDP, dimethacrylate resins, HEMA, Vitrebond copolymer (acrylic acid and itaconic acid), filler, ethanol, initiators, silane, and water	1. Adhesive is applied to the prepared tooth for 20 s. 2. The adhesive layer is dried for 10 s. 3. Light cure for 10 s.

Table 2: The microtensile bond strengths (µTBS) of the adhesive systems (MPa). *The same superscript letters in the columns and rows indicate no significant differences (*P*>0.05)

	n	Control group µGBD (MPa)	After 60,000 cycles of mechanical loading µGBD (MPa)
Gluma Bond Universal (GBU)	20	12.4350 ± 6.79181^{a}	10.1600 ± 4.97609^{a}
Clearfil Universal Bond (CUB)	20	9.4100 ± 5.46124^{a}	12.0400 ± 5.55274^{a}
Tetric N-Bond Universal (TBU)	20	14.9550 ± 5.02703^{ab}	17.3000 ± 4.92865^{bc}
All-Bond Universal (ABU)	20	22.4150 ± 9.61097^{cd}	16.0100 ± 7.74460^{ab}
Single Bond Universal (SBU)	20	23.8150 ± 7.64449^{cd}	28.5450 ± 7.39420^{d}

Figure 2: Placing the samples prepared in the study in a microtensile testing machine

strength (23.81 ± 7.64 MPa), while CUB had the weakest bond strength (9.41 ± 5.46 MPa); there was a statistically significant difference between these adhesive systems ($P < 0.05$). There was no statistically significant difference between the µTBS values of CUB, GBU, and TBU ($P > 0.05$) and between ABU and SBU ($P > 0.05$).

In the mechanical load cycling (aging) group, SBU showed the greatest bond strength (28.54 ± 7.39 MPa), while GBU had the weakest bond strength (10.16 ± 4.97 MPa). In terms of µTBS, there was no statistical difference between CUB and GBU and between TBU and ABU ($P > 0.05$).

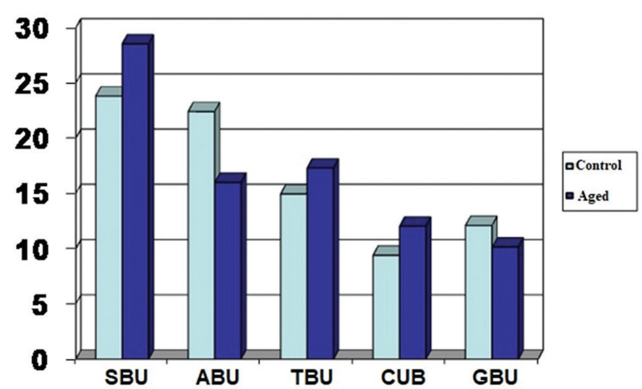

Figure 3: The microtensile bond strengths of the tested universal adhesive systems

The ANOVA revealed that both the adhesive system and the mechanical load cycling (aging) had a significant effect on the µTBS values ($P < 0.05$). The results of Tukey's HSD test showed that the µTBS values of ABU in the mechanical load cycling group were significantly lower than the µTBS values of the control group ($P < 0.05$). However, there were no significant differences between the mean µTBS values of the mechanical load cycling group and the control groups for the other adhesive systems ($P > 0.05$).

Table 3: Failure modes of the tested universal adhesive systems

	n	Adhesive-type failures	Mixed-type	Cohesive in composite	Cohesive in dentin
GBU control	20	20	0	0	0
GBU mechanical load cycling	20	20	0	0	0
CUB control	20	20	0	0	0
CUB mechanical load cycling	20	20	0	0	0
TBU control	20	20	0	0	0
TBU mechanical load cycling	20	20	0	0	0
ABU control	20	20	0	0	0
ABU mechanical load cycling	20	20	0	0	0
SBU control	20	16	3	0	1
SBU mechanical load cycling	20	14	3	0	3

The distribution of the failure modes among the universal adhesive systems is shown in Table 3. According to the microscopic analysis, except for the SBU control group and mechanical load cycling group, all groups showed 100% adhesive-type failures. The SBU mechanical load cycling group showed 70% adhesive-type failures, while the SBU control group showed 80% adhesive-type failures. Both SBU groups also showed mixed-type and cohesive-type failures [Table 3].

Discussion

In this study, the *in vitro* bonding performances of five universal adhesives (CUB, GBU, SBU, ABU, and TBU) were evaluated using the μTBS test before and after mechanical load cycling (aging). There are few studies on these universal adhesives because they are relatively new and contain different functional monomers.

Although clinical trials are used to evaluate the success of newly developed dental adhesives, the results are generally obtained over a long period of time.[1,7] Well-planned, randomized, controlled clinical trials are considered to be the standard for evaluating the success of dental adhesives.[23] However, when the clinical observations are concluded, usage of a dental adhesive may be restricted or completely eliminated. For this reason, it is necessary to apply laboratory tests based on estimating the long-term clinical behavior of dental adhesives. Therefore, in this study, we compared the success of the universal dental adhesives using laboratory tests.

Universal adhesive systems have been developed as single-step self-etch adhesives, although they can also be used with different techniques (i.e. total-etch, self-etch, and selective-etch) in clinical applications.[24] In our study, five universal adhesive systems were used in self-etch mode on dentin surfaces. The mean μTBS values of the control groups and the mechanical load cycling group of the five universal adhesives were statistically significantly different from each other. Therefore, the null hypotheses that mechanical loading does not decrease the μTBS values of the dentin tissue surface and that there is no difference between the five universal adhesive on the dentin μTBS values were rejected.

It has been reported that the 10-MDP monomer contained in universal adhesive systems chemically bond to hydroxyapatite crystals and forms a nanolayer that further increases the mechanical strength at the interface.[12] In addition, the accumulation of stable 10-MDP-Ca salts throughout the nanolayer increases the bond strength.[13] All the universal adhesive systems used in our study contained 10-MDP. The greatest μTBS was observed in the SBU group; the value was significantly higher than the values in the other control groups except ABU. In addition to 10-MDP, SBU contains the polyhydrous copolymer of Vitrebond, also known as polyalkenoic acid copolymer. One study reported that more than 50% of the carboxyl groups in this copolymer were bound with hydroxyapatite and replaced with phosphate ions by ionic bonds with calcium ions.[25] The additional calcium chelation can contribute to the stability and life of the dentin–adhesive interface.[24] Therefore, it can be assumed that the high bonding value of the SBU group may be related to the inclusion of polyalkenoic acid copolymer (Vitrebond copolymer) in its formulation. Our study, as well as other studies, found that the self-etch bonding values of SBU in dentin were higher than in other universal adhesives.[14,26]

In addition to 10-MDP, universal adhesive systems may contain functional acidic monomers, such as phenyl-P and 4-MET acid. It was reported that none of these monomers showed bonding values that were similar to 10-MDP.[9] The calcium salts formed by the functional acidic monomers are not resistant to dissolution and, thus, they are not hydrolytically stable.[25] It has been reported that 4-MET acid adhesives exhibit lower bonding values when the bonding strength is evaluated after a long aging period in dentin bonding systems with different contents to dentin.[27] In our study, GBU and CUB had the weakest μTBS. Due to the presence of 4-MET acid in GBU and the different composition of CUB, it can be assumed that the monomers degraded and affected the bonding values. Chen *et al.* also showed that CUB has a lower μTBS than other universal adhesives.[28]

Unlike other universal adhesive systems, TBU contains D3MA and MCAP. D3MA allows a reaction to take place between the composites or adhesive resin cements and the weaker polar monomers.[15] MCAP is a carboxylic acid functional polymer that reacts with and adheres to hydroxyapatite; the polymeric backbone or chain contains many carboxylic acid groups that allow multiple bonds to the tooth surface.[15] In our study, there was no additional increase in the μTBS values of TBU compared with the other universal adhesive systems.

The composition of universal adhesive systems, including fillers and non-fillers, can vary according to brands. ABU and GBU do not contain fillers. In this study, there was a reduction in the μTBS values of ABU and GBU groups after the mechanical load cycling (meaning significantly in ABU). The addition of filler prevents the adhesive layer from becoming too thin.[29] Due to oxygen inhibition, incomplete polymerization can occur if the adhesive layer is too thin.[30] Thus, the addition of filler to universal adhesive systems provides a stable hybrid layer that avoids degradation, which can occur in the interface after mechanical load cycling.

In this study, the mechanical loading caused a reduction in the bond strength because the applied load cycles caused the restorations to deform. The applied force may have caused micro- or nanospacings or fractures and cracks between the dentin surface and the adhesive layer; in addition, plastic deformations may have occurred in the adhesive layer itself. In our study, the only statistically significant decrease in the μTBS value that was caused by mechanical loading occurred in ABU. However, Farias *et al*. did not observe a decrease in the bonding strength of ABU in their mechanical loading study.[14] Differences in experimental conditions, sample preparation, and loading conditions may explain the conflicting results.[31,32] In addition, because the pH of the ABU that we used in our study was higher than the pH of the other universal adhesive systems, the decrease in μTBS could be due to the insufficient infiltration of the adhesive or to the presence of a hybrid layer that may have been more easily deformed during mechanical loading. The presence of 10-MDP, along with the chemical affinity of residual hydroxyapatite, may have contributed to the increased mean μTBS after aging.[9,33] Considering the present results, mechanical load cycling had no deleterious effect on μTBS of the new universal adhesives except ABU; moreover, the bonding effectiveness of the new universal adhesives in self-etch mode is sufficient for clinical use. The results of this *in vitro* study can be supported by future clinical studies that may provide clear findings.

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Conflicts of interest

There are no conflicts of interest.

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