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Three different adhesive systems; three different bond strength test methods

DIGDEM EREN[1], ÖZDEN ÖZEL BEKTAŞ[1] & ŞEYDA HERGÜNER SISO[2]

[1]*Department of Restorative Dentistry, Faculty of Dentistry, Cumhuriyet University, Sivas, Turkey, and* [2]*Department of Restorative Dentistry, Faculty of Dentistry, Bezmialem University, İstanbul, Turkey*

Abstract

Objective. The aim of this study was to evaluate the efficacy of the microtensile, microshear and shear bond strength test methods to assess the bond strength of two self-etch adhesives and one etch&rinse adhesive on dentin. ***Materials and methods:*** Seventy-five extracted human molars were ground to expose their flat dentin surfaces and randomly assigned to one of three groups according to the type of test method (15 for microtensile, 15 for microshear, 45 for shear). Each of these groups was then assigned to three sub-groups according to the bonding systems (Clearfil SE Bond, Kuraray; G Bond, GC; Prime&Bond NT, Dentsply) used. Then, 15 specimens were prepared for each sub-group according to the test method employed ($n = 15$). After being stored in distilled water at 37°C for 24 h, the specimens were placed in a universal testing machine for three test methods and stressed at a crosshead speed of 0.5 mm/min. Mean bond strengths were analyzed using Kruskall-Wallis and Mann-Whitney U-tests at a significance level of $p < 0.05$. ***Results.*** The microtensile test had the highest bond strength ($p = 0.046$). Clearfil SE Bond and Prime&Bond NT produced significantly higher values than the G Bond in the microtensile bond test ($p < 0.05$), whereas no significant differences were found among the adhesives in the microshear bond test ($p > 0.05$). For the shear test, Clearfil SE Bond showed higher bond strength than Prime&Bond NT and G Bond ($p < 0.05$). ***Conclusion:*** Bond strength to dentin depends on the material and the test method used.

Key Words: *adhesive systems, microtensile bond strength, microshear bond strength, shear bond strength*

Introduction

Adhesive strength plays an important role in determining the clinical success of dental restorations [1,2]. Whereas long-term clinical trials are the ultimate test to evaluate the longetivity of dental restoration [3], they are difficult to perform because they take a long time. In addition, dental companies often introduce new adhesive systems prior to study completion [4]. Also, clinical trials cannot determine the true reason for failure given the simultaneous impact of diverse stresses on restorations within the aggressive oral cavity [3]. Therefore, laboratory bond strength tests are commonly used to compare adhesive systems. These tests can be readily used for qualitative comparisons between materials under similar test conditions [4,5].

In order to measure the bonding effectiveness of adhesives to enamel and dentin, diverse methodologies can be used [6]. These tests are categorized as tensile or shear bond strengths. In laboratory tests, bond strength is calculated as the initial mechanical load that generates the final fracture divided by the simple, geometrically defined, cross-sectional area of the bond [7]. Depending upon the size of the bond area, they are also categorized as macro-or micro tests [8].

Shear bond strength tests have been widely used, primarily because of their relative simplicity compared to tensile bond strength tests. In the latter it is difficult to align the specimen in the testing machine without creating deleterious stress distribution [9,10].

Nevertheless, the validity of expressing bond strength in terms of nominal stress has been questioned due to the heterogeneity of the stress distribution at the bonded interface [11–13]. Moreover, cohesive failure of both the composite and the dental substrate is a common occurrence, precluding an accurate assessment of the interfacial bond

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Correspondence: Özden Özel Bektaş, Cumhuriyet University, Faculty of Dentistry, Department of Restorative Dentistry, Sivas, Turkey.
Tel: +90346 2191300 2791. Fax: +90346 2191237. E-mail: ozdenozel@hotmail.com

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strength [4]. Therefore, microtensile and microshear test methods which have smaller test area dimensions have been developed in order to differentiate adhesives that produce higher bond strengths [14–16].

A microtensile bond strength methodology that uses small specimens with bond surfaces < 1 mm^2 was introduced by Sano et al. [14] in 1994. In the microtensile test, after the bonding procedure, additional specimen processing or actual preparation is required, rendering the test more laborious and technique-sensitive. However, a long list of advantages is typically ascribed to the conventional tensile test when compared to macro-bond-strength testing, including better economic use of teeth (with multiple microspecimens originating from one tooth), control of regional differences (e.g. peripheral vs central dentin) and distribution at the true interface (avoiding cohesive failure in tooth substrate or composite) [14,15].

The microshear test was developed by Shimada [16]. It combines the ease of manipulation with the ability to test several specimens per tooth. The very fine composite build-up (cylinder) with a typical diameter of 0.7 mm in combination with a relatively thick adhesive layer may, however, result in considerable bending and variable and non-uniform loading conditions [8,17]. According to numerous authors [16–19] who used this test method, it allows for the testing of small areas, thus permitting a regional mapping or depth profiling of different substrates. In addition, it involves preparing multiple specimens from the same tooth, as in microtensile tests, but without sectioning procedures [10].

Given that there is insufficient data in the literature comparing different methods, in order to assess the bond strength of adhesives to tooth structure, the aim of the present study was to evaluate different adhesive systems applied to dentin, using microtensile, microshear and shear strength tests. The null hypothesis was that the bond strength of different adhesive systems is independent of the test methods.

Materials and methods

Seventy-five non-carious human molars, which were extracted for orthodontic reasons, were used within 2 months of extraction. They were stored at 4°C in saline solution containing 0.1% thymol. In order to make handling easier during the sample of tooth preparation, a plastic ring mold was filled with an autopolymerizing resin. Further the root surface was embedded in the acrylic resin.

To prepare the dentin surfaces, the occlusal surface of third molar was removed with a slow-speed saw (Isomet; Buehler Ltd, Lake Bluff, IL), thereby exposing a flat mid-coronal dentin surface. The smear layer on the occlusal dentin was standardized using 600 grit silicon carbide paper. These samples were randomly assigned to one of three groups according to the type of test method: Specifically, 15 teeth were used for the microtensile bond strength test, 15 teeth were used for the microshear bond test and 45 teeth were used for the shear bond test. Then, each of these groups was assigned to three sub-groups according to the bonding systems [Prime&Bond NT (PB), Clearfil SE Bond (SE), and G Bond (GB)] used. Bonding and filling procedures recommended by the manufacturer were followed (Table I).

Microtensile test

After preparing the tooth surfaces, adhesive systems were applied according to the manufacturer's recommendations. Then, two increments of ~ 2 mm in thickness of resin composites were placed on the bonded surfaces. Each of the two resin composite increments were light cured, with a halogen curing

Table I. Materials used and restorative procedures completed.

Materials	Composition	Technique	Manufacturer
Clearfil SE Bond	*Primer*: 10- MDP, 2-HEMA hydrophilic dimethacrylate,dl Camphorquinone N,N-Diethanol-p-toluidine, Water *Bond*: MDP, Bis-GMA, HEMA, Hydrophobic dimethacrylate, dl-Camphorquinone, N,N-Dietanol-p toluidin, microfiller	*Primer*: Apply and allow to stand for 20 s in air gently *Bond*: Apply and gently air thin, light cure for 10 s	Kuraray, Japan
G Bond	4-methacryloxyethyltrimelletic acid (4-MET), phosphoric acid ester monomer, filler, acetone, water	Bond apply and allowed to remain for 10 sec, gently air dry for 5 sec, 10 sec light cure	GC, Japan
Prime&Bond NT	%35 phosphoric acid PENTA, UDMA, acetone, methacrylated resin monomer, fumed silica filler	Acid apply for 15 s, rinse for 15 s, gently air dry for 5 s Bond apply and allowed to remain for 5 s gently air-thin, 10 s light cure	Dentsplay-De Trey, Germany
Venus	Bis-GMA,TEGDMA, Ba Al F silicate glass, silicon dioxide	40 s light cure	Heraeus Kulzer, Germany

light (Hilux; Benlioglu Dental, Turkey) for 40 s. After being stored in distilled water at 37°C for 24 h, each tooth was vertically sectioned under copious water irrigation in a mesiodistal direction through the composite build up and dentin to produce a series of 0.7 mm thick slabs. They were then rotated 90° and serially sectioned again in a buccal-lingual direction to produce ~ 0.7 × 0.7 × 8 mm sticks. The teeth were sectioned using a low-speed diamond saw. A total of 15 specimen sticks were prepared from fiveteeth for each sub-group and individually tested for microtensile bond strength ($n = 15$).

Special flat stainless steel grips were made to fit a Bencor Multi-T (Danville Engineering Company, San Ramon, CA), which was held in a testing machine (Lloyd LF Plus, Ametek Inc, Lloyd Instruments, Leicester, UK). A small drop of a cyanoacrylate adhesive (Zapit, Dental Ventures of America, Corona, CA) was placed at the extreme ends of each specimen stick. Each stick was then carefully positioned on the device with slight pressure. Moreover, an accelerator (Zapit, DVA) was spread on either grip. Bonded sticks were subjected to microtensile testing at a crosshead speed of 0.5 mm/min until they fractured.

Microshear test

After applying the adhesive system to the dentin surface according to the manufacturer's recommendations, a small piece of polyethylene tubing with an internal diameter of 0.7 mm and height of 1 mm, was firmly placed on the uncured resin. Then, the adhesive resin was light cured according to the manufacturer's instructions. A composite resin was put into the bonded tube and light cured with the halogen curing light for 40 s. Three resin cylinders were bonded on each surface. In this manner a total of 15 specimens were prepared for each sub-group ($n = 15$). Next, the teeth were cut horizontally in order to obtain specimens that were ~ 1 mm thick. After being stored in 37°C water for 24 h the samples were attached to the testing device (Bencor Multi-T) with a cyanoacrylate adhesive, which was placed in a universal testing machine for shear bond testing. A thin wire with a diameter of 0.2 mm was looped around the resin cylinder, making contact with half of the cylinder base and held flush against the resin/tooth interface. Force was applied to each specimen at a crosshead speed of 0.5 mm/min until failure occurred.

Shear test

Forty-five teeth were randomly assigned to three subgroups ($n = 15$) according to the type of adhesive system. Adhesive systems were applied to each subgroup according to the manufacturer's instructions and polymerized. Next, the resin composite was built up on the dentin of each specimen by packing the material into a cylindrically shaped plastic apparatus with an internal diameter of 2.34 mm and a height of 3 mm (Ultradent Products, South Jordan, UT). Excess composite was carefully removed with an explorer and the specimens were light cured with halogen for 40 s. In this way, 15 specimens were prepared for each sub-group. All specimens were stored in a moisture medium at 37°C for 24 h. Bond strength was tested using a Universal Testing Machine (Instron, USA) with a notched blade attached to a compression load traveling at a crosshead speed of 0.5 mm/min. A force parallel to the tooth surface through the composite and tooth interface was applied.

Maximum loads at bond failure were recorded in Newtons (N) and bond strengths were calculated in megapascals (MPa). Mean bond strengths were analyzed using Kruskall-Wallis and Mann-Whitney U-tests at a significance level of $p < 0.05$.

Results

The mean values obtained in each experimental group are shown in Tables II and III. Comparison among tests showed significant differences ($p < 0.05$). Whereas the microtensile test had the highest bond strength, the shear test showed the lowest bond strength (see Table II).

When the microtensile test was used, Clearfil SE Bond and Prime&Bond showed significantly higher microtensile bond strength than that of the G Bond ($p = 0.044$ and $p = 0.042$, respectively). When the microshear test was used, no statistical differences were found among the adhesive systems ($p = 0.213$). When the results of the shear test were evaluated, Clearfil SE Bond showed a higher bond strength than Prime&Bond NT and G Bond ($p = 0.001$ and $p = 0.003$, respectively).

Discussion

The results of the current study revealed that bond strengths of adhesive systems were dependent

Table II. Mean and standard deviation of the microtensile, microshear and shear bond strength values for the pooled data of the groups test methods.

	Bond strength (Mean ± SD)
Microtensile	27.77 ± 5.86^a
Microshear	24.04 ± 5.40^b
Shear	17.40 ± 6.20^c
	KW = 82.06 $p = 0.001$ $p < 0.05$

Means with different letters are statistically significantly different at $p < 0.05$.
KW, Kruskal-Wallis test.

Table III. Mean and standard deviation of the microtensile, microshear and shear bond strengths values for three dental adhesives.

	Microtensile (Mean ± SD)	Microshear (Mean ± SD)	Shear (Mean ± SD)
Clearfil SE Bond	28.90 ± 5.32^{a}	25.42 ± 6.06^{c}	25.21 ± 10.19^{d}
G Bond	26.47 ± 8.03^{b}	21.73 ± 5.65^{c}	12.92 ± 4.64^{e}
Prime&Bond NT	28.46 ± 4.25^{a}	24.87 ± 4.54^{c}	14.30 ± 3.92^{e}
	KW = 6.17 $p = 0.046$ $p < 0.05$	KW = 3.08 $p = 0.213$ $p > 0.05$	KW = 17.56 $p = 0.001$ $p < 0.05$

Means with different letters are statistically significantly different at $p < 0.05$.
KW, Kruskal-Wallis test.

upon the test methods. Thus, the null hypothesis was rejected.

According to our test results, macro shear test values were lower than those found for both microshear and microtensile tests. This finding is consistent with those of previous researchers [14,20]. The macrotest with an adhesive interface ~ 7 mm^2 as encountered in shear and tensile tests delivered lower bond strength values than their equivalent microtest with an adhesive interface ~ 1 mm [3,21]. Sano et al. [14] explained that the small adhesive interface used in microtests contained fewer defects compared to larger specimens. Thus, microbond strengths tend to be much higher because the defect concentration is lower [1,4,14,15,22].

Considering the microtest results, microshear bond strengths showed lower values than microtensile bond strengths, which is consistent with the findings of another study [23]. The differences in the results of microbond strengths are thought to have arisen from the stress concentration at the adhesive interface [5]. The stress concentration is much more severe in specimens loaded in shear, compared to tension. Also, in the tensile test, stresses are more homogeneous [11].

If a bond strength test is capable of measuring higher values, an increase in the sensitivity of the test makes it capable of detecting subtle differences between groups [24]. In this study, the statistical differences between groups were detected with microtensile test results as opposed to the microshear test.

On the contrary, El Zohairy et al. [25], who compared the effect of microtest methods on bond strength to enamel, reported that the microshear test was capable of differentiating between strong and very strong adhesives. Also, Ishikawa et al. [26] evaluated the microtensile and microshear bond strength and found similar bond strength results in the case of dentin. However, in the case of enamel, they reported that the microtensile test detected no differences, although significant variations were found with the microshear bond strength test. Enamel is naturally brittle and fragile. As a result, it can easily crack, especially along the enamel prisms, which should be avoided. The trimming produced free enamel, which might have caused a reduction in bond strength during the microtensile test.

Not only the tooth substrate but also other factors, such as type of cavity, regional variations, type of resin tested [24] and material handling [27] might effect the bond strength test values. Therefore, all factors should be considered before choosing a bond test method.

In the microshear test, using such a small diameter tube made it difficult to pack the resin composite into the tube. In addition bubble inclusions and peripheral marginal gaps were often observed. This led to premature failure [22]. This may have affected the bond strengths.

El Zohairy et al. [25] claimed that a direct comparison between test methods is impractical due to the fundamental differences that exist between test methodologies such as direction of forces andcross-sectional area. It is important to evaluate the ability of each test to rank dental adhesives.

Taking the tests into account, they all ranked the adhesive systems similarly despite the lack of statistically significant differences in some groups. The microtensile bond test ranked the adhesives as follows: SE = PB > GB. The microshear bond test ranked as: SE = PB = GB. Finally, the shear bond strength test ranked them as follows: SE > PB = GB.

Preparation for adhesive treatment involves the superficial dissolution of dental hard substance by phosphoric acid (etch-and-rinse technique) or by acidic monomers (self-etching technique). In the etch-and-rinse technique, the adhesive is applied after the rinse step to impregnate the morphological features exposed by phosphoric acid with polymerizable monomers. In the self-etching technique, features exposed by the action of acidic monomers are simultaneously infiltrated with adhesive [28]. In this way, these systems offer a simpler clinical application than the etch-and-rinse technique [29]. In addition, the development of one-step self-etching resulted in more simplified systems. They combine the etchant, primer and adhesive in one bottle [30,31]. Self-etch adhesive systems can generally be classified as either a one-step (all-in-one) adhesive system or a two-step adhesive system, based on whether or not a bonding agent is applied [32].

Some authors suggest that etch-and-rinse adhesives have higher bond strength than self-etching adhesives [29,33]. On the contrary, Kiremitçi et al. [34] showed that self-etching adhesives provide higher bond strength than etch-and-rinse adhesives. In this study, according to the all the test methods' results, two-step self-etching adhesive Clearfil SE bond showed the highest bond strength values. The discrepancies between the results of these studies are due to the comparison of different branded adhesives.

A literature search was conducted for the years between 1998–2009 to identify research on the bond strength measurements of resin composite to dentin using four tests: shear, tensile, microshear and microtensile. Data from all six adhesives were pooled from four tests and their rankings were compared. The result of a previous study have shown that two-step self-etching adhesive Clearfil SE Bond always ranked among the top products [21]. Walter et al. [35] attributed the good bond strength values of Clearfil SE Bond to its ingredients. It contains the hydrophilic monomer MDP, which has been shown to have a significantly stronger chemical bond to hydroxyapatite.

Previous studies have shown that a two-step adhesive system exhibited relatively higher bonding performance of resin to tooth than a one-step self-etching adhesive system [36–38]. Correspondingly, in this study, the one-step self-etch adhesive (GB) showed a lower bond strength than the two-step self-etch adhesive (SE) in all test methods evaluated. Also, the results of Okado et al. [39] and Burrow et al. [40], in which they evaluate the same trademarks of adhesive systems, were in agreement with findings of the present study. G-Bond is a HEMA-free adhesive resin. In a recent study, phase separation among adhesive compositions was confirmed as droplets entrapped during solvent evaporation from HEMA-free adhesives. This phenomenon could be explained by the evaporation of solvents such as ethanol and acetone, which affected the balance of solvents and resin monomer and caused water to separate from other compositions of the adhesive [41]. Spherical blisters within the resin film may be the outcome of residual, free water, not completely evaporated and entrapped at the interfacial level. The convergence of small blisters into larger ones tends to produce honeycomb structures that may jeopardize the bonded interface [42].

Within the limitations of this study, it can be concluded that bond strength to dentin depends on the material and the test method used. On the other hand, all bond strength tests showed that the two-step self-etch adhesive produced higher bond strengths to dentin than the one-step self-etch adhesive. Nevertheless, additional clinical studies are needed to further evaluate the efficacy of all systems.

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