Original Article

Strength and Durability of a New Adhesive Bond to Superficial Dentin Using Etch and Rinse and Self-Etch Systems: An In Vitro Study

S. Manafi [1], A. Eskandari zadeh [2], N. Shademan [1], M. Mofidi [1], M. Norozy [3].

[1] Assistant Professor, Department of Operative Dentistry, School of Dentistry, Kerman University of Medical Sciences, Kerman, Iran
[2] Associated Professor, Department of Operative Dentistry, School of Dentistry, Kerman University of Medical Sciences, Kerman, Iran
[3] DDS, MS, Specialist in Aesthetic and Operative Dentistry, School of Dentistry, Qom University of Medical Sciences, Qom, Iran

Abstract

Background and Aim: Data regarding the efficacy of universal adhesives are limited. This study aimed to assess the durability of a universal adhesive bond to superficial dentin using etch and rinse and self-etch modes.

Materials and Methods: In this in vitro, experimental study, 32 sound molars were randomly divided into two groups of Scotchbond Universal adhesive with two-step etch and rinse mode and Scotchbond Universal adhesive with one one-step self-etch mode as recommended by the manufacturer. Coronal part of teeth was restored with composite. The teeth were mounted in acrylic blocks and prepared according to the trimming protocol. The specimens were then randomly divided into two groups of immediate and 5000 thermal cycles and were then subjected to tensile load at a crosshead speed of 1mm/minute. Data were analyzed using two-way ANOVA.

Results: The interaction effect of the two independent variables on microtensile bond strength was not significant (P=0.957). Bond strength in etch and rinse group was significantly higher than that of self-etch group (P<0.001). Bond strength in thermocycling subgroups was significantly higher than that of immediate subgroups (P=0.034).

Conclusion: Etch and rinse mode was superior to self-etch mode for use of Scotchbond Universal, and thermocycling increased the bond strength of this adhesive.

Key Words: Dentin, Universal Adhesive, 10-MDP, Aging, Tensile Strength

Corresponding author:
M. Norozy, DDS, MS, Specialist in Aesthetic and Operative Dentistry, School of Dentistry, Qom University of Medical Sciences, Qom, Iran

M_Norozi@kmu.ac.ir

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Introduction

In contemporary dentistry, use of composite resins is increasing such that in some countries, composite resins have completely replaced mercury-containing amalgam [1]. Since composite resins bond to tooth structure, their main cause of failure is loss of retention and marginal misfit [2]. Bond to enamel is based on micromechanical interlocking of resin and etched enamel, which is reliable and durable. Bond to dentin is more complex due to structural properties such as high water content, presence of tubules, positive pressure of fluid and high organic content [3]. Adhesives used for direct composite restorations include three-step and two-step etch and rinse systems and one-step and two-step self-etch adhesives [4,5]. For etch and rinse adhesives, mismatch between the demineralization depth and resin penetration depth may leave some nanometer-scale three-dimensional canals allowing microleakage and nano-leakage. Nano-leakage of water may cause softening of adhesive resins and hydrolysis of exposed collagen in the hybrid layer [6]. It has been shown that collagen hydrolysis extensively compromises the long-term bond strength of etch and rinse adhesive systems [7].

In self-etch adhesives, depth of dentin demineralization and penetration depth of resin monomers may not match, which is due to the simultaneous function of acidic monomers in these adhesives and their penetration to dentin. Most one-step self-etch adhesives include ionic monomers with phosphate acidic or carboxylic groups, hydrophilic monomers, water and organic solvent in one bottle. Thus, these adhesives are highly hydrophilic and have higher water sorption. Water sorption by adhesive polymers causes swelling of polymer, its softening and weakening of polymer network. Water sorption by these adhesives enhances degradation of adhesive-dentin interface after long-term water storage [6]. Evidence shows that following water sorption, water tree and voids form and phase separation occurs at the interface in long-term use of single bottle self-etch adhesives (due to their high water content) causing bond degradation [7].

Considering the controversy in professional judgments of clinicians regarding the best adhesive strategy and its steps, some manufacturers developed one bottle adhesives, which enable etch and rinse and self-etch applications. These adhesives are named universal, multi-purpose or multi-mode [8]. Scotchbond universal adhesive by 3M is among these adhesives and the manufacturer claims that it can be applied on dentin in self-etch or etch and rinse mode. Its chemical formulation includes water, alcohol solvent, HEMA, Vitrebond copolymer, MDP acidic monomer and silane. Thus, it yields high durable bond strength in both modes. It has been stated that hybrid layer in use of phosphoric acid is thicker than that in self-etch mode. Based on the advantages and limitations mentioned for etch and rinse and self-etch adhesives, this study aimed to assess the strength and durability of a new adhesive to superficial dentin in etch and rinse and self-etch modes.

Materials and Methods

This in vitro experimental study was conducted on 32 surgically extracted impacted human third molars. The teeth were not carious and were immersed in 0.2% thymol solution at 4°C for less than one month. Occlusal enamel was cut using a diamond disc (Top Dent, Gothenburg, Switzerland) and low speed hand piece under water coolant parallel to the occlusal surface such that a smooth dentin surface was obtained. Exposed occlusal dentin was polished with a 600-grit silicon carbide paper measuring $25cm^2$and moistened with water. Each tooth was polished for 60 seconds [4,9] with circular motion to obtain a thin standard smear layer [10,11]. The teeth were randomly divided into two groups of 16.

Group 1: Scotchbond Universal adhesive (3M ESPE, St. Paul, MN, USA) in two-step etch and rinse mode

Group 2: Scotchbond Universal adhesive (3M ESPE, St. Paul, MN, USA) in one-step self-etch mode

In group 1, dentin surface was etched with 34% phosphoric acid (Scotch Universal etchant) for 15 seconds according to the manufacturer's instructions and was then rinsed by water spray for 10 seconds and dried with cotton pellet such that a moist shiny dentin surface was obtained. One layer of adhesive was then applied by a micro-brush on moist dentin and rubbed for 20 seconds. It was then air sprayed gently for 5 seconds to evaporate the solvent followed by 10 seconds of light curing (Elipar, 3M ESPE, St. Paul, MN, USA) with a light intensity of $600mW/cm^2$. The output light was constantly controlled by a radiometer. In group 2, one layer of Scotchbond adhesive was applied by a micro-brush on dentin surface for 5 seconds and rubbed for 20 seconds followed by gentle air spray for 5 seconds and 10 seconds of light curing [5].

Coronal part of teeth in both groups was restored with three layers of nano-hybrid composite with B1 shade (Filtek Z250 XT, 3M ESPE, St. Paul, MN, USA). Composite was applied in 2mm thick increments using a Williams probe and each layer was cured for 40 seconds. The etch and rinse and self-etch groups (n=16) were randomly divided into two subgroups (n=8) for immediate testing after 24 hours of storage and aging by thermocycling. The teeth were then mounted in clear cold-cureacrylic blocks (Acropars, Tehran, Iran) such that they were completely embedded in acrylic resin. Acrylic blocks were transferred to CNC cutting machine (Nemo Phanavaran Pars, Mashhad, Iran) and sectioned in mesiodistal direction with 1mm thickness using a low speed diamond saw under water coolant. Also, the device was digitally programmed to apply a vertical section in buccolingual direction as well and thus, specimens with a cross sectional area of 1×5 mm^2 with 12 mm length were obtained (Figure 1). In each group, about 6±1 specimens were obtained of each third molar tooth. According to the trimming protocol, specimens were prepared in the form of hourglass measuring 0.8±0.2 $mm^2$with 008 diamond bur.

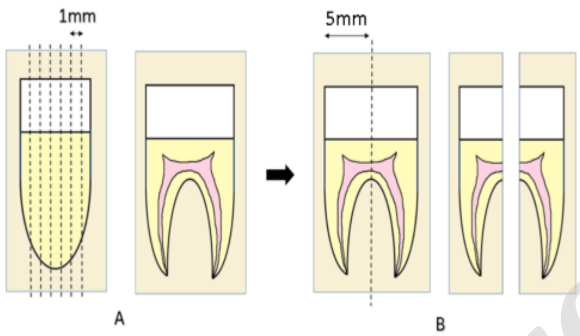

Figure 1. Schematic view of making sticks of teeth with composite core in transparent acrylic resin

- **A.** Sections with 1mm thickness from mesial to distal and its buccal view
- **B.** Perpendicular section and obtaining sticks with a cross sectional area of 1×5 mm^2 with approximate length of 12mm

Specimens prepared of eight teeth in etch and rinse group and eight teeth in self-etch group were immersed in distilled water and incubated at 37°C for 24 hours. This group served as the control, no thermocycling group, which was immediately subjected to microtensile bond strength test. The thermocycling group (n=8) was subjected to 5000 thermal cycles between 5-55°C [2] with 20 seconds of dwell time and 20 seconds of transfer time [12]. The bonding interface area in each specimen was measured by a digital caliper with 0.01 mm accuracy and reported in square-millimeters. Load at fracture was recorded in Newtons (N) and divided by the bonding surface area in square-millimeters to obtain microtensile bond strength in megapascals (MPa).

Each specimen was then attached to the jig of microtensile tester (Bisco, Schaumburg, IL, USA) by cyanoacrylate glue and microtensile load was applied at a crosshead speed of 1mm/minute [3,5,6,9]. Mode of failure was determined by two observers under a stereomicroscope at×40 magnification. Mode of failure was determined to be adhesive (at the dentin-adhesive or adhesive-composite interface), cohesive (in composite or dentin) and mixed. Two-way ANOVA was applied to assess the effect of self-etch or etch and rinse mode and different storage times on bond strength.

Results

Specimens with preload failure were subjected to statistical analysis with minimum bond strength. Table 1 shows descriptive statistics of the mean microtensile bond strength and standard deviation in different groups. As shown in Table 1, Scotchbond universal application mode (etch and rinse and self-etch) significantly affected the microtensile bond strength (P<0.001). Scotchbond universal in etch and rinse mode yielded significantly higher microtensile bond strength than self-etch mode.

A significant difference was noted between the immediate and aged groups in terms of microtensile bond strength (P=0.034). It means that in both groups, thermocycling increased the bond strength. Two-way ANOVA showed that the interaction effect of the two factors on bond strength was not significant (P=0.957). In other words, the effect of mode of adhesive application on the mean bond strength was not affected by duration of storage of samples. The frequency distribution of modes of failure for etch and rinse modein immediate and aged groups was 47% and 45% mixed failure and 59% and 67% adhesive failure for immediate and aged self-etch groups, respectively.

It should be noted that percentage of preload failure in etch and rinse mode of group 1 with immediate testing was about 13%. In aged group, this rate was 7%. In group 2 or self-etch group, this rate was 58% in immediate and 48% in aged

Table 1. Mean and standard deviation of microtensile bond strength in groups based on two-way ANOVA (Newtons)

Group/Testing time	Immediate	Aged
Etch and rinse	36.9817±16.8674 ^{A*} (n=52)	41.6791±16.4110 ^{Ab} (n=43)
Self-etch	16.9674±13.1177 ^{Ba} (n=53)	21.9085±17.0902 ^{Bb} (n=54)

*** Mean values with dissimilar letters in columns and rows indicate statistically significant differences**

subgroup. Considering the preload failure of about half of the specimens in both immediate and aged subgroups in self-etch mode, eight other samples were prepared with Scotchbond Universal in self-etch mode prior to microtensile test as described in the materials and methods section. The results showed 53% failure prior to loading in this new group (self-etch), which was in line with the results of primary testing.

Discussion

Based on the results, application of Scotchbond universal in etch and rinse mode showed significantly higher microtensile bond strength compared to the self-etch mode; based on this finding, the primary hypothesis was refuted.

Multi-mode or universal adhesives revolutionized adhesive dentistry. They are simplified adhesives supplied in one bottle. According to the manufacturer, they can be used in etch and rinse and self-etch modes [13]. Self-etch adhesives, depending on the pH (mild, moderate, strong) are different in penetration depth, morphology of dentin-adhesive interface and bond strength [10,14,15]. Shallower etching pattern and less micromechanical retention are among the common concerns regarding the use of mild self-etch adhesives [16]. Evidence shows that single-step self-etch adhesives have lower efficacy than two-step self-etch adhesives and etch and rinse systems due to the following:

1. They provide a very thin coat, a great part of which may be comprised of oxygen inhibited layer and thus, adhesive layer may have low polymerization.

These adhesives are very susceptible to phase separation due to evaporation of solvent and after polymerization, they serve as a permeable barrier [17]. The adhesive used in our study was Scotchbond Universal, which according to the manufacturer, has a pH of 2.7, which is classified as a self-etch one-step adhesive with ultra-mild acidity. In our study, Scotchbond Universal was rubbed on dentin for 20 seconds as recommended by the manufacturer similar to previous studies [4,9]. To standardize the specimens, adhesive was applied in one step in adequate amount to cover the entire dentin surface in coronal third of third molars. Based on the pH of this adhesive and the results of bond strength test, it may be assumed that application of a single layer of adhesive with ultra-mild acidity results in incomplete penetration of acidic monomers into the smear layer and its partial dissolution and consequent minimal penetration into dentin beneath the smear layer [12].

Perdiago et al. [9] and Munoz et al. [4] found no significant difference in immediate bond strength of Scotchbond Universal with self-etch and etch and rinse modes. Difference between their results and ours may be due to the possible difference in the amount of adhesive applied in self-etch mode (more than one layer). Addition of adhesive on the material buffered by the smear layer compensates for the insufficient acidity of monomers for penetration into the smear layer and underlying dentin. Additionally, by an increase in thickness of the protective layer created by the single-step adhesive, polymerization is enhanced.

Taschner et al, [18] in 2012 evaluated the effect of additional etching on bond strength of one step self-etch adhesives. The results showed that Adper Easy Bond (3M ESPE) with a pH of 2.7 applied after phosphoric acid etching of dentin for 15 secondsyielded higher bond strength after 24 hours and six months of water storage compared to the no phosphoric acid etching group. The same group of researchers in another study [19] suggested phosphoric acid etching prior to the use of self-etch one-step adhesives with ultra-mild acidity. It should be noted that many studies have questioned the efficacy of simple one-step self-etch adhesives such as Scotchbond Universal due to their complex formulation and high amount of solvent as well as incomplete solvent evaporation [13].

In our study, the immediate microtensile bond strength of Scotchbond Universal in etch and rinse and self-etch mode had a significant difference with that after thermocycling; which means that bond strength increased by thermocycling and the second hypothesis was accepted. In our study, bond strength in thermocycling subgroups was significantly higher than that of immediate subgroups, which means that our second hypothesis was accepted. This finding was in line with that of previous studies [2,5,11,20].

Marchesiet al, [5] in 2013 reported that immediate bond strength of Scotchbond Universal in etch and rinse mode (dry and moist) and also in self-etch mode was significantly lower than its delayed bond strength after six months of water storage. The difference between their results and ours may be due to different methods of aging. Wagner et al, [21] in 2014 assessedthe durability of All-Bond Universal adhesive by Bisco. According to the manufacturer, it contains 10-MDP. They concluded that 5000 thermal cycles could not decrease the bond strength of adhesive. Inoue et al, [22] in 2005 concluded that microtensile bond strength of adhesives containing 10-MDP does not decrease significantly even after 100,000 thermal cycles.

Based on these results, it may be concluded that thermal cycles more than 10,000 [22] or water storage longer than six months [5] are required to degrade the interface of adhesives containing 10-MDP such as Scotchbond Universal. This explains why no reduction in bond strength was noted in our study after 5000 thermal cycles in the two modes.

Regarding no reduction in bond strength and even its increase after aging by thermocycling, it should be mentioned that the higher the partition coefficient measured by log P value in an adhesive, the more hydrophobic the adhesive would be. The log P value of Bis-GMA is 5.1 and that of MDP, 4-MET and HEMA is 4.0, 1.7 and 0.26, respectively [23]. This explains lower water sorption by Scotchbond universal, containing 10-MDP in thermocycling process. Also, Scotchbond Universal due to the presence of MDP in its formulation can chemically bond to dentin such that an effective chemical bond is formed between MDP and hydroxyapatite remaining around collagen fibrils. It increases the mechanical strength of the adhesive interface, and the MDP-ca salt in hybrid layer increases the durability of bond [4,5].

In a review study by Liu et al, [24] on limitations of bond to dentin and strategies for prevention of bond degradation, it was stated that hydrolysis of hydrophilic resin components, which have not been adequately polymerized is one reason for bond degradation. No reduction in bond strength and even its increase after thermocycling can be explained by continuation of thermal polymerization of incompletely polymerized resin components.

High preload failure rate in self-etch group compared to etch and rinse mode in our study may explain the bond strength test results as well. Regarding the mode of failure, the results showed that etch and rinse group in both immediate and aged subgroups had the highest frequency of mixed failure (about 50%). In self-etch group, both immediate and aged subgroups showed the highest frequency of adhesive failure (over 50%). Based on the bond strength results, failure mode in both etch and rinse and self-etch groups was expected.

Conclusion

Based on the results of this study showing significantly higher bond strength of Scotchbond Universal in etch and rinse mode compared to its self-etch mode, it may be concluded that etch and rinse mode is superior to self-etch mode for use of Scotchbond Universal.

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