

Microtensile Bond Strength of Composite to Enamel Using Universal Adhesive with/without Acid Etching Compared To Etch and Rinse and Self-Etch Bonding Agents

Hoda Pouyanfar¹, Elaheh Seyed Tabaii², Samaneh Aghazadeh³, Seyyed Pedram Tabatabaei Navaei Nobari⁴, Mohammad Moslem Imani^{5*}

¹Department of Restorative Dentistry, Dental Faculty, Kermanshah University of Medical Sciences, Kermanshah, Iran;
²Orthodontic Department, Qom University of Medical Sciences, Qom, Iran; ³Private Practice, Tehran, Iran; ⁴Students Research Committee, Kermanshah University of Medical Sciences, Kermanshah, Iran; ⁵Department of Orthodontics, Kermanshah University of Medical Sciences, Kermanshah, Iran

Abstract

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***Correspondence:** Mohammad Moslem Imani. Department of Orthodontics, Kermanshah University of Medical Sciences, Kermanshah, Iran. E-mail: mmoslem.imani@yahoo.com

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AIM: Considering the recent introduction of universal adhesives and the controversy regarding the use/no use of etchant prior to their application, this study sought to assess the microtensile bond strength of composite to enamel using universal adhesive with/without acid etching compared to three-step etch and rinse, two-step etch and rinse and two-step self-etch bonding agents.

METHODS: This in vitro, an experimental study was conducted on 80 extracted sound human molars in five groups (16 each): Scotchbond Universal adhesive (3M) with/without prior etching, Adper Scotchbond Multi-Purpose, Single Bond and Clearfil SE Bond. Etching was performed with 37% phosphoric acid for 20 seconds followed by rinsing and drying. The bonding agent was then applied and light-cured. The e-lite composite was bonded to surfaces and light-cured. The teeth were then mounted, sectioned and subjected to microtensile bond strength test in a universal testing machine. The mode of failure was, determined under a stereomicroscope. Data were analysed using one-way ANOVA followed by Tukey's test.

RESULTS: Universal adhesive with prior etching yielded the highest bond strength ($P = 0.03$). Pairwise comparisons showed that the bond strength of this group was significantly higher than that of universal adhesive without prior etching ($P = 0.04$). No other significant differences were noted ($P > 0.05$). The modes of failure were significantly different among the groups ($P = 0.003$).

CONCLUSION: Enamel etching with phosphoric acid can significantly increase the bond strength to universal adhesive. Universal adhesive without prior etching provided the bond strength as high as that provided by etching and rinse and self-etch bonding agents.

Introduction

At present, dental adhesive systems are highly popular among dental clinicians due to their ability to bond to enamel and dentin and enabling conservative cavity preparation. The composition of dental adhesive systems undergoes constant development, and new products are continuously introduced to the market [1] [2]. Bonding to enamel is easier, stronger and more durable than that to dentin due to higher mineral content and lower water content

of the enamel compared to dentin [3]. Achieving a strong and durable bonding between the restoration and tooth structure is a prerequisite for a successful restoration [3]. On the other hand, bond failure at the tooth-restoration interface causes marginal discoloration, postoperative tooth hypersensitivity, secondary caries and pulp involvement [4] [5]. The mechanism of bonding of dental adhesive systems to the dental substrate is mainly based on the replacement of the lost minerals with resin monomers, resulting in a micromechanical interlocking of polymer in dental substrate [6]. Bonding agents mediate the

bonding of composite to enamel and dentin. Resin bond to enamel is durable and predictable and is based on penetration of resin monomers into microscopic porosities of the enamel surface caused by acid etching and subsequent formation of resin tags [7]. To increase the bond strength to the dental substrate, some modifications have been made in dental adhesives regarding their chemical composition, mechanism of bonding, number of bottles (treatment steps) and their application technique, which affect their clinical efficacy. As a result, several generations of bonding agents are now available in the dental market. The two-step etches and rinses bonding agents include two steps of acid etching and application of bonding agent. In this generation of bonding agents, the primer and bonding agent are supplied together in one bottle [8]. Demand for simplification of adhesive application resulted in the development of self-etch adhesives. The two-step self-etch adhesives do not require a separate etching step and contain advanced acidic monomers such as 4-META and 10-MDP, which confer further hydrophilicity to these adhesives compared to previous generations.

To ensure the etching capability of monomers, water is also present as an ionising agent in the composition of self-etch adhesives [9]. In two-step, self-etch adhesives, etching and priming of dentin and enamel are performed simultaneously (due to the use of acidic primers). These systems have greatly simplified the adhesive application process since they do not require rinsing and drying. As a result, the risk of over-drying or excessive moisture is no longer present [8] [10] [11]. The major superiority of the 7th generation dentin bonding agents over the 6th generation is their single-step application. These bonding agents have one-step application to minimise technical sensitivity, further facilitate the bonding process and prevent the problems related to incomplete penetration of resin tags into the porosities created by etching. As a result, these systems are extremely easy to use and save time [10].

Universal adhesives are used in dentistry in self-etch, etch and rinse or selective etching modes. They can bond to methacrylate-based restorative materials, cement, sealants, dentin, enamel, glass-ionomer and indirect restoration substrates including metals, alumina, zirconia and other ceramics. They all contain acidic monomers and have a similar application pattern as that of self-etch systems. On the other hand, nano-adhesives contain nano-fillers and can form a very strong bond to dentin and enamel. Bonding systems containing nano-fillers are known as universal systems and are applied in self-etch mode. Some studies have compared different generations of bonding agents and have reported controversial results [12] [13] [14]. The quality of bonding of coronal restoration to tooth structure depends on the type of bonding agent used.

Considering the recent introduction of universal adhesives and the controversy regarding the use/no use of etchant prior to their application with regard to their bond strength, this study aimed to assess the microtensile bond strength of enamel to composite following the application of a universal adhesive with/without prior acid etching, a three-step etch and rinse bonding agent, a two-step etch and rinse bonding agent and a two-step self-etch bonding agent.

Material and Methods

This in vitro, an experimental study was performed on 80 extracted sound human molars. The study was approved in the Ethics Committee of Kermanshah University of Medical Sciences (Code: KUMS.REC.1395.426). Minimum sample size was calculated to be 16 in each of the five groups according to a study by Joseph et al. [15], assuming the mean bond strength of 3.35 ± 1.58 MPa for two-step self-etch adhesive and 34.93 ± 2.54 MPa for universal adhesive with 95% confidence interval and 90% study power using PASS version 21.0 software (a total of 80 samples). A total of 80 permanent sound molars extracted within the past 6 months for reasons unrelated to this study were selected using convenience sampling. The teeth were immersed in 0.5% chloramine T solution at room temperature for one week for disinfection. The teeth were then stored in distilled water at room temperature until the experiment [16]. The teeth were inspected under a light microscope to ensure the absence of caries and cracks. The teeth were randomly divided into five groups ($n = 16$) as follows:

Group 1: The buccal/labial enamel surface was etched with 37% phosphoric acid gel (Ultradent, South Jordan, UT, USA) for 20 seconds followed by 10 seconds of rinsing with water spray and 5 seconds of drying with air spray. Adper Scotchbond Multi-Purpose (3M ESPE, St. Paul, MN, USA) primer was applied on the surface, and after gentle air spray, Adper Scotchbond Multi-Purpose bonding agent was applied. Light curing was performed using a LED light curing unit (Demetron, Kerr, Orange, CA, USA) with a light intensity of 700 mW/cm^2 . Before curing, the output power of light curing unit was calibrated using a radiometer. Elite AA composite (Bisco Dental Products, Schaumburg, IL, USA) was then applied in two 2.5 mm-thick increments, and each layer was polymerized for 20 seconds. A mould measuring $9 \times 9 \times 5$ mm was used to standardize the composite thickness in this group.

Group 2: Enamel surface was etched, rinsed and dried as explained in group 1. Single Bond (3M ESPE, St. Paul, MN, USA) bonding agent was then applied in two layers and cured using a LED light

curing unit. Elite AA composite (Bisco Dental Products, Schaumburg, IL, USA) was then applied in two 2.5 mm-thick increments, and each layer was polymerized for 20 seconds. A mould measuring 9 x 9 x 5 mm was used to standardise the composite thickness.

Group 3: Two layers of Clearfil SE Bond primer were first applied on the enamel surface and dried with gentle air spray for 5 seconds. One layer of Clearfil SE Bond bonding agent was then applied on the surface and cured using a LED light curing unit. Elite AA composite (Bisco Dental Products, Schaumburg, IL, USA) was applied in two 2.5 mm-thick increments, and each layer was polymerised for 20 seconds. A mould measuring 9 x 9 x 5 mm was used to standardise the composite thickness.

Group 4: Scotchbond Universal adhesive (3M ESPE, St. Paul, MN, USA) was applied on the surface of samples without etching and cured using a LED light curing unit. Elite AA composite (Bisco Dental Products, Schaumburg, IL, USA) was applied in two 2.5 mm-thick increments, and each layer was polymerised for 20 seconds. A mould measuring 9 x 9 x 5 mm was used to standardise the composite thickness.

Group 5: The enamel surface was first etched with 37% phosphoric acid gel, and then Scotchbond Universal adhesive (3M ESPE, St. Paul, MN, USA) was applied and cured using a LED light curing unit. Elite AA composite (Bisco Dental Products, Schaumburg, IL, USA) was applied in two 2.5 mm-thick increments, and each layer was polymerised for 20 seconds. A mould measuring 9 x 9 x 5 mm was used to standardise the composite thickness.

Table 1 shows the composition of adhesives used in this study. The teeth in each group were mounted in acrylic resin perpendicular to the horizon and parallel to each other and composite was subsequently bonded to them as described earlier. After immersion in distilled water at room temperature for 24 hours, the teeth were sectioned into slices measuring 1 x 1 mm with 8 mm height by a low-speed diamond disc of a microtome (Isomet, Buehler Ltd., Bluff, IL, USA) such that the disc was perpendicular to the tooth surface. The sections of each group were separately immersed in distilled water and incubated at 37°C and 100% humidity for 24 hours.

Table 1: Composition of adhesives used in this study

Adhesive	Composition
Clearfil SE Bond (Kuraray, Tokyo, Japan)	Primer: water, MDP, HEMA, CQ, DET, hydrophilic DMA Bond: MDP, bis-GMA, HEMA, hydrophobic DMA, CQ, DET, silanated colloidal silica
Adper Scotchbond Multi-Purpose (3M ESPE, St. Paul, MN, USA)	10-MDP methacrylate resin, HEMA, Ethanol, Water, Polyacrylic Acid Copolymer, Silane, Fillers, Initiators
Single Bond Universal Adhesive (3M ESPE, St. Paul, MN, USA)	Adhesive: MDP phosphate monomer, methacrylate resins, HEMA, silane methacrylate-modified polyalkenoic acid copolymer, filler, ethanol, water, initiators
Scotchbond Universal adhesive 3M	MDP, bis-GMA, HEMA, ethanol, water, initiators
MDPB: 12-methacryloyloxydodecylpyridinium bromide; bis-GMA: bisphenol A diglycidylmethacrylate; HEMA: 2-hydroxyethyl methacrylate; MDP: 10-methacryloyloxydecyl; dihydrogen phosphate; DMA: dimethacrylate; DET: N,N-diethanol p-toluidine; CQ: camphorquinone.	

For measurement of microtensile bond strength, each sample was fixed to the plate of a universal testing machine (Z020, Zwick GmbH & Co. KG, Germany) using cyanoacrylate glue. The load was applied at a crosshead speed of 0.5 mm/minute until failure. The load at failure was recorded in Newtons (N).

The interface area of the broken piece was measured by a gauge. The load at failure in Newtons was divided by the enamel/composite interface area in square millimetres (mm²) to obtain the bond strength in megapascals (MPa).

The samples were then inspected under a stereomicroscope (Olympus, SZX9, Iran) at (x 10) magnification to determine the mode of failure. The mode of failure was classified into three groups of cohesive within the tooth structure, cohesive within the composite and mixed (Figure 1).

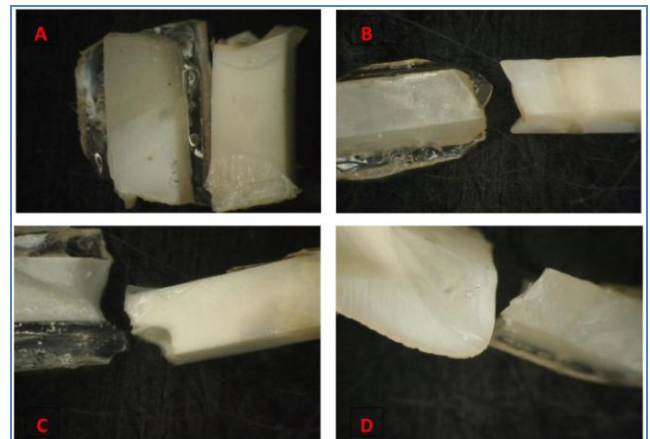


Figure 1: A) Cohesive failure in tooth structure in a universal adhesive group with an etchant; B) cohesive failure of the composite in a universal adhesive group with an etchant; C) cohesive failure of the composite in the universal adhesive group without etchant; D) cohesive failure in tooth structure in the universal adhesive group without etchant

Data were analysed using SPSS version 21 (SPSS Inc., IL, USA). Normal distribution of data was assessed using the Shapiro-Wilk test. Homogeneity of variances was assessed using the Levene's test. One-way ANOVA was applied to assess the difference in microtensile bond strength of the groups. Pairwise comparisons were carried out using Tukey's test. The modes of failure were compared among the groups using the chi-square test. P < 0.05 was considered significant.

Results

According to the Shapiro-Wilk test, the microtensile bond strength data were normally distributed (P > 0.05). The assumption of homogeneity

of variances according to the Levene's test was met ($P = 0.17$).

Maximum microtensile bond strength was obtained by the use of universal adhesive after etching while the minimum microtensile bond strength was noted in the use of universal adhesive without etchant (Table 2). According to one-way ANOVA, the difference in microtensile bond strength of the groups was significant ($P = 0.03$).

Table 2: Mean microtensile bond strength in the five groups (n=16)

Bonding agent	Mean	Standard deviation	Minimum	Maximum	Median
Scotchbond Universal 3M with etchant	65.75b	32.13	20.8	134.8	60.1
Adper Scotchbond 3M	45.81ab	19.82	15.07	84.3	41.85
Single Bond 3M	45.52ab	21.84	10.2	79.5	45.45
Clearfil SE Bond	44.91ab	15.92	23.3	76.7	46.65
Scotchbond Universal 3M without etchant	42.75a	19.79	11.16	80.0	38.6

Mean values with lowercase letters indicate no significant difference in pairwise comparisons.

Thus, Tukey's test was applied for pairwise comparisons (Table 3) and showed that only the difference in microtensile bond strength of universal adhesive with the etchant and universal adhesive without etchant was significant ($P = 0.04$). No other significant differences were noted in pairwise comparisons ($P > 0.05$).

Table 3: Pairwise comparison of groups regarding microtensile bond strength

First bonding agent	Second bonding agent	Mean difference	P-value
Single Bond 3M	Clearfil SE Bond	0.61	1.0
	Universal adhesive with an etchant	20.23	0.09
	Universal adhesive without etchant	2.77	0.99
Clearfil SE Bond	Adper Scotchbond	0.29	1.0
	Universal adhesive with an etchant	20.84	0.07
	Universal adhesive without etchant	2.16	0.99
Universal adhesive with an etchant	Adper Scotchbond	0.91	1.0
	Universal adhesive without etchant	22.99	0.04
	Adper Scotchbond	19.93	0.09
Universal adhesive without etchant	Adper Scotchbond	3.06	0.99

According to the chi-square test, the difference in modes of failure among the groups was significant ($P = 0.003$) such that cohesive failure within the composite had a significantly higher frequency in Clearfil SE Bond and universal adhesive without etchant groups while cohesive failure in tooth had a higher frequency in universal adhesive with etchant and Scotchbond groups (Table 4).

Table 4: Frequency of the modes of failure in the groups

Mode of failure	Universal adhesive with an etchant	Scotchbond	Single Bond	Clearfil SE Bond	Universal adhesive without etchant
In tooth	9 (56.3%)	9 (56.3%)	8 (50.0%)	0	4 (25.0%)
In composite	7 (43.8%)	7 (43.8%)	8 (50.0%)	16 (100%)	12 (75.0%)

The frequency of fractures in teeth and composite was the same in the Single Bond group (Figure 2).

Discussion

This study assessed the microtensile bond strength of enamel to composite following the use of a universal adhesive with/without prior etching, three-step etch and rinse (Scotchbond), a two-step etch and rinse (Single Bond) and two-step self-etch (Clearfil SE Bond) bonding agents. The results showed that the microtensile bond strength was significantly higher in the universal adhesive with prior etching group (65.75 MPa).

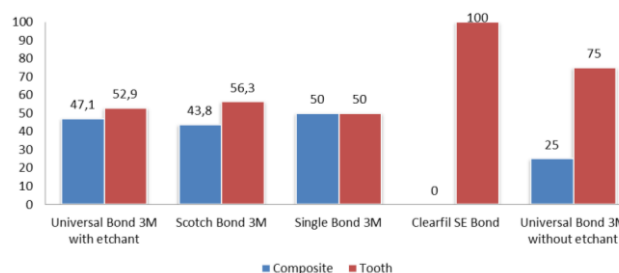


Figure 2: Frequency percentage of different modes of failure in the five groups

Pairwise comparisons showed that this group had a significant difference with universal adhesive without prior etching group. No other significant differences were noted in the bond strength among the groups. The results showed that enamel etching with phosphoric acid before the application of universal adhesive significantly increased the bond strength of enamel to composite (compared to other groups), which indicates that acid etching before the application of different bonding agents is still an acceptable method to increase the bond strength of enamel to composite. On the other hand, a universal adhesive with no prior enamel etching yielded the bond strength as high as that provided by three-step etch and rinse, a two-step etches and rinse and two-step self-etch systems. This indicates that despite the elimination of the etching step, universal adhesive yielded the bond strength as high as that of other systems.

Acid etching is a commonly used method to roughen the enamel surface and increase the bond strength of adhesive materials to enamel. Significantly higher bond strength in the universal adhesive with prior acid etching group is probably due to the formation of porosities in the enamel surface and penetration of resin into the porosities. Evidence shows that resin tags can be as long as 15-20 μ at the resin-acid etched enamel interface [17] [18]. On the

other hand, evidence shows that acid etching creates a honeycomb pattern in the enamel surfaces, which results in micromechanical retention [19]. Acid etching removes about 10 μ from the enamel surface and creates a 5-50 μ m thick porous layer [19].

On the other hand, application of self-etch adhesives on the enamel is associated with some concerns [20] [21]. The superficial etching pattern of the enamel and reduction of micromechanical retention can negatively affect the bond strength and durability [22]. Also, it is not known whether application of self-etch adhesives with moderate pH on the enamel can provide adequate mechanical and chemical resistance in the oral environment as do the etch and rinse adhesives. Further investigations are warranted to answer this question.

In line with our study, van Landuyt et al., [23] indicated that enamel surface preparation by acid etching significantly increases the bond strength. However, it has been reported that acid etching decreases the bond strength to dentin [24]. New self-etch adhesives are mainly categorised as mildly acidic adhesives, and enamel preparation by acid etching before their application can significantly increase their bond strength [25]. Thus, it should be noted that simplifying the process of adhesive application by eliminating the etch and rinse steps (as in universal adhesives) although enhances the bonding process, does not necessarily result in higher clinical success rate. Further studies are required to elucidate this topic better.

Joseph et al., [15] compared the microtensile bond strength of Clearfil SE Bond two-step self-etch adhesive, Adper Easy One 7th generation bonding agent and Futurabond universal adhesive and reported that universal adhesive had the highest bond strength followed by the two-step self-etch bonding agent. The 7th generation bonding agent ranked last regarding the bond strength. This finding was in agreement with our results since universal adhesive with prior etching yielded the highest bond strength in our study.

On the other hand, Souza-Saroni et al., [26] evaluated the microtensile bond strength of Clearfil Liner Bond 2V, Prime and Bond NT/NRC, Single Bond and All Bond 2 to enamel and reported that they all provided almost similar bond strength values. In our study, all adhesives yielded equal bond strength values to enamel except for universal adhesive with prior etching. High bond strength provided by universal adhesive can be due to the presence of nano-size cross-linkers in its composition. Clearfil SE Bond had high bond strength close to that of other groups (except for universal adhesive with prior etching). This can be due to the presence of 10-MDP in its composition, which enhances its chemical bond to hydroxyapatite crystals [27].

Clearfil SE Bond is a two-step self-etch adhesive with a mildly acidic pH (1.8). Some authors

consider it as the gold standard for self-etch adhesives [28]. Two-step self-etch adhesives such as Clearfil SE Bond have separate bottles for resin and primer and therefore are more hydrophobic. Thus, they can provide high bond strength in contrast to one-step self-etch adhesives. Favourable in vitro and clinical results related to the application of this adhesive can be attributed to its double bonding mechanism. Its mild pH can result in the formation of a micromechanical bond by creating a thin and uniform hybrid layer. It can resist debonding forces and shear loads applied during shear bond strength testing. It contains 10-MDP functional monomer and can, therefore, form a stable chemical bond with hydroxyapatite. It is resistant to hydrolytic degradation and seals the restoration margins for a long period [29].

Single Bond is a two-step etch and rinse adhesive that provided the bond strength as high as that of other groups in our study. It contains polymerising resin monomers dissolved in acetone or ethanol. It is reportedly suitable for bonding to enamel [30], and our results confirmed this statement.

Formulation of adhesive systems plays an important role in the performance and clinical service of dental materials. Universal and self-etch adhesives are generally less acidic and therefore have lower efficacy for demineralisation of the mineral phase of enamel and subsequent provision of micromechanical retention [31]. Type and amount of solvent and composition and percentage of monomers in adhesive systems as well as thinning agents can all affect the bond strength. The amount of filler and percentage of mass load are also variable in different bonding agents. The manufacturers do not disclose the exact composition of their products. Moreover, information regarding the rate of shrinkage and hardness of adhesives after polymerisation is limited [25].

Universal adhesive with/without prior acid etching was used in this study. Universal adhesives are self-etch and dual-core and have a pH of 2.2 to 3.2. Generally, self-etch universal adhesives with mild or very mild etching capability may not be able to provide adequately high bond strength to enamel [32]. Despite some concerns in this regard, the bond strength of universal adhesives has been reported to be acceptably high [33]. Our results suggested selective etching of enamel with phosphoric acid to increase the bond strength of the universal adhesive to enamel. This has also been suggested by another study [32]. However, some concerns exist in this respect. For example, the risk of excessive etching of dentin still exists due to the inability to precisely control the area and subsequent reduction of the bond strength to dentin [33].

Evidence shows that bond strength to enamel or dentin more than 20 MPa results in mainly cohesive failure in the dental substrate or composite [9] [34]. In our study, all failures were within the composite or

tooth. In Clearfil SE Bond, all fractures were within the composite and only in Scotchbond group, the percentage of fractures in tooth structure was slightly higher than that within the composite.

In the present study, the bond strength was measured using microtensile bond strength test. Application of microtensile load results in better stress distribution at the adhesive interface compared to conventional tensile or shear loads and yields more accurate results with less diversity [35] [36].

This test enables better stress distribution due to the smaller interface area, which was 1 mm² in our study. However, the bond strength tests, in general, are only suitable for ranking of adhesives because many factors such as masticatory loads, pH alterations and thermal changes are present in the oral environment and affect the bond strength of adhesives to tooth structure. Thus, the results of bond strength tests in vitro cannot well predict the performance of adhesives in the clinical setting [37]. Clinical studies are required to cast a final judgment in this regard. Also, the bond strength of different adhesives to dentin should be compared in future studies.

In conclusion, within the limitations of this study, the results showed that phosphoric acid etching of the enamel before the application of universal adhesive yielded the bond strength significantly higher than that of other groups. Universal adhesive without prior etching yielded the bond strength as high as that of two-step etch and rinse and two-step self-etch bonding agents. This finding highlights the optimal efficacy of universal adhesive in the provision of optimal bond strength with the simplified application.

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Author's contribution

HP and MMI conceptualised this study, designed the methodology for data collection, collected and analysed the data, and wrote the manuscript. EST and SA extensively supported the

development of the study concept, data analysis, and the writing, editing and finalising of the manuscript. SPTNN assisted in data entry and analysis. All authors read and approved the final manuscript.

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