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Bond strength of one-step self-etch adhesives and their predecessors to ground versus unground enamel

A.Ruya Yazici[1]
Zeren Yildirim[1]
Atila Ertan[2]
Gül Ozgunaltay[1]
Berrin Dayangac[1]
Sibel A Antonson[3]
Donald E Antonson[3]

ABSTRACT

Objective: The aim of this study was to compare the shear bond strength of several self-etch adhesives to their two-step predecessors to ground and unground enamel.

Methods: Seventy-five extracted, non-carious human third molar teeth were selected for this study. The buccal surfaces of each tooth were mechanically ground to obtain flat enamel surfaces (ground enamel), while the lingual surfaces were left intact (unground enamel). The teeth were randomly divided into five groups according to the adhesive systems (n=15): one-step self-etch adhesive - Clearfil S[3] Bond, its two-step predecessor - Clearfil SE Bond, one-step self-etch adhesive - AdheSE One, and its two-step predecessor - AdheSE, and a two-step etch-and-rinse adhesive - Adper Single Bond 2(control). After application of the adhesives to the buccal and lingual enamel surfaces of each tooth, a cylindrical capsule filled with a hybrid composite resin (TPH) was seated against the surfaces. The specimens were stored in distilled water at 37°C for 24 hours, followed by thermocycling (5°C-55°C/500 cycles). They were subjected to shear bond strength test in a universal testing machine at a crosshead speed of 1.0 mm/minute. The data were compared using a two-way ANOVA, followed by Bonferroni test at P<.05.

Results: All adhesives exhibited statistically similar bond strengths to ground and unground enamel except for the etch-and-rinse adhesive that showed significantly higher bond strengths than the self-etch adhesives (P<.05). No significant differences in bond strength values were observed between ground and unground enamel for any of the adhesives tested (P=.17).

Conclusion: Similar bond strengths to ground and unground enamel were achieved with one-step self-etch adhesives and their predecessors. Enamel preparation did not influence the bonding performance of the adhesives tested. (Eur J Dent 2012;6:280-286)

Key words: Bond strength; self-etch adhesives; etch and rinse adhesives; enamel

[1] Department of Restorative Dentistry, School of Dentistry, Hacettepe University, Ankara, TURKIYE
[2] Department of Prosthetic Dentistry, School of Dentistry, Hacettepe University, Ankara, TURKIYE
[3] Department of Restorative Dentistry, School of Dental Medicine, University at Buffalo, State University of New York, Buffalo, USA.

Corresponding author: Dr. Ruya Yazici
Department of Restorative Dentistry, School of Dentistry, Hacettepe University, Ankara, TURKIYE
Tel: +90 312 3052270
Fax:+90 312 3113438
Email: ruyay@hacettepe.edu.tr

INTRODUCTION

Adhesive dentistry has developed rapidly with the introduction of new adhesive systems. Although etch and rinse adhesives are still the most reliable and effective approach to achieve efficient and stable bonding to enamel, self-etch adhesives seem to be gaining momentum because they are more user friendly with fewer steps and less technique sensitivity.[1] While a separate adhesive resin should be applied after self-etch primer in two-step self-etch systems, continuing the trend toward simplification, one-step self-etch adhesives that incorporate the classical steps of etching, priming, and bonding into one solution have become increasingly popular.[2] An additional advantage of the one-step self-etch adhesives from the dentist's perspective is that they require shorter chair time.

The bonding effectiveness of self-etch adhesives to dentin has been well documented.[3,4] However, their performance on enamel bonding is questionable. In etch-and-rinse adhesive systems, the bonding of resin to enamel surfaces is a durable and reliable clinical procedure with the use of phosphoric acid etching. Acid etching causes dissolution of hydroxyapatite, resulting in regular microporosities that increase the surface area and surface energy.[5] However, the etching effect of self-etch adhesives is related to the acidic functional monomers that interact with the mineral component of tooth substrate.[6,7] These monomers are generally phosphoric acid or carboxylic acid esters with a pH higher than that of phosphoric acid.[8,9] Self-etch adhesives are less aggressive than the phosphoric acid used in etch-and-rinse adhesives.[10,11] The high hydrophilic nature of acidic monomers in one-step self-etch adhesives might compromise enamel bond strength and there are conflicting results about the bonding effectiveness of one- and two-step self-etch adhesives to enamel.[3,12-14] The surface preparation method significantly affects the nature of the smear layer and thus the interaction of self-etch adhesives.[15] Adhesion to unprepared enamel is even more challenging. It has been reported that the presence of a prismless layer in unground enamel negatively influenced the etching morphology of self-etch adhesives.[14,16] The aprismatic layer is less conducive to bonding due to its highly mineralized structure and more inorganic material content.[17-19] Therefore, this structure might present a challenge especially in terms of the bonding capacity of self-etch adhesives to unground enamel. Low enamel bond strengths have been reported for self-etch adhesives, especially when the enamel was left intact.[13,20,21] On the other hand, there are many clinical applications on unground enamel such as sealant applications, diastema closures, tooth recontouring, and bonding of orthodontic devices that need adequate bond strength.

The aim of this study was to compare the shear bond strength of one-step self-etch adhesives and their two-step predecessors to ground and unground enamel using an etch-and-rise adhesive system as the control. The null hypothesis tested in the present study was 2-fold: (1) the enamel preparation would not influence bond strength values, (2) the bond strengths of one-step self-etch adhesives would not result in bond strengths different from those obtained with their two-step predecessors.

METHODS AND MATERIALS

Seventy-five extracted, non-carious human third molars were stored in 0.01% thymol solution at 4°C and used within one month of extraction. The buccal surfaces of each tooth were mechanically ground with water-cooled #600-grit sandpaper to obtain flat enamel surfaces (ground enamel), while the lingual surfaces were left intact (unground enamel). The lingual enamel surface was marked to outline the flattest area for bonding. The teeth were then inserted into a resin block using a cubical mold, leaving the upper portion of the crowns exposed. The teeth were randomly divided into five groups according to the adhesive systems being used (n=15): Clearfil S^3 Bond, its two-step predecessor - Clearfil SE Bond, a one-step self-etch adhesive - AdheSE One, its two-step predecessor - AdheSE, and a two-step etch-and-rinse adhesive - Adper Single Bond 2 (control). The adhesives were applied to the buccal (ground) and lingual (unground) enamel surfaces of each tooth according to the manufacturers' instructions and polymerized (Table 1). A hybrid composite resin (TPH, Dentsply, Konstanz, Germany) was packed into a plastic cylindrical capsule 2 mm in diameter and 2 mm in height placed in contact with the enamel surface. The excess composite at the capsule-enamel interface was removed with an explorer. The composite resin was light-cured for 40 s using a Quartz-tungsten-halogen light-curing unit (Hilux, Benlioglu, Ankara, Turkiye). Light intensity was monitored by a curing radiometer at a level over 500 mW/cm^2 throughout the study. The specimens were stored in distilled water at 37°C for 24 hours, followed by thermocycling between 5°C and 55°C for 500 cycles. Samples were tested in shear mode using a shear knife-edge testing apparatus in a universal testing machine (Instron Corporation, Canton, MA, USA) at a crosshead speed of 1.0 mm/minute. Shear bond strength values in MPa were calculated from the peak load at failure divided by the specimen surface area. Bond strength data were compared using a two-way ANOVA at a significance level of $P<.05$. Post hoc comparisons of means were performed with Bonferroni test.

RESULTS

The mean shear bond strengths and standard deviations in MPa are shown in Table 2. Two-way ANOVA revealed that the bond strength results were significantly influenced by the adhesive type ($P=.00$) but not by enamel preparation ($P=.17$). The interaction between adhesive systems and enamel preparation was also not statistically significant different ($P=.99$). The bond strength of Adper Single Bond 2 to ground and unground enamel was

Table 1. Adhesive systems, batch number, composition and application mode.

Adhesive Systems	Composition	Mode of Application
Adper Single Bond 2 Two-step etch-and-rinse (3M, ESPE, St. Paul, MN, USA) Batch # 51202	HEMA, Bis-GMA, ethanol, methacrylate fuctioanl copolymer of polyacrylic acid, nano-filler, photoinitator	Apply Scotchbond Etchant to tooth surface for 15s. Rinse for 10s and blot dry. Apply two consecutive coats of adhesive for 15s with gentle agitation. Gently air thin for 5 s. Light cure for 10s
Clearfil SE Bond Two-step self-etch (Kuraray, Osaka, Japan) Batch #41491 (pH=1,8)	Primer: water, MDP, HEMA, CQ, hydrophilic DMA Bond: MDP, HEMA, hydrophilic DMA, Bis-GMA, microfiller	Apply primer for 20s and gently air dry for 2s, apply one coat of adhesive, gently air dry for 2 s, light cure for 10s.
Clearfil S3 Bond One-step self-etch (Kuraray, Osaka, Japan) Batch # 41117 (pH=2,4)	Water, MDP, Bis-GMA, HEMA, hydrophobic DMA, CQ, ethyl alcohol, silanated colloidal silica	Dispense one drop of liquid into well. Apply to enamel for 20s. Strong stream of air to dry and light cure for 20s.
AdheSE Two-step self-etch (Ivoclar Vivadent, Schaan, Liechtenstein) Batch # K11435 (pH= 1,7)	Primer: acrylic ether phosphonic acid, bisacrylamide, water, CQ, stabilizers Bond: Bis-GMA, GDMA, HEMA, fumed silica, CQ, tertiary amine, stabilizers	Apply primer and brush for 15s. Dry with mild air. Apply bond and dry with mild air and light cure for 10s.
AdheSE One One-step self-etch (Ivoclar Vivadent, Schaan, Liechtenstein) Batch # K14311 (pH= 1,5)	Derivates of bis-acrylamide, water, bis-meth-acrylamide dihydrogen phosphate, amino acid acrylamide, hydroxy alkyl methacrylamide, silicon dioxide	Apply a single layer adhesive to enamel, brush it into the enamel for 30s. Strong stream of air dry and light cure for 10s.

MDP: 10-methacryloyloxydecyl dihydrogen phosphate; HEMA: 2-hydroxyethylmethacrylate; Bis-GMA: bisphenol A glycidyl methacrylate.

Table 2. Means and Standard Deviations of enamel bond strength values (MPa±SD).

Adhesive Systems	Ground enamel	Unground enamel
Adper Single Bond 2	37.3 ± 7.3^a	35.1 ± 12.2^a
Clearfil SE Bond	22.7 ± 9.7^b	21.44 ± 9.0^b
Clearfil Tri-S Bond	22.05 ± 6.6^b	20.0 ± 6.8^b
AdheSE	25.9 ± 12.9^b	24.2± 12.8^b
AdheSE One	24.15 ± 10.9^b	21.79 ± 7.7^b

Groups with the same superscript are not statistically different (P>.05).

significantly higher than the shear bond strength of the other adhesives tested (P<.05). The one-step self-ecth adhesives and their two-step predecessors exhibited similar bond strength values to ground and unground enamel (P>.05). When the bond strengths of the ground and unground enamel were compared for each individual adhesive, no statistically significant differences were found (P>.05).

DISCUSSION

The present study compared the bonding performance of one-step self-etch adhesives and their two-step predecessors on ground and unground enamel using an etch-and-rinse adhesive system as the control, under shear bond test. The bond strength of the etch-and-rinse adhesive, Adper Single Bond 2, was higher than that of the two- (Clearfil SE Bond and AdheSE) and one-step self etch adhesives (Clearfil S[3] and AdheSE One) to ground and unground enamel. The shear bond strengths of all the self-etch adhesives to ground enamel were similar to those to unground enamel. So, the first null hypothesis should be accepted. As in the present study the bond strengths of one-step self-etch adhesives and their two-step predecessors were similar, regardless of the enamel substrate, we should accept the second null hypothesis.

The result was not surprising as the application of an additional acid etching step has been recommended in most studies in order to improve the enamel bond strengths of self-etch adhesives.[22,23] However, this additional step will defeat the original purpose of self-etch adhesives. Our results concur with studies that reported that self-etch adhesives produce bond strengths to enamel lower than do etch-and-rinse adhesives.[20,21] On the other hand, Tay et al[24] found no difference among the bond strengths of aggressive self-etch adhesives and etch-and-rinse systems to unground enamel. Another study reported that the bond strength of self-etch adhesives to ground enamel was inferior to that of etch-and-rinse adhesives.[25] Kanemura et al[13] compared the tensile bond strengths of etch-and-rinse systems versus self-etch systems to ground and unground enamel surfaces. While they found that bond strengths to ground enamel did not differ significantly between adhesive systems, in the case of bonding to unground enamel, self-etch adhesives showed lower bond strength values. Perdigao et al[20] compared the enamel bond strength of self-etch adhesives with that of corresponding etch-and-rinse adhesives from the same manufacturer. They reported that most of the etch-and-rinse adhesive systems bonded better to enamel than their corresponding self-etch adhesives; only some of the adhesives tested resulted in lower bond strengths to intact enamel than to ground enamel.

There are conflicting results about the bonding effectiveness of self-etch adhesives to ground and unground enamel. Most studies reported that self-etch adhesives showed lower bond strengths on unground enamel.[13,14,20,26,27] They attributed this result to the hypermineralized nature and high content of fluoride of unground enamel, which make it less accessible to self-etch adhesives. Senawongse et al[21] compared the microshear bond strengths of an etch-and-rinse adhesive system (Single Bond), a two-step self-etching system (Clearfil SE Bond), and a one-step system (One-Up Bond F) to intact and ground enamel surfaces. The bond strengths on ground enamel were significantly higher than those on intact enamel except for the group that was bonded with the etch-and-rinse adhesive system. They also found that the two-step adhesives showed significantly higher bond strengths than did the one-step adhesive. Ibarra et al[12] assessed the microtensile bond strength of two self-etch adhesives, Clearfil SE Bond and Prompt L-Pop, and a conventional adhesive system, Scotchbond Multi-Purpose, to ground and unground enamel. They found that surface preparation and adhesive type had no influence on resin composite enamel bond strength. In another study,[16] the enamel surface preparation did not influence the microtensile bond strength of two-step self-etch adhesives. However, bond strength values were lower when the enamel was left intact for one-step self-etch adhesives. Pivetta et al,[28] found that the enamel pretreatment did not provide an increase in the resin–enamel bond strength of etch-and-rinse and self-etch adhesives. Sengun et al,[29] evaluated whether mechanical alteration of the enamel surfaces with air abrasion and bur abrasion techniques could enhance the bonding performance of a three step and a self-etching adhesive resin system to enamel. It was found that bond strengths values of three-step adhesive were significantly higher than bond strengths of two-step self-etch adhesive for all types of enamel. While bond strength of three step adhesive system did not differ according to the enamel preparation methods, bond strengths obtained with SE Bond to air abraded and bur abraded enamel surfaces were higher than bond strengths to unprepared enamel surfaces. Loguercio et al[26] analyzed the bond strength values (MTBS) of a mild, a moderate, and a strong two-step self-etch system and two etch-and-rinse adhesive systems to unground and ground enamel. The mean values in the SiC paper and diamond bur groups were similar and greater than that in the unground group only for the moderate self-etch systems. Reis et al,[27] evaluated the bond strengths of one-step self-etch systems to unground, bur-cut or SiC-roughened enamel. The bond strength values in the SiC paper and diamond bur groups were similar and higher than the unground group. They also found that one-step self-etch adhesives showed higher bond strengths on ground enamel. The most of the studies mentioned above used either microtensile or microshear tests. So we cannot directly compare our results with these studies.

Enamel adhesion depends on surface roughness, which affects the degree of mechanical anchorage. Therefore, it might be thought that the etching effect of the self-etch adhesive on the enamel surfaces has an important role in producing high bond strength to enamel. However, de Munck et al[3] stated that etching aggressiveness is not entirely correlated with bonding effectiveness but the adhesive resins' properties is. The authors found that some mild and intermediately strong self-etch adhesives approach the etch-and-rinse adhesives, in contrast to the strong self-etch adhesives. There is a lack of correlation between depth of etch and enamel-resin bond strength.[30] Moreover, Hobson and McCabe[31] reported that for strong bond strength values the ideal etch pattern is not essential. In a recent study, it was concluded that pH is not the only parameter necessary for achieving a good bond.[32] Ibarra et al[33] evaluated the enamel surface and interface morphology of self-etch and etch-and-rinse adhesives after bonding to ground and unground enamel. They did not observe large differences between ground and unground enamel surfaces either in their surface etching effect or in the ultrastructural morphology of the adhesive interface. In the present study, the acidity of AdheSE One was 1.5, which was the highest. However, its bond strength values did not differ from those of the other adhesives with low acidity. Since Clearfil S^3 Bond is weakly acidic, with a pH of 2.4, which is higher than that of the other adhesives tested, it might be expected that it would result in lower enamel bond strengths. However, the differences between the bond strengths' of self-etch adhesives were insignificant.

In most studies it was found that two-step self-etch adhesives give results superior to those of one-step systems when bonding to enamel.[19,31,34] Perdigao et al[35] compared the microtensile bond strengths of one-step self-etch adhesives, two-step self-etch adhesive and an etch-and-rinse adhesive as a control. They found that the control yielded the highest mean bond strengths for both ground and unground enamel. While bonds made with Clearfil S^3 resulted in significantly higher mean bond strengths than with its two-step predecessor (Clearfil SE Bond) on unground enamel, no difference was observed between these two adhesives to ground enamel. Our results are in line with a study[32] that compared the bond strengths of all-in-one adhesives with a two-step self-etch adhesive. It was found that some of the all-in-one adhesives produced similar bond strengths. Britta et al,[36] also reported that one-step and two-step self-etch adhesives showed similar bond strength values to unground enamel. Ishikawa et al[37] evaluated the micro-tensile and micro-shear bond strengths of self-etch adhesives to enamel and dentin. While Clearfil SE Bond produced significantly higher values than the one-step self-etch adhesive Clearfil S^3 in the micro-shear bond test to enamel, no significant differences were found among the adhesives in the micro-tensile bond test. Therefore, it might be concluded that the bond strength values vary according to the test methodology. The reason why we obtained results different from those of previous studies might be the shear bond test that we used in our study. Another reason might be related to the composition and mechanical properties of the adhesives. Both S^3 and its predecessor SE contain 10-methacryloxydecyl dihydrogen phosphate (MDP) with two hydroxyl groups that have a chemical chelation property to calcium.[1,38] This may lead to similar bond strength values. One-step self-etch adhesives contain higher concentrations of acidic monomer, which makes them more aggressive in terms of etching capacity and also insensitive to the characteristics of enamel substrate. AdheSE One is a newly marketed one-step self-etch adhesive and to the best of our knowledge, no study has been published about its bonding effectiveness to enamel. AdheSE One is regarded as an intermediately strong self-etch adhesive. Its etching capacity is based on phosphonic acid acrylates. AdheSE One also does not include any solvent that might induce phase separation and precipitation of adhesive components. In the present study these compositional features might have led to the similar bonding effectiveness of AdheSE One and its predecessor on enamel.

It seems appropriate to emphasize that further research is required to confirm these results, in which other types of adhesives and other methods that simulate degradation of the adhesive interface should be performed. Moreover, the findings of this study were developed from *in vitro*, therefore, clinical investigations are needed to confirm these results and provide clinical recommendations.

CONCLUSION

Within the limitations of this *in vitro* study it can be concluded that:

1- Enamel preparation did not influence the bonding performance of the adhesives.

2- The one-step self-etch adhesives and their predecessors gave similar bond strengths to both ground and unground enamel.

3- The etch-and-rinse adhesive showed higher bond strengths than one- or two-step self-etch adhesives regardless of the enamel preparation.

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