the Journal of Contemporary Dental Practice
at www.thejcdp.com
Volume 10 Number 2 March 1, 2009

Effect of Multiple Consecutive Applications of One-step Self-etch Adhesive on Microtensile Bond Strength

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Abstract

Aim: The aim of this study was to evaluate the effects of multiple consecutive coatings of a one-step self-etch adhesive system (Clearfil Tri-S Bond) on the microtensile bond strength (MBS) to dentin.

Methods and Materials: Nine caries-free human lower third molars were sectioned perpendicular to the long axis to expose the mid-coronal dentin. The teeth were separated into three experimental groups (n=3) according to the number of adhesive resin coats applied. In Group 1 one layer of Clearfil Tri-S Bond was applied according to the manufacturer's instructions. In Group 2 and Group 3 the adhesive was applied in two and three layers, respectively. In these two groups the first layer was applied according to the manufacturer's instructions but the second and third layers of adhesive were not light cured after application. After the placement of the composite, the teeth were sectioned to obtain approximately 1 mm^2 beams for testing. Eighteen beams were prepared for each group using the 'non-trimming' method. The MBS values of all specimens were tested, and fracture modes were then determined using a stereomicroscope.

Results: The mean MBS values (in MPa) of Group 1 (one coat of the adhesive) was significantly lower (p=0.04) than those of Group 3 (three applied coats of the adhesive). There were no statistically significant differences between Groups 1 and 2 and Groups 2 and 3.

Conclusion: Within the limitations of this *in vitro* study the application of multiple coats of a one-step self-etch adhesive may provide an increase in bond strength compared to the application of only one coat of adhesive.

Clinical Significance: Three consecutive coats of one-step self-etching adhesive application can improve MBS.

Keywords: Dentin bonding, microtensile bond strength, MBS, one-step self-etch adhesive

Citation: Arisu HD, Eligüzeloğlu E, Üçtaşli M, Ömürlü H. Effect of Multiple Consecutive Applications of One-step Self-etch Adhesive on Microtensile Bond Strength. J Contemp Dent Pract 2009 March; (10)2:067-074.

Introduction

Bonding to enamel is widely accepted as being clinically very reliable. The acid-etch technique has proven to produce an ideal surface morphology for bonding. However, dentin bonding is still a challenge due to the wet tubular ultra structure and heterogeneous, partially organic composition of dentin matrix.[1-3] It is generally accepted dentin bonding relies primarily on micro-mechanical interaction, similar to enamel bonding, mediated by the permeation of resin monomers into acid-etched dentin.[4,5] The entanglement of polymerized adhesive resin with collagen fibrils and residual hydroxyapatite crystals generates an interfacial structure called the 'hybrid layer' or 'resin-dentin inter-diffusion zone'.[5]

Dentin bonding resins were originally formulated with seperate etchants, primers, and adhesives, and these systems have increased the acceptance and reliabilty of dentin bonding.[6-9] Because these systems require dentin conditioning and priming steps prior to adhesive application,[1,10] clinical success with these systems is thought to be technique sensitive and dependant on material-related factors.[11-13] To overcome these shortcomings, single application bonding systems that combine the function of self-etching primer and adhesive have been developed.[14-16] These are usually referred to as "one-step self-etching adhesives".

The advent of one-step self-etch adhesives have improved the efficiency in clinical procedures by simplifying procedural steps and reducing the complexity of the technique.[17,18] Although low technique sensitivity and consistent performance are expected to be achieved with one-step self-etch adhesives due to their simplified application procedures, some previous studies have identified some concern regarding the performance of these adhesives[19] such as reduced dentin bond strengths relative to earlier formulations.[20-22]

Bond strength testing methods are commonly used for the assessment of bonding effectiveness of adhesive resins. In traditional bond strength tests specimens with a large surface area (7-12 mm^2) exhibit an uneven stress distribution at the resin-dentin interface, and 80% of the failures occur cohesively in dentin during testing. In comparison, the microtensile bond strength (MBS) test method using smaller sized specimens (1 mm^2) offers several advantages including optimal stress distribution at the resin/dentin interface, the prevention of cohesive failures in dentin, and provides the opportunity to measure regional differences in resin-dentin bond strength. Because of these advantages, MBS testing is considered the most credible method of evaluating dentin adhesion.[23,24]

Although there have been studies on the effects of multiple coatings adhesive resin systems have on nanoleakage, microtensile, and shear bond strength, the aim of this *in vitro* study was to evaluate the effects of multiple consecutive coatings of a one-step self-etch adhesive system on the MBS to dentin. The null hypothesis tested was that multiple consecutive applications of adhesive have no effect on MBS.

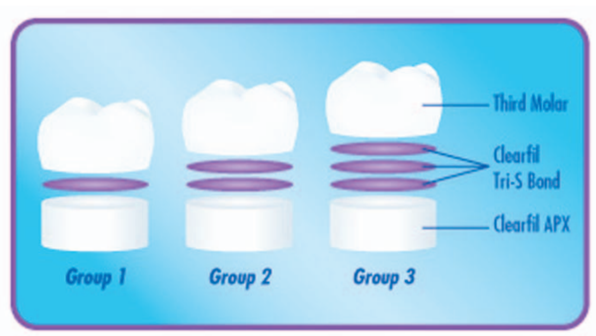
Methods and Materials

Nine caries-free human lower third molars, stored in saline solution for a maximum of one month, were used in this study. Each tooth was sectioned perpendicular to its long axis, using a slow speed diamond saw under water coolant, to expose mid-coronal dentin. Each surface was ground with 600-grit silicone carbide paper under running water for 60 seconds just before the bonding procedure to create a smear layer on dentin. The teeth were separated into three experimental groups (n=3) according to the number of adhesive resin coats applied to the dentin.

Test Material and Consecutive Coating

Clearfil Tri-S Bond is a one-step self-etch adhesive resin (Kuraray Medical, Osaka, Japan) chosen for this study. It consists of the following ingredients:

- Water
- 10-methacryloyloxydecyl dihydrogen phosphate (MDP)
- Bis-phenol A diglycidylmethacrylate (Bis-GMA)
- 2-hydroxyethyl methacrylate (HEMA)
- Hydrophobic dimethacrylate, camphorquinon
- Ethyl alcohol
- Silanated colloidal silica



Group 1 had one coat of adhesive applied to the dentin surface using a sponge for 20 seconds then dried for 5 seconds with high-pressure air and light cured for 10 seconds with a halogen light curing unit (Elipar Freelight II, Dentsply, Kontstanz, Germany) according to the manufacturer's instructions.

In Group 2 and Group 3 the adhesive was applied in two and three layers, respectively. In these two groups the first layer was applied according to the manufacturer's instructions but without light curing. They were light cured for 10 seconds after the second and third layers of adhesive application.

Following the bonding procedures, five 1 mm thick increments of a hybrid restorative resin composite (Clearfil APX, Kuraray Medical, Osaka, Japan) were built up and individually photopolymerized for 40 seconds. After the restorative procedure, the samples were stored in distilled water at 37°C for 24 hours.

Microtensile Bond Strength (MBS) Test

The samples were sectioned into 1 mm thick slabs perpendicular to the adhesive interface using a slow speed water-cooled diamond saw. The slabs were sectioned again in a perpendicular direction to create beams with approximately 1 mm^2 cross sections (Figure 1). Eighteen beams were prepared for each group (six beams from each tooth) using the 'non-trimming' method. The cross sectional areas of each specimen were measured with a caliper and recorded.

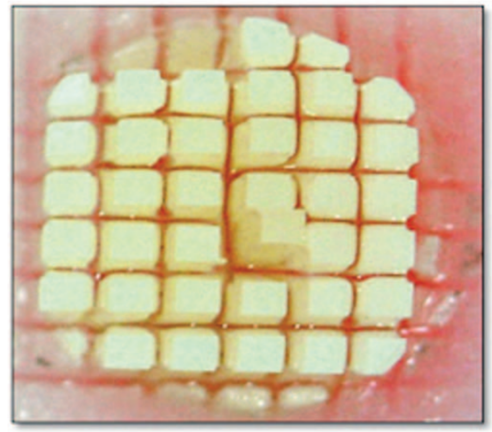


Figure 1. A 1 mm^2 specimen preparation for the microtensile bond test.

The beams were attached to the flat grips of a microtensile testing device (Micro Tensile Tester, T-61010K, Bisco, Inc., Schaumburg, IL, USA) using cyanoacrylate resin (Zapit, Bisco, Inc., Schaumburg, IL, USA) and were tested until failure. The data were recorded in Newtons and later converted to megapascals.

After testing, the specimens were removed from the fixtures and the fracture sites were evaluated for fracture mode (adhesive or cohesive) determination using a stereomicroscope (30X). Failures were classified as follows:[25]

- Adhesive failure, if the fracture site was entirely within the adhesive.
- Mixed failure if the fracture site continued from the adhesive into either the resin composite or dentin.
- Cohesive failure if the fracture occurred exclusively within the resin composite or dentin.

The MBS of groups was analyzed using a one way analysis of variance (ANOVA) and Tukey post hoc tests for a 95% coefficient interval. The Pearson's correlation coefficient was calculated to determine the influence the number of coats of adhesive had on MBS's.

Results

The descriptive statistics of MBS values (in MPa) of groups are shown in Table 1.

The mean MBS value after application of one coat of the adhesive (Group 1) is significantly lower (p=0.04) than after application of three coats of the adhesive (Group 3). There were no statistically significant differences between Groups 1 and 2 and Groups 2 and 3 (Figure 2).

The distribution of failure modes as determined by stereomicroscopic analysis is presented in Table 2. Bond failures were mostly of the adhesive or mixed modes rather than cohesive failures.

Bivariate correlation showed a significant and positive correlation between the number of coats and MBS values of one-step self-etch adhesive to dentin (Pearson correlation coefficient = 0.478 and significance (2-tailed) = 0.001).

Discussion

Numerous studies[26-28] have reported bond strengths of Clearfil Tri-S Bond using the microtensile bond test for only one coat of adhesive application as the manufacturer recommended. Perdigao et al.[26] reported the MBS of Clearfil Tri-S Bond was 27.8±13.2 MPa. The results of the present study also revealed the MBS of Clearfil Tri-S Bond to be 28.18±8.86 MPa after application of one coat (Group 1). The highest values for bond strengths were achieved in Group 3 following three consecutive applications (38.24 MPa). The relationship between the depth of demineralization and the extent of resin monomer penetration is the key to creating a high quality hybrid layer. Poor infiltration of the adhesive resin into the demineralized dentin leaves nano-spaces in the hybrid layer[29,30] making such a region potentially susceptible to degradation from oral fluids.[31] In the case of one-step self-etch adhesives, penetration of the adhesive into dentin occurs simultaneously with demineralization of the mineral component

Table 1. Descriptive statistics of MBS values (in MPa) of groups.

Groups	N	Mean	Std. Deviation	Minimum	Maximum
Group 1	18	28.179	8.8595	16.4	39.8
Group 2	18	33.114	6.1011	21.3	42.6
Group 3	18	38.236	8.2403	24.3	49.0

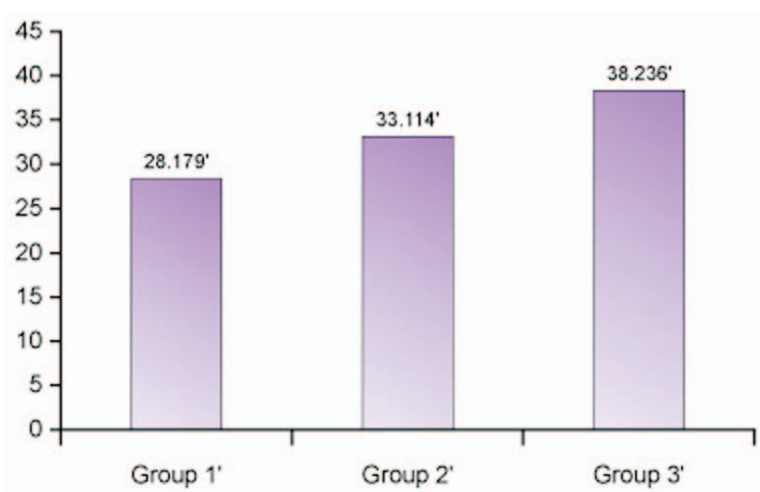


Figure 2. The bars represent the mean MBS values of groups, and the horizontal lines are representing the means for groups in homegeneous subsets for α= 0.05.

Table 2. Distrubution of failure modes.

Groups	Adhesive failure	Mixed failure	Cohesive failure in resin composite	Cohesive failure in dentin
Group 1	16 (88.8%)	1 (5.5%)	1 (5.5%)	0 (0%)
Group 2	15 (83.3%)	2 (11.1%)	0 (0%)	1 (5.5%)
Group 3	16 (88.8%)	1 (5.5%)	1 (5.5%)	0 (0%)

of the dentin. When the resin fails to completely infiltrate deeper portions of the demineralized dentin, the bond between resin and dentin might be weakened. Etching the dentin and faciliating the monomer are the diffusion control phenomenon that requires time for the adhesive to penetrate into dentin. A longer adhesive application time might create a deeper demineralized dentin layer with the resin monomer penetrating into the total depth of the etched, demineralized dentin. A study by el-Din and Abd el-Mohsen[32] reported longer resin application times increased resin-dentin bond strength, presumably due to increased resin infiltration into the hybrid layer. The use of multiple applications of adhesives without curing, as was done in the present study, allows more time for inward diffusion of adhesive monomer. The duration of dentin primer application to etched dentin surfaces has been reported to have an effect on the dentin bond strength of three-step bonding systems.[33] Hashimoto et al.[34] reported the repeated application and subsequent solvent evaporation may promote improved resin infiltration within the exposed collagen fibers. The increased extent of resin impregnation into collagen caused by the consecutive coating method might remove residual water, thereby, improving resin infiltration and cross-linking of the adhesive comonomers within the hybrid layer.

Clearfil Tri-S Bond contains a significant amount of water and solvent in the adhesive container which are expected to be removed by air blowing after application of the adhesive. Because of the detrimental effects water and remaining solvents have on bonding performance as indicated by other studies[35-37] it could be assumed one of the reasons for the differences between bonding performance of one coat and multiple coats was the water and solvent content of the adhesive layer after air-drying. Sadr et al.[38] and Ikeda et al.[35] found the air-drying time of the solvent-containing agent affected bonding performance. In the present study air-drying was repeated when multiple consecutive coats were applied. An additional 5 seconds of drying was carried out for the application of two coats of adhesive and an additional 10 seconds for the application of three coats of adhesive to the specimens.

Use of all self-etching products resulted in a thin hybrid layer. Some investigators believe the thin layer is due to a decrease in acid concentration as it infiltrates the dentin and encounters moisture.[39,40] The reduced thickness should not decrease the adhesive properties of the material because demineralization of approximately 2 μm is sufficient for good bond strength.[41] Pashley et al.[42] and Silva et al.[43] demonstrated an increased bond strength using the technique of applying multiple layers of adhesive then curing each successive layer. When each successive layer is light cured, the adhesive layer becomes thicker without changing the quality of the hybrid layer. The thicker adhesive layer may increase bond strength by improving stress distrubution via increased elasticity of the thicker adhesive layer. However, the increased bond strength using multiple coatings of adhesive without light curing between each layer in the present study might be due to increased resin infiltration into the demineralized dentin and increased removal of residual water rather than the increased thickness of the overlying adhesive layer.

Conclusion

Within the limitations of this *in vitro* study the application of multiple coats of a one-step self-etch adhesive may provide an increase in bond strength compared to the application of only one coat of adhesive.

Clinical Significance

Three consecutive coats of one-step self-etching adhesive application can improve MBS.

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