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Laboratory Research

Effect of Placement Agitation and Placement Time on the Shear Bond Strength of 3 Self-etching Adhesives

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Clinical Relevance

Agitation improves dentin bond strength at certain application times.

SUMMARY

This study measured the shear bond strength (SBS) of 3 self-etching bonding agents to enamel and dentin with and without agitation at 3 different application times. The null hypotheses tested were that agitation and application time have no effect on bond strength. Occlusal surfaces of 180 recently extracted caries-free human molars were wet ground with 600 grit wet-dry silica carbide abrasive paper to obtain a flat enamel surface. The teeth were divided into 18 groups of 10 teeth. Three self-etching bonding agents, Clearfil SE BOND (Kuraray America), Xeno III (Dentsply) and AdheSE (Ivoclar-Vivadent) were applied using application times of 10, 20 or 30 seconds with or without agitation, thinned with a gentle stream of air and cured for 10 seconds, according to manufacturers' directions. Z100 (3M ESPE) composite, A2 shade, was placed over the cured adhesive and cured for 40 seconds. The samples were stored in distilled water at room temperature until testing. The samples were tested in shear to failure with a 1-mm/minute crosshead speed. After enamel shear bond strength testing, the teeth were again ground with 400 and 600-grit wet-dry SiC paper to obtain a flat dentin surface. The protocol used for preparing the enamel bond test samples was repeated, and the teeth were stored until testing in distilled water at room temperature. The samples were again tested in shear at a 1-mm/minute crosshead speed. Values were converted to MPa and data analyzed for intergroup differences using ANOVA and Tukey post-hoc tests. Agitation did not improve enamel SBS for any of the materials tested, but there was a significant difference in enamel SBS among materials: Clearfil SE Bond shear bond strength was greater than Xeno III, which was greater than AdheSE. At 10 seconds application time on dentin, agitation improved the Clearfil SE Bond SBS and, at 20 seconds application time on dentin, agitation significant-

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DOI: 10.2341/05-52

ly improved SBS to dentin for all systems tested. Agitation had no affect when the adhesive was applied to dentin for 30 seconds. Clearfil SE Bond SBS to dentin was significantly higher than the other self-etching adhesives tested except at 10 seconds without agitation.

INTRODUCTION

Proper adhesive placement is critical for the success of bonded restorations. New generations of adhesives have decreased the number of application steps to simplify placement, reduce application time and increase consistency of bond strength. However, there is some question whether this simplification results in clinical success. The newest adhesives have replaced the total-etch, moist-surface technique with one- or two-step systems that combine the etching step with monomer infiltration to create a shallow hybrid layer.[1] Combining the etching and monomer infiltration steps may allow self-etching bonding agents to more fully replace the tooth structure removed by the etching process with adhesive monomer/polymer. Acidic monomers in the primers of self-etching adhesives etch enamel and dentin and modify or dissolve the smear layer. Since primer is not rinsed off, salts from the etching process, remnants of the smear layer and residue from a previous restoration are incorporated into the bonding layer.[1] Since etching and priming occur simultaneously, the moist tooth surface required with total-etch adhesives is not needed to enhance penetration of the primer monomer.[2] Self-etching systems, therefore, do not present the risk of over-etching or over-drying, as with total-etch adhesives.[3]

Some studies have recommend agitation for bonding systems, using phosphoric acid etching, to provide a consistent etch and enhance the interaction between the bonding agent and tooth structure.[4] Agitation keeps fresh etchant in contact with tooth structure, disperses air trapped in the etchant and mixes etching byproducts into the solution for more efficient removal. Jacobsen and Söderholm reported that, with some total-etch systems, primer agitation during placement increases bond strength to dentin.[5]

Active agitation of self-etching bonding agents may improve bonding by a mechanism similar to that of total-etch systems; enhancing the interaction of acid monomers with tooth structure and dispersing etching byproducts into the hybrid layer. As with total-etch adhesive systems, altering the application time of self-etching bonding agents may affect bond strength.[4,6] If the application time is too short, the acid monomer may not adequately etch and penetrate tooth structure and, as a consequence, may produce poor bond strength.

This study measured the shear bond strength of 3 self-etching bonding agents to enamel and dentin with and without agitation at 3 different application times. The null hypotheses tested were that agitation and application times have no effect on bond strength.

METHODS AND MATERIALS

The occlusal surfaces of 180 recently extracted caries-free human molars were wet ground progressively with 120, 400 and 600-grit wet-dry silica carbide abrasive paper to obtain a flat enamel surface. The teeth were divided into 18 groups of 10 teeth. Three self-etching bonding agents, Clearfil SE Bond (Kuraray America, New York, NY, USA), Xeno III (Dentsply, York, PA, USA), and AdheSE (Ivoclar-Vivadent, Amherst, NY, USA), were applied using application times of 10, 20 or 30 seconds with or without agitation. Table 1 lists the manufacturers' directions for adhesive application.

Table 1: *Manufacturers' Directions for Adhesive Application*

	AdheSE	Clearfil SE Bond	Xeno III
Tooth Surface	Dry	Dry	Dry, do not dessicate
Application	Vigorously scrub a generous amount of primer onto enamel surfaces for 15-seconds	Dispense necessary amount of Primer into a well, immediately before application	Shake Liquid A bottle 2-3 times
	Vigorously scrub a generous amount of primer onto dentin surfaces for 15-seconds	Apply Primer to cavity walls and leave in place for 20 seconds	Dispense 1 drop of liquid A and Liquid B into a mixing well
	Disperse Primer with a strong stream of air until the mobile film disappears	Evaporate volatile ingredients with a mild air stream, avoid pooling	Mix for 5 seconds
	Apply Bonding Agent to thoroughly coat conditioned surfaces	Dispense necessary amount of Bond into a well	Apply generously onto preparation surfaces
		Apply Bond to cavity	Leave for at least 20 seconds
Drying	Disperse Bonding Agent with a weak stream of air to eliminate pooling	Make bond film as thin as possible with a gentle air stream	Gentle stream of air for at least 2 seconds until there is no flow
Curing	10 seconds	10 seconds	At least 10 seconds

After the bonding agents were applied, they were thinned with a gentle stream of air and cured for 10 seconds, according to manufacturers' directions, using an Avante curing light (Pentron, Wallingford, CT, USA), with an output of 1230mW/cm^2. Two-millimeter columns of Z100 (3M ESPE, St Paul, MN, USA) composite, A2 shade, were placed over the cured adhesive and cured for 40 seconds with the same visible light curing unit. The samples were then stored in distilled water at room temperature until testing. The samples were placed in an Instron model 4411 testing machine (Instron, Canton, MA, USA) and tested in shear to failure with a 1-mm/minute crosshead speed. Values were converted to MPa and the data analyzed for intergroup differences.

After enamel shear bond strength testing, the teeth were again ground with 400-grit, then 600-grit wet-dry SiC paper to obtain a flat dentin surface. The protocol used for preparing the enamel bond test samples was repeated; the teeth were then stored in distilled water at room temperature until testing. The samples were again tested in shear at a 1-mm/minute crosshead speed. The values were converted to MPa and the data analyzed for intergroup differences using ANOVA and Tukey post-hoc tests.

Mean shear bond strength (SBS) to enamel and dentin was compared for 3 materials (AdheSe, Clearfil SE Bond and Xeno III) under 2 conditions: agitation (present or absent) and application time (10, 20 and 30 seconds) using analysis of variance (ANOVA). A full 3-factor ANOVA model with factors for material, application time, agitation and their interactions was initially fitted. The enamel bond groups exhibited no significant interactions; thus, a reduced main effects ANOVA model was used to test for differences in group level means. Tukey's HSD was used to compare mean SBS among materials. Since the agitation time factor is quantitative, comparisons of mean SBS among the time points was performed using linear contrasts. In particular, the 10-second time point was compared to the 20-second and 30-second time points for a linear trend effect.

For the dentin groups, significant interactions with time were detected; therefore, separate 2-way ANOVA models were used to test for differences in group level means at each time. The 5% significance level was used in all hypothesis tests. The GLM procedure of The Statistical Analysis System, Version 9.0, was used to analyze the ANOVA models.

RESULTS

Enamel bond strength results are summarized in Table 2. In the full 3-factor ANOVA model, agitation was not significant (p=0.6907) and was removed from the model. In the final 2-factor ANOVA model that included factors for material and application time, both factors were highly significant (p<0.001). The 3 bonding materials differed in SBS (p<0.0001); Clearfil produced the greatest strengths, averaging 20.0 MPa, followed by Xeno III and AdheSE, with a mean SBS of 12.7 and 10.3 MPa, respectively. The overall comparison of SBS at all application times was marginally significant (p=0.0577). Comparing the 10-second to the 20- and 30-second application times indicated a significant increase in SBS at 30 seconds (SBS: 17.8 MPa) as compared with 10 seconds (SBS: 15.7 MPa; p=0.0178).

In an evaluation of shear bond strength to dentin (Table 3), material and agitation significantly interacted at 10 seconds (p=0.0006). Differences in mean shear bond strength across brands depended on whether or not agitation was used. At 10 seconds, no significant differences in mean SBS were found (p=0.1019) in samples without agitation. With agitation, Clearfil SE Bond had significantly higher mean SBS than Xeno III and AdheSE (p<0.0001). Agitation gave no significant benefit with AdheSE or Xeno III (p=0.8779 and p=0.930, respectively) at 10 seconds but significantly improved SBS for Clearfil SE Bond (p=0.0014). When applied for

Table 2: *Results of Enamel Shear Bond Testing in MPa with Standard Deviation for Each Group*

Application Time	10 Seconds		20 Seconds		30 Seconds	
Agitation	Yes	No	Yes	No	Yes	No
Clearfil SE Bond	20.0 ± 4.3	22.9 ± 6.1	21.5 ± 4.6	19.1 ± 4.8	23.2 ± 6.0	20.7 ± 4.7
Xeno III	14.6 ± 4.4	13.0 ± 5.4	15.9 ± 4.2	20.4 ± 4.7	17.3 ± 6.7	17.7 ± 2.9
AdhesSE	12.9 ± 4.6	10.7 ± 3.8	13.1 ± 3.3	12.0 ± 6.3	14.1 ± 4.2	13.9 ± 2.5

Table 3: *Results of Shear Bond Testing to Dentin (MPa) with Standard Deviation for Each Group*

Application Time	10 Seconds		20 Seconds		30 Seconds	
Agitation	Yes	No	Yes	No	Yes	No
Clearfil SE Bond	23.5 ± 6.4	14.6 ± 4.4	23.4 ± 9.0	19.1 ± 6.1	20.4 ± 6.0	19.6 ± 7.4
Xeno III	13.3 ± 4.0	17.2 ± 5.9	12.8 ± 2.9	9.1 ± 4.0	11.1 ± 4.4	13.1 ± 3.8
AdhesSE	11.6 ± 4.1	11.9 ± 5.7	12.7 ± 4.6	6.1 ± 4.4	8.5 ± 3.7	10.2 ± 4.4

20 seconds, agitation gave significantly higher SBS (p=0.0009), and Clearfil SE Bond had significantly higher SBS than the other 2 adhesive systems (p<0.0001). At 30 seconds, agitation had no significant effect, but Clearfil SE Bond gave significantly higher SBS than the other 2 materials (p<0.0001).

DISCUSSION

This study found no significant difference in SBS to enamel with or without agitation (Figure 1). Miyazaki and others examined the SBS to enamel when self-etching bonding agents were applied with and without agitation and reported improved bond strength to enamel with agitation with Imperva Fluorobond, MacBond II and Unifil Bond.[7] However, they found no significant difference in enamel bond strength with agitation for Clearfil SE Bond.

In this study, agitation improved SBS to dentin with Clearfil SE Bond at 10 and 20 seconds but not at 30 seconds (Figure 2). Agitation improved SBS to dentin for Xeno III and AdheSE only at the 20-second application time. Miyazaki and others found that primer agitation improved bond strength to dentin for 2 total-etch systems, but the result was not statistically significant.[4] Chan and others found bond strength to dentin with a thick smear layer increased significantly with agitation and, on SEM evaluation, they noted passive application resulted in a hybridized smear layer over the dentin hybrid layer, while agitation resulted in the smear layer being completely dissolved or dispersed into the adhesive.[8]

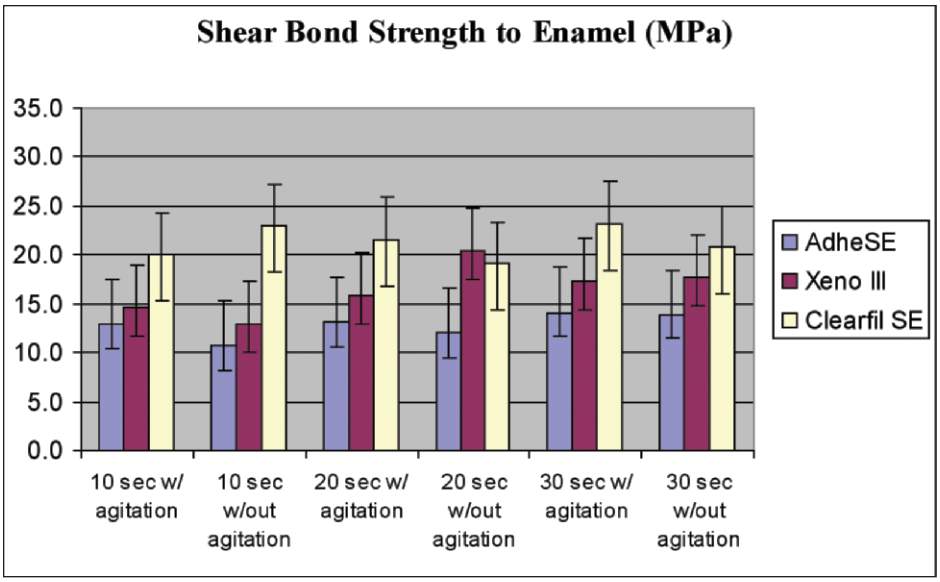

Figure 1. *Enamel shear bond test results graph (MPa).*

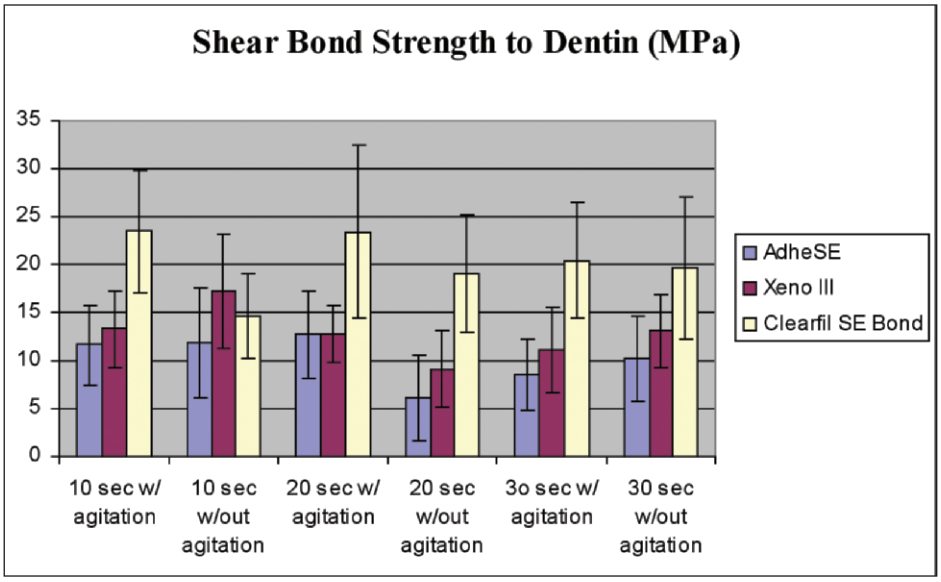

Figure 2. *Dentin shear bond test results graph (MPa).*

The results from other studies verify the bond strength of Clearfil SE Bond. Inoue and others[9] compared single bottle total-etch systems, 1- and 2-bottle self-etch systems and an etch prime and bond system and found that 2-bottle self-etch adhesives were similar in bond strength to single bottle total-etch systems but weaker than the 3-step total-etch system.

Using the classification system proposed by Van Meerbeek and others[1] for the pH of bonding agents, Xeno III and AdheSE are intermediately strong (each with a primer pH of 1.4) and Clearfil SE Bond primer is mild (pH 1.9). Clearfil SE Bond, the mild self-etching, 2-step adhesive produced the highest bond strengths in this study.

Clinical success with adhesives depends on proper manipulation of the material. Several studies have demonstrated the effect of operator variability in the placement of dental adhesives.[10-12] Errors in technique lower bond strength.[13-15] Miyazaki and others[16] demonstrated that inadequate primer drying time decreased bond strength for self-etch adhesives (especially water-based systems, such as Clearfil SE Bond), and they noted that technique-sensitive factors could undo the

benefits of simplified adhesive systems. They reported that each of the tested systems had an optimal drying time.

Further study is needed to refine these techniques to ensure long-term clinical success with self-etching adhesive systems. Variables in products, including the pH of acidic monomers, the solvent used and adhesive monomer composition mean that one application method may not work with all products.

CONCLUSIONS

1. Agitation did not improve enamel SBS for any of the self-etching bonding agents tested.
2. There was a significant difference in enamel SBS among materials: Clearfil SE Bond shear bond strength was greater than Xeno III, which was greater than AdheSE.
3. At 10 seconds application time on dentin, agitation improved Clearfil SE Bond SBS.
4. At 20 seconds application time on dentin, agitation significantly improved SBS to dentin for all systems tested.
5. Agitation had no affect when the adhesive was applied to dentin for 30 seconds.
6. Clearfil SE Bond SBS to dentin was significantly higher than the other self-etching adhesives tested, except at 10 seconds without agitation.

(Received 11 April 2005)

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