

Bond Strength of Different Adhesive Systems to Dental Hard Tissues

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Clinical Relevance

Bond strength to dental hard tissues was dependent upon the type of adhesive system used and varied with respect to tooth regions.

SUMMARY

This study compared the shear bond strengths of different adhesive systems to enamel and dentin of different depths. The adhesive systems used were: Single Bond one-bottle total-etch; AQ Bond one-step self-etching, Clearfil SE Bond two-step self-etching and Tyrian SPE/One-step Plus two-step self-etching.

Eighty extracted non-carious human mandibular molars were mounted in self-curing resin and the occlusal surfaces were ground with a mechanical grinder to obtain flat occlusal enamel surfaces. After applying the adhesive systems, a plastic tube was attached to the enamel surfaces. The tube was filled with a universal hybrid resin composite, which was then polymerized. The specimens were stored in water at 37°C for 24 hours. Shear bond testing was carried out using an Instron Universal testing machine with a crosshead speed of 1 mm/minute. The occlusal enamel of the 80 teeth was removed to determine the bond strengths of the adhesives to superficial dentin. To obtain deep dentin, the same teeth were ground deep towards the pulp, with the remaining dentin thickness approximately 0.7 mm. The adhesives and restorative material were then applied to the prepared dentin surfaces following the methodology described above. For occlusal enamel surfaces, the bond strength of Single Bond was significantly higher than the other adhesives. Single Bond, Clearfil SE Bond and Tyrian SPE/One-step

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Plus performed equally when bonded to superficial dentin; the lowest bond strength was obtained with AQ Bond. On deep dentin, the highest bond strengths were obtained with Clearfil SE Bond.

For each adhesive system, shear bond strength to enamel was statistically higher than it was to either superficial or deep dentin. There were no significant differences between shear bond strengths to superficial versus deep dentin, regardless of adhesive.

INTRODUCTION

The most currently used adhesive system classification is based on the number of steps necessary for clinical application and on interaction with dental hard tissues. Total-etch adhesive systems, which remove the smear layer with phosphoric acid and combine the functions of primer and adhesive in one bottle, have been widely used.¹ Although long-term clinical success has been achieved with total-etch systems,²⁻³ the demand for simplified application has increased, resulting in the development of self-etching adhesive systems. The bonding mechanism of self-etching adhesive systems is based on the simultaneous etching and priming of enamel and dentin without rinsing, forming a continuum in the substrate and incorporating smear plugs into the resin tags.⁴⁻⁵ A self-etch approach involves either a one- or two-step application procedure. Moreover, depending on etching aggressiveness, self-etching adhesives can be subdivided into strong, moderate and mild.⁶⁻⁷ The bond strength of self-etching adhesives to dentin was found to be almost equal to total-etch adhesives.⁸⁻⁹ However, there is some concern about the bonding effectiveness of self-etching adhesives to enamel. While several studies showed that total-etch adhesives yielded enamel bond strength values superior to self-etching adhesives,¹⁰⁻¹² others have reported that there was no significant difference in enamel bond strengths between these two types of adhesive systems.¹³⁻¹⁸

The reliability of dentin adhesives is dependent upon the quality of the dentin. Variation in dentin depth can have a significant influence on the bond strength of adhesive systems, as the density and number of tubules play important roles in adhesion.¹⁹⁻²¹ In a study investigating the bond strength of self-etching adhesives to enamel, superficial and deep dentin, no statistically significant differences between adhesive bond strength values were observed for superficial and deep dentin.²² On the other hand, in another study, self-etching and one-bottle adhesives exhibited significantly lower bond strengths when bonded to deep dentin.²³

A number of studies have investigated the bonding ability of adhesive systems to either enamel, dentin or

both. Although such results are of great value for comparative purposes, in clinical situations, the depth of dentin differs in cavity preparation. Most clinically prepared cavities are complex in design and include not only areas of exposed enamel and superficial dentin, but also deep dentinal areas. Since many different adhesive systems are on the market today, it is desirable to use adhesive systems that produce high, uniform bond strengths to all of these dental hard tissues. As there seems to be few studies related to how these adhesive systems bond to enamel, superficial and deep dentin, the bond strength of adhesives to all these dental hard tissues is a subject in need of investigation. Therefore, the objectives of this study were to: 1) evaluate the shear bond strength of different adhesive systems (Single Bond, a one-bottle total-etch adhesive; AQ Bond, a one-step self-etching adhesive; Clearfil SE Bond, a two-step self-etching adhesive; Tyrian SPE/One-step Plus, a two-step self-etching adhesive) to enamel and dentin of different depths and 2) to determine the difference in bond strengths between these adhesive systems.

METHODS AND MATERIALS

Eighty extracted non-carious human mandibular molars, which had been stored for less than four weeks in 0.2% thymol, were selected and cleaned. The teeth were mounted in self-curing resin. The occlusal surfaces were ground with 120-grit abrasive on a water cooled Ecomet grinder (Buehler, Lake Bluff, IL, USA) to obtain a flat enamel surface and subsequently polished for 30 seconds with wet 240-, 400- and 600-grit silicon carbide abrasive paper. The teeth were then randomly assigned to four groups (n=20), and one of the adhesive systems was applied. Four commercially available adhesive systems were examined: a one-bottle total-etch adhesive (Single Bond, 3M ESPE, St Paul, MN, USA), a one-step self-etching adhesive (AQ Bond, Sun Medical, Shiga, Japan) and two different two-step self-etching adhesives (Clearfil SE Bond, Kuraray Co Ltd, Osaka, Japan and Tyrian SPE/One-step Plus, BISCO, Inc, Schaumburg, IL, USA). The adhesives were used according to the manufacturers' instructions. Table 1 lists the composition of these adhesives and instructions for their use.

Following application of the adhesives, a plastic tube (3 mm in diameter and 3 mm in length) was seated securely against the flattened enamel surface. A universal hybrid resin composite, TPH (Dentsply DeTrey, Kostanz, Germany), was incrementally placed into the tube and polymerized for 40 seconds using a light-curing unit (Hilux, Benlioglu Dental, Ankara, Turkey) with a light intensity above 500mW/cm². The light output of the curing unit was monitored with a light meter (Curing Radiometer Model 100; Demetron Corp, Danbury, CT, USA).

Table 1: Composition and Application Procedures of the Adhesive Systems Used in the Study

Adhesive	Composition	Procedure
Single Bond	Etchant: 35% phosphoric acid Adhesive: HEMA, dimethacrylate, Bis-GMA, ethanol, water	Apply acid for 15 seconds, rinse and dry gently. Apply two coats, dry gently, 10 seconds light cure.
AQ Bond	Adhesive: 4-META, UDMA, acetone, water AQ Sponge: Sodium p-toluensulfinate	Apply three coats using a saturated AQ sponge, dry gently, 10 seconds light cure.
Clearfil SE Bond	Primer: HEMA, MDP, hydrophilic dimethacrylate, water Adhesive: MDP, Bis-GMA, HEMA, hydrophilic dimethacrylate, microfiller	Apply SE-Primer for 20 seconds, dry gently, apply SE-Bond, dry gently, 10 seconds light cure.
Tyrian SPE/One-Step Plus	Primer A: Thymol blue, ethanol, water Primer B: AMPS, bisMEP, TPO, ethanol One Step Plus: Bis-GMA, BPDM, HEMA, CQ p-dimethylaminobenzoic acid (co-initiator), acetone, 8.5% glass fillers	Apply mixture of Primer A and B with continuous rubbing for 10 seconds. Then apply two coats of the bonding resin One-Step Plus, dry gently, 10 seconds light cure.

4-META: 4-metacryloxyethyl trimellitate anhydride; UDMA: urethane dimethacrylate; MDP: 10-methacryloyloxydecyl dihydrogen phosphate; HEMA: 2-hydroxyethylmethacrylate; Bis-GMA: (1-methylethylene)bis[4,1-phenyleneoxy(2-hydroxy-3,1-propanediyl)]bismethacrylate; AMPS: 2-acrylamido-2-methylpropane-sulfonic acid; BisMEP: Bis(2-(methacryloyloxy)ethyl) phosphate; BPDM: biphenyl dimethacrylate; TPO: 2,4,6-[trimethylbenzoyl]diphenylphosphine oxide; CQ: camphorquinone

The specimens were stored in water at 37°C for 24 hours. Shear bond strengths were determined using a Universal Testing Machine (Model 4444, Instron Corporation, Canton, MA, USA) at a crosshead speed of 1 mm/minute. Shear bond strengths in MPa were calculated by dividing the maximum force that induced failure by the bonded area.

After measuring bond strength values of the adhesive systems to enamel, the occlusal enamel of the same teeth was removed with a water-cooled slow-speed diamond saw (Isomet, Buehler) to determine the bond strengths of the adhesives to superficial dentin. After exposing the superficial dentin, flat dentin surfaces were polished with 600-grit silicon carbide paper under running water to create a uniform surface and smear layer. The adhesives and restorative material were then applied to the prepared dentin surfaces following the methodology described above.

To reach deep dentin, the same teeth were deeply ground towards the pulp with 120-grit abrasive on a water-cooled Ecomet grinder (Buehler), with the remaining dentin thickness approximately 0.7 mm from the exposed dentin surfaces. The remaining dentin thickness was verified with periapical radiographs using double-packet Kodak Ektaspeed dental X-ray film (Eastman Kodak Co, Rochester, NY, USA). These radiographs were taken with a Planmeca Prostyle

Table 2: The Mean Shear Bond Strength Values and Standard Deviations in MPa

	AQ One-step Self-etching Adhesive	CSE Two-step Self-etching Adhesive	TSPE Two-step Self-etching Adhesive	SB One-bottle Total-etch Adhesive
Occlusal Enamel	16.1 ± (8.46)	19.8 ± (6.56)	17.5 ± (3.70)	28.6 ± (8.41)*
Superficial Dentin	11.0 ± (3.22)*	15.7 ± (4.00)	13.7 ± (3.89)	14.9 ± (4.52)
Deep Dentin	10.3 ± (4.35)	16.7 ± (3.12)*	11.7 ± (4.10)	2.9 ± (3.47)

For each row, * indicates statistically significant difference ($p < 0.05$)
For each column, brackets indicate no statistically significant difference ($p > 0.05$)

Intra (Helsinki, Finland) intraoral radiography unit that was operated at 63kVp, 8mA. The adhesives and restorative material were then applied to the prepared deep dentin surfaces following the methodology previously described.

Bond strength data were statistically analyzed using two-way ANOVA to determine the effects of different adhesive systems on different dental hard tissues and the interactions between these two factors on the shear bond strengths. Differences between the groups were analyzed using one-way ANOVA/Tukey HSD post-hoc test ($p < 0.05$).

RESULTS

The mean shear bond strength values and standard deviations in MPa are shown in Table 2.

Two-way ANOVA revealed that the bond strength results were significantly influenced by the different adhesive systems ($F=18.73$; $p=0.00$) and dental hard tissues ($F=51.8$; $p=0.00$). The interaction of these factors was also statistically significant ($F=6.8$; $p=0.00$).

Results of the comparisons of adhesive system vs adhesive system and dental hard tissues vs dental hard tissues are shown in Tables 3 and 4, respectively.

Significant differences in bond strengths to enamel were found between the adhesive systems ($p=0.00$). The bond strength of Single Bond one-bottle total-etch adhesive to enamel was statistically higher than other tested adhesives.

The one-step self-etching adhesive AQ Bond showed significantly lower bond strength to superficial dentin than did the two-step self-etching adhesives Clearfil SE Bond, Tyrian SPE/One-step Plus and Single Bond one bottle total-etch adhesive. There were no statistical differences between the two two-step self-etching adhesives and the one-bottle total-etch adhesive bond strengths to superficial dentin ($p<0.05$).

For deep dentin, Clearfil SE Bond exhibited significantly higher bond strengths than the other tested adhesives.

For each adhesive system, shear bond strength to enamel was statistically higher than to either superficial or deep dentin ($p<0.05$).

There were no significant differences between shear bond strengths to superficial versus deep dentin, regardless of adhesive ($p>0.05$).

DISCUSSION

Enamel adhesion by means of acid etching has become an accepted technique in restorative dentistry. Phosphoric acid conditioning of dental enamel causes preferential dissolution of interprismatic enamel, allowing micro-mechanical retention by adhesive resins. While traditionally 30%–40% phosphoric acids have generally been used in total-etch adhesive systems,⁷ self-etching adhesives are composed of acidic monomers rather than phosphoric acid.²⁴ The mild aggressiveness of these acidic monomers could result in minor modifications and less enamel loss, which, in turn, could affect resin adaptation.²⁵ In a morphological study evaluating the surface of etched enamel, Perdigão and others²⁶ reported that the application of self-etching adhesive primer did not result in as deep an enamel etching pattern as did the application of phosphoric acid.

While self-etching adhesives show shallow etching patterns, in most studies, their bond strengths to enamel were found to be similar to total-etch adhesive systems.¹³⁻¹⁸ In another study comparing self-etching adhesives bond strength to enamel, only Clearfil SE Bond achieved high composite-to-enamel bond strength, which was similar to the total-etch bonding systems.²⁷

These results are supported by enamel bond strength studies in which total-etch adhesives were proven to be superior to self-etching adhesives.¹⁰⁻¹² De Munck and others¹⁰ reported that one-step self-etching adhesives produced lower bond strengths than two-step self-etching adhesives. In the current study, although two-step self-etching adhesives produced higher bond strengths, they did not significantly differ from one-step self-etching adhesive.

When the current study compared the dentin bonding effectiveness of self-etching adhesives to total-etch adhesives, it was found that AQ Bond one-step self-etching adhesive produced significantly lower bond strengths to superficial dentin. The other two-step self-etching adhesives were comparable to total-etch adhesive. The reason for the low bond strength values obtained by AQ Bond could be its relatively higher pH (2.5), which might have been incapable of etching superficial dentin. Another reason could be related to its hydrophilic properties. It is known that one-step self-etching adhesives are more hydrophilic than two-step self-etching adhesives⁷ and they attract more water. As it is difficult to evaporate water from these adhesives, water will rapidly diffuse back from the bonded dentin into the adhesive resin and, subsequently, a lower mechanical strength results.²⁸ Recent reports indicate that one-step self-etching adhesives do not perform well in bond strength measurements.^{10,29-30}

Table 3: *p-values for Comparison: Adhesive vs Adhesive for Each Dental Hard Tissue (the underlined p -values are <0.05 and indicate significant difference)*

Enamel	CSE	TSPE	SB
AQ	0.368	0.923	<u>0.000</u>
CSE		0.748	<u>0.001</u>
TSPE			<u>0.000</u>
Superficial Dentin			
AQ	<u>0.002</u>	<u>0.044</u>	<u>0.012</u>
CSE		0.365	0.924
TSPE			0.743
Deep Dentin			
AQ	<u>0.000</u>	0.637	0.138
CSE		<u>0.000</u>	<u>0.012</u>
TSPE			0.754

Table 4: *p-values for Comparison: Dental Hard Tissues vs Dental Hard Tissues for Each Adhesive Material (the underlined p -values are <0.05 and indicate significant difference)*

Dental Hard Tissues vs Dental Hard Tissues	Adhesive Systems			
	AQ	CSE	TSPE	SB
Enamel vs Superficial Dentin	<u>0.019</u>	<u>0.026</u>	<u>0.008</u>	<u>0.000</u>
Enamel vs Deep Dentin	<u>0.007</u>	<u>0.015</u>	<u>0.000</u>	<u>0.000</u>
Superficial Dentin vs Deep Dentin	0.924	0.791	0.258	0.521

The authors of the current study found that, for deep dentin, Clearfil SE Bond showed statistically higher bond strengths than the other adhesive systems tested. This might be attributable to the two hydroxyl groups in the MDP (10-methacryloyloxydecyl dihydrogen phosphate) molecule, which chelates calcium.³¹⁻³² Moreover, hydrophilic 10-MDP monomer improves wetting of the moist tooth surface.³¹⁻³³ Miyazaki and others³⁴ reported that a 10% filler content in adhesives was necessary to increase bond strength. The presence of fillers may produce a sufficiently thick resin film that stabilizes the hybrid layer and provides an elastic buffer zone that compensates for shrinkage stress during polymerization.⁸ Also mentioned is the effect of filled adhesive being more evident on deep dentin.³⁵⁻³⁶ Clearfil SE Bond is a highly filled adhesive, which might explain its higher bond strength values.

It might be expected that bond strengths to deep dentin would be reduced, because the water content of deep dentin is greater than superficial dentin due to the greater diameter and number of tubules per unit area.³⁷⁻³⁸ Tay and others³⁹ reported that the higher water content in deep dentin might dilute the organic solvents of some adhesive systems, causing monomers to leave the soluble phase and form resin globules in water. Recent adhesive systems have improved their bonding ability to deep dentin, developing a circumferential hybrid layer that anchors the tag to the adjacent intertubular dentin.⁴⁰

In the current study, depth of dentin appeared to have only a small affect on bond strength. Although the bond strength value of deep dentin was lower than superficial dentin, no statistically significant difference was observed. Burrow and others²⁰ also reported that dentin depth had no influence on bond strength. They pointed out the importance of the quality of the resin-impregnated layer to obtaining high bond strengths.

Toledano and others⁸ investigated the bond strength to superficial and deep dentin of five adhesive systems. They found that Single Bond and Clearfil SE Bond performed equally when bonded to superficial dentin; they also showed similar bond strength to both dentin depths. Similar to the findings of this study, for deep dentin, Sattabanasuk and others²³ found that one-bottle adhesive systems produced significantly lower values than Clearfil SE Bond.

In this study, statistically higher bond strengths were attained with the Clearfil SE Bond system on deep dentin. Pereira and others⁴¹ reported low bond strength of one-bottle total-etch adhesives to dentin over the pulpal horn region. They also mentioned that water perfusion occurs by removing the smear layer during etching and that rinsing might affect bond strength, even without pulpal pressure. In the current study, when Single Bond was used, residual water, which was left behind

after the etching and rinsing steps, might have negatively affected bond strength values.

In a study that evaluated microtensile bond strengths of self-etching adhesives to dentin, Kenshima and others⁴² found that Tyrian SPE showed the lowest bond strength. They reported that its solvent content is higher than other self-etching adhesives, so that it can promote complete ionization of the acidic monomers. They attributed the comparably lower bond strength value of Tyrian SPE to the higher acetone content of One-Step Plus, which is placed over the self-priming solution. Cho and Dickens⁴³ investigated the varying acetone content of different single solution dentin bonding agents and their effects on microtensile bond strength of resin composites and found that lower acetone concentration improved the integrity of the adhesive interface. In this study, Tyrian SPE, which was the most acidic self-etching adhesive tested, provided lower bond strength values than Clearfil SE Bond.

Dentin is known to be a less favorable substrate than enamel for resin bonding due to its high organic content, the presence of fluid and the odontoblastic process in dentin tubules, and the presence of a smear layer on prepared surfaces.⁴⁴ This was confirmed in the current study for all the adhesive systems that demonstrated higher bond strength values for enamel.

The majority of published bond strength tests are performed using enamel or dentin extracted from human and bovine teeth; only a few studies are available that evaluate the shear bond strength of these teeth simulating dentin tubule fluid flow. The authors speculate that these bond strength values would change in vital teeth. Therefore, long-term clinical trials are needed to evaluate the clinical relevance of these *in vitro* results.

CONCLUSIONS

Within the limitations of this *in vitro* study, bond strengths were found to be dependent upon the type of adhesive system used, and they varied with respect to tooth regions.

While enamel bond strengths were greater than dentin for all the adhesive systems tested, no significant difference was observed between superficial and deep dentin bond strengths.

The enamel bonding of the Single Bond one-bottle total-etch adhesive system was significantly greater than the self-etching adhesive systems. One-bottle total-etch and self-etching adhesive systems displayed significantly different bond strengths at both dentinal depths.

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A clinical course in conservative gold castings, mentored by Dr Richard V Tucker, will be held June 18-22, 2007 at the University of Washington Dental School. For course information, contact Dr Dennis Miya, (206) 244-1618 or at dmichi@aol.com.

Faculty Positions



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Operative Dentistry Home Page



We hope all our readers will take advantage of the information available by accessing our Internet home page. Our address is: <http://www.jopdent.org/>

The home page contains a buttons that, hopefully, will lead you to answers to any questions you may have related to *Operative Dentistry*. These are:

Journal: leads to information on the Editorial Staff and Editorial Board; a complete index of journal volumes; a compilation of direct gold references; highlights of the current issue and a detailed look at published Editorials.

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News: announcements of interest to our readers, including meeting information, advertised faculty positions, and upcoming CE courses.

Authors: complete instructions for contributors to the journal.

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Correction

In *Operative Dentistry* **32-2** 166-172, Bond Strength of Different Adhesive Systems to Dental Hard Tissues, AR Yazici, Ç Çelik, G Özgünlaltay & B Dayangaç, an error was inadvertently made. In the fifth column of Table 2 on pg 168, the bond strength value of Single Bond (SB) one-bottle total etch adhesive to deep dentin was written as 2.9; whereas, it should have been listed as 12.9.