In Vitro Evaluation of Shear Bond Strength of Self Etching Primers to Dentin

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Abstract

Objectives: To evaluate and compare the shear bond strength of four self etching primer adhesives to dentin. Materials & Methods: A total of 75 extracted human maxillary and mandibular molars were selected for the study. The teeth were divided into 5 groups of 15 teeth each, Group A-AdheSE (Ivoclar Vivadent), Group B-Adper prompt (3M ESPE), Group C- i bond (Heraeus-Kulzer), Group D-XenoIII (Dentsply, De Trey) Group E-Single bond (3M ESPE) was used and served as control. All the adhesives were applied according to the manufacturer's instructions. Composite post was built on these bonded surfaces using Z-100 hybrid composite. The teeth were subjected to thermocycling for 500 cycles between 5°C to 55°C. The teeth were then mounted on universal testing machine and fractured under a shearing load, applied at a speed of 0.2mm/min. The readings were noted, tabulated and shear bond strength calculated in Mega Pascal (Mpa) units. Results: There was significant difference in the mean shear bond strength of the four self etching primers, adhesives tested. Shear strength values were in the range of 16.57 to 21.73 Mpa. Xeno III gave the highest mean of shear bond strength whereas Adhe SE showed the lowest value of shear strength. Conclusion: Based on the results of the study, it can be concluded that contemporary self etching primer adhesives bond successfully to dentin. Moreover the bonding ability of Self Etching Systems seems to be comparable to the conventional Total Etch Systems.

Key words: Shear bond strength, Dentin bonding, Self etching primers

Introduction

Adhesive dentistry is a rapidly evolving discipline. It has revolutionized restorative dental practice during the past 30 years. Recent advances in resin adhesives and restorative materials as well as an increased demand for esthetics, have stimulated a great increase in the use of resin based composites in anterior and posterior teeth. Improved adhesive materials have made resin based composite restorations more reliable and long lasting. Well placed composite restorations provide an excellent alternative to traditional posterior restorations.

Adhesive restorations reinforce the weakened tooth structure by effectively transmitting and distributing the functional stresses across the bonding interface. Adhesion also reduces micro leakage at restoration tooth interface resulting in lesser clinical problems such as postoperative sensitivity, marginal staining and recurrent caries all of which otherwise jeopardize the clinical longevity of the restoration. Adhesive technique also allows deteriorating restorations to be repaired and debonded restorations to be replaced with minimum or no additional loss of tooth structure.

The principles of adhesive dentistry date back to 1955 when Buonocore, using techniques of industrial bonding, postulated that acids could be used as a surface treatment before application of the resins. In the late 1960's, Buonocore suggested that it was the formation of resin tags that caused the principal adhesion of the resins to acid etched enamel. The idea that resin penetrates the micro porosities of etched enamel and results in a micromechanical bond is well accepted today. But as Buonocore suggested in 1963, adhesion of our restorative materials to dentin has proved to be more elusive.

The technique of total etch, though, gave good bond strength and showed improved retention of restorations, but was still technique sensitive in terms of multiple steps involved in the bonding process. The most critical step was washing off the etchant and ensuring that the exposed collagen is maintained in a moist state, but not so wet as to affect the bond. It has been shown that too much water can cause some bonding resins to undergo complex phase separation and lose their efficacy in forming a reliable bond. Conversely, over drying leads to incomplete resin infiltration of the collapsed collagen scaffold and reduced bond strength.

Hence, self etching primers were developed in an attempt to simplify the bonding procedures and to prevent discrepancies between the depth of dentin demineralized by the acid and the ability of the primer to penetrate this demineralized layer. The self etching primers are designed to etch through smear layers into the underlying dentin. They utilize weaker acids that have been shown to remove partially the smear layer, maintain the smear plugs and to create thin hybrid layers. These systems act by simultaneous conditioning, demineralizing and infiltrating both the enamel and dentin. The smear layer is altered but not removed and rinsing is not indicated. As acidic monomers are responsible for etching
and bonding, the depth of demineralization is equal to the
depth of penetration of the monomers and clinically this
corresponds to a reduced change of postoperative sensitivity.
In addition to simplifying the bonding procedure and
reducing the working time, the rinsing and drying steps were
eliminated. Hence the need for an ideal dentin wetness
condition reduces the negative influences of these steps on
establishing adhesion.

A very recent development in the field of adhesive dentistry,
is the introduction of self-etching primer adhesive. A further
development of the concept of self etching primers, the self
etching adhesives or self etching primer adhesives was
recently introduced. These materials have incorporated all
the components of bonding systems (acidic conditioner,
hydrophilic primer and hydrophobic adhesive resin) into one
bottle and are the first true 'one step agents'. This takes
simplification of the bonding procedure a step further ahead.
Currently, there are several self etching systems available but
little is known about their capacity to adhere to dental hard
tissues. Since bond strength testing is used as a screening tool
to help understand and predict the clinical behaviors of
adhesives, this invitro study was designed to investigate and
compare the shear bond strength (SBS) to dentin achieved
with several self etching primer/adhesive systems.

Materials and Methods
In this in-vitro study the following four self etching primer/
adhesives were tested for their shear bond strength to dentin
and the results were compared with single bond (total etch
adhesive).

Group A : Adhe SE dentin adhesive (Ivoclar vivadent co.)
Group B : Adper prompt dentin adhesive (3M ESPE)
Group C : i bond dentin adhesive (Heraeus Kulzer)
Group D : Xeno III dentin adhesive (Dentsply DeTrey)
Group E : Single bond dentin adhesive (3M ESPE)

A total of seventy five non carious intact extracted human
permanent maxillary and mandibular molars were selected
for the study. The teeth were ground on a model trimmer to
remove coronal enamel and expose adequate underlying
dentinal surface for bonding. The exposed dentinal surface
was abraded with series of medium grit silicon carbide paper
(320, 400, 600 grit Silicon carbide) in wet conditions. The
teeth were randomly divided into 5 groups of 15 teeth each
(Fig.1), that differed by the adhesive system used. All the
teeth were bonded according to the manufacturer's
instruction; a plastic cylindrical matrix of 4x5 mm dimension
was placed securely on the dentinal surface. The matrix was
filled with Z-100 hybrid composite resin (3M). The
composite was placed in 2 increments and each increment
was cured for 40 sec and additional 20 sec after removal of the
plastic matrix.

Fig.1: Abraded teeth ready for composite bonding.

Group A: Adhe SE Dentin Adhesive
The dentinal surface of the tooth to be bonded was gently
dried by blotting the excess water (moisture) on the surface
with cotton pellet or tissue paper. Adequate amount of Adhe
SE primer was then applied to the dentinal surface with a
micro applicator brush tip. The primer was brushed onto the
total etch adhesive surface for not less than 30 sec. Any excess amount of
primer left on the surface was dispersed with moisture and oil
free compressed air until mobile liquid film disappeared.
Adhe SE Bond was then applied on the primed surface with a
fresh applicator tip. It was polymerized for 10 sec using light
cure unit. The light emission window was held as closely as
possible to the material.

Group B: Adper Prompt Dentin Adhesive
The dentinal surface of the tooth to be bonded was gently air
dried until there was no water visible on the surface. Then 1
drop of liquid from bottle A and 1 drop from bottle B were
dropped in the mixing well and both the components were
mixed thoroughly with a disposable applicator tip until clear
yellowish solution was formed. This adhesive solution was
then applied to the dentin surface with an applicator brush. The
adhesive was massaged on the surface for 1 sec applying

Fig.2: Universal triaxial testing machine with specimen mounted.
pressure. Then the adhesive was dried thoroughly using a gentle stream of air. Again a second coat of adhesive was applied and gently air-dried to thin film. The adhesive was then polymerized for 10 sec using a light cure unit.

**Group C: i bond Dentin Adhesive**
The dentinal surface was gently air dried as mentioned previously. The adhesive was dispensed in a mixing well, and was applied to the dentinal surface with an applicator tip. 2-3 coats of adhesive was applied consequently. The adhesive was slightly agitated with the brush tip for 30 sec to improve diffusion. The adhesive was then gently air dried and light cured for 20 sec.

**Group D: Xeno III Dentin Adhesive**
After drying the dentinal surface gently, equal amount of liquid from bottle A and bottle B were disposed in the mixing well and mixed thoroughly for about 5 sec with the applicator tip. The adhesive was then applied with the same applicator tip to entire dentinal surface and was left undisturbed for 20 sec. It was then uniformly spread with gentle stream of air and light cured for 10 sec.

**Group E: Single Bond Dentin Adhesive**
In this group, the dentinal surface was acid etched with 35% phosphoric acid for a period of 15 sec and rinsed with water for further 15 sec. Excess water was removed by using a tissue paper so that the surface was left visibly moist. Then the single bond dentin adhesive was applied in 2 consecutive coats, gently air dried with oil free compressed air from air syringe and then light cured for 10 sec.

The matrices were removed from the teeth by slitting them with a Bard Parker blade along its length after the composite was set. The specimens were then stored in saline solution at room temperature for 24 hours. After 24 hours, the teeth were subjected to thermo cycling for 500 cycles between 5°C to 55°C (+ 1°C) with a dwell time of 30 sec.

The specimens were then mounted on custom fixture for determination of shear bond strength using Universal testing machine (Fig.2). A knife edged chisel (0.5 mm in cross section) was used to deliver the shearing force. The shearing load was applied at a speed of 0.2 mm/min until fracture of the material occurred. The shearing force was noted and shear bond strength was calculated and recorded in Mega Pascal units.

**Results:**
Results derived from the study are tabulated in Table 1. Statistical analysis of the shear bond strength of five groups of dentin adhesives was done by one way analysis of variance (ANOVA) and the results obtained are shown in Table 2.

<table>
<thead>
<tr>
<th>Source of variation</th>
<th>SS</th>
<th>df</th>
<th>MS</th>
<th>F</th>
<th>p-value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Between groups</td>
<td>272.2</td>
<td>4</td>
<td>68.05</td>
<td>21.007</td>
<td>2.07X10^-11*</td>
</tr>
<tr>
<td>Within groups</td>
<td>226.756</td>
<td>70</td>
<td>3.2394</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>498.956</td>
<td>74</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Where: SS- sum of squares, df- degree of freedom, MS- Mean sum of squares, F- Statistic F, *p-value <0.05 = Statistically Significant

The very small value of p indicated that there was no homogeneity among the means of the five groups. Hence statistical comparison of the shear strength of five groups was done individually using students unpaired 't' test and the values of p were tabulated as shown in Table 3.

<table>
<thead>
<tr>
<th>Groups</th>
<th>p-value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Group A &amp; B</td>
<td>0.000194*</td>
</tr>
<tr>
<td>Group A &amp; C</td>
<td>0.7263</td>
</tr>
<tr>
<td>Group A &amp; D</td>
<td>1.77X10^-8*</td>
</tr>
<tr>
<td>Group A &amp; E</td>
<td>0.000081*</td>
</tr>
<tr>
<td>Group B &amp; C</td>
<td>0.001686*</td>
</tr>
<tr>
<td>Group B &amp; D</td>
<td>0.003951*</td>
</tr>
<tr>
<td>Group B &amp; E</td>
<td>0.6911</td>
</tr>
<tr>
<td>Group C &amp; D</td>
<td>2.5X10^-8*</td>
</tr>
<tr>
<td>Group C &amp; E</td>
<td>0.001572*</td>
</tr>
<tr>
<td>Group D &amp; E</td>
<td>0.000334*</td>
</tr>
</tbody>
</table>

*p-value <0.05 = Statistically Significant

**Discussion**
The growing demand for esthetic restorations and the alleged toxicity of silver amalgam have stimulated intensive research focused on amalgam alternatives. Successful adhesion to hard tooth tissue is mandatory for the restoration of teeth with tooth coloured materials such as direct or indirect resin composites, ceramic inlays and veneers.

The polymerization shrinkage of resin composites generates stress between bonded restoration and tooth, therefore shrinkage still remains the major antagonist to durable adhesion of resin composites. Good marginal seal guarantees gap free margins and prevents microleakage, recurrent caries and pulpal irritation.

Since its introduction, the enamel etch technique has
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provided an ideal surface for reliable bonding performance using adhesive resins. Success with approaches of bonding to dentin, however, have been less reliable due to the characteristics of the dentin substrate, including high organic content, tubular structure variations and the presence of outward fluid movement.\textsuperscript{11} Despite these difficulties dentin bonding has become more successful with the development of new dentin adhesive systems over the last 10 years.\textsuperscript{11}

The use of self etching primers and adhesives is a recent approach towards the simplification of bonding techniques. This approach does not require rinsing and can be done in a two step method, combining the etching and priming functions, or in a one step method, combining etching, priming and bonding functions. The rationale behind the use of the self etching systems is the formation of continuity between tooth surfaces and adhesive material, which is accomplished by the simultaneous demineralization and penetration of its agents. This could be an advantage compared to the claimed technique sensitivity of conventional total etch dentin bonding agents.\textsuperscript{12}

Currently, many studies have been conducted to evaluate the bond strength of resin composite to enamel and dentin by using different bonding agents. Shear bond strength test is a simple evaluation procedure used to test the adhesion of dental adhesives.\textsuperscript{12} In vitro bond strength tests are useful and essential for predicting the performance of new adhesive systems and possible correlation with clinical issues, however in-vitro investigations are not capable of predicting clinical success.\textsuperscript{12}

According to the results obtained from the present study, it was observed that group D (Xeno III) gave the highest mean of shear bond strength compared to all the other groups. Group B (Adper prompt) gave bond strength values similar to that of group E (single bond) control group. Where as group A and group C gave lowest means of shear bond strength and the means were not statistically different from each other. The highest mean of shear bond strength were obtained with group D, i.e. (Xeno III dentin adhesive). See Table 1.

The results of dentin shear bond strength with Xeno III in this study are in agreement with the literature.\textsuperscript{13,14} These results may be related to its very low pH value (<1) assuring a better monomer penetration to the critical mode of application of this system and by the fillers in the adhesive composition, enhancing the bond strength to dentin. Adper prompt self etch adhesive gave bond strength values similar to that of single bond control group. Adhe SE, a self etching primer gave the lowest mean shear bond strength. Similar low bond strength values were obtained with i-bond adhesive. i-bond all in one self etch adhesive gave bond strength similar to Adhe SE and differed significantly from the control group.

The main objective of bond strength test is to establish a demonstrative value for how strong the bonding of an adhesive system is to dental hard tissues. When composites are bonded the volumetric shrinkage that occurs under polymerization generates stresses on the bonded opposing walls in box like cavities. It has been stated that composite bond strength should be as high as 17 to 20 Mpa to resist this shrinkage stress.\textsuperscript{15}

The in vitro methods used for evaluation of dentin adhesive have varied from one laboratory to another and wide variations in bond strength values are reported.\textsuperscript{16} The variations in the values of in vitro bond strength indicate not only the complex nature of the testing procedures but also the sensitivity of handling and manipulation of these systems and the composite restorative material. See Table 2.

One among the many factors that may be responsible for large variations in shear bond strength values is the quality and structure of the dentin itself.\textsuperscript{16} Dentin factors affecting adhesion include the smear layer, dentinal tubule density, dentinal tubule length, size and content and sclerotic changes in the dentin.\textsuperscript{16}

The result of the present study showed that there was significant difference in the in vitro dentin shear bond strength among the self etching primer adhesives tested, but there is no common factor which accounts for the differential performance of the systems tested. While in vitro testing is not a definitive predictor of clinical behavior, the Xeno III system generated values higher to, and Adper prompt system gave values similar to that of single bond total etch system that has had a long history of clinical success. See Table 3.

However due to the inherent limitation of an in vitro study, the bonding and sealing ability of these self etching adhesive systems to dentin warrant further investigation.

**Conclusion**

The result of the study showed statistically significant difference in the shear bond strength to dentin of the four self etching primer adhesives tested. Xeno III adhesive gave the highest mean of shear bond strength. Adper Prompt self etch adhesive showed values similar to single bond total etch system. Also Adhe SE and i-bond generated lower values of shear strength and differed significantly from the control group.

Based on the results of this study it appears that contemporary
self etching primer adhesives bond to dentin successfully. Moreover the bonding ability of self etching systems seems to be comparable to the conventional total etch system.

However, further long-term clinical evaluations are obviously necessary to confirm these observations and to decide if these systems can be seen as a good and adequate alternative to 5th generation products.

Reference

Source of Support: Nil.
Conflict of Interest: None Declared.