Study of Shear Bond Strength of Two Adhesive Resin Systems

Mohamed Atta Gowida1*, Ahmed Yehia Ashour2, Seham Ahmed Hanafy3, Waleed Abdel-Maguid El-mahy4 and Wegdan M Abdel-Fattah5

1Senior Registrar at the Conservative Dentistry Department, Faculty of Dentistry, Alexandria University, Egypt
2Professor of Conservative Dentistry, Faculty of Dentistry, Alexandria University, Egypt
3Professor of Dental Biomaterials, Faculty of Dentistry, Alexandria University, Egypt
4Professor of Conservative Dentistry, Faculty of Dentistry, Alexandria University, Egypt
5Isa professor of conservative dentistry, faculty of dentistry, alexandria university, Egypt

*Corresponding Author: Mohamed Atta Gowida, Senior Registrar at the Conservative Dentistry Department, Faculty of Dentistry, Alexandria University, Egypt.

Received: May 27, 2016; Published: June 07, 2016

Abstract

Aim: The aim of this study was to evaluate shear bond strength of two adhesive resin systems self-etching primer (Clearfil SE Bond) and self-etching adhesive (One-Up Bond F).

Materials and Methods: Thirty non-carious extracted human permanent molars teeth prepared in thirty acrylic molds were used in this study. The dentin surface of each specimen was wet ground with a medium grit sandpaper disk and then 600 grit silicon carbide abrasive paper. Fifteen specimens were used for each of the bonding agent systems, each specimen was bonded with hybrid composite resin (Filtek Z250) and applied to the treated dentinal surface through a plastic mold. All specimens were placed into water for one week. All specimens were shear tested to failure at a crosshead speed of 5 mm/min until fracture occurred using a universal testing machine.

Results: The two-step self-etching primer (Clearfil SE Bond) showed higher dentin shear bond strength than one step self-etching adhesive (One Up Bond F). There was a statistically significant difference between the two test adhesives (p<0.0001*).

Keywords: Self-Etching primer; Clearfil SE Bond; resin; One Up Bond F; Polymerization

Introduction

Simplification of adhesive resin systems aim to reduce technique-sensitivity by reducing the number of clinical steps involved. Self-etch adhesives are applied to the tooth surface before resin composite placement, to ensure maximum adhesion by improving monomer penetration into the hydrophilic dentin substrate, and to improve wettability of the tooth surface by the resin components [1].

Single-step self-etch adhesive systems form a continuous layer by demineralization of the superficial dentin substrate, followed by resin monomer penetration into the etched dentin [2]. Previously, the application of single-step self-etch adhesive to dentin resulted in retention of a smear layer and insufficient bond strength [3]. By contrast, higher bond strengths were obtained for two-step self-etch primer adhesive systems through the introduction of a submicron resin tag [4].

Self-etching priming agents which serve simultaneously as conditioner and primer without being rinsed off have been introduced as dentin adhesive systems [5]. The reactive molecules in these self-etching/self-priming systems are esters from bivalent alcohols with methacrylic acid and phosphoric acid or derivative [6]. The combined self-etching, self-priming adhesive agent applied to a dentinal surface penetrates the substrate via three-dimensional, reticulate channels formed by the self-etching primer [7]. Demineralization and monomer infiltration of the dentin take place simultaneously, thereby creating a hybrid layer with no need for separately applied acid etching and priming [8].

However, only spares data have been published concerning the bonding potential of more recently introduced self-etching/self-priming adhesive systems [9].

The bond strength provided by adhesive systems is the force per unit of area required to break a bonded assembly with failure occurring in or near the adhesive surface. The purpose of shear bond strength test is to establish a numeric value in order to determine how strong that bond was [10].

With the development of the new bonding systems, more details about bonding to tooth structure needs to be evaluated [11].

The aim of this study was to evaluate shear bond strength of two adhesive resin systems Self-etching primer (Clearfil SE Bond) and Self etching adhesive (One-Up Bond F).

Materials and Methods

Thirty caries free extracted human permanent molars were cut 2 mm apical to the cemento-enamel junction. Each tooth was then embedded to the cervical line in a prefabricated copper cylinder (20 mm long and 14 mm inside diameter) (Figure 1), oriented with the long axis of the tooth perpendicular to the base of the mold. A soft mix of auto-polymerizing acrylic resin was used to fill the mold. The resin was poured into the mold to the level of the cemento-enamel junction of the tooth. The occlusal surfaces of the teeth were reduced on a dental model trimmer so that the occlusal groove patterns were removed. This produced a flat dentin surface perpendicular to the specimen base (Figure 2).

Dentin surface preparation

The dentin surface of each specimen was wet ground with a medium grit sand paper disk then 600 grit silicon carbide abrasive paper to create a uniform smooth surface. The surface was then dried with a steam of oil free compressed air for 10 seconds. Teeth with their acrylic mold were divided into two groups of fifteen each according to the type of bonding agent used. The specimens were classified into two groups (n = 15) fifteen for each according to the bonding type used (Table 1). Self-etching adhesive (One Up Bond F) were applied to the group1 and self-etching primer (Clearfil SE Bond) were applied to the group 2 according to manufacturer instructions and light cured. A plastic cylindrical mold (5 mm in diameter and 3 mm in height) was centered and firmly held perpendicular to the prepared surface of each tooth.

The prepared dentin surfaces of the embedded teeth were pretreated within the confines of the plastic molds; the plastic mold was over filled with hybrid composite resin (Filtek Z250) incrementally, and pressed to avoid air bubbles. Curing of the specimens was done using the light cure unit. Following polymerization, all specimens were placed into water for one week.

Shear bond strength testing

All specimens were secured in a specially designed holder to a universal testing machine*** (Figure 3).

Citation: Mohamed Atta Gowida., et al “Study of Shear Bond Strength of Two Adhesive Resin Systems”. EC Dental Science 4.5 (2016): 862-868.
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The upper plateau of which contacted a copper rod placed across the occlusal surface of the test teeth for each of the two groups. This was bearing its force on the restoration, so that the plastic cylinder was at 90 degrees to the vertical plane (Figure 4).

<table>
<thead>
<tr>
<th>Products</th>
<th>Characteristics</th>
<th>Composition</th>
<th>Application protocol</th>
<th>Batch number</th>
<th>Manufacturer</th>
</tr>
</thead>
<tbody>
<tr>
<td>One Up Bond F</td>
<td>Self-etching adhesive</td>
<td><em>Bonding A</em>: Water; MMA, HEMA, coumarin dye, methacryloyloxyalkyl acid phosphate, MAC-10, <em>Bonding B</em>: multifunctional methacrylic monomer; fluoroalumino-silicate glass; photo initiator (aryl borate catalyst).</td>
<td>After mixing of one drop of agent A and agent B, agitate the pink liquid for 10 s on dentin; gently air blast Light cure for 20s.</td>
<td>U0022Y2 5053010110</td>
<td>J. morita USA</td>
</tr>
<tr>
<td>Clearfil SE Bond</td>
<td>Self-etching primer</td>
<td><em>Primer</em>: MDP, HEMA, hydrophilic Dimethylacrylate, di-Camphoquinone, N, N-Diethanol-P-toluidine, water. <em>Bond</em>: MDP, BIS-GMA, HEMA, Hydrophobic dimethylacrylate, di-Camphoquinone, N, N-Diethanol-P-toluidine, silanated colloidal silica</td>
<td>Apply primer for 20 s. Mild air stream. Apply bond. Gentle air stream. Light cure for 20 s</td>
<td>352</td>
<td>Kurary Medical Inc., Okayama, Japan</td>
</tr>
<tr>
<td>Filtek Z 250</td>
<td>hybrid composite resin</td>
<td>Bis-GMA, UDMA, Bis-EMA, zirconia/silica (60%, 0.6 µm)</td>
<td>Incrementally 1 mm thickness and 40 s light cure.</td>
<td>5cc 1370A3</td>
<td>3M ESPE, USA</td>
</tr>
</tbody>
</table>

Abbreviations: MMA: methyl methacrylate; HEMA: 2-hydroxyethyl methacrylate; MAC-10: methacryloyundecane dicarboxylic acid; MDP: 10-methacryloyloxydecyl dihydrogen phosphate; BIS-GMA: bisphenyl glycidyl methacrylate; UDMA: urethane dimethacrylate; BIS-EMA: Bisphenol A polyethylene glycol diether dimethacrylate; S: seconds; µm: micron; mm: millimeter

Table 1: Adhesives and hybrid composite resin used in the study.

All specimens were shear tested to failure at across head speed of 5 mm/min until fracture occurred. The diameter of each specimen was recorded, and the load was recorded in kilograms.

The shear bond strength was calculated by dividing the force at which failure occurred by the bonding area.

The shear bond strength was calculated by the following equation;

Shear bond strength (in kg/cm²) = l/a

l = breaking load (in kg)
a = area of composite dentin interface in cm²

Area = 3.14r²

The shear bond strength values in kg/cm² were converted to MPa.

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Statistical Analysis
Mann Whitney test was used for comparison between unpaired signed ranks test.

Results
The mean shear bond strength values for the (One Up Bond F) and (Clearfil SE Bond) were $8.98 \pm 1.41 \text{ MPa}$ and $13.7 \pm 2.16 \text{ MPa}$ respectively (Table 2) and (Figure 5).

<table>
<thead>
<tr>
<th></th>
<th>One Up Bond F</th>
<th>Clearfil SE Bond</th>
</tr>
</thead>
<tbody>
<tr>
<td>Maximum value</td>
<td>11.7</td>
<td>17.2</td>
</tr>
<tr>
<td>Minimum value</td>
<td>6.8</td>
<td>8.5</td>
</tr>
<tr>
<td>Mean</td>
<td>8.98</td>
<td>13.7</td>
</tr>
<tr>
<td>Standard deviation</td>
<td>1.41</td>
<td>2.16</td>
</tr>
<tr>
<td>U</td>
<td>8.69</td>
<td></td>
</tr>
<tr>
<td>P</td>
<td>0.00001*</td>
<td></td>
</tr>
</tbody>
</table>

*Table 2: Statistical analysis of shear bond strength (MPa) of the two test groups.

Also, there was a statistically significant difference between the two test adhesives using Mann Whitney with $U = 8.69$ and $p < 0.0001^*$.  

Discussion
Shear bond strength is one of the criteria that affect clinical success rate of dental materials. Adequate bond strength is important to prevent opening of margins by polymerization shrinkage. Dislodgment of a filling is inconvenient to both patient and dentist. Shear bond strength tests are useful in understanding the failures stress rates and assessing whether these forces also exist in clinical situation, since dental practices require materials with a reliable adhesion to tooth structure. As an indicator of material retention ability, the value of shear bond strength can be measured \textit{in vitro} [12].

The results of shear bond strength test of the present study showed that there was significant difference between the self-etching primer and self-etching adhesive, (Clearfil SE Bond) showed higher shear bond strength than (One Up Bond F). This coincides with the results of Sensi, \textit{et al.} [13], Naughton and Latta [14], John, \textit{et al.} [15], Bradna, \textit{et al.} [16], and Yu, \textit{et al.} [17] who found that the two step self-etching adhesives has higher shear bond strength than one step self-etching adhesives.

The adhesive system Clearfil SE Bond is characterized as a self-etch primer with pH about 1.9. After its application, the acid is totally neutralized by phosphate ions during demineralization. On the other hand, One Up Bond F, which has a pH around 1.0, provided lower dentin shear bond strength than Clearfil SE Bond.

In spite of the importance of pH values to understand the efficacy of self-etching adhesive systems, it is not possible to consider that the pH alone is responsible for the performance of the adhesive systems since their composition is an important factor of influence at the interfacial aspect [18].

It has been reported that Clearfil SE Bond produce good adhesion to dentin, with bond strength comparable to the three-step etch-and-rinse adhesive [19]. One possible explanation is that Clearfil SE Bond contains hydrophilic acidic monomer MDP (10-methacryloyloxydecyl dihydrogen phosphate) as the functional monomer MDP causes minimal dissolution of the smear plugs, which reduces dentin permeability and is capable of forming strong ionic bonds with calcium salts [20]. This contributes to the long term durability of the resin/dentin interface [21].

The minimal dissolution of the smear plugs at the tubular openings reduces dentin permeability through tubular pathways. In addition, it is able to demineralize and permeate into the sub surfaces of intact dentin below the smear layer. According to Kubo et al, the presence of two hydroxyl groups in the 10-MDP structure may facilitate its chelation with the calcium ions of dentin and enamel, and thus help reduce permeability [22]. Yoshida, et al. have also shown evidence of chemical bonding between MDP and hydroxyapatite structure [20].

The primer also contains hydrophilic dimethacrylate to promote wetting while the bonding agent has hydrophobic dimethacrylate and Bis-GMA to promote adhesion to the resin material. Previous studies have shown that these agents demonstrate good adhesion to both dentin and enamel. The One Up Bond F adhesive system uses a mixture of methacrylated phosphoric acid monomer and MAC-10 (a 10-carbon chain monomer with two carboxylic acid ends) to aggressively demineralize dentin [23].

One Up Bond F etches, primes and bonds simultaneously, so it is called an all-in-one adhesive system. These products produce smear layer dissolution, and dentin self-controlling demineralization by pH modifications during their application. Since the primer or primer/adhesive itself is the substance performing demineralization, it is understood that the entire demineralized dentin thickness will be filled with adhesive monomers [24].

The cured layer of a single-step adhesive might act as a permeable membrane that allows water diffusion from the dentin to the interaction zone between the adhesive and composite [25].

Single-step self-etching adhesives have hydrophilic low molecular weight resin monomers that can infiltrate relatively deeply into the etched tooth substrate. Water movement across the cured adhesive layer might occur in the presence of low-molecular-weight-resin monomers, allowing the diffusion of water from the hybridized dentin to the adhesive surfaces. Water diffusion into the bonded interface causes the resinous components to swell and become plasticized. Single-step self-etch systems have relatively thin interaction zone between dentin and adhesive, less than that for adhesive systems utilizing strong acid with a thicker interaction zone [26].

**Conclusion**

The two-step self-etching primer (Clearfil SE Bond) showed higher dentin shear bond strength than one step self-etching adhesive (One Up Bond F).

**Bibliography**

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