Effect of sodium hypochlorite gel on shear bond strength of one-bottle adhesive systems

Ana Karina Barbieri Bedran de Castro¹
Cristiane Mariote Amaral¹
Gláucia Maria Boni Ambrosano¹
Luiz André Freire Pimenta²
¹Graduate Students, Department of Restorative Dentistry, School of Dentistry of Piracicaba, University of Campinas (UNICAMP), Brazil.
²Associate Professor, Department of Restorative Dentistry, School of Dentistry of Piracicaba, University of Campinas (UNICAMP), Brazil.

Abstract

The aim of this study was to determine the effect of 10% NaOCl-gel on the dentin shear bond strengths (SBS) of three hydrophilic adhesive systems. One-hundred and twenty bovine incisors were mounted, polished with 320 to 600 - grit Al₂O₃ paper and randomly divided in 6 groups (n=15): G1 - Single Bond applied according to manufacturer’s instructions; G2 – 10% NaOCl gel + Single Bond; G3 - Prime & Bond 2.1 used according to manufacturer’s instructions; G4 - 10% NaOCl-gel + Prime & Bond 2.1; G5 - Gluma One Bond used according manufacturer’s instructions; G6- 10% NaOCl-gel + Gluma One Bond. In all groups, the dentin was etched, washed and lightly dried. After acid conditioning, groups 2, 4 and 6 received one drop of 10% NaOCl-gel, applied with a dwell time of 60 seconds, washed and blot dried. After the adhesive systems were applied and light-cured, a restorative composite resin was inserted in a teflon bipartite matrix and cured. The SBS were measured in a universal test machine at a cross-head speed of 0.5mm/min. The mean values were analyzed by two-way ANOVA and Tukey’s tests (± £0.01). The SBS values (expressed in decreasing order, MPa ± SD) were as follows: G6=14.71 ± 4.38; G1=14.26 ± 3.95; G3=12.94 ± 5.20; G2= 12.84± 5.06; G4=10.24 ± 2.04; G5=10.14 ± 2.77. Conclusion: Collagen removal significantly increased the SBS of Gluma One Bond, but did not affect the SBS values of the other adhesives. The influence of 10% NaOCl gel in bond strength values may be dependent on the adhesive systems applied.

Key Words:
adhesive systems, collagen removal, shear bond strength
**Introduction**

The integrity of the bond between dentin and resin adhesive systems has important implications for clinical dentistry in improving the success of composite resin restorations. Thus, several studies have been developed by manufacturers and researchers to find materials and innovative techniques for obtaining better adhesion and, consequently, success, in adhesive restorative treatment.

The presence of a rich organic collagen zone at the surface of conditioned dentin has been shown to be important for the interaction with hydrophilic adhesive resin, resulting in the formation of a "hybrid layer". However, it has been observed that poor infiltration of adhesive resin into the collagen-rich area of the demineralized dentin leaves gaps in the hybrid layer that, after long-term exposure to water, are vulnerable to degradation.

In 1994, Gwinnet observed that the collagen zone offered no direct, quantitative contribution to the interfacial bond strength. Since then, the treatment of the tooth structure with sodium hypochlorite to remove exposed collagen fibers from demineralized dentin, and the influence of the collagen rich demineralized zone on bond strength, has been studied. Improvement in adhesion to demineralized and deproteinized dentin has been related to the interaction with hydrophilic adhesive resin, resulting in the formation of a "hybrid layer". However, it has been observed that poor infiltration of adhesive resin into the collagen-rich area of the demineralized dentin leaves gaps in the hybrid layer that, after long-term exposure to water, are vulnerable to degradation.

Almost all studies evaluating the influence of sodium hypochlorite used it in solution form, but for a clinical application, as like phosphoric acid, the use of gel form would permit a controlled application. Therefore, this study was conducted to determine the effects of an experimental 10% NaOCl gel on the shear bond strength of three adhesive systems in dentin. The null hypothesis to be tested was that the treatment of dentin with NaOCl gel would not compromise dentin bonding.

**Material and Methods**

**Specimen preparation**

One hundred and twenty freshly extracted bovine incisor teeth were collected, cleaned and stored in 2% formaldehyde buffered solution. A piece from the coronal facial surface was sectioned in each tooth with a 3/4 inch diameter PVC ring, parallel to the base of the ring. The rings were then filled with self-curing polyester resin to obtain 5-6 mm areas of flat standardized middle dentin surfaces. Teeth were then stored in distilled water at 37°C.

**Bonding Procedure**

The specimens were randomly assigned to six groups (n = 20). Before the surface treatment, a 3mm circular area was left uncovered (as a bonding site) by placing a piece of vinyl tape with a 3 mm-diameter punched hole over the dentin. The adhesive systems used in this study, their batch numbers and components are described in Table 1. Composite resin TPH Spectrum (Dentsply De Trey Weybridge, UK) was used to complete the bonding procedure. The restoration of the specimens was performed following an arbitrary sequence. The groups received the following treatments:

**Group 1** – The dentin surface was etched with 37% phosphoric acid for 15s, rinsed with water for 15 seconds, and dried with absorbent paper. Next, two consecutive coats of Single Bond were applied, lightly air-dried for 2 seconds, and light cured (Optilux 500 Demetron-Kerr Corp., Danbury, CT, USA) for 10 seconds.

**Group 2** – The bonding procedure was the same as in Group 1, with the exception that after the phosphoric acid treatment, the surface was treated with one drop of 10% NaOCl gel (FGM, Joinville, SC, Brazil) which was applied with a dwell time of 60 seconds, rinsed with water for 30 seconds and dried with absorbent paper.

**Group 3** - The dentin surface was etched with 37% phosphoric acid for 15s, rinsed with water for 15 seconds, and dried with absorbent paper. Next, a coat of Prime & Bond 2.1 was applied, left to rest for 30 seconds, lightly air-dried for 2 seconds and light-cured for 10 seconds. Another coat of the adhesive was applied, lightly air-dried for 2 seconds and light-cured for 10 seconds.

**Group 4** - The bonding procedure was the same as in Group 3, with the exception that after the phosphoric acid treatment, the surface was treated with one drop of 10% NaOCl gel which was applied with a dwell time of 60 seconds, rinsed with water for 30 seconds and dried with absorbent paper.

**Group 5** - The dentin surface was etched with 37% phosphoric acid for 20s, rinsed with water for 15 seconds, and dried with absorbent paper. Next, two consecutive coats of the adhesive were applied, lightly air-dried for 5 seconds and light-cured for 20 seconds.

**Group 6** - The bonding procedure was the same as in Group 5 with the exception that, after the phosphoric acid treatment, the surface was treated with one drop of 10% NaOCl gel which was applied with a dwell time of 60 seconds, rinsed with water for 30 seconds and dried with absorbent paper. A 3-mm diameter and 5 mm high bipartite teflon ring mold was clamped to the dentin surfaces such that the mold was positioned over the treated dentin. The mold was filled (TPH Spectrum) and light-cured for 40 seconds, and then light-
cured again for an additional 40 seconds after removing the mold. The light intensity was measured periodically by a radiometer (Demetron/Kerr Corp. - Danbury, CT, USA.) and ranged from 550 to 580 mW/cm². The specimens were stored in distilled water, at 37°C, for 7 days.

**Bond strength test**

Each specimen was mounted in a custom apparatus attached to an universal testing machine (EMIC Ltda São José dos Pinhais, SP, Brazil.) with the dentin surface parallel to the machine’s trajectory. A compressive load was applied using a steel knife-edge placed over the specimens so that the force of the shear was applied directly on the bond interface. The specimens were loaded to fail at a crosshead speed of 0.5 mm/minute. Means and standard deviations were calculated with units expressed in MPa.

**Statistical Analysis**

The data were subjected to two-way analysis of variance (ANOVA). Multiple comparison Tukey’s test (α=0.01) was chosen to verify differences in means of possible interactions (adhesive system vs. treatment).

**Results**

The Two-Way ANOVA pointed a statistically significant interaction ($P_{value}=0.00113$) between the adhesive systems and treatment - with or without NaOCl gel. With regard to the factor adhesive system, no significant differences could be observed between the different treatments for the adhesives Single Bond and Prime & Bond 2.1 adhesives. The adhesive system, Gluma One Bond, demonstrated a significant increase in bond strength when 10% NaOCl gel was applied (The statistical differences are expressed in Table 2 by capital letters).

With regard to the factor treatment, differences were found among the adhesive systems applied without 10% NaOCl and after 10% NaOCl gel application (the statistical differences are expressed in Table 2 by small letters). When the adhesives were applied after the acid-etch conditioning, Single Bond presented statistically significantly higher values of bond strength than Gluma One Bond and Prime & Bond 2.1, no significant differences were observed between these latter two adhesives.

The association of 10% NaOCl gel treatment with the adhesive systems demonstrated statistically significant differences when Gluma One Bond was compared with Prime & Bond 2.1 and Single Bond, the gel treatment did not significantly affect the other two adhesives.

**Discussion**

Considerable improvement in dentin adhesion has occurred in recent years with the introduction of the “total etch” technique and hydrophilic primers. For a dentin adhesive system to be effective, the ability to thoroughly infiltrate the collagen network and partially demineralized zone is required to encapsulate the collagen and hydroxyapatite crystallites at the front of the demineralized dentin, and to produce a well-polymerized durable hybrid layer.

---

**Table 1. Characteristics and main components of the adhesive systems tested.**

<table>
<thead>
<tr>
<th>Adhesive systems</th>
<th>Manufacturer</th>
<th>Components</th>
<th>Batch no.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Single Bond</td>
<td>3M Dental Products St. Paul, MN, USA</td>
<td>Bis-GMA, HEMA, water, ethanol, polyalkenoic, acid copolymer</td>
<td>9CY</td>
</tr>
<tr>
<td>Prime &amp; Bond 2.1</td>
<td>Dentsply de Trey Weybridge, UK</td>
<td>PENTA, cetilamine hydrofluoric, elastomeric di-methacrylate resin, acetone, photoinitiators, estabilizers</td>
<td>49329</td>
</tr>
<tr>
<td>Gluma One Bond</td>
<td>Heraeus-Kulzer South Bend, IN, USA</td>
<td>4-META, acetone, photoinitiators</td>
<td>105623</td>
</tr>
</tbody>
</table>

**Table 2. Shear bond strength means values and Standard deviations (SD) in MPa.**

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Single Bond</th>
<th>Prime &amp; Bond 2.1</th>
<th>Gluma One Bond</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Mean</td>
<td>SD</td>
<td>Mean</td>
</tr>
<tr>
<td>Control</td>
<td>14.26 a A</td>
<td>3.95</td>
<td>12.94 ab A</td>
</tr>
<tr>
<td>NaOCl gel</td>
<td>12.84 ab A</td>
<td>5.06</td>
<td>10.24 b A</td>
</tr>
</tbody>
</table>

*Statistical differences are expressed by different small letters in rows, and by different capital letters in columns (α=0.01).*
When phosphoric acid is applied, the smear layer is removed, clearing the dentin tubules and collagen fibers, which is essential for the formation of the hybrid layer\textsuperscript{3,15-16}. However, over-drying the etched dentin surface could promote the collapse of collagen fibers\textsuperscript{17-18}, contributing to the incomplete penetration of the primer and adhesive, thus leaving the region of the dentin tubules not completely filled\textsuperscript{19} and vulnerable to degradation\textsuperscript{6,16-18}. Hydrolysis of these bands of exposed collagen unprotected by resin (“non-hybridized collagen”) could occur with long-term exposure to water, leading to deterioration of the adhesive between the resin and dentin, resulting in decreased bond strength\textsuperscript{4,10}.

Some authors have suggested that the hybrid layer might not be important for the mechanism of adhesion between bonding materials and dentin\textsuperscript{7,8}. An increase in adhesion to demineralized and deproteinized dentin tissue was related by Inai et al.\textsuperscript{2} in a study where the presence of micro-roughness on the dentin surface, exposed after collagen removal, was observed by SEM. In our study, two materials (Single Bond and Prime & Bond 2.1) did not present any statistically significant difference after NaOCl gel treatment compared with acid etch treatment alone. In contrast, the use of NaOCl gel in association with Gluma One Bond increased the bond strength values when compared to the control group. These findings do not agree with those of Perdigão et al.\textsuperscript{11} who observed a progressive decrease in shear bond strength for Single Bond and Prime & Bond NT when a drop of 10% NaOCl gel was applied. According to the manufacturer of NaOCl gel used in our study, this product is instable and a variation in the concentration of NaOCl in the gel could oscillate results. For reliable results, the manufacturer recommends the use of 10% NaOCl gel within 15 days of fabrication. Thus, care should be taken for adequate storage and use of NaOCl gel.

All current dental adhesive systems are designed to be hydrophilic, containing resin monomers dissolved in acetone, water, ethanol, or some combination of these solvents\textsuperscript{1}. It is considered that acetone and alcohol effectively displace water and therefore are better facilitators of resin primer infiltration into the collagen network in comparison to water-based adhesive systems\textsuperscript{19}. The results obtained in our study show that the acetone-based adhesive, Gluma One Bond, when applied after demineralization and deproteinization, showed increased SBS values when compared with the control group. However, Prime & Bond 2.1 which is also an acetone-based adhesive did not demonstrate difference in SBS when the dentin was demineralized and deproteinized. The correlation between the acetone-based systems and high values of bond strength on demineralized and deproteinized dentin demonstrated divergent results when compared with demineralized collagen exposed dentin, depending on the acetone-based adhesive employed\textsuperscript{2,9,11-13}. For some water-based and ethanol-based adhesives, a number of studies have reported decreases in bond strength but have also shown increases and no difference in strength when compared to a control group\textsuperscript{2,7,9-12}. Other factor that could interfere in adhesion is the hydrophilic monomer employed. The monomer 4-META has been shown to produce excellent adhesion when the dentin is pre-treated with 10% citric acid solution containing 3% ferric chloride in as opposed to citric acid or phosphoric acid\textsuperscript{5}. Ferric chloride has been shown to interfere with the collapse of the remineralized dentin\textsuperscript{1}. While the presence of a collapsed dentin would create a barrier for total dentin infiltration, it could be speculated that the high bond strength values achieved with 4-META based adhesive (Gluma One Bond) used on demineralized and deproteinized could be a result of the absence of collagen fibers, leading to better dentin penetration.

In view of previous studies and the current investigation it could be postulated that the demineralized and deproteinized bonding technique is extremely sensitive and that factors such as adhesive systems composition, depth (superficial vs. deep dentin) and type (human vs. bovine teeth) of dental substratum and the wettability of the dentin surface (wet vs. dry) are determinant factors that could influence adhesion. In conclusion, the use of NaOCl gel significantly increases the shear bond strength of the Gluma One Bond adhesive system.

The hydrophilic monomer, 4-META, could influence bond strength values depending on the surface treatment. Demineralization and deproteinization of dentin is a sensitive adhesive technique and could be influenced by various factors.

References