The ART of GIC proximal restorations in primary teeth
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Chapter 8

Microshear Bond Strength of Flowable GIC to Caries-Affected Dentin

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Submitted
Abstract

Aim: The purpose of this study was to evaluate the microshear bond strength (μSBS) of a high-viscosity glass-ionomer cement (GIC) using two different powder/liquid mixing ratio, to sound and caries-affected dentin.

Methods: Sixty bovine incisors were ground to obtain flat buccal dentinal surfaces and polished with 600 SiC paper for 60 s. Specimens were allocated into four groups (n = 15) according to substrate: sound and caries-affected dentin (pH-cycling for 14 days), and the powder/liquid ratio of the high-viscosity GIC: conventional (1:1) and flowable (1:2). Polyethylene tubes with internal diameter of 0.76 mm were placed over the pre-treated dentin and filled up with GIC (Ketac™ Molar Easy Mix, 3M ESPE). The μSBS test (1 mm/min) was performed after 24h of water storage at 37°C. Failure mode was evaluated with a stereomicroscope (X400). Data were analyzed with two-way ANOVA and Tukey’s post hoc tests (α=0.05).

Results: We observed no significant differences between conventional (5.3±1.6 MPa) and flowable (5.4±1.4 MPa) GIC (p=0.99), regardless of the substrate. Sound dentin (6.3±1.1 MPa) showed higher bond strength values than caries-affected dentin (4.4±1.0 MPa) (p<0.0001). For all groups, adhesive/mixed failure prevailed.

Conclusion: The powder/liquid ratio does not affect the μSBS, and caries-affected dentin leads to lower μSBS than sound dentin.
Introduction

The actual scenario of minimal intervention dentistry is characterized by a better understanding of the caries process and less invasive treatments (1, 2). One of the most investigated minimal intervention techniques in the last decades is the Atraumatic Restorative Treatment (ART). It is based on caries removal using only hand instruments and restoring the cavity with an adhesive restorative material (3).

Glass-ionomer cement (GIC) is the material of choice for ART because of its properties, such as ability to chemically bond to enamel and dentin, biocompatibility with dental and periodontal tissues, fluoride release and uptake, and a similar coefficient of thermal expansion to tooth structure (4). Despite their satisfactory results in occlusal-ART restorations (5-10), the same results were not reported in proximal-ART restorations (11-14).

GIC presents some adverse mechanical properties as a relative low fracture strength and high occlusal wear rate in comparison to amalgam and resin composite (15, 16). To improve their clinical performance up to the level that they could be used as a permanent restoration in posterior teeth, the high-viscosity or condensable GICs were developed for ART, with better mechanical properties by increasing the powder/liquid ratio (17, 18). However, the high-viscosity of the material hampers its handling, insertion into the cavity and adaptation to the cavity walls. The thick consistency of the material usually leads to air bubble inclusion during the insertion of the material into the cavity (19). The consistency may also contribute to an incorrect adaptation to the tooth surface, reducing the strength of the material and making its adaptation at the cervical margin more difficult (20-24).

Previous studies have suggested the use of a low viscous or flowable layer of GIC before the insertion of a conventional layer of high-viscous GIC (two-layer technique) to minimize the adverse effects related to margin adaptation. Recent research showed that the two-layer technique presented less microleakage and improved adaptation to the cavity walls in proximal cavities compared to the conventional one-layer technique (25). Furthermore, higher microtensile bond strength values were observed for the two-layer technique when applied to sound compared to caries-affected dentin (26). However, since GICs are brittle materials and tend to have a higher number of cohesive fractures in a microtensile bond strength test than in a microshear bond strength test, their reported bonding effectiveness may be more dependent on the mechanical test itself than on their actual interactions with the substrate (27). Once caries-affected dentin is the predominant substrate in cavities prepared according to ART, it is, therefore, relevant to evaluate whether a flowable GIC would have any positive influence in its bond strength, in particular to caries affected-dentin and by microshear bond strength test. We investigated the microshear bond strength of a flowable and a conventional GIC to sound and caries-affected dentin. Our null-hypothesis was that there is no difference in microshear bond strength of a flowable and a conventional GIC bonded to sound and caries-affected dentin.

Materials and methods

Teeth Selection and Preparation

Sixty freshly extracted bovine incisors were stored in 0.5% aqueous chloramine at 4°C. The root portion of each tooth was removed using a low speed water-cooled diamond
saw in a cutting machine (Labcut 1010, Extec Co, Enfield, CT, USA) and the crowns were embedded in self-curing acrylic resin (JET Clássico®, São Paulo, BR). The buccal surfaces were ground with 120-grit SiC paper until a flat dentin surface was obtained. Dentin surfaces were polished with 600-grit SiC paper for 60 s, under running water, to standardize the smear layer.

The teeth were randomly assigned to four experimental groups according to the experimental groups, based on: substrate - sound or caries-affected dentin; and GIC powder/liquid mixing ratio - conventional GIC (1:1) and flowable GIC (1:2). This resulted in a 2 x 2 factorial experimental design with 15 teeth in each subgroup. Composition, batch number and manufacturer’s instructions of the GIC are summarized in Table 1.

Artificial caries induction

Thirty teeth were submitted to cariogenic challenge by pH cycling. The cervical portion was sealed with epoxy resin (Araldit Hobby, Ciba Especialidades Químicas Ltda, São Paulo, BR) and received two layers of acid-resistant nail polish (Colorama Maybelline Ltda, São Paulo, BR). The teeth were individually submitted to 14 cycles of 8 hours in 10 mL of demineralizing solution (2.2 mM CaCl₂, 2.2 mM NaH₂PO₄, 0.05 M acetic acid adjusted to pH 4.8 with 1 M KOH) and 16 hours in 10 mL of remineralizing solution (1.5 mM CaCl₂, 0.9 mM NaH₂PO₄ and 0.15 mM KCl adjusted to pH 7.0) at room temperature and without agitation (28). The solutions were changed every cycle and at each interval the teeth were washed with deionized water, and dried with absorbent paper.

Bonding and Restorative Procedures

The surface pre-treatment was done with the diluted liquid of the GIC (29). Polyethylene tubes (Micro-bore® Tygon S-54-HL Medical Tubing, Saint-Gobain Performance Plastics, Akron, OH, USA) with an internal diameter of 0.76 mm and 1.0 mm height were placed on the bonded surface, filled up with GIC, covered with a matrix strip and gently pressed with a glass slide. Conventional GIC groups were mixed according to manufacturer instructions (Table 1), and flowable GIC groups were obtained from the mixing of two liquid drops to one powder portion. A thin layer of petroleum jelly was applied to the exposed GIC surface to avoid water uptake and loss. The bonding and restorative procedures were carried out by a previously trained operator at room temperature.

After 24 hours storage in distilled water at 37°C, the polyethylene tubes were removed by cutting it into two hemi-cylinders using a surgical blade, resulting in cylindrical specimens with a cross-sectional area of approximately 0.45 mm². Specimens were examined using a stereomicroscope at X10 magnification (Discovery V20, Zeiss, Berlin, GE) for interfacial defects. Specimens with interfacial gaps, bubble inclusion or other defects were excluded from the test and replaced.

Microshear bond strength test (µSBS)

One by one, the specimens were attached to a universal testing machine (Kratos Industrial Equipment, Cotia, BR). A thin steel wire (0.20 mm diameter) was looped flush between the load cell projection and the GIC cylinder making contact with the lower half-circumference of the cylinder and touching the dentin surface. A shear load was applied at a crosshead speed of 1.0 mm/min, until failure occurred. The cylinder was kept in line with the center of the load cell and the wire loop was parallel to the load cell movement direction and to the bonding interface. The fracture load was recorded and the bond strength was expressed in MPa.
**Failure mode**

The failure mode of debonded specimens was determined at X400 magnification using a stereomicroscope and classified as adhesive/mixed failure (presence of dentin or GIC adjacent to interface) or cohesive (failure in dentin or GIC).

<table>
<thead>
<tr>
<th>Table 1 – GIC; characteristics, general composition, manufacturer, batch number and manufacturer’s instructions</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Ketac Molar Easy Mix</strong></td>
</tr>
<tr>
<td>Characteristics</td>
</tr>
<tr>
<td>High-viscosity glass-ionomer cement</td>
</tr>
<tr>
<td>Composition</td>
</tr>
<tr>
<td>Powder: Al-Ca-La fluorsilicate glass, 5% copolymer acid (acrylic and maleic acid)</td>
</tr>
<tr>
<td>Liquid: Polyalkenoic acid, tartaric acid, water</td>
</tr>
<tr>
<td>Manufacturer</td>
</tr>
<tr>
<td>3M/ESPE</td>
</tr>
<tr>
<td>Seeefeld, GE</td>
</tr>
<tr>
<td>Batch Number</td>
</tr>
<tr>
<td>386216</td>
</tr>
<tr>
<td>Manufacturer’s instructions</td>
</tr>
<tr>
<td>Conditioning of the substrate:</td>
</tr>
<tr>
<td>- Apply the liquid for 10 s.</td>
</tr>
<tr>
<td>- Rinse with copious amounts of water.</td>
</tr>
<tr>
<td>- Blot excess water with cotton pellet.</td>
</tr>
<tr>
<td>Dosing:</td>
</tr>
<tr>
<td>- 1 spoonful with leveled powder surface;</td>
</tr>
<tr>
<td>- 1 drop of liquid.</td>
</tr>
</tbody>
</table>

**Statistical analysis**

The experimental unit in the current study was the tooth. Thus, the mean of μSBS values of all specimens from same tooth were averaged for statistical analysis. Normal distribution of bond strength data and equality of variances were assumed after Kolmogorov-Smirnov and Levene’s tests, respectively. The μSBS means were analyzed with two-way ANOVA and Tukey post hoc test. The significance level was set at \( p<0.05 \). All statistical analysis were performed using SPSS V18 (SPSS Inc., Chicago, IL, USA) for Windows.

**Results**

Microshear bond strength means (MPa) and standard deviations for all experimental groups are summarized in Table 2. Conventional GIC showed similar microshear bond strength values to the ones obtained by the flowable GIC \( (p=0.99; F=0.022) \), regardless to substrate. The factor substrate was statistically significant \( (p<0.0001; F=50.514) \). Sound dentin showed higher microshear bond strength values than caries-affected dentin. The interaction between these two factors revealed no significance \( (p=0.646; F=0.213) \), indicating that the bond strength values of GIC were not dependent upon the substrate.
Microshear Bond Strength of Flowable GIC to Caries-Affected Dentin

Table 2 - Microshear bond strength means (MPa) and standard deviations for all experimental groups.

<table>
<thead>
<tr>
<th>Powder/liquid ratio GIC</th>
<th>Sound dentin</th>
<th>Caries-affected dentin</th>
</tr>
</thead>
<tbody>
<tr>
<td>Conventional GIC</td>
<td>6.4(0.9)^a,A</td>
<td>4.3(0.9)^a,A</td>
</tr>
<tr>
<td>Flowable GIC</td>
<td>6.3(1.3)^a,A</td>
<td>4.5(1.0)^a,A</td>
</tr>
</tbody>
</table>

*Different lowercases indicate statistically significant difference between the columns (substrate).
**Equal uppercases indicate no statistically significant difference between the rows (GIC) (p<0.05).

The percentages of failure mode and pre-testing failures observed are showed in Table 3. There was a predominance of adhesive/mixed failures, whereas no cohesive failures in dentin were verified.

Table 3 - Failures mode and pre-testing failures for experimental groups (%).

<table>
<thead>
<tr>
<th>Adhesive/mixed</th>
<th>Cohesive in GIC</th>
<th>Pre-testing failures</th>
</tr>
</thead>
<tbody>
<tr>
<td>Conventional GIC to sound dentin</td>
<td>67 (29)</td>
<td>5 (2)</td>
</tr>
<tr>
<td>Flowable GIC to sound dentin</td>
<td>80 (35)</td>
<td>7 (3)</td>
</tr>
<tr>
<td>Conventional GIC to caries-affected dentin</td>
<td>70 (31)</td>
<td>9 (4)</td>
</tr>
<tr>
<td>Flowable GIC to caries-affected dentin</td>
<td>67 (28)</td>
<td>7 (3)</td>
</tr>
</tbody>
</table>

Discussion

In this study we investigated the microshear bond strength of a flowable and a conventional GIC to sound and caries-affected dentin. We found similar performance for the flowable GIC and the conventional GIC, regardless the substrate. Although the change in powder/liquid ratio may modify other mechanical properties of the material, it seems not to affect the bond strength to dentin. The null-hypothesis was, therefore, rejected.

We observed that both conventional and flowable GIC were influenced by the substrate condition, with lower bond strength values for caries-affected dentin. This substrate presents some difference in morphological and chemical characteristics such as the higher degree of porosity, which can result in minor mineral content and might lead to a deeper demineralization layer (30, 31). This demineralized zone might not be fully infiltrated by the GIC, resulting in exposed collagen fibers in the hybrid layer and reduction of the bond strength. Caries-affected dentin also contains calcium-phosphate crystals, which are less soluble to acid conditioner than sound dentin. This less soluble structure is likely to...
require stronger acids to solubilize the mineral phase and, consequently, an adequate material infiltration associated to high bond strength is more difficult to be obtained in caries-affected dentin (32, 33).

A previous study (25) showed that a low viscous layer of GIC inserted prior to a conventional layer reduces the microleakage and improves adaptation to the cavity walls in proximal cavities in primary teeth. According to these results, it was speculated that a higher number of cross-links and a better wettability could be obtained with flowable GIC, resulting in a better adhesion to tooth structures. Since for the flowable GIC we used two drops of the liquid, the higher polyacrylic acid concentration could result in more substrate demineralization. As a consequence, a better micromechanical bonding by infiltration of the fluid GIC could be obtained, which should contribute to the longevity of the restorative procedure. Our results do not confirm this hypothesis, as we did not find difference in bond strength between the flowable and the conventional GIC.

Lenzi et al. (26) evaluated the microtensile bond strength of another high-viscosity GIC (Fuji IX) and found no difference between flowable and conventional viscosities, which corroborates with our results. Nevertheless, they observed a better performance of the flowable GIC in sound than in caries-affected dentin, diverging from our results, as we found that the µSBS values of the two-layer GIC were independent of the substrate. They also observed more premature failures in caries-affected dentin, while we found similar distribution of pre-testing failures among all groups. The difference observed between these two studies might be attributed to the materials composition or to the design of test performed (27).

Literature reports that, since contemporary materials have high bond strength values, "microtests" are more accurate than "macrotests", as shear and tensile bond strength tests (34, 35). In the case of brittle materials like the GICs, the microtensile test may not be the most appropriate test as specimens confection can create micro-cracks on the material. These cracks may propagate and lead to cohesive fracture in low loading values.

Bonifácio et al. (27) evaluated the adhesion of GICs to sound dentin with microshear and microtensile bond strength test and observed that the different tests resulted in different material’s ranking and different failure patterns. The microshear bond strength test showed predominance of adhesive/mixed failures, which is a desirable failure in bond strength tests as it may measure more accurately the strength in the adhesive interface (36). Accordingly, it seems to be reliable to use this method to evaluate the bond strength of GICs.

We used bovine dentin for bond strength evaluation, since it has been used as a substitute of human teeth in several research evaluating bonding mechanisms (37-39). Despite some anatomical differences, evidence in literature showed similar micromorphology’s characteristics between human and bovine teeth such as number and diameter of tubules per mm², and collagen matrix, confirming the validity of the use of bovine teeth to adhesion studies (37, 40, 41).

Within the limitations of this laboratory study, we can conclude that the presence of caries-affected dentin result in lower bond strength values than sound dentin, and that the powder/liquid ratio has no effect on the microshear bond strength. A flowable layer of GIC employed before the use of a conventional layer could be an alternative to ART that does not jeopardize the bonding to dentin. Further studies could complement the current comprehension of the behavior of a flowable GIC layer in ART treatments. In order to
investigate whether this more fluid layer of GIC can lead to different interface degradation over time, long-term evaluations may be, specially, desired. The drawbacks of the two-layer technique as higher material costs and a more time consuming technique should be also considered.
References


