Micro-shear Bond Strength of Composite Resin to Glass Ionomer Cement Using an Alternative Method to Build up Test Specimens

Abstract

Context: Despite the relevance of the sandwich technique, there are still doubts about the best adhesive strategy and surface treatment for glass ionomer cements (GICs). Aim: The aim of this study is to evaluate the best surface treatment for GIC to ensure an effective and durable adhesion to resin, through micro-shear test, using an alternative method to build up test specimens. Subjects and Methods: Eighty GIC samples were divided into eight groups (n = 10) according to five surface treatments (none, etching, air drying, grinding, and grinding plus etching) and according to the adhesive system (conventional or self-etch). Five starch tubes were positioned on each sample, and a flowable composite was inserted generating 50 resin test bodies per group and a total of 400 tested areas. All specimens were submitted to the micro-shear test: half immediately and half after thermal cycling (10,000 cycles of 20 s each/5°C and 55°C). All samples were analyzed to evaluate fracture. Representative samples were also analyzed by scanning electron microscopy and energy dispersive spectroscopy. Data were analyzed with two-way ANOVA and Tukey’s honest significant difference post hoc test (P <.05). Results: The bond strengths in the thermal cycled specimens were lower and showed a statistically significant difference (P = 0). The “grinding” groups showed the highest bond strength. Conclusions: The alternative method to build up test specimens was effective and easy to execute. Grinding of the GIC surface, which is not normally performed before the use of the adhesive system, represented the best option of surface treatment.

Keywords: Adhesives, composite resins, glass ionomer cements, shear strength

Introduction

Glass ionomer cement (GIC) is systematically studied with constant evolution. Currently, some studies have combined the GIC with other materials (as bioactive glass) to achieve better properties. [1] New GICs with a high resistance used in pediatric dentistry and atrumatic restorative treatment present important improvements with regard to mechanical strength and setting time. The GIC has advantageous properties that are not achieved by other materials and are useful for resolving some challenges that the adhesive technique with composite resin still presents. Among these, properties include the adherence to dental tissues without the need of the tissue hybridization step, high biocompatibility, fluoride release, coefficients of thermal expansion and contraction similar to dentin, low shrinkage, as well as the advantages of having an acceptable cost and single insertion into the cavity. [2-4]

Among the many applications of the material in the oral cavity, the GIC can be used alongside resin composites in restorations known as the “sandwich.” The combined use of GIC and composite resin is widely indicated in dental clinical procedures. The technique has been documented in the literature for 30 years. However, certain questions about the best adhesive technique to use to adhere resin to the GIC still remain, and new technical variations emerged with the advent of self-etching adhesives. According to Li et al., [5] the bond strength between the two materials is limited by the low cohesive strength of the GIC and also by the minimal chemical bond between them due to the different chemical reactions that each one undergoes.

Different forms of treatment of the GIC surface before the insertion of the resin material at the same visit (immediate sandwich) were suggested in the literature to increase the bond strength between the materials. However, despite the frequent

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use of the sandwich technique, Kasraie et al.\(^{[6]}\) state that over the past three decades, few studies have evaluated the adhesion between the GIC and composites using different adhesive systems.

Based on this, it is necessary to evaluate the best surface treatment of the GIC to ensure an effective and durable adhesion with composite resins. Until now, however, no study evaluated all the treatment strategies available in the literature, varying the most current adhesive systems and using simulated aging to predict the clinical effectiveness of a restoration.

Thus, the aim of this study was to mechanically evaluate the influence of different adhesive strategies on the GIC samples before insertion of the composite resin. The micro-shear test was the method used to evaluate the bond strength between the materials in two stages: immediately and after aging by thermal cycling.

### Subjects and Methods

The GIC (Ionofil Plus, VOCO GmbH, Cuxhaven, Germany) was manipulated, inserted into acrylic artificial cavities (0.2 cm depth × 1 height × 1 cm width), and pressed with a Mylar strip and a glass slide to protect the material and ensure a smooth surface. The setting time of the material (6 min) was respected before the surface treatment.

Then, the specimens were divided according to the different treatment of the GIC surface and according to the adhesive’s system used. Eighty samples of GIC were divided into eight treatment groups \((n = 10)\) as listed in Table 1.

A modified technique was used to build resin specimens for micro-shear testing [Figure 1]. After the surface treatment and before the adhesive application, five starch tubes (internal diameter of 1.25 mm, external diameter of

| Groups | Surface treatment | Steps of surface treatment |
|--------|-------------------|-----------------------------|
| CA     | Conventional adhesive (One Coat Bond SL, Coltène, Altstätten, Switzerland) | Adhesive application - 20 s and light cure - 30 s |
| SE     | Single-step self-etching adhesive (Single Bond Universal, 3M ESPE, Seefeld, Germany) | Adhesive application - 20 s and light cure - 10 s |
| ACA    | Air drying + conventional adhesive | Air blast - 30 s - 40 psi Adhesive application - 20 s and light cure - 30 s |
| ASE    | Air drying + single-step self-etching adhesive | Air blast - 30 s - 40 psi Adhesive application - 20 s and light cure - 10 s |
| ECA    | Acid etching + conventional adhesive | 37% phosphoric acid - 30 s, wash 60 s, and dry with absorbent paper Adhesive application - 20 s and light cure - 30 s |
| GCA    | Grinding + conventional adhesive | Cylindrical diamond point (PM61G) at low speed for 15 s Adhesive application - 20 s and light cure - 30 s |
| GSE    | Grinding + single-step self-etching adhesive | Cylindrical diamond point (PM61G) at low speed for 15 s Adhesive application - 20 s and light cure - 30 s |
| GECA   | Grinding + acid etching + conventional adhesive | Cylindrical diamond point (PM61G) at low speed for 15 s 37% phosphoric acid - 30 s, wash - 60 s, and dry with absorbent paper Adhesive application - 20 s and light cure - 30 s |

![Figure 1: Technique used to build resin specimens for micro-shear testing in GECA and GECAT group. (a) Glass ionomer cement was inserted into acrylic artificial cavities; (b) grinding the glass ionomer cement surface with cylindrical diamond point; (c) acid etching with 37% phosphoric acid in all glass ionomer cement surface; (d) starch tube stabilized in position with a gingival barrier; (e) adhesive application within each starch tube followed by insertion of the high-flow resin composite; (f) starch tubes softened by the moisture were easily removed with a manual instrument](image)

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Ramos, et al.: Micro-shear bond strength of composite resin to GIC

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3 mm, and 2.0 mm in height) were positioned on each sample according to a previous area selection. These tubes delimited the adhesive interface’s area and worked as conformers for insertion of the high-flow resin composite. They were stabilized in position with a gingival barrier (TopDam, FGM, Joinville, SC, Brazil). The flowable composite (Natural Flow, New DFL, Rio de Janeiro, RJ, Brazil) was inserted within each starch tube and light-cured for 40 s. Five resin specimens were prepared on each GIC sample, generating 50 per group and a total of 400 tested areas.

The samples were stored in distilled water at room temperature for 48 h. The starch tubes softened by the moisture and could be easily removed with a manual instrument.

Half of the specimens in each group were submitted to a micro-shear test in a universal testing machine (EMIC DL 500, Curitiba, Paraná, Brazil) with a 50 kgf load cell at a crosshead speed of 0.5 mm/min. The other half was subjected to aging in a thermal cycler (OMC200b, Odeme, Luzerna, SC, Brazil) (10,000 cycles in 5°C and 55°C water baths with 20 s dwell times were used to simulate a period of 1 year) and then the micro-shear test. Thus, eight groups were divided into 16 groups, half immediate and the other half thermal cycled.

All samples were analyzed in the stereomicroscope (Mitutoyo MSM-414 L, Sul Americana, Suzano, SP, Brazil) at ×10 magnification to evaluate the interfacial fracture and classified as adhesive, cohesive, or mixed.

To validate this analysis, representative samples of each group were covered with gold and observed in a scanning electron microscope (SEM-HITACHI TM3030Plus Tabletop Microscope, Tokyo, Japan) and analyzed by energy dispersive spectroscopy (EDS-HITACHI TM3030Plus Tabletop Microscope, Tokyo, Japan) to determine the composition of the materials.

Two-way ANOVA followed by Tukey’s honest significant difference post hoc test for parametric comparison of means was used to compare the means, with a significance level of 5% (P < 0.05). Statistical analysis was performed using SPSS 22 (IBM SPSS Statistics 22, IBM Corp., Armonk, NY, USA).

Results

Micro-shear bond strength (mean and standard deviation) was calculated and analyzed statistically [Table 2]. The bond strength values in the thermal cycled stage groups were lower and showed a statistically significant difference from the immediate stage groups (P = 0). The mean bond strength of 9.02 MPa in the immediate stage group dropped to 4.49 MPa in the thermal cycled stage group.

Regarding the fracture, there were more cohesive failures at the immediate stage in groups that had higher bond strength values. However, after thermal cycling, a larger number of adhesive fractures were detected in all groups. Mixed fractures were not prevalent. These results may be best observed in Figure 2.

For SEM and EDS analysis, two samples were prepared for materials’ characterization: one containing only GIC [Figure 3a] and one in which the adhesive system was applied on the GIC [Figure 3b]. On a surface with an adhesive fracture, the EDS analysis denoted similar composition to the surface of the GIC. The adhesive fracture and GIC images were also very similar [Figure 4a].

Table 2: Mean and standard deviation of micro-shear bond strength represented in MPa. The groups are divided according to the surface treatment of glass ionomer cements and the stage (immediate and thermal cycled)

| Immediate stage | CA     | SE     | ACA    | ASE    | ECA    | GCA    | GSE    | GECA   |
|-----------------|--------|--------|--------|--------|--------|--------|--------|--------|
| Mean (SD)       | 7.30 (1.67) | 8.45 (2.66) | 9.52 (2.35) | 9.75 (2.90) | 8.87 (2.96) | 9.30 (2.70) | 9.70 (2.55) | 9.28 (2.72) |
| Statistical difference | a, g | a, b | b | b | a, b | b | b | b |
| Thermal cycled stage | CAT   | SET   | ACAT  | ASET  | ECAT  | GCAT  | GSET  | GECAT  |
| Mean (SD)       | 2.65 (0.35) | 4.01 (0.81) | 3.11 (0.75) | 3.22 (0.53) | 4.61 (1.16) | 5.84 (1.11) | 5.33 (1.02) | 7.13 (1.31) |
| Statistical difference | c | c, d, e | c, d | c, d | d, e | e, f, g | e, f | a, f, g |

Tukey post hoc test for parametric comparison of means identified the homogeneous subsets. Different letters indicate statistical difference (P<0.05). SD=Standard deviation
This indicates that the fracture occurred between the adhesive and the GIC, and the adhesive was detached after the test. On a surface with a cohesive failure, a fracture in the GIC body was observed, exposing particles of glass and matrix and a fracture in the resin body [Figure 4b]. The EDS analysis for the chemical characterization of the components was done delimiting the two materials, and the chemical composition was consistent. On a surface with a mixed fracture, the EDS analysis showed the presence of chemical elements of both materials [Figure 4c].

EDS analysis on the GIC surface showed high indexes of silicon (Si), aluminum (Al), and fluorine (F), which is typical of this material [Figure 5]. On a GIC surface covered by adhesive, it was possible to observe an increase in the percentage of carbon (C), characteristic of a resinous polymer, and a lower percentage of aluminum and fluorine.

**Discussion**

Treatment of a GIC surface after setting and before placing resin in sandwich restorations is still a topic that requires research.[4, 7-9] Various treatments have been proposed in the literature to increase the mechanical retention between the materials.

In the present study, two adhesive systems were tested (two-step etch-and-rinse and one-step self-etching) in different situations of surface treatment and also for isolated application. Overall, the self-etching adhesive showed slightly better performance although statistically, this difference was not significant.

The acid etching of the GIC surface showed a bond strength value fairly similar to the group in which only self-etching adhesive was applied. This result suggests that the use of a self-etching adhesive can be a substitute for the etching procedure, reducing the clinical steps, and eliminating the washing step of the GIC. However, after statistical analysis, this difference was not significant. Therefore, the two procedures could be used effectively, according to the study of Pamir et al.[4]

In the air-drying group, the bond strength values were high (immediate stage) and similar to those found in the grinding group. The same happens in the study of Subrata and Davidson,[10] in which the shear strength values were similar in the groups with air drying, surface grinding, and phosphoric acid etching. However, after thermal cycling, the resistance decreased significantly. In the literature, there are no reports of samples which have undergone air drying and were then thermally cycled simulating the aging process. Thus, it is suggested that thermal changes, contact with moisture, and differences between coefficients of thermal expansion of the materials can change the dimensional stability and reduce their bond strength. It is necessary to remember that thermal cycling presents intrinsic limitations simulating the clinical reality. This is particularly relevant when considering the proportion of the interfacial area of exposed specimens during thermal cycling and of the interfacial area in a real clinical situation with a conventional restoration.

Mangum et al.[11] claim that the bond is enhanced by surface roughness and that the use of an instrument (diamond point) produces more roughness than etching a smooth surface. This statement was confirmed in this study. In the two different stages (immediate and thermal cycled) evaluated, the bond strength values were higher in the grinding group compared to the etching group. In the current study, it was observed that the groups that had a surface treatment that created greater surface roughness obtained the highest results compared to the groups that only had the adhesive system applied. This fact agrees with the findings of the study of Subrata and Davidson,[10] which claim that the acid etching, the grinding, and the air drying of the GIC surface have a significant effect on bond strength.

Commonly, a polyethylene tube is used as a former for insertion of the composite resin. However, removal of the
The results of this study suggest studying variations in the methodology for the use of the polyethylene tube and its effect on the bond strength. Based on this, the present study has relied on a new methodology that uses starch tubes as a matrix instead of polyethylene tubes. First, all surface treatment steps are performed and then the tube is positioned and fixed. Second, the adhesive system was applied only within the internal diameter of the tube. The starch tube is easily removed because it softens after some hours of immersion in water. The removal can be done with a manual instrument without generating stress in the adhesive interface. This fact is extremely important to not generate interference in the bond strength values. Tedesco et al. also used a starch tube as a former to insert the resin composite. In the modified technique employed, the tube has been used to delimit the area of application of adhesive. This prevents the occurrence of an adhesive surface extending beyond the composite resin specimen diameter, which could lead to measurements changed in micro-shear assay.

Scherrer et al. affirm that care should be taken when interpreting the failure mode. In the present study, representative samples were also observed under SEM to validate the analysis of the fracture. Furthermore, analysis of the chemical composition of the substrates was done using EDS to differentiate the materials that were present in the fractured surface, as was done in the study of Kandaswamy et al. Scherrer et al. suggest studying variations in the methodology for the use of the polyethylene tube and its effect on the bond strength. Based on this, the present study has relied on a new methodology that uses starch tubes as a matrix instead of polyethylene tubes. First, all surface treatment steps are performed and then the tube is positioned and fixed. Second, the adhesive system was applied only within the internal diameter of the tube. The starch tube is easily removed because it softens after some hours of immersion in water. The removal can be done with a manual instrument without generating stress in the adhesive interface. This fact is extremely important to not generate interference in the bond strength values. Tedesco et al. also used a starch tube as a former to insert the resin composite. In the modified technique employed, the tube has been used to delimit the area of application of adhesive. This prevents the occurrence of an adhesive surface extending beyond the composite resin specimen diameter, which could lead to measurements changed in micro-shear assay.

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