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Microtensile bond strength of composite resin and glass ionomer cement with total-etching or self-etching universal adhesive

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ABSTRACT

Purpose: To assess the effect of acid etching (AE) and adhesive systems on the microtensile bond strength of conventional glass ionomer cement (GIC) and a nanofilled composite resin.

Materials and methods: Specimens of conventional GIC (RIVA, Self-cure, SDI) were prepared in a bipartite Teflon mold and randomly assigned (n = 12) to **G1**- GIC + Single Bond 2 (SB2) (3M-ESPE); **G2**- GIC + Acid etching (AE) (37% phosphoric acid, Condac, FGM) + SB2; **G3-** GIC + Single Bond Universal (SBU) (3M-ESPE); and **G4**- GIC + AE + SBU. The adhesive systems and the composite (Filtek Z350XT, 3M-ESPE) were inserted into the mold. After 7 days stored in a humid environment, the specimens were sectioned into five slices (Isomet 1000, Buehler). Hourglass slices were trimmed and subjected to microtensile bond strength testing (BISCO®; Schaumburg, USA) with 0.5 mm/min crosshead speed. Data were analyzed by two-way ANOVA and the Tukey test (SPSS 17.0, $\alpha = 5\%$).

Results: The microtensile bond strength (MPa) means (standard deviation) were G1 = 9.46(3.79), G2 = 6.27(3.21), G3 = 9.35(3.91), and G4 = 10.13(3.53). G2 differed significantly from the other groups ($p < 0.001$). G1, G3 and G4 were not significantly different from each other ($p > 0.05$). There were 83% mixed fractures, 9.5% cohesive and 7.5% adhesive.

Conclusion: GIC etching promoted higher microtensile bond strength with universal adhesive than with a total-etch adhesive system. Acid etching is not necessary to enhance the universal adhesive bond strength and negatively affected the bond strength of the total-etch adhesive system. Without etching the GIC, there is no difference in microtensile bond strength between the adhesive systems.

1. Introduction

Several restorative techniques have been suggested to reduce the effects of the polymerization shrinkage stress of composite resins (CR). The sandwich restorative technique is an alternative choice to reduce marginal microleakage and secondary caries and to prevent the clinical failure of composite restorations. A substantial part of CR is replaced with glass ionomer cement (GIC) [10] that chemically bonds to enamel and dentin; exhibits thermal expansion similar to hard dental tissues, a low elastic modulus, and biocompatibility; and releases fluoride [10,16,19].

The microshear bond strength is higher for the self-etch adhesive [2], but other findings show no significant difference between a total-etch 2-step adhesive and a one-step self-etch adhesive [13]. Regarding the GIC type, the bond strength of resin modified glass ionomer cements (RMGICs) is higher than conventional GIC, and it can be enhanced with 30 s acid etching [13]; but the bond strength of CR and GIC is significantly higher for self-etch primer employed on unset GIC [7,8].

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[2] Performed the microtensile bond strength testing and fracture mode assessment, contributed substantially to discussion.
[3] Performed the statistical tests, wrote the paper, contributed substantially to discussion.
[4] Contributed substantially to experimental design and discussion, proofread the manuscript
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Single Bond Universal, similar to other self-etch adhesives, has acidic monomers that simultaneously condition and prime the dental substrate. Methacryloxydecyl phosphate (MDP) is the functional monomer that chemically bonds to the calcium of hydroxyapatite (Hap), forming calcium phosphate, thus reducing the technique sensitivity and steps in clinical performance. It seems to be a good option for the immediate sandwich technique, as it is not necessary to rinse. Mild self-etch adhesives and GIC interact only superficially with the enamel and dentin and hardly dissolve Hap crystals, but rather keep them in place within a thin submicron hybrid layer. 10-MDP bonds through its phosphate group to Hap and forms a regularly Ca-monomer nano-layered structure on the Hap surface [17].

This study aims to assess the effect of acid etching, total and self-etch adhesive systems on the microtensile bond strength between a conventional encapsulated GIC and a composite resin. The null hypothesis tested was that the adhesive type and etching do not affect the microtensile bond strength of composite resin and glass ionomer cement.

2. Materials and methods

2.1. Experimental design

This in vitro study was designed in randomized complete blocks with 12 specimens in each group. The independent variables were the adhesive system (Adper Single Bond 2 and Single Bond Universal) and the acid etching (with or without). The response variable was the microtensile bond strength in MPa.

Forty-eight initial specimens were prepared and randomly divided into four groups ($n = 12$):

Group 1: GIC RIVA encapsulated self-cure + Adper Single Bond 2.

Group 2: GIC RIVA encapsulated self-cure + Acid etching (10 s) + Adper Single Bond 2.

Group 3: GIC RIVA encapsulated self-cure + Single Bond Universal.

Group 4: GIC RIVA encapsulated self-cure + Acid etching (10 s) + Single Bond Universal.

The specimens were prepared using a bipartite Teflon mold containing compartments for the conventional GIC (6.0 mm width, 2.0 mm height, 7.0 mm length) and composite (6.0 mm width, 6.0 mm height, 7.0 mm length) insertion.

The self-cure RIVA GIC capsules were manipulated according to manufacturer instructions for 10 seconds on the Ultramat 2 (SDI Limited, Bayswater, Australia) device. The capsule content was inserted in the mold, with a polyether strip to planify the surface. According to each treatment, the GIC surface received or did not receive acid etching with a 37% phosphoric acid (Acid gel, Dentalville do Brasil LTDA., Joinville, Brazil) for 10 seconds, followed by rinsing and the respective bonding system - Adper Single Bond 2 (3 M ESPE, St Paul, USA) (Total-etch bonding agent) or Single Bond Universal (3 M ESPE, St Paul, USA) (Self–etch bonding agent) - and lightcuring for 20 seconds with 1200 mW/cm^2 (LED Radii-cal, SDI Limited, Bayswater, Australia). The irradiance of the light curing device was measured using a radiometer (RD-7, Ecel Indústria e Comércio LTDA, Ribeirão Preto, Brazil). Increments of 2 mm of a nanofilled composite resin (Filtek Z350XT, 3 M ESPE, St Paul, USA) were inserted and lightcured for 20 seconds on the GIC. The blocks were removed from the mold, the GIC surface was protected with RIVA coat (SDI Limited, Bayswater, Australia), and then they were stored in an incubator with a 100% humid environment in 37 °C for 7 days. Table 1 shows the materials, manufacturers and chemical composition.

2.2. Microtensile bond strength test

After 7 days, each block was sliced with a precision saw with a diamond blade (Isomet 1000, Buehler, Lake Buff, USA) with a 200 rpm speed. Five slices were obtained of each initial specimen (resulting in 240 final specimens), which were trimmed in an hourglass shape with a 3216 F diamond bur (KG Sorensen, Cotia, Brazil) on the adhesive interface between the GIC and composite resin.

Each hourglass specimen was fixed to a modified device using a cyanoacrylate bonder (Super Bonder Loctite, Henkel, Düsseldorf, Germany), then attached to the universal testing machine (BISCO, Schaumburg, USA) and subjected to the microtensile test with a crosshead speed of 0.5 mm/min. The microtensile bond strength was recorded in newtons and transformed into MPa by dividing by the adhesion area in mm^2. The cross-sectional adhesion area was measured using a digital caliper (MIP/E-104-1, Mitutoyo Sul Americana LTDA, Santo Amaro, Brazil) with 0.01 mm accuracy.

2.3. Optical microscopy analysis

After the microtensile bond strength test, the specimens were evaluated in a stereoscopy magnifying glass (Carl Zeiss, Oberkochen, Germany) with a 40-fold increase to assess the fracture pattern. The specimen fracture patterns were classified as adhesive (failure at the composite resin interface), cohesive (failure at the GIC) or mixed (partially adhesive and cohesive), and the percentage of each group was recorded. The failure modes were classified by two different evaluators, followed by a repetition 15 days later. The intra- and inter-evaluator results were compared (Kappa ≥ 0.8). The specimens lost during trimming were included, and each was considered as a zero value for statistical analysis.

2.4. Statistical analysis

The normal distribution and homogeneity of variance of the data were analyzed with Kolmogorov-Smirnov and Levene's tests. Data were analyzed by two-way ANOVA and the Tukey test by a blinded person. All statistical analyses were performed using the statistical software SPSS 17 (Statistical Product and Service Solutions, SPSS, Chicago, USA). A confidence level of 5% was adopted for all tests.

3. Results

Two-way ANOVA showed significant effects of the factors acid etching ($p = 0.001$), adhesive system ($p = 0.029$) and interaction ($p < 0.001$). The results of the Tukey test are shown in Table 2. The means (standard deviation) of the microtensile bond strength (MPa) of the experimental groups demonstrated that G2 was significantly different from the other groups ($p < 0.001$), while G1, G3 and G4 were not significantly different from each other ($p > 0.05$).

The fracture mode assessment showed that in a total of 188 (excluding 52 lost specimens), there were 156 specimens with mixed fractures (83%), 18 with cohesive fractures (9.5%), and 14 with adhesive fractures (7.5%) (Figs. 1–3). Table 3 shows the fracture pattern in each group, in percentages.

4. Discussion

The null hypothesis tested was rejected since the adhesive type and etching affected the microtensile bond strength of the composite resin and glass ionomer cement. The total-etch adhesive system Adper Single Bond 2 with acid etching promoted lower microtensile bond strength than the self-etch adhesive Single Bond Universal with or without acid etching for the adhesion between the composite resin and glass ionomer cement.

From a clinical standpoint, the use of a cavity lining has a weakening effect on the overall strength of the restoration, resulting in more fracturing of composite restorations [16]. The bond strength between GIC and dentin is only 25% of the strength of composite resin [5]. However, the biomimetic principle (replacement of the whole tissue or part of it using materials that can reproduce the original tissue

Table 1
Materials, manufacturers and chemical composition.

Materials and manufacturers	Chemical composition
RIVA self-cure (SDI Limited, Bayswater, Australia)	Fluoroaluminosicilate glass (90–95%)
	Polyacrylic acid (5–10%)
RIVA coat (SDI Limited, Bayswater, Australia)	Acrylic monomer (100%)
Filtek Z350-XT shade A3B (3 M ESPE, St Paul, USA)	Filler: 59.5 vol% combination of aggregated zirconia/silica cluster filler with primary particle sizes of 5–20 nm, and nonagglomerated 20 nm silica filler. Polymeric matrix: Bis-GMA, Bis-EMA, UDMA, TEGDMA
Single bond universal (3 M ESPE, St Paul, USA)	Bis-GMA, 2-hydroxyethyl methacrylate decamethylene dimethacrylate, ethanol, water, silane-treated silica, 2-propenoic acid 2-methyl-, reaction products with 1, 10-decanediol and phosphorous oxide (P_2O_5), copolymer of acrylic and itaconic acid, camphorquinone, dimethylaminobenzoate (-4), toluene
Adper single bond 2 (3 M ESPE, St Paul, USA)	Bis-GMA, HEMA, polyacrylic acid, poly(itaconic acid), water, ethanol, dl-camphorquinone, silica (10% wt)
Acid gel (Dentalville do Brasil LTDA., Joinville, Brazil)	37% phosphoric acid, thickener, dye and deionized water

Table 2
Means (standard deviations) of microtensile bond strength (MPa) of groups.

	Mean (standard deviation) of microtensile bond strength (MPa)
G1	9.46 (3.79)^b
G2	6.27 (3.21)^a
G3	9.35 (3.91)^b
G4	10.13 (3.53)^b

Identical superscript letters indicate no statistical difference ($p < 0.001$)

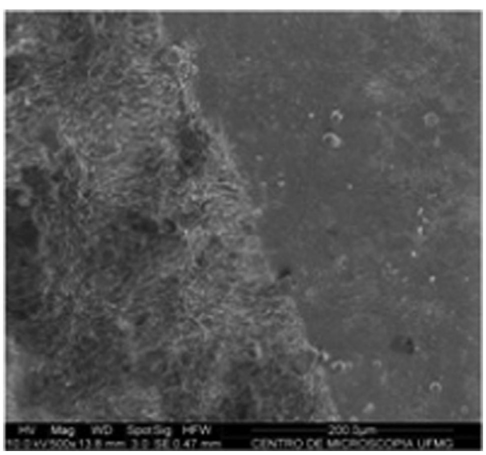

Fig. 1. Mixed fracture.

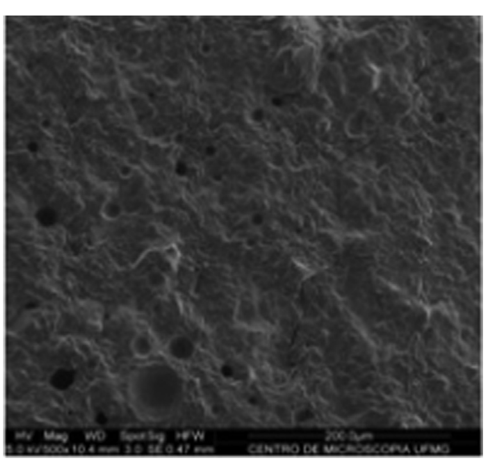

Fig. 2. Cohesive fracture.

characteristics as well as possible) makes GIC the best substitute for dentin [4]. According to the manufacturer, the GIC used in this study has a flexural strength very similar to dentin.

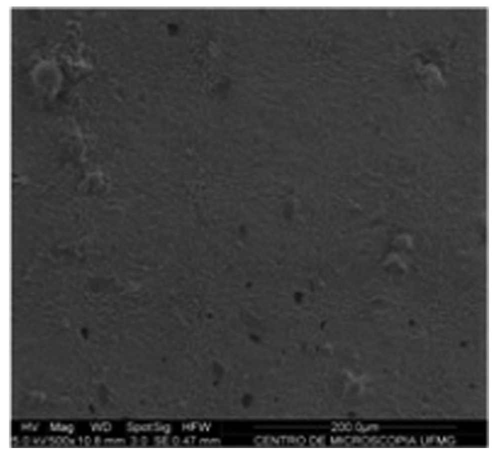

Fig. 3. Adhesive fracture.

Table 3
Percentage of fracture mode assessment among groups.

	Cohesive	Adhesive	Mixed
G1	38.5%	0	61.5%
G2	60%	0	40%
G3	64.3%	0	35.7%
G4	38.5%	7.7%	53.9%

The bond strength between CR and GIC depends on the tensile strength of the GIC (given by the powder/liquid ratio), bonding agent viscosity *versus* wetting ability on the GIC surface, volumetric change on the composite resin during polymerization and difficulty in packing and adapting the CR to the GIC without the incorporation of voids [11]. The adhesion between conventional GIC and composite resin is limited due to the lack of chemical adhesion between these materials. Moreover, other reasons for failure can be related to the GIC water sensitivity and loss of part of the structure after acid etching [7]. Water in the initial setting can dissolve the weak calcium polyacrylate chains [8]. Water plays an important role in the GIC setting reaction, acting as the reaction solvent, as otherwise the acid would not be able to react. It is also the final reaction product and is responsible for the decrease in cement stiffness, which explains the sensitivity to humidity in the initial phases of the setting [12].

Cohesive failure occurs primarily due to the relatively weak shear and tensile strength of the GIC [20]. The random distribution to the groups was performed to minimize the effect of specimen preparation on the resulting bond strength values. The specimens were made with a 2 mm height of GIC and 6 mm of CR to minimize the cohesive failure in the GIC, which has a compressive strength of 270 MPa 7 days after initial setting, while the composite resin Filtek Z350XT has approximately 400 MPa, both values given by the manufacturers. The bond strength depends not only on the resistance to failure but also on the presence of defects such as air bubbles in the specimens [3]. Bond strength data from mechanical tests represents a statistical distribution of defects (discontinuity, defects, voids, gaps and residual solvent). The smaller the specimen, the lower the chance of having large defects that can lead to failure, and thus it will have a higher apparent strength [1]. The hourglass-shaped specimen seems to fail under low stresses compared to the beans or dumb-bells, due to the high stress concentration induced in the adhesive layer during the trimming. The stress concentrates near the hourglass edges, making this area more prone to initiating failure [9,15]. However, other authors have found that the hourglass specimens show higher average bond strength compared to the other shapes and found no difference in the values of bond strength between bean and dumb-bell shaped specimens [6,14].

The highest value of microtensile bond strength of the universal adhesive with previous etching may be due to the chemical union of 10-MDP and calcium ions from GIC. Self-etching and self-adhesive bonding systems have hydrophilic and hydrophobic components that simultaneously demineralize and infiltrate into the tooth surface. The 10-MDP functional monomer in Single Bond Universal chemically binds to calcium salts, forming stable calcium phosphate, with a superficial descaling effect. Self-etching adhesives of moderate pH interact only with the superficial enamel and dentin, do not dissolve the hydroxyapatite crystals and keep them in a hybrid layer [18]. However, there was no additional effect of etching GIC prior to Single Bond Universal compared to specimens without etching.

Adper Single Bond 2 without previous etching promotes higher microtensile bond strength between CR and conventional GIC than when applied with etching. This result confirms that the acid etching treatment is not necessary for the immediate sandwich technique to enhance the bond strength promoted with the total-etch adhesive system but actually damages bonding. Acid etching can dissolve and disorganize the conventional GIC matrix, decreasing the adhesive wetting surface. Acid etching glass ionomer cement is a critical clinical step and increases the risk of excessive cement degradation and involuntary acid-dentin interaction [21]. Since GIC is sensitive to water on the initial setting, [12] according to our results, the suitable approaches for the immediate sandwich technique are applying universal adhesive or total-etch adhesive without etching the GIC layer.

Of the 240 hourglass shape final specimens, 52 (21.5%) were lost. It was observed that 18% of losses occurred among the acid-etched specimens. The loss among the groups that did not receive acid etching may be due to the diamond bur vibration during the specimen preparation, compromising the bonding on the adhesive interface GIC-CR. A relatively high pre-test failures percentage (27%) was found during hourglass trimming of microtensile specimens but it has been reported elsewhere [1]. However, the brittle nature of glass-ionomer cement and resin-based composite requires a uniform interface between the grip components and the specimen. Furthermore, non-trimming technique does not create a defined test region with a uniform stress state [1]. Spontaneous interfacial debonded specimens are usually not addressed in the papers. The majority of the articles reporting pre-test failures included them as zero values in the statistical analysis, whereas 30% reported the number of pre-test failures but not include them in the statistical analysis [14].

In our study, 9.5% of the fractures were predominantly cohesive. Cohesive fractures are clinically undesirable because they mean a higher chance of microleakage on the GIC-tooth interface. If failure occurs, it would be better that it occur in the GIC-CR interface, as seen in 7.5% of the specimens (adhesive fracture).

Since the conventional glass ionomer cement does not bond to the composite resin, we found important to evaluate the micromechanical bonding strength using different adhesives, not varying the resin composite or GIC. Future studies should include a group that provides increased surface area without acid, for example grit-blasting or grinding to roughen the surface, in order to exclude, or otherwise, the effect of acid (or water contamination) on the setting reaction of GIC.

Within the limitations of this study, we can conclude that GIC etching promoted higher microtensile bond strength with the universal adhesive than with the total-etch adhesive system. Acid etching is not necessary to enhance the universal adhesive bond strength and negatively affected the bond strength of total-etch adhesive system. Without etching the GIC, there is no difference in microtensile bond strength between the adhesive systems.

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Clinical relevance

The suitable approaches for the immediate sandwich technique are to use the universal adhesive or total-etch adhesive system without etching the conventional glass ionomer cement layer to achieve higher microtensile bond strength to the composite resin.

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