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EFFECT OF THE ADHESIVE ON THE BOND STRENGTH BETWEEN GLASS IONOMER CEMENT AND COMPOSITE RESIN

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ABSTRACT

The growing demand for aesthetic restorations in the posterior region has become a fundamental part of dental practice, reflecting the increasing interest in aesthetic materials. However, finding a restorative material that exhibits ideal characteristics similar to dental structure has always been a challenge in dentistry. In pursuit of these properties, techniques that combine the physical and chemical aspects of different materials are being explored to ensure the success of long-lasting aesthetic restorations in the posterior region. This study investigates the "sandwich" technique, which combines glass ionomer cement and composite resin, through *in vitro* shear strength tests. The objective was to evaluate the effectiveness of this technique with different types of adhesive systems. A total of 360 adhesions were performed on the surface of resin-modified glass ionomer cement, Riva (SDI, Australia), divided according to the adhesive material: Scotchbond™ Universal Plus 3M™, Adper™ Single Bond 2 3M™, Adper™ Scotchbond™ 3M™. Additionally, the influence of phosphoric acid conditioning on the resin-modified glass ionomer cement and artificial aging through immersion of the specimens in a humid environment for 6 months were evaluated. It was concluded that, after storage, a reduction in shear strength values was observed for the group using the Universal adhesive when phosphoric acid was applied. The other groups did not show a reduction in values, regardless of the phosphoric acid application on the resin-modified glass ionomer cement. After 6 months of storage, an increase in cohesive fractures was observed.

Keywords: Dentin-bonding agents, glass ionomer cements, shear strength.

INTRODUCTION

In recent years, there has been a consistent increase in the demand for aesthetic adhesive treatments in dentistry.[1,2] In response to this demand, an alternative restorative technique known as the sandwich technique emerged, aiming to address the challenges related to the polymerization shrinkage of composite resin.[3] This technique involves the application of glass ionomer cement (GIC) before the composite resin, thus reducing the amount of resin used in direct restorations.

However, despite advances in methacrylate-based materials, they still face challenges such as high polymerization shrinkage and high thermal expansion coefficient, which can lead to marginal microleakage, postoperative sensitivity, and pulpal irritation.[1,4,5]

GIC stands out as a viable alternative due to its properties that are compatible with dental structure, including low thermal expansion coefficient, effective chemical bonding to enamel and dentin even under humid conditions, and its anticariogenic action due to fluoride release.[5-7] It is important to highlight that glass ionomer cement will release significant amounts of fluoride in the initial days due to the initial superficial washout effect, further enhancing its effectiveness in caries prevention.[8] Conventional GIC has limitations in terms of chemical bond strength with composite resin, which may affect the success of the sandwich technique.[2] A great alternative to improve adhesion would be the use of resin-modified GIC, which incorporates photoactivated methacrylate and 2-HEMA or bisphenol-A-glycidyl methacrylate, combining the mechanical properties of conventional GIC with the adhesion of composite resin.[1-3]

The sandwich technique, by combining GIC and composite resin, offers significant advantages, improving marginal quality, resistance to deformation, and reducing postoperative sensitivity.[4,5] Moreover, improved adhesion between GIC and composite resin can be achieved through prior acid conditioning, increasing the roughness of GIC and promoting better micromechanical retention. It is important to avoid moisture contamination during the phosphoric acid washing process.[1,10] Although the sandwich technique has its advantages, some studies highlight challenges such as fractures in the restoration.[11] The low chemical bonding between conventional GIC and composite resin may negatively influence the longevity of the restoration, but the use of resin-modified GIC can improve this adhesion.[1,3]

One approach to strengthen the bond between GIC and composite resin is acid conditioning for 30 seconds, which increases microretention in the restored area.[2] However, the subsequent washing process may introduce moisture contamination, potentially dissolving the sodium polyacrylate chains and altering the chemical properties of the material.[1] To overcome this problem, the use of self-etching adhesives, which eliminate the washing step, emerges as an effective solution. Studies highlight the superiority of self-etch adhesives in terms of bond strength.[2,3,12]

Additionally, universal adhesives tend to provide higher bond strength, while acid conditioning may negatively impact adhesion with total-etch adhesive systems.[2,5] Bond strength tests play a crucial role in evaluating the bonds between materials in dentistry, especially sensitive substrates such as glass ionomers and enamel.[13]

This study aimed to evaluate and compare the adhesion between resin-modified GIC and composite resin using different types of adhesive systems, both conventional and self-etching, after 24 hours and 6 months of storage in a humid environment. The results provide important insights for clinical practice and the development of more effective approaches in aesthetic adhesive dental restorations.

MATERIALS AND METHODS

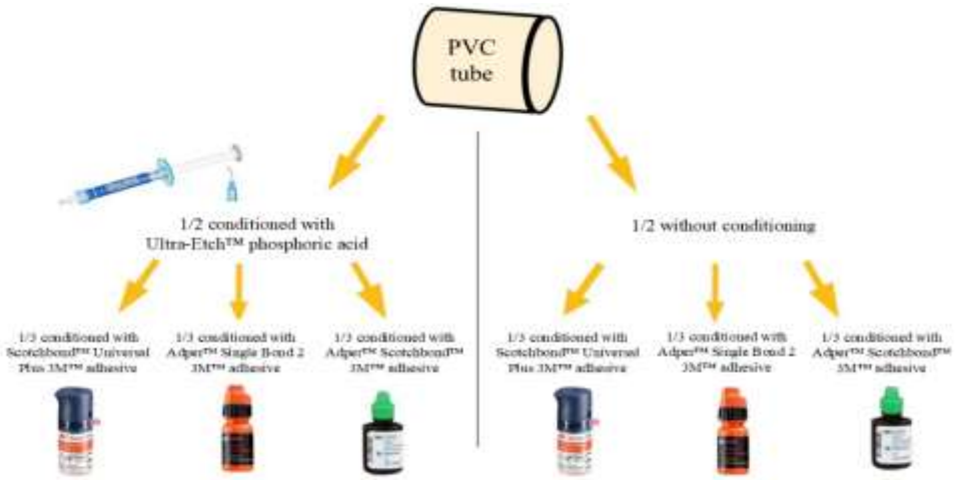
Within the scope of this study, specific materials were employed to conduct the proposed dental study. The following materials were used: resin-modified glass ionomer cement RIVA LIGHT CURE (SDI, Australia); the microhybrid composite resin Filtek™ Z100 (3M ESPE, St. Paul, USA); the phosphoric acid Ultra-Etch™ (Ultradent Products, Inc., South Jordan, USA), essential for the acid conditioning process; and three types of dental adhesives with different compositions: Scotchbond™ Universal Plus, Adper™ Single Bond 2, Adper™ Scotchbond™ Adhesive, all from 3M ESPE (St. Paul, USA). Regarding the polymerization processes, the photopolymerizer VALO™ (ULTRADENT, USA) was used, operating in Standard mode at $1000 mW/cm^2$. The resin-modified glass ionomer cement, RIVA (SDI, Australia), was mixed in the Ultramat 2 amalgamator (SDI Limited, Bayswater, Australia) and carefully placed into self-polymerizing acrylic resin molds JET – powder and liquid (Clássico, Brazil) with RIVA applicator (SDI, Australia). Subsequently, resin increments were placed into polyurethane molds (Elastic Separator Ring, MORELLI, Brazil) with a 4mm diameter, following the established technical protocols for the procedure.

This in vitro research was conducted using a total of 360 specimens for the experimental condition. For the preparation of the specimens, split molds made of Polyvinyl Chloride (PVC) were used, with dimensions of 10 mm in diameter and 1.5 mm in thickness. These molds were carefully filled with self-polymerizing acrylic resin Jet, creating a specific space for the accommodation of resin-modified glass ionomer cement (GIC) using a maxicut drill. The preparation of the specimens followed a meticulous process, ensuring precision in measurements and uniformity of the samples. Through the controlled insertion of the acrylic resin into the molds, a structural compartment was created that allowed the subsequent introduction of resin-modified GIC, ensuring ideal conditions for the analyses proposed in this study.

The procedure for handling the RIVA glass ionomer cement strictly followed the manufacturer's guidelines. After being subjected to the Ultramat 2 amalgamator for 10 seconds, the contents of the capsule were carefully placed into the mold using the RIVA applicator. This approach aimed to ensure precise access and reduce the likelihood of internal bubble formation. Then, polymerization was performed with the VALO photopolymerizer, operating in Standard mode at an intensity of 1000mW/cm^2 for 20 seconds, along with a polyester strip (K-DENT, Brazil) to standardize the surface of the GIC.

After one week, all glass ionomer cement discs underwent standardized surface wear using silicon carbide sandpaper discs with 400 and 600 grit. This procedure aimed to create a uniform and leveled surface on the discs. The sanding process was manually performed by a single operator for one minute, ensuring consistency in the results. The discs were then divided into different conditioning groups according to the proposed treatment for the surface of the glass ionomer cement. They were either conditioned or not with 37% phosphoric acid Ultra-Etch™ for 15 seconds, followed by rinsing, drying, and the application of the respective adhesive system. The adhesive activation was done with the VALO photopolymerizer in Standard mode at an intensity of 1000mW/cm^2 for 20 seconds. The independent variables were represented by the different adhesive systems used: Scotchbond™ Universal Plus, Adper™ Single Bond 2, Adper™ Scotchbond™ Adhesive (all 3M™), as well as the conditioning with 37% phosphoric acid (with or without). This study involved 360 specimens, which were prepared and randomly distributed into six different conditioning groups, as described above (Figure 1).

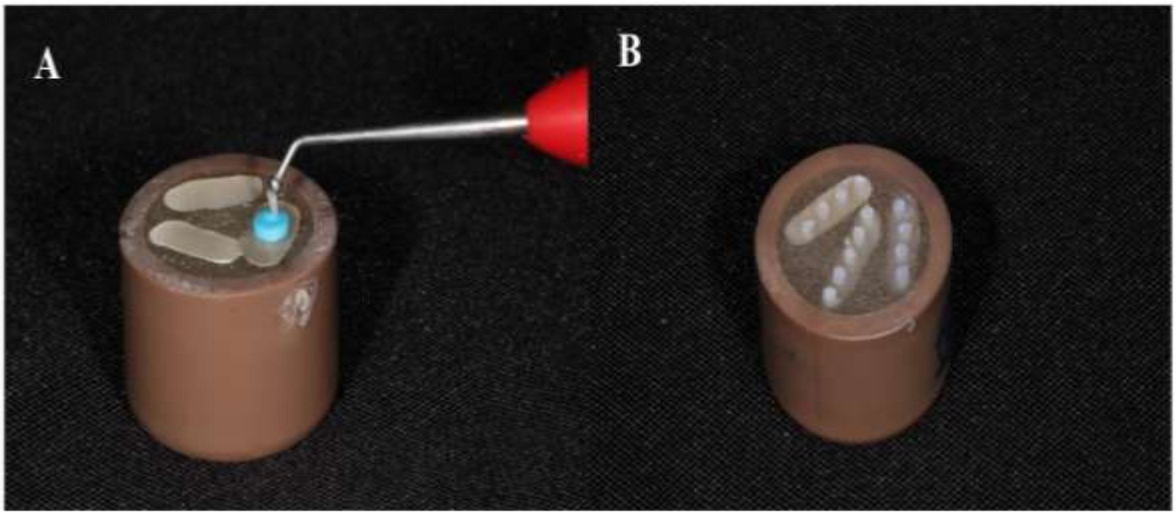
Figure 1: Distribution of the conditioning protocols. Created by the author.



Source: Original work (2025)

Subsequently, increments of the microhybrid composite resin Z100 were placed over the RIVA GIC layer using 4mm diameter polyurethane molds (Elastic Separator Ring – MORELLI). These molds played a crucial role in standardizing and shaping the composite resin increments, which were photopolymerized for a period of 20 seconds. After complete polymerization, the elastics were removed, giving the composite resin the desired shape and ensuring consistency in the application process (Figure 2).

Figure 2: (A) Resin increments on a 4mm polyurethane mold; (B) After polymerization and removal of the elastic, the increments were standardized. Created by the author.



Source: Original work (2025)

The adhered specimens were carefully stored in a container under controlled humidity conditions, maintaining an environment with 100% relative humidity using distilled water. After a 24-hour period, half of the groups (groups 1 to 6) were subjected to shear strength testing. Meanwhile, the other half of the groups (groups 7 to 12) were kept in distilled water at 37°C for 6 months, with the water being changed weekly. These groups were as follows:

- **Group 1:** RIVA GIC with pre-conditioning using 37% phosphoric acid Ultra-Etch™ and Scotchbond™ Universal Plus 3M™ (n=30);
- **Group 2:** RIVA GIC with Scotchbond™ Universal Plus 3M™ (n=30);
- **Group 3:** RIVA GIC with pre-conditioning using 37% phosphoric acid Ultra-Etch™ and Adper™ Single Bond 2 3M™ (n=30);

- **Group 4:** RIVA GIC with Adper™ Single Bond 2 3M™ (n=30);
- **Group 5:** RIVA GIC with pre-conditioning using 37% phosphoric acid Ultra-Etch™ and Adper™ Scotchbond™ 3M™ (n=30);
- **Group 6:** RIVA GIC with Adper™ Scotchbond™ 3M™ (n=30);
- **Group 7:** RIVA GIC with pre-conditioning using 37% phosphoric acid Ultra-Etch™ and Scotchbond™ Universal Plus 3M™ (n=30);
- **Group 8:** RIVA GIC with Scotchbond™ Universal Plus 3M™ (n=30);
- **Group 9:** RIVA GIC with pre-conditioning using 37% phosphoric acid Ultra-Etch™ and Adper™ Single Bond 2 3M™ (n=30);
- **Group 10:** RIVA GIC with Adper™ Single Bond 2 3M™ (n=30);
- **Group 11:** RIVA GIC with pre-conditioning using 37% phosphoric acid Ultra-Etch™ and Adper™ Scotchbond™ 3M™ (n=30);
- **Group 12:** RIVA GIC with Adper™ Scotchbond™ 3M™ (n=30);

After the 24-hour and 6-month periods, each PVC block was fixed in the universal testing machine (Emic 500, Brazil), equipped with a 500 Newton load cell, for shear strength testing at the interface between the resin-modified GIC and composite resin. These tests were conducted by two operators in the Engineering Laboratory at the Pontifical Catholic University of Minas Gerais, using the available equipment and resources for analysis and evaluation of the bonding properties of the materials under study. Of the 360 composite resin specimens used, only 12 were lost during the study. Of these, three were due to operator errors during machine handling, while the remaining nine were found loose in the storage container for the 6-month samples. Although the percentage of loss was relatively low, the three cases resulting from operational error were not included as zero values in the statistical analysis (they were later replaced), while the nine cases were considered as zero values for this analysis.

After performing the shear strength test, the specimens were examined using the SteREO Discovery. V8 microscope (Carl Zeiss, Oberkochen, Germany) at 3.2x magnification to analyze the resulting fracture patterns. This analysis was conducted by two operators simultaneously, seeking consensus on the type of fracture observed under optical microscopy. Thus, fracture patterns were classified into three distinct categories: adhesive (indicating failure at the composite resin interface), cohesive (indicating failure in the resin-modified GIC), or mixed (representing a partially adhesive and cohesive failure).

RESULTS

After verifying the normality of the data, they were subjected to a three-way Analysis of Variance (ANOVA), followed by the Tukey *post hoc* test for multiple comparisons between groups. The results obtained were compiled and presented in Table 1, displaying the results of the three-way ANOVA along with the corresponding interaction values between the variables.

Table 1: Three-way Analysis of Variance. Created by the author.

	Sum of Squares (SS)	df	Mean Square (MS)	F	p
Adhesive	552.09	2	276.05	10.1915	< .001
Acid	2.08	1	2.08	0.0767	0.782
Aging	123.68	1	123.68	4.5664	0.033
Adhesive ✻ Acid	182.71	2	91.36	3.3728	0.035
Adhesive ✻ Aging	194.25	2	97.12	3.5858	0.029
Acid ✻ Aging	2.96	1	2.96	0.1092	0.741
Adhesive ✻ Acid ✻ Aging	169.55	2	84.77	3.1298	0.045
Residuals	9425.83	348	27.09		

df: degrees of freedom;
Source: Original work (2025)

Table 2 presents the shear strength values after 24 hours. This table shows the data for the resin-modified glass ionomer cements subjected to phosphoric acid conditioning and without this treatment. When phosphoric acid was applied to the resin-modified glass ionomer cement, the Scotchbond™ Universal Plus 3M™ adhesive demonstrated the best performance, significantly differing from the other groups. On the other hand, in the absence of this conditioning, the adhesives exhibited similar shear strength.

Table 2: Values in MPa and standard deviation of the groups tested at the 24-hour period (groups conditioned or not with phosphoric acid). Created by the author.

	Universal 24h	Single Bond 2 24h	Scotch Bond MP 24h
With acid conditioning	13.2 (6.07) Aa	8.06 (5.21) Ba	6.60 (4.07) Ba
Without acid conditioning	10.07 (5.47) Aa	9.19 (4.63) Aa	7.87 (4.66) Aa

Tukey test applied after three-way analysis of variance. Different uppercase letters in a row indicate statistically significant differences by Tukey's test ($p<0.05$). Different lowercase letters in a column indicate statistically significant differences by Tukey's test ($p<0.05$).
Source: Original work (2025)

After the 6-month aging period in distilled water (Table 3), no statistically significant differences were observed between the experimental conditions. The values obtained were similar in relation to both the type of adhesive and the treatment applied to the resin-modified glass ionomer cement in the specimens stored during this period.

Table 3: Values in MPa and standard deviation of the groups tested after a 6-month period (groups conditioned or not with phosphoric acid). Created by the author.

	Universal 6 months	Single Bond 2 6 months	Scotch Bond MP 6 months
With acid conditioning	8.67 (4.26) Aa	9.00 (5.77) Aa	6.13 (5.03) Aa
Without acid conditioning	8.97 (5.33) Aa	6.67 (4.48) Aa	9.31 (6.62) Aa

Tukey's test applied after three-way analysis of variance. Different uppercase letters in a row indicate statistically significant differences by Tukey's test ($p<0.05$). Different lowercase letters in a column indicate statistically significant differences by Tukey's test ($p<0.05$).
Source: Original work (2025)

Tables 4 and 5 were designed to highlight the comparisons between groups after the aging period.

In Table 4, it is observed that the Scotchbond™ Universal Plus 3M™ adhesive showed a decrease in shear strength values (from 13.2 to 8.67) after artificial aging, specifically when the acid conditioning technique was used.

Table 4: With acid conditioning in both test periods (24 hours and 6 months). Created by the author.

	Universal	Single Bond 2	Scotch Bond MP
24 hours	13.2 (6.07) Aa	8.06 (5.21) Ba	6.60 (4.07) Ba
6 months	8.67 (4,26) Ab	9.00 (5.77) Aa	6.13 (5.03) Aa

Tukey's test applied after three-way analysis of variance. Different uppercase letters in a row indicate statistically significant differences by Tukey's test ($p<0.05$). Different lowercase letters in a column indicate statistically significant differences by Tukey's test ($p<0.05$).
Source: Original work (2025)

On the other hand, the other materials maintained consistent values after the storage period. However, this observation was not repeated when the ionomer surface was not treated with phosphoric acid (Table 5). When no acid application was performed, all groups were considered statistically similar, even after artificial aging.

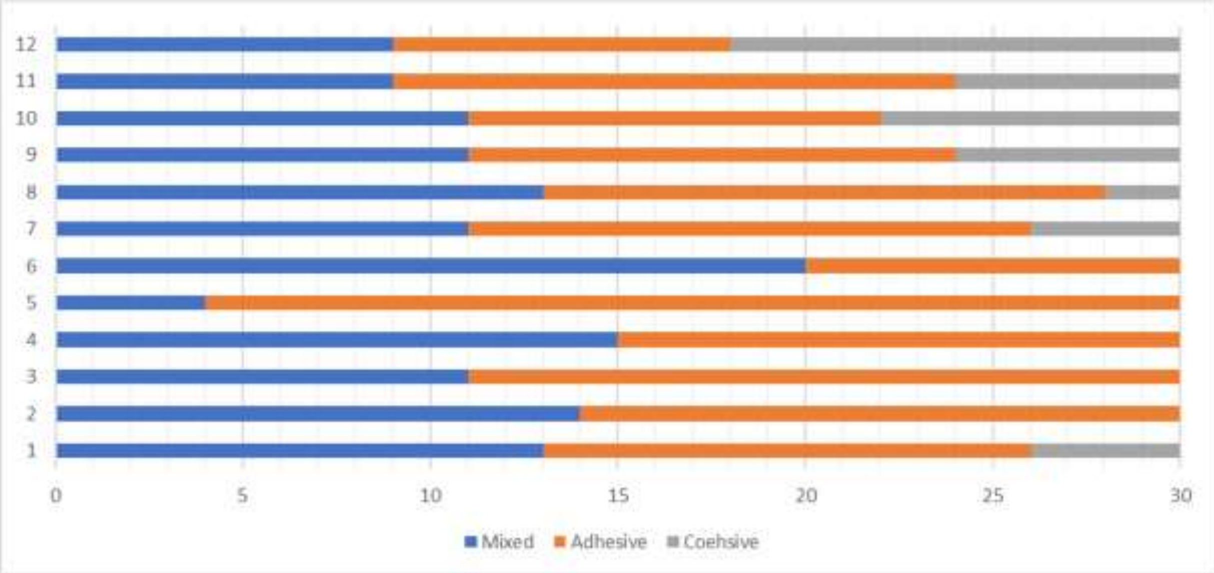
Table 5: Without acid conditioning in both test periods (24 hours and 6 months). Created by the author.

	Universal	**Single Bond 2**	**Scotch Bond MP**
24 hours	10.07 (5.47) Aa	9.19 (4.63) Aa	7.87 (4.66) Aa
6 months	8.97 (5.33) Aa	6.67 (4.48) Aa	9.31 (6.62) Aa

Tukey's test applied after three-way analysis of variance. Different uppercase letters in a row indicate statistically significant differences by Tukey's test ($p<0.05$). Different lowercase letters in a column indicate statistically significant differences by Tukey's test ($p<0.05$).
Source: Original work (2025)

After conducting the shear strength test, the specimens were carefully examined using the SteREO Discovery. V8 microscope (Carl Zeiss, Oberkochen, Germany) to analyze the resulting fracture patterns. This analysis was carried out by two operators, seeking a more comprehensive and consistent conclusion, allowing for the classification of fracture patterns into three distinct categories: adhesive (indicating failure at the composite resin interface), cohesive (indicating failure in the resin-modified GIC), or mixed (representing a partially adhesive and cohesive failure), as shown in Figure 3.

Figure 3: Analysis of fracture pattern of the groups. Classification of fracture patterns into three distinct categories: adhesive (indicating failure at the composite resin interface), cohesive (indicating failure in the GIC), or mixed (representing a partially adhesive and cohesive failure). Groups 1-6 were stored for 24 hours, and groups 7-12 were stored for 6 months. Created by the author.

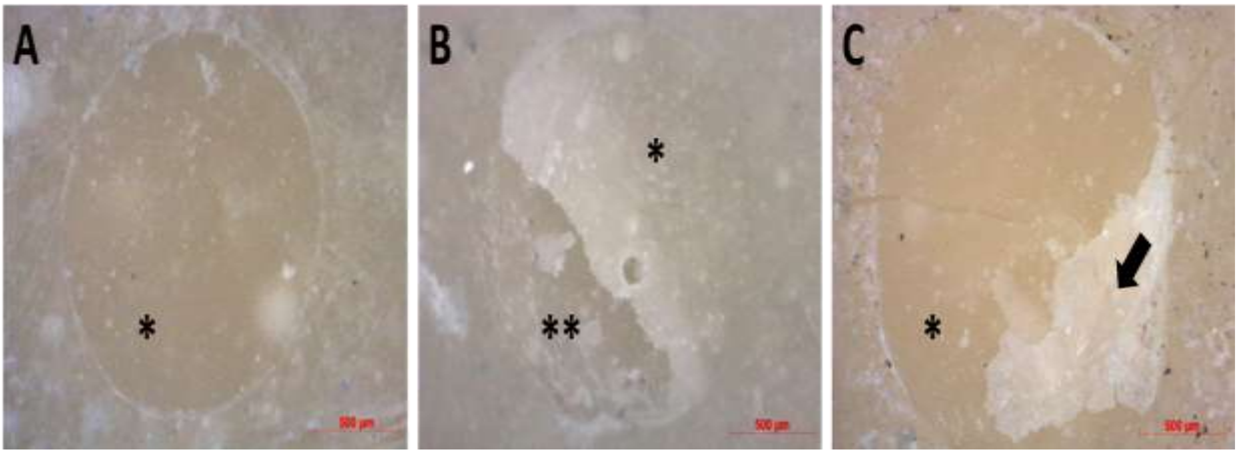


Source: Original work (2025)

It is observed that there is a predominance of mixed and adhesive fractures at the beginning of the study with 24 hours of storage. After six months of storage, cohesive fractures emerge more frequently (Figure 3, groups 7 to 12), although they do not reach predominance, as shown in the graph in Figure 3.

In Figure 4, optical microscopy images of the 3 fracture patterns observed can be seen.

Figure 4: Optical microscopy images of the 3 fracture patterns observed. Image "A" shows the fracture pattern classified as adhesive. The asterisk () indicates the exposed GIC surface after the displacement of the composite resin specimen. Image "B" shows the type of fracture classified as cohesive in GIC, where the single asterisk () and double asterisk (**) indicate the two sides of the wedge-shaped fracture of the GIC. Image "C" shows the fracture considered as mixed, with the presence of a piece of composite resin (arrow) and the GIC surface. Created by the author.



Source: Original work (2025)

DISCUSSION

In response to the demand for aesthetic treatments, alternative restorative techniques, such as the sandwich technique, have been proposed to overcome challenges such as polymerization shrinkage of composite resin. This approach involves the application of glass ionomer cement (GIC) before composite resin, thereby minimizing the use of the latter in cavities to be restored by the direct method.[1,2]

Using GIC before composite resin restoration emerges as an alternative to improve clinical success. This is because GIC has properties that resemble the dental structure, such as a low thermal expansion coefficient similar to that of dentin, the ability to form a chemical bond to enamel and dentin even under wet conditions, biocompatibility, and anti-caries action due to fluoride release.[5-7] It has been reported that materials that flex with the tooth, such as GIC, reduce the transfer of stress to the dental structure during flexion.[14] Additionally, GIC has a thermal expansion coefficient similar to dentin, which improves tensile strength and, consequently, the adhesion between GIC and composite resin.[15] Despite this, the bond strength between GIC and dentin is only 25% of that of composite resin.[6] In situations involving oblique loads, the stress generated at the interface of the restoration with GIC is even higher and is a determining factor for the high failure rates observed clinically in class V restorations.[16]

Although the use of 37% phosphoric acid in the sandwich technique raises concerns among many dentists, the causes of possible failures are not yet fully understood. It is believed that the washing process of phosphoric acid may alter the physical properties of glass ionomer cement (GIC), breaking calcium polyacrylate chains.[1,10] However, in our analysis, the use or absence of phosphoric acid did not show statistically significant differences in shear strength (Tables 2 and 3), regardless of the evaluation time (24 hours or 6 months of storage). The same result had been reported previously.[17] In contrast, another study concluded that acid etching can improve this bond when compared to no etching.[18]

In the present study, it was observed that the Scotchbond™ Universal Plus adhesive showed a statistically significant reduction in shear strength values (from 13.2 to 8.67) after aging when acid conditioning was applied. This was not observed when the GIC surface was not treated with 37% phosphoric acid. In these cases, a reduction in adhesive failures was noted, while cohesive failures increased in the groups subjected to the aging process (Figure 3). It is speculated that the increase in cohesive failures in GIC may result from the material's own aging and hydrolytic degradation. Thus, it can be inferred that the GIC/composite resisted shear stress more effectively than the internal structure of the GIC. This constitutes one of the limitations of the shear strength measurement methodology, something that could also be observed if the force were tensile or compressive, as the bond of the materials was not properly tested due to the failure of one side of the adhesion.

Although notable shear strength was found with the self-etching adhesive Scotchbond™ Universal Plus after pre-conditioning with 37% phosphoric acid compared to other adhesives subjected to the same pre-conditioning, no significant difference was observed when GIC was used with Scotchbond™ Universal Plus, regardless of pre-conditioning with 37% phosphoric acid. This adhesive system contains the 10-MDP monomer, recognized for its ability to form a chemical bond with the calcium ions of GIC, creating a stable calcium phosphate compound that has a superficial demineralizing effect. However, it is speculated that the adhesive layer created may be more acidic than the other adhesives. The hypertonic environment created may have allowed greater water absorption in the area, contributing to the reduction in shear strength values.

Another study employed a different mechanical treatment methodology, sandblasting with aluminum oxide. As a result, sandblasting followed by adhesive conditioning showed higher bond strength to conventional GIC, resin-modified GIC, flowable composite, and Cention-N (Ivoclar, Switzerland).[19]

Clinically, the progressive loss of adhesion over time may be influenced by several factors, such as malocclusion and hyperfunction. In these cases, the teeth undergo excessive flexion, which contributes to the reduction of adhesion between dentin and the restoration. These occlusal forces can lead to the formation of gaps at the margins of the restoration, and it is important not to underestimate the impact of these forces.[20] Therefore, it is crucial to consider that the results of the present study should be interpreted with caution, based on its limitations, as they do not fully replicate the complexity of real dental structure and salivary composition. Additionally, various factors, some of which have already been mentioned, may contribute to clinical failure, such as repeated dynamic stress on the restoration, thermal stresses and variations, parafunctional habits, and poor dietary choices.

CONCLUSIONS

Within the limitations of the study, it was concluded that after storage, there was a reduction in shear strength values for the group that used the Universal adhesive when phosphoric acid was applied. The other groups did not show a reduction in values, regardless of the application of phosphoric acid on the resin-modified glass ionomer cement (GIC). After 6 months of storage, there was an increase in the number of cohesive fractures.

Declaration of Interest

The authors report no conflict of interest, financial, or otherwise.

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