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Effect of self-etching dual-curing universal adhesive system application on bond strength of dentin resinous liners dentin

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ABSTRACT

Purpose: The aim of this study was to evaluate the influence of previous application of an adhesive system on bond strength of resinous liner materials to dentin. **Methods:** Ninety bovine incisors crowns had a 6 × 6 mm area of dentin exposed, with minimum of 2 mm thickness. They were embedded in acrylic resin, and the dentin was polished with P600 SiC sandpaper for 30 s to standardize the smear layer. The specimens were divided into 6 groups (n = 15) according to the application or not of a self-etching system (Futurabond U – Voco) and the type of resinous liner used: A+Ionoseal (adhesive and Ionoseal – Voco); Ionoseal (Ionoseal only); A+Vitrebond (adhesive and Vitrebond – 3M/ESPE); Vitrebond (Vitrebond only); A+Ionosit (adhesive and Ionosit – DMG) and Ionosit (Ionosit only). Adhesives were used following manufacturer's instructions, and the liner materials were applied inside a 2-mm-depth matrix and light-cured for 20 s. The bond strength was measured by microtensile test, using a universal testing machine with a cross-speed of 1 mm/min. Data were analyzed using one-way ANOVA and Tukey's test ($p < 0.05$). **Results:** The adhesive system application increased bond strength of all liners tested. Ionoseal presented the highest bond strength when the adhesive system was used and exhibited similar performance to Vitrebond without adhesive. Ionosit without adhesive showed the smallest bond strength compared with the other liners tested. **Conclusion:** The application of an adhesive system prior to the use of the resinous liners improved the bond strength to dentin and should be preconized.

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Introduction

Liners are materials applied over dentin as a thin layer, aiming to seal the cavity floor and walls, promoting pulp protection from the influx of bacteria and irritants from restorative procedures.[1] These materials can be used to fill large cavities, due to their rheological and mechanical properties such as low viscosity and elastic modulus. Those characteristics allow them to improve not only stress distribution over dentin during masticatory functions, but also the marginal integrity of composite restorations.[2,3]

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Glass ionomers have been used as liners for more than 40 years [4] due to properties such as: thermal expansion coefficient similar to dentin; bacteriostatic action, due to fluoride release; low shrinkage; and the ability to bond to the tooth structure by calcium chelation.[2] However, they present high sensitivity to humidity, low mechanical strength and wear resistance, and are unsuitable as a restorative material in areas of direct occlusal stress.[5] Although some studies show that the use of a glass ionomer liner improves shrinkage stress distribution and decreases cuspal deflection in large cavities,[6,7] others show that this does not improve the adhesion of composite resin to dentin.[8,9] Moreover, material cohesive failures have been observed with some liners.[10,11] Failures in adhesion result from the formation of cracks and imperfections in the adhesive interface, which tends to increase when teeth are submitted to masticatory stress. These can lead to secondary caries, stained margins, and loss of retention,[1,2] justifying the need for reinforcement of the liner–dentin interface by using adhesive systems.

Resin-modified glass ionomer cements were introduced as an attempt to improve the properties of glass ionomer cements, maintaining its clinical advantages. These materials contain polyacids, photocurable monomers, silanized glass, and water, with the ability to set the acid–base reaction of the glass ionomer, fluoride release, and ion exchange during bonding.[12]

Nevertheless, in deep cavities, in which liner materials are indicated, the use of phosphoric acid would be an aggressive action, since it opens the dentin tubules, which are sometimes not completely sealed by the monomers. This leaves a pathway for leakage of toxic products and bacteria, causing pulp irritation due to the proximity of the cavity floor and the top of the pulpal chamber.[13] In these cases, self-etching adhesive systems might represent an alternative for improvement of the bond strength at the dentin–liner interface, since they present acidic monomers, which are less aggressive than phosphoric acid and do not open the tubules.[14,15]

The aim of this study was to evaluate the influence of a previous application of a self-etching dual-curing universal adhesive system on the bond strength of resinous liner materials to dentin. The null hypothesis tested is that the use of an adhesive system does not improve the bond strength of resinous liner materials over the dentin surface.

Materials and methods

Specimen preparation

Ninety freshly extracted, non-damaged bovine incisors were stored in deionized water, under refrigeration, until required. The root portion was removed using a high-speed cutting machine (Nevoni, São Paulo, SP, Brazil) with a diamond disk (Dremel, Breda, Netherlands). To expose the dentin portion, the buccal surfaces were ground flat with P400-SiC sandpaper (Extec Corp, Enfield, CT, USA) in a polishing device (Panambra, São Paulo, SP, Brazil) until a 6 × 6 mm area was obtained. After that, access to the pulpal chambers was opened with high-speed diamond burs. This allowed for the measurement of the dentin thickness, which should be a minimum of 2 mm.

The standardized specimens were embedded in acrylic resin (Jet, Clássico, São Paulo, SP, Brazil) in a cylindrical silicone mold (Rodhorsil, Clássico, São Paulo, SP, Brazil), with the exposed dentin surface facing down. After the curing of the acrylic resin, the cylindrical

Table 1. Composition and batch number of all materials tested.

Material	Type	Manufacturer	Batch number	Composition
Futurabond U	Universal Adhesive System	VOCO	1,308,047	HEMA, BIS-GMA, HEDMA, MDP, UDMA, Catalyst (≤2.5%)
Ionoseal	Compomer	VOCO	1,234,214	Dimethacrylates, silicates, pigments, catalyst system, BIS-GMA, UDMA
Vitrebond	Resin-Modified Glass Ionomer	3M/ESPE	N391282	*Liquid*: resin-modified polyalkenoic acid, HEMA, water and initiators (including camphorquinone) *Powder*: HEMA, BIS-GMA, water, initiators and a fluoroaluminosilicate glass
Ionosit	Compomer	DMG	673,180	Glass ionomer in a matrix of polymerizable oligo- and polycarbonic acids and other light-cure dental resins. Contain fluoride zinc ions and acrylate resin. Filler: 55%vol, size of 0.02–6 µm

specimens were positioned in the polishing device and the smear layer was standardized using P600-SiC sandpaper for 30 s.

Experimental groups division

Specimens were divided into six groups according to the application or not of a self-etching bonding system and the type of resinous liner applied ($n = 15$):

- **A+IONOSEAL** – Application of the self-etching adhesive Futurabond U (VOCO GmbH; Cuxhaven, Germany) associated with the light-cured liner Ionoseal (Voco);
- **IONOSEAL** – Application of Ionoseal directly on the dentin;
- **A+VITREBOND** – Application of Futurabond U associated with the light-cured liner Vitrebond (3M/ESPE; St Paul, MN, USA);
- **VITREBOND** – Application of Vitrebond directly on the dentin;
- **A+IONOSIT** – Application of Futurabond U associated with the light-cured Ionosit (DMG GmbH; Hamburg, Germany); and
- **IONOSIT** – Application of Ionosit directly on the dentin.

The self-etching dual-curing universal adhesive Futurabond U was applied actively over the surface for 20 s, followed by a blow of air for 5 s and light curing for 10 s, following the manufacturer's instructions. Table 1 shows the composition of all the materials tested. The liner materials were applied inside a 6 × 6 × 2 mm flexible matrix in a single 2-mm layer increment and light-cured for 20 s. After that, the matrix was removed and each side of the block was additionally light-cured for 20 s, using a light curing unit with a light power density of 600 mW/cm^2 (XL 3000, 3M/ESPE, St. Paul, MN, USA). The specimens were stored in deionized water at 37 °C for 24 h.

Bond strength test

The specimens were longitudinally sectioned using a low-speed diamond saw (Labcut 1010, Extec Corp., Enfield, CT, USA), under water cooling at 300 rpm, to obtain bonded sticks with a cross-sectional area of approximately 1 mm^2. Each specimen provided about nine sticks, which were immediately placed and glued in L2500 specimen holder. To reduce the cyanoacrylate glue dryness time used to hold the specimens at the holder, an accelerator was applied over it (Loctite 7452). After this procedure, the specimens were immediately tested. All sticks that debonded during specimen preparation (failed during cutting) were recorded but not included in the statistical analysis. Only sticks that failed during the microtensile test were noted and used in the statistical analysis. From the valid sticks of each specimen, the mean bond strength values were calculated and used in the statistical analysis.

The cross-sectional area of each stick was measured with a digital caliper (Absolute Digimatic, Mitutoyo, Tokyo, Japan) and recorded for subsequent calculation of the bond strength. Each bonded stick was glued to the L2500 specimen holder (Erios, São Paulo, SP, Brazil) using cyanoacrylate glue (454 Loctite; Itapevi, SP, Brazil) and submitted to a tensile test. The analyses were performed using a universal testing machine (DL200MF, EMIC, São José dos Pinhais, PR, Brazil), at a speed of 1 mm/min and using the 10 kg load cell, following the rules ISO/TS 11405 (2003).

After the test, all sticks were examined by a single observer using stereomicroscope at 50× magnification, and failures were classified as cohesive in dentin (CD), cohesive in liner material (CL), adhesive (A), or mixed (M).

Statistical analysis

The bond strength data were examined for normality assumption using Kolmogorov–Smirnov test (5%) and analyzed by one-way ANOVA ($p < 0.05$), followed by Tukey's test. Failure analysis was made using χ^2 test with 5% significance.

Results

The results of one-way ANOVA for bond strength evaluation revealed significant differences among the groups ($p = 0.000$, $df = 5$, $F = 88.15$). The results of Tukey's test are presented in Table 2. The application of an adhesive system prior to the use of a resinous liner material increased the bond strength values, improving its adhesion to dentin for all the materials tested. Although Ionoseal and Vitrebond presented similar bond strength values when the adhesive system was not applied, its application improved the bonding performance of Ionoseal more than the other liners tested. All sticks from the Ionosit group presented adhesive failure during the cutting procedure, making it impossible to test the bond strength of this group, which was considered zero. Figure 1 shows the failure distribution after the microtensile test, and χ^2 test revealed a significant difference among the failure types for all groups ($p < 0.0001$).

Table 2. Mean (MPa) and standard deviation (SD) of bond strength for the groups tested.

Type of adhesive	Mean (MPa)
Adhesive + IONOSEAL	27.43 (±4.48)^{A}
Adhesive + VITREBOND	20.70 (±5.17)^{B}
Adhesive + IONOSIT	19.90 (±2.76)^{BC}
IONOSEAL	16.37 (±4.46)^{CD}
VITREBOND	12.87 (±3.25)^{D}
IONOSIT	0.00 (±0.00)^{E}

Note. Sets with different letters are statistically different ($p < 0.05$).

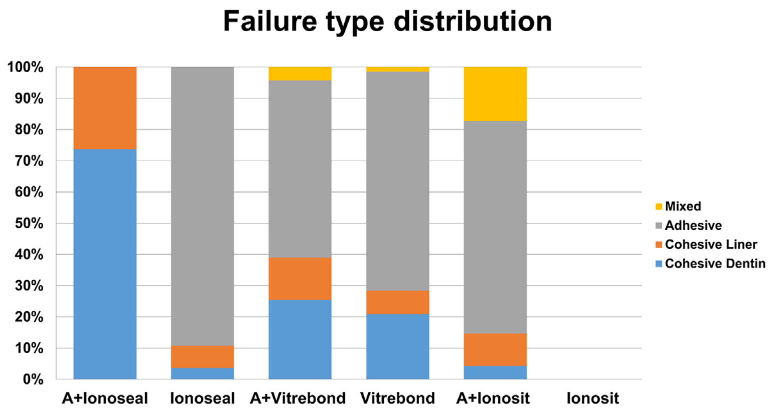

Figure 1. Failure distribution of liner materials tested.

Discussion

The null hypothesis tested was rejected, as the use of a self-etching dual-cure adhesive system improved the bond strength of resinous liner materials to the dentin surface for all liners tested. While the adhesive failures were predominant in almost all the groups tested (Figure 1), the failure analysis in this study indicates that the use of the adhesive system improved the bond strength in the dentin/liner interface, increasing the cohesive failures in the dentin and liner materials. The higher number of cohesive failures in dentin for the A+Ionoseal group can be related to the chemical compatibility between them, making the bonding of the liner with the adhesive layer stronger than the cohesive strength of the dentin. The application of an adhesive system prior to the use of a resin-modified glass ionomer liner might be a viable option to improve its bond strength to dentin.

Although the chemical interaction between ionomers and the calcium-rich layer from the tooth known as calcium chelation is clinically accepted, Kakaboura el al. observed failures between some liners and the dentin smear layer, suggesting the need for reinforcement of the dentin/liner interface.[10] This can be performed by conditioning dentin with polyacrilic acid before application of a resin-modified glass ionomer cement (RMGIC) as the liner.[9] Alternatively, improvement in the bond strength of liners over dentin can be achieved after acid etching and use of a two-step adhesive.[16] This study showed better behavior of the liners tested when the dual curing self-etching adhesive system was used. The Futurabond U adhesive tested in this study contains MDP, an acidic monomer [17] responsible for modifying the smear layer and demineralizing dental tissues, creating an hybrid layer.[18] As self-etching systems dismiss the use of phosphoric acid, they can be applied over dentin even in deep cavities, resulting in lower cytotoxic effects to pulp cells.[19]

The key to understand the influence of the adhesive system in the bond strength of the liner materials tested might be in their composition. The Vitrebond liner tested is classified as a RMGIC, because it presents an acid–basic reaction for setting, associated with resinous monomers responsible for the light curing. The polyalkenoic acid is responsible for the bonding properties, for reacting with hydroxyapatite, and displacing and replacing surface phosphate and calcium.[20] The water in these cements is responsible for ion transportation during the cement reactions. If water is lost, these reactions stop and the final mechanical properties of the cement are harmed. According to the manufacturer, it is recommended that the liner be applied directly over the dentin, without a previous treatment. On the other hand, Ionoseal is considered a polyacid-modified composite resin or compomer. It presented the highest bond strength values, whether associated with an adhesive system or not. It presents resinous acidic monomers similar to the ones found in self-adhesive resin cements that are used to achieve demineralization and bonding to the tooth surface. These monomers are predominantly methacrylate monomers with either carboxylic acid groups or phosphoric acid groups, which are responsible for demineralizing dentin surface and promoting stable bonding with calcium, forming a stable salt.[21]

In relation to Ionosit, which is also a compomer, all specimens failed during the stick sectioning, showing that the bonding between this material and dentin is very weak. However, when associated with an adhesive system, Ionosit presented bond strength values similar to A+Vitrebond, and almost 70% of adhesive failure. Therefore, this compomer liner showed a behavior much more like a composite than glass ionomer materials. The polycarbonic acids present in its composition, declared by the manufacturer, probably did not react sufficiently with the dentin surface to promote self-adhesive properties. As a consequence of the poor bonding performance of liner materials, the formation of gaps between them and the dentin pulpal wall is very probably, due to its debonding,[10] allowing for the occurrence of microleakage. The manufacturer recommends the previous application of some adhesive system over the dentin, before the use of this material, probably because of its low self-adhesive properties.

Usually conventional glass ionomer liner materials are applied to decrease the shrinkage stresses in Class I or II composite resin restorations, in the so-called 'sandwich' technique. This technique aims to reduce microleakage and improve the marginal adaptation of the restorations by decreasing the volume of composite resin used.[22–24] As resin-modified glass ionomers and compomers contain resinous monomers incorporated into their matrix, they also present shrinkage during the curing process, similar to composites.[25,26] The main problem associated with the shrinkage is the stress formation in the tooth–material interface, which can lead to gap formation due to the tendency for the restoration to be pulled away from the cavity wall.[27] Because of these gaps, microleakage can occur and cause the restoration to fail.[26,28] The use of the adhesive system as proposed in this study might increase the bond strength of the dentin/liner interface, and avoid the formation of these gaps and, consequently, microleakage.

Due to their resinous composition, the bonding of these liners to a composite is expected to be high. After its application, an oxygen-inhibited layer will allow its chemical bonding to the resinous monomers of the composite. In fact, a study proposed the use of an adhesive system between the liner and composite to improve the bonding between them.[29] Based on that, it is questioned whether the set liner–composite would rip the liner material off from the dentin. Therefore, the use of an adhesive system prior to the application of the liner could also help to improve the bonding between the material and dentin, avoiding this problem. Actually, some manufacturers already recommend this previous application, as is the case of Ionosit. However, this is not a standard procedure for all materials, creating doubts about what would be the best decision.

Despite all limitations of an *in vitro* analysis, this study shows that the prior application of an adhesive system to glass ionomer liners increased their bond strength to dentin. However, careful interpretation of the results should be taken, as the setup used did not include the placement of composite resin over the samples, as is mandatory in clinical situations. The choice for this different setup was made to avoid the creation of a second adhesive interface between the liner and the composite, which might lead to confusing results. Also, bovine incisors were used, which can be different from human teeth in vital situations, although they can be considered adequate substitutes for human teeth due the lack of substrate availability for research nowadays.[30,31] Additional *in vivo* studies should be performed to confirm that the application of adhesive before the liners improves the clinical performance of composite restorations.

Conclusions

Regardless of the limitation of this study, it can be concluded that the use of an adhesive system before the application of a liner material increased the bond strength for all materials tested. Associated with an adhesive system, Ionoseal showed the highest mean bond strength, and no significant differences were observed between Vitrebond and Ionosit. However, without the application of adhesive, Ionosit did not present any measurable bond to the dentin.

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