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Bond strength of a composite resin to glass ionomer cements using different adhesive systems

Resistência de união de uma resina composta a cimentos de ionômeros de vidro utilizando diferentes sistemas adesivos

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Resumo

Introdução: Os cimentos de ionômero de vidro são frequentemente usados como base ou forramento cavitário, previamente ao material restaurador. **Objetivo:** Avaliar a resistência de união da resina composta a diferentes cimentos de ionômero de vidro, utilizando sistemas adesivos convencional simplificado e auto-condicionantes. **Material e método:** Foram utilizados três cimentos (Ketac Molar Easymix, Vitremer e Vitrebond), a resina Filtek Z350 XT e os sistemas adesivos Adper Single Bond 2, Clearfil SE Bond e Adper Easy One. Como controle negativo, a resina foi aplicada sobre o cimento sem a utilização de sistema adesivo. Cavidades (4 mm de diâmetro e 2 mm de profundidade), confeccionadas em blocos acrílicos, foram preenchidas com os cimentos ionoméricos (n=12/grupo). Na superfície foi delimitada uma área de 1mm de diâmetro, aplicado o sistema adesivo e confeccionado um espécime de resina composta com 1 mm de altura. Após 24 horas de armazenamento (37 °C e 100% de umidade), foi realizado o ensaio de microcisalhamento. Os dados foram analisados usando ANOVA a dois fatores, e teste de Tukey para comparação entre grupos (α=0,05). **Resultado:** Os sistemas adesivos melhoraram significativamente a resistência de união resina/cimento de ionômero de vidro (p≤0,001). Não houve diferença significativa na resistência de união quando os sistemas adesivos auto-condicionantes foram comparados com o convencional simplificado, com exceção no Vitrebond onde o Clearfil SE Bond determinou maior resistência de união quando comparado com o Adper Single Bond 2 (p=0,003). **Conclusão:** Os sistemas adesivos auto-condicionantes constituem uma boa opção para estabelecer a união entre resina composta e cimento de ionômero de vidro.

Descritores: Cimentos de ionômeros de vidro; adesivos dentinários; resinas compostas; resistência ao cisalhamento.

Abstract

Introduction: Glass ionomer cements are often used as a base or cavity lining prior to restorative material. **Objective:** To evaluate the bond strength of a composite resin to different glass ionomer cements, when using a two-step conventional and self-etching adhesive systems. **Material and method:** Three glass ionomer cements (Ketac Molar Easymix, Vitremer and Vitrebond), the composite resin Filtek Z350 XT and the adhesive systems Adper Single Bond 2, Clearfil SE Bond and Adper Easy One were used. As negative control, resin was bonded to cement without using an adhesive system. Holes (4 mm diameter, 2 mm deep) prepared in acrilic bloks were filled with the glass ionomer cements (n=12/group). On the surface, an area of 1mm in diameter was delimited, the adhesive system was applied, and a specimen of composite resin with 1 mm height was made. After 24 hours storage (37 °C and 100% humidity), the microshear test was performed. Data were analyzed using two-way ANOVA and Tukey test for comparison between groups (α=0.05). **Result:** The adhesive systems significantly improved the bond strenght of composite resin to glass ionomer cements (p≤0.001). There was no significant difference in bond strength when self-etching adhesive systems were compared with the simplified etch-and-rinse adhesive, except for Vitrebond where Clearfil SE Bond determined higher bond strength when compared to Adper Single Bond 2 (p=0.003). **Conclusion:** Self-etching adhesive systems are a good option for establishing the bond between the composite resin and the glass ionomer cement.

Descriptors: Glass ionomer cements; dentin-bonding agents; composite resins; shear strength.

INTRODUCTION

The increasing demand for aesthetic restorations and the evolution of composite resins (CR) have encouraged the use of these materials in posterior teeth. However, when they are associated with adhesive systems in deep dentin without a protection of the pulpodentin complex, they are an important factor contributing to pulpal irritation[1,2].

Glass ionomer cement (GIC) lining prior to restoration seems to increase clinical success, because this procedure associates the low coefficient of thermal expansion, biocompatibility[3] and anticariogenic activity[4,5] of GIC with the aesthetic and fracture resistance properties of composite resins. However, the lack of chemical bonding between composite resin and conventional GICs may interfere in the final properties of the restoration, and consequently, its longevity[6]. To improve the GIC/composite resin bond, the use of resin-modified glass ionomer cements may be indicated[3,7].

Another method to optimize the bond GIC/composite resin is to perform phosphoric acid etching on the GIC surface, which increases the retentive microporosities and consequently improves the micromechanical retention[8]. Nonetheless, moisture contamination during setting of the GICs may cause dissolution of the calcium polyacrylate chains altering their physical properties[9], therefore it is advisable to wait for the initial setting to occur before carrying out the acid etching and washing[6,10].

The use of self-etching adhesive systems may solve this problem, since they do not require the washing step[6]. In addition, one or more carboxylic or phosphate groups may be incorporated into these self-etching adhesive systems, and studies have shown that they exhibit enamel and dentin bond strength similar to that of total acid etching (etch and rinse) adhesive systems[11,12]. Although these adhesive systems have shown promising results, to date few studies have evaluated the composite resin bond to glass ionomer cements when using these materials.

Thus, the objective of this study was to evaluate the bond strength of a composite resin and both conventional and resin modified glass ionomer cements when using a simplified etch-and-rinse and self-etching adhesive systems. The null hypothesis was that the type of adhesive system did not interfere with the bond strength of a composite resin to different glass ionomer cements.

MATERIAL AND METHOD

Materials

The materials used in this study are shown in Table 1.

Experimental Design

The Groups were distributed according to the variation factors, totalizing 12 levels of variation (Table 2):

Table 1. Commercially available materials used in the study

Trade name/ Manufacturer	Main Components (% in weight)	Powder/ Liquid ratio	PTR/TIS
Ketac Molar Easy Mix (3M ESPE, St. Paul, MN, USA)	Powder: glass powder (fluor-alumino-silicate crystals), polyacrylic acid	2.9:1	5 min
	Liquid: water, polyethylene polycarbonic acid, tartaric acid		
Vitremer (3M ESPE, St. Paul, MN, USA)	Powder: treated glass (fluorine-alumino-silicate glass), potassium persulfate	2.5:1	40 s
	Liquid: copolymers of acrylic and itaconic acids, water; HEMA		
Vitrebond (3M ESPE Dental Products, St. Paul, MN, USA)	Powder: fluorine-alumino-silicate glass, diphenyl iodine chloride	1.4:1	30s
	Liquid: copolymers of acrylic and itaconic acids, HEMA, water		
Filtec Z350 (3M ESPE, St. Paul, MN, USA)	Bis-GMA, UDMA, Bis-EMA, PEGDMA, TEGDMA, silanized ceramics, zirconia and silanized silica, BHT <5%	--	20s
Scotchbond Etchant (3M ESPE, St. Paul, MN, USA)	Phosphoric acid 35%	--	--
Adper Single Bond 2 (3M ESPE, St. Paul, MN, USA)	Bis-GMA, HEMA, diurethane dimethacrylate, copolymer of polyalkenoic acid, camphorquinone, water, ethanol and glycerol 1.3 dimethacrylate, 10% by weight of silica nanoparticles	--	10s
Clearfil SE Bond (Kuraray Co., Ltda., Osaka, Osaka, Japan)	Primer: HEMA, hydrophilic dimethacrylate, MDP, N, N-diethanol p-toluidine, camphorquinone, water		10s
	Adhesive: Bis-GMA, HEMA, hydrophobic dimethacrylate, MDP, camphorquinone, N, N-diethanol-p-toluidine, silanized colloidal silica		
Adper Easy One (3M ESPE, St. Paul, MN, USA)	Ester phosphoric methacrylate, Vitrebond copolymer, nanoparticles, ethanol, water, dimethacrylates, HEMA, initiators		10s

PTR: photoactivation time recommended by the manufacturer; TIS: waiting time for initial setting; HEMA: 2-hydroxyethyl methacrylate; Bis-GMA: Bisphenol A diglycidyl ether dimethacrylate; UDMA: Diurethane dimethacrylate; BIS-EMA: Bisphenol A polyethylene glycol diether dimethacrylate; PEGDMA: Polyethylene glycol dimethacrylate; TEGDMA: triethylene glycol dimethacrylate; BHT: 2,6-di-tert-butyl-p-cresol; MDP: 10-methacryloyloxy methacrylate.

Table 2. Distribution of the study groups according to material and type of bond surface treatment

Glass Ionomer Cement	Surface Treatment	Description	n
KM	no treatment	Ketac Molar Easymix + Filtek Z350 XT	12
	SB	Ketac Molar Easymix + acid conditioning +Adper Single Bond 2 + Filtek Z350 XT	12
	EO	Ketac Molar Easymix + Adper Easy One + Filtek Z350 XT	12
	CSE	Ketac Molar Easymix + CSE primer + CSE Bond + Filtek Z350 XT	12
VM	no treatment	Vitremer + Filtek Z350 XT	12
	SB	Vitremer + acidic conditioning + Adper Single Bond 2 + Filtek Z350 XT	12
	EO	Vitremer + Adper Easy One + Filtek Z350 XT	12
	CSE	Vitremer + CSE primer + CSE Bond + Filtek Z350 XT	12
VB	no treatment	Vitrebond + Filtek Z350 XT	12
	SB	Vitrebond+ acidic conditioning + Adper Single Bond 2 + Filtek Z350 XT	12
	EO	Vitrebond + Adper Easy One + Filtek Z350 XT	12
	CSE	Vitrebond + CSE primer + CSE Bond + Filtek Z350 XT	12

n: number of specimens; KM: Ketac Molar Easymix; VM: Vitremer; VB: Vitrebond; SB: Adper Single Bond 2; EO: Adper Easy One; CSE: Clearfil SE Bond.

1 - Material: high viscosity GIC (Ketac Molar Easymix - 3M ESPE, St. Paul, MN, USA); resin-modified GIC for restoration (Vitremer - 3M ESPE, St. Paul, MN, USA) and resin-modified GIC for base/lining (Vitrebond - 3M ESPE, St. Paul, MN, USA);
2 - Type of GIC surface treatment: no treatment, simplified etch-and-rinse adhesive system (Adper Single Bond 2 - 3M ESPE, St. Paul, MN, USA), two-step self-etching adhesive system (Clearfil SE Bond 2 - 3M ESPE, St. Paul, MN, USA), and one-step self-etching adhesive system (Adper Easy One - 3M ESPE, St. Paul, MN, USA).

Preparation of the Specimens

For specimen preparation, blocks (20 mm high, 18 mm in diameter) containing three holes (4 mm in diameter, 2 mm deep) in the upper surface, were made of acrylic resin (OrtoClas, Artículos Odontológicos Clássico Ltda. Ind. Bras, São Paulo, SP, Brazil).

The holes were filled with the GICs which were manipulated in the powder/liquid ratio (by weight) recommended by the manufacturers and inserted by means of a Centrix syringe (DFL, São Paulo, SP, Brazil), in a single increment. After application of the material, a polyester matrix strip, followed by a glass plate and a 500 g weight were placed on its surface for 30 seconds to ensure a flat, smooth surface. Resin-modified glass ionomer cements Vitremer and Vitrebond, were photoactivated for the time recommended by the manufacturer by using a photocuring unit (UltraLed, Dabi Atlante Inds., Medical Odontology Ltda, Ribeirão Preto, SP, Brazil) previously evaluated for light intensity (450 ± 10 mW / cm^2). For the conventional glass ionomer cement (Ketac Molar Easymix), the time required for the initial setting (5 min) was waited. The matrix/GIC set was stored at 37 °C with 100% humidity for 24 h.

In the second stage of specimen preparation, the GIC surfaces were sanded with 320 grit abrasive paper to simulate wear with a diamond drill. The bond area was delimited by means of resistant double-sided adhesive tape (Tectape, Manaus, AM, Brazil) with a central perforation 1.0 mm in diameter, made by means of an adapted rubber sheet perforator (model Ainsworth, Wilcos do Brasil Indústria e Comércio Ltda., Petrópolis, RJ, Brazil). The dentin adhesive systems: Adper Single Bond 2, Clearfil SE Bond and Adper Easy One were applied on the GICs in accordance with the respective manufacturers' recommendations and then were photoactivated.

A plastic microtube (1.0 mm internal diameter and 2.0 mm high) (Tygon, Norton Performance Plastic Co, Cleveland, OH, USA) was fitted with the internal area coinciding with the area bounded by the adhesive tape and was used as a matrix for preparing the specimens. The composite resin Filtek Z350 XT was inserted into the microtube in a single increment, with the aid of a clinical probe (Duflex, Rio de Janeiro, RJ, Brazil) and photoactivated for 20 seconds as recommended by the manufacturer. As a control group, composite resin was applied on the glass ionomer cements without any previous surface treatment.

The specimens were stored at 37 °C with 100% humidity for 48 hours. After this period, the plastic microtube was removed with the help of a nº.15 scalpel blade (Embramed, São Paulo, SP, Brazil) and the specimens were observed under a stereoscopic magnifying glass at approximately 50x magnification to certify the absence of defects at the bond interface.

Microshear Bond Strength Test

The mechanical microshear test was performed in a mechanical testing machine (DL-Digital line, EMIC, São José dos Pinhais, PR, Brazil), previously adjusted for tensile forces, with a load cell of 100 N. To perform the test, the specimen was positioned in the machine in line with the extension of the load cell, a metal wire 0.2 mm in diameter was loosely adapted to simultaneously bring the load cell and composite resin cylinder as closely as possible to the bond interface.

Traction movements were made at a speed of 0.5 mm/min. The tests started by means of a specific computerized program (Tesc-Test Script, EMIC Equipamentos de Ensaio Ltda, São José dos Pinhais, PR, Brazil) and proceeded until fracture. The maximum stress values in MegaPascal supported by the union composite resin/GIC were recorded.

Fracture Pattern Analysis

After the microshear test, the specimens were stored in closed recipients at room temperature. The failure type was identified by examination under a stereomicroscope (Mod SZX7, Olympus, São Paulo, SP, Brazil), at approximately 50x magnification. The failures were classified as: adhesive (failure at composite resin/GIC interface), cohesive (failure within GIC or composite resin), or mixed (combination of adhesive and cohesive failure).

Statistical Analysis

To evaluate the influence of using the different adhesive systems on the bond strength of composite resin to the different GICs, the normality and the homoscedasticity of the data were initially observed. Based on these assumptions, a parametric test (two-way ANOVA) was used, and the Tukey post-test was applied to identify the differences between the groups. The level of significance adopted for decision making was 5% ($\alpha = 0.05$).

The fracture patterns were descriptively analyzed.

RESULT

The means and standard deviations of bond strength are presented in Table 3. The use of adhesive systems significantly improved the bond strength of the composite resin to GICs ($p \leq 0.001$), and bond strength was influenced by the type of glass ionomer cement and adhesive system ($p < 0.001$).

When bonding was produced on a conventional glass ionomer cement (KM), the CSE adhesive system showed bond strength values similar to SB (p=0.072) and higher than EO (p=0.005). For bonding produced on VM, adhesive systems did not influence the bond strength results ($p \geq 0.126$), while for VB, the CSE adhesive system showed bond strength values higher than SB (p=0.003) and similar to EO (p=0.287). The EO and SB adhesive systems did not show statistically significant difference for the different GICs ($p > 0.05$).

Table 3. Bond strength of composite resin (Filtek Z350 XT) to different GICs, according to the surface treatment (adhesive system) applied

Adhesive system	Bond strength (MPa) GIC		
	KM	VM	VB
no adhesive system	7.41(2.53)*Bc**	12.88(4.13) Aa	4.08(1.10) Bc
SB	13.87(4.21) ABab	16.90(6.17) Aa	10.94(2.50) Bb
CSE	18.44(6.52) Aa	18.00(6.99) Aa	15.33(3.25) Aa
EO	11.08(3.58) Bbc	18.85(3.01) Aa	13.21(2.71) Bab

KM: Ketac Molar Easymix; VM: Vitremer; VB: Vitrebond; SB: Adper Single Bond 2; CSE: Clearfil SE Bond; EO: Adper Easy One; *Values represent mean (standard deviation); **Different capital letters denote statistically significant difference in the comparison between GICs and different lower case letters denote statistically significant difference in the comparison between adhesive systems (ANOVA and Tukey test, $p \leq 0.05$).

The distribution of failure types observed, considering the GICs and adhesive systems used, is presented as a percentage of occurrences in Figure 1. Adhesive failures were identified in 100% of cases when no surface treatment of GIC was performed before placement of the composite resin. No cohesive fracture of the composite resin was observed. For conventional GIC, Ketac Molar Easymix, irrespective of the adhesive system used, 50% of the failures observed were cohesive within GIC, 27.5% were adhesive and 22.5% mixed. In the resin-modified GICs, the failures were distributed as follows: Vitremer (47.9% adhesive, 27.1% cohesive within the material and 25% mixed); and Vitrebond (44.7% adhesive, 23.7% cohesive within the material and 31.6% mixed failure). Adhesive systems had a significant influence on the failure type (Figure 1).

DISCUSSION

Our findings showed that bond strength of the composite resin to different glass ionomer cements was influenced by adhesive system used. Few studies have evaluated the bond strength of composite resin to glass ionomer cements when different types of dentin adhesive systems were used. Sá et al.[13] compared the bond strength of composite resin to different conventional GICs, by using three adhesive systems, and either performing etching with 37% phosphoric acid, or not, before their application. They

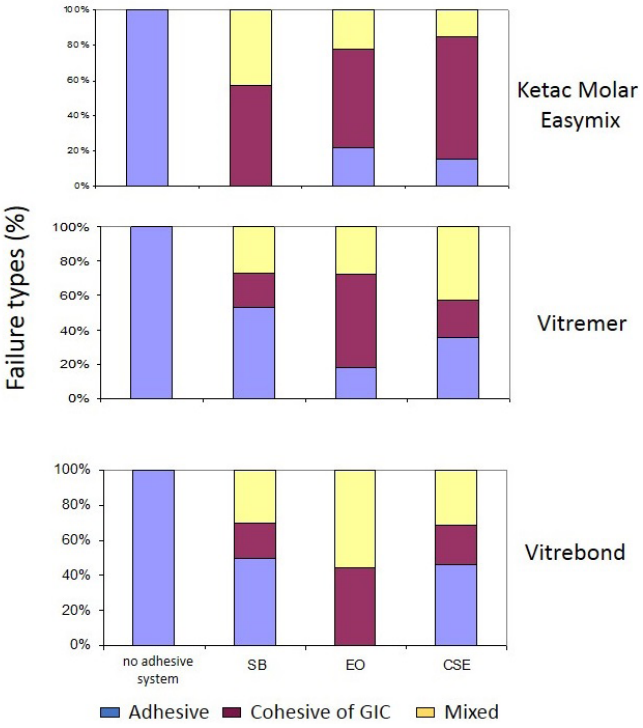

Figure 1. Failure types (%) observed after microshear bond strength test according GIC and adhesive system used. GIC= Glass Ionomer Cement; SB= Adper Single Bond 2; EO= Adper Easy One; CSE = Clearfil SE Bond.

concluded that acid etching was dispensable for both base/lining and restoration GICs. In our study, the bond strength between composite resin and GICs when using a simplified etch-and-rinse and self-etching adhesive systems improved significantly when compared with application of the composite resin directly onto the GIC. Similar results were observed by Kundabala et al.[14]. According to Hinoura et al.[15] adhesive systems improved the wettability of the GIC, and consequently, improved the bond to the composite resin.

Pamir et al.[16] comparing a simplified etch-and-rinse and a single-step self-etching adhesive did not observe significant difference in bond strength of a composite resin to the conventional and resin-modified GICs . In the present study, we found no significant difference between the single-step self-etching (EO) and simplified etch-and-rinse (SB) adhesive systems. But there was difference between CSE (two-step self-etching adhesive) and SB only when the glass ionomer VB was used as base. Although the statistically significant difference was not always present, self-etching adhesive systems determined higher mean bond strength values between composite resin and resin-modified GICs than the conventional adhesive system. Kundabala et al.[14] and Zhang et al.[17] also observed higher values of bond strength when using self-etching adhesive systems. This may have been due to a lower viscosity of self-etching adhesive systems, which determines a lower angle of contact with the surface of the GICs, resulting in better wettability, and consequently, better bonding between composite resin and GICs[18].

Venkateshbabu et al.[19], studying the bonding capacity of composite resin to GIC when using strong self-etching adhesives (pH=1.0), intermediate strength (pH=1.4) and weak (pH=2.2) adhesives, concluded that weak self-etching adhesive systems significantly improved bond strength when compared with other adhesive systems. When strong acids are used higher cation neutralization and formation of fragile structure salts occur, which adversely affect the bond strength.

In this study, as in the study of Zhang et al.[17], the only significant difference in composite resin/GIC bond strength between two-step (pH 2.0) and one-step (pH 2.3) self-etching adhesive systems was observed when they were applied on conventional GIC. According to these authors[17], the cohesive type of failure of conventional GIC was relatively common and when this type of failure occurs, the real strength of the interfacial bond between the GIC and composite resin must not being evaluated, since the cohesive strength of GIC's acts as a limiting factor in bond strength tests.

When the composite resin was applied directly onto the GIC, we obtained low bond strength values and 100% of the failures that occurred were adhesive. When the bond of the composite resin to the conventional GIC was established using adhesive systems 50% of the fractures observed were cohesive of the GIC. According to Gupta, Mahajan[10], the bond strength between composite resin and conventional GIC was reduced by the low cohesive strength of the GIC and the absence of chemical bonding, due to the different setting reactions occurring in these materials. Sneed, Looper[20], considered that the bond strength between composite resin, adhesive system and GIC was greater than the cohesive force of the GIC. Moreover, when the etch-and-rinse adhesive system was used on the conventional GIC, phosphoric acid etching could dissolve surface charged particles[21,22] creating a zone of fragility that resulted in cohesive failure of the material, possibly resulting in lower bond strengths[17,22].

According to Kerby, Knobloch[23], resin-modified glass ionomer cements exhibit significantly higher cohesive strength when compared with conventional GICs. In the present study, resin-modified glass ionomer cement (Vitremer) showed the highest bond strength to composite resin. These results suggested that because this material exhibited a higher cohesive strength, the evaluation reflected higher real bond strength values at the composite resin/adhesive/GIC interface. According to Kundabala et al.[14] due to the presence of resinous monomers in their composition, resin-modified GICs showed a chemical bond to the composite resin, which favoured the increase in bond strength.

In this study, when the composite resin bond to the resin-modified GICs was established with the use of the conventional adhesive system, about 80% of the fractures were of the adhesive or mixed type. Whereas, when the self-etching adhesive systems were used, cohesive of GIC and mixed failures prevailed.

Although only CSE adhesive system showed significant difference in bond strength when compared with SB, there was considerable difference in behaviour with regard to the type of failure that occurred during the microshear test when the simplified etch-and-rinse and self-etching adhesive systems were considered. Therefore, further studies are needed to evaluate the longevity of composite resin/GIC bonding when using conventional and self-etching adhesive systems.

CONCLUSION

The authors verified that the composite resin/GIC bond strength improved significantly with the use of adhesive systems. Although there was no significant difference in bond strength when self-etching adhesive systems were compared with the simplified etch-and-rinse adhesive, Clear Fil SE bond determined the highest bond strength values. Therefore, self-etching adhesive systems are a good option for establishing the bond between the composite resin and the glass ionomer cement.

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CONFLICTS OF INTERESTS

The authors declare no conflicts of interest.

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