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Bond strength evaluation of composite resin bonded to glass ionomer cements after different periods of setting

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ABSTRACT

This study investigated the time elapsed after setting of glass ionomer cements on the bond strength to composite resin restorations. Bovine incisors received cavity preparations on the buccal surface (6 mm × 6 mm × 2 mm) and the specimens were tested according to cement type (conventional and resin-modified) and time elapsed before performing the restorations: GC10m: conventional glass ionomer cement and 10 min time elapsed after setting; GC24h: conventional cement and 24 h after setting; GC7d: conventional cement and 7 days after setting; GRM10m: resin-modified glass ionomer cement and 10 min after setting; GRM24h: resin-modified cement and 24 h after setting; and GRM7d: resin-modified cement and 7 days after setting. Specimens were subjected to micro-shear testing and the data were analyzed by Analysis of Variance and Tukey's test ($p=0.05$). Bond strength of restorations performed on conventional cement after 10 min of time elapsed presented the lowest mean values and differed statistically from values at 24 h and 7 days. Resin-modified cement after 24 h presented the highest mean values and differed statistically from values at 10 min and 7 days. The time elapsed after setting of glass ionomer cement may interfere in the bond strength to composite restorations.

1. Introduction

Dental adhesive resin systems, used to enhance retention and reduce microleakage of composite resin restorations, when applied in deep dentin, can penetrate into the dentinal tubules and induce pulp inflammation and lack of dentin regeneration [1–3]. The humidity present in this area inhibits complete polymerization of the adhesive, and exposure to partially polymerized adhesive induces apoptosis of approximately 40–50% odontoblast-like cells and undifferentiated pulp cells after 12 h [4]. A histological evaluation of exposed human dental pulps capped with different adhesives showed that the pulp response varied from acute inflammatory cell infiltrate with varying degrees to necrosis, indicating that these materials should not be applied directly to the pulp [3,5–7].

The majority of the substances released by resin-based dental restorative materials are able to cause cytotoxic and genotoxic effects and irreversible disturbance of basic cellular functions, such as cell proliferation, enzyme activities, membrane integrity, and cell morphology, metabolism and viability. Signaling pathways involved in the immune response, tissue homeostasis and repair are also affected [1,2,8]. Understanding outcomes of the interaction between a dental material and tooth tissue is important, not only in terms of biocompatibility, but also of the potential for the material to modulate the tissue response [1,9].

Glass ionomer cements (GICs) are extensively used as lining materials in composite restorations because they enable composites to be attached to dentin with the glass ionomer cement functioning as a biocompatible intermediate layer with low cytotoxic effects [1,9,10]. Since its introduction in dental practice, many attempts have been made to improve the strength of GIC, for

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example, by incorporating a secondary filler such as metal powder (e.g., silver–tin, gold, platinum, palladium, stain-less steel), fibers (e.g., carbon, glass) [11–13], or by modifying the system with a resin component, with a separate setting mechanism to form a so-called resin-modified GIC (RMGIC) [14,15].

Conventional GICs are formed by an acid-base reaction between a degradable aluminosilicate glass and an aqueous solution of polyalkenoic acid. The acid attacks and degrades the aluminosilicate glass structure, releasing calcium and aluminum cations. These cations are then chelated by the carboxylate groups and crosslink with the polyalkenoic acid chains. This crosslinking reaction is a continuous process evident by the increase in mechanical properties of the cement with time [16]. Resin-modified glass ionomers were introduced as a hybrid between conventional resin composites and glass ionomers [17]. These materials polymerize by up to three mechanisms: (1) an acid–base reaction between the polyacrylic acid and the fluoroaluminosilicate glass; (2) a photo-initiated free-radical reaction between methacrylate monomers; and (3) a chemically-initiated reaction between methacrylate monomers remaining after photo-initiation [18–20].

It has been reported that the continuous acid-base reaction of the GICs enhances the performance level of these materials, increasing the mechanical properties of the cement with time [21]. The aim of the present study was to evaluate the influence of time elapsed after setting reaction of conventional and resin-modified glass ionomer cements on the bond strength of composite resin restorations. The null hypothesis tested was that there is no influence of the elapsed time of setting on bond strength of resin restorations.

2. Materials and methods

Commercial brand names, chemical composition and material manufacturers are presented in Table 1. In order to obtain specimens for the micro-shear bond strength test, the experimental set-up shown in Fig. 1 was carried out [22].

Thirty freshly extracted bovine incisors that were refrigerated in a solution of 0.05% thymol (LabSynth Produtos para Laboratórios Ltda., Diadema, SP, Brazil) for no longer than three months after extraction and placed in distilled water for 24 h before beginning the experiment. They were examined at ×10 magnification (Carl Zeiss, Oberkochen, West Germany) to ensure that there were no cracks, defects, or caries. The specimens were prepared using a flexible diamond disc at low speed (n.7020—KG Sorensen, Barueri, SP, Brazil) under water cooling to remove the tooth root. The pulp chamber was cleaned using manual instruments.

After root section, the crowns were embedded in acrylic resin (Vipi, Pirassununga, SP, Brazil) and cavity preparations measuring 6.0 mm × 6.0 mm × 3.0 mm were made on the buccal surface using a carbide bur (n.330—KG Sorensen, Barueri, SP, Brazil) adapted to a high-speed handpiece. Each bur was replaced after being used for five cavity preparations. The cavity preparations were accomplished under copious water irrigation and concluded using manual cutting instruments.

The glass ionomer cements were manipulated according to the manufacturers' instructions, and inserted in the cavity preparations. Materials were covered with mylar strips, and a constant and uniform load of 250 g was applied, using a custom-made device for 2 min for the conventional GIC (Vitro Fil, DFL, Rio de Janeiro, RJ, Brazil) or during the photoactivation (40 s) of the RMGIC (Vitro Fil LC, DFL, Rio de Janeiro, RJ, Brazil). The materials were randomly divided into six experimental groups and tested according to glass ionomer time elapsed after setting:

- GC10m: 10 min after setting;
- GC24h: 24 h after setting;
- GC7d: 7 days after setting;
- GRM10m: 10 min after setting;
- GRM24h: 24 h after setting;
- GRM7d: 7 days after setting.

The composite restorations were made waiting the time elapsed after glass ionomer setting. The specimens belonging to groups GC10m and GRM10m were not stored, and the restorations were performed after the time elapsed.

Samples belonging to groups GC24h, GC7d, GRM24h and GRM7d were stored in artificial saliva at 37 °C after surface protection with a dental varnish (Varnal—Biodinâmica, Ibiporã, PR, Brazil) and the restorations were performed after the determined period. The buccal surfaces were wet-ground with 180, 220, 400 and 600-grit SiC abrasive papers using a polishing machine (APL-4, Arotec, Cotia, SP, Brazil) to remove 1.0 mm of the tooth structure and the glass ionomer creating a smooth, flat surface.

To perform the resin restorations the glass ionomer surface was treated with 37% phosphoric acid (Adper Scotchbond—3M/ESPE, St. Paul, MN, USA) for 15 s and rinsed for 15 s. Absorbent paper was used to remove the excess moisture (Snack, Melhoramentos Papéis Ltda., Caieiras, SP, Brazil). The adhesive system (Single Bond 2—3M/ESPE, St. Paul, MN, USA) was applied on the glass ionomer surface, according to the manufacturer's instructions. Customized 0.5 mm-thick elastomer molds with cylinder-shaped orifices (1.2 mm in diameter), were placed on the tooth surfaces, allowing delimitation of the bonding area. After photo-activation of the bonding agent for 10 s (LED light curing unit, Coltolux LED—Coltène/Whaledent, Cuyahoga Falls, OH, USA), the orifices were filled with composite resin (Filtek Z250—3M/ESPE, St. Paul, MN, USA) and photo-activation was performed again for 40 s. The output irradiance of the curing unit was 850 mW/cm^2, confirmed with a digital power meter (Ophir Optronics, Danvers, MA, USA). After polymerization, the specimens were stored in deionized water at 37 °C for 24 h. All resin cylinders were checked

Table 1
Materials tested—commercial brand names, manufacturer, batch number, and composition*.

Material	Composition	Batch number
Adper Single Bond 2 (3M/ESPE, St Paul, MN, USA)	Silane treated silica (nanofiller). Bis-GMA, HEMA. Dimethacrylate, methacrylate functional copolymer of polyacrylic and polytaconic acid. Water. Ethyl alcohol	9UU
Filtek Z250 (3M/ESPE, St Paul, MN, USA)	Silane treated ceramic. BISEMA. UDMA. BISGMA. TEGDMA	N148344BR
Vitro Fil (DFL, Rio de Janeiro, RJ, Brazil)	Powder—strontium aluminum silicate, dehydrated polyacrylic acid, and iron oxide Liquid—polyacrylic acid, tartaric acid and distilled water	09121362
Vitro Fil LC (DFL, Rio de Janeiro, RJ, Brazil)	Powder—strontium aluminum silicate, excipients, activators and iron oxide Liquid—2-hydroxyetil methacrylate, polyacrylic and tartaric acid solutions, benzoyl peroxide and camphorquinone	09020210

* According to manufacturer's information.

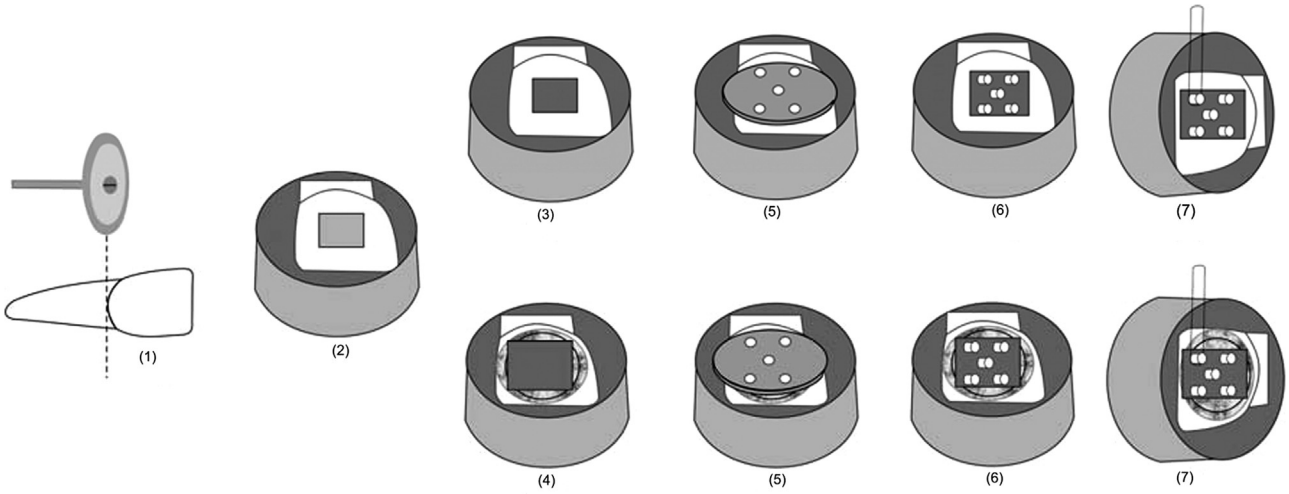

Fig. 1. Experimental design of the study. (1) Removal of the tooth root; (2) teeth embedded in acrylic resin showing cavity preparation—6.0 mm × 6.0 mm × 3.0 mm; (3) cavity preparation filled with glass ionomer and showing flat surface—GC10m and GRM10m; (4) cavity preparation filled with glass ionomer and showing flat surface after being wet-ground—GC24h, GC7d, GRM24h and GRM7d; (5) elastomer mold with cylinder-shaped orifices positioned on the surface; (6) resin cylinders after polymerization; (7) micro-shear test.

at 40 × magnification. Those presenting flaws, irregularities or bonding defects were eliminated.

For the micro-shear test, a thin steel wire (0.2 mm in diameter) was looped around each cylinder and aligned with the bonding interface. The test was conducted in a universal testing machine (model AG-IC; Shimadzu, Kyoto, Japan), at a crosshead speed of 0.5 mm/min until failure. Micro-shear bond strength calculations were made using the following equation: $s=L/A$, where s is the ultimate shear strength (MPa), L is the shear loading at the moment of failure (N), and A is the bonding area (mm^2). For each group, five specimens were tested, consisting of five cylinders per specimen. The cylinders were considered as experimental unit.

Bond strength data were submitted to an exploratory analysis that showed the data could be evaluated by parametric tests due to normal distribution (Kolmogorov–Smirnov test and Shapiro–Wilk test—$\alpha=0.05$) and homogeneity (Levene's test—$\alpha=0.05$) indicating the use of one-way analysis of variance (ANOVA) and Tukey post-hoc test at a 5% level of confidence. The fractured specimens were examined by optical microscopy at 200 × magnification. Failure modes were classified as follows: adhesive failure (Mode 1), cohesive failure within glass ionomer (Mode 2), cohesive failure within composite resin (Mode 3), or mixed failure involving bonding agent and glass ionomer (Mode 4).

3. Results

Table 2 shows the mean micro-shear bond strengths and standard deviations for the experimental groups. One-way ANOVA revealed significant difference among the conventional cement groups ($p=0.0001$) and resin-modified cement groups ($p=0.0083$).

Considering the conventional cement groups it was observed that the bond strength of the restorations performed after the time elapsed of 10 min presented the lowest mean values (4.20 MPa), and differed statistically from the values at 24 h (7.58 MPa) and 7 days (9.59 MPa). No statistical difference was observed between these two groups.

Resin modified glass ionomer cement stored for 24 h before the composite restorations presented the highest mean values (29.44 MPa) and differed statistically from the values at 10 min (19.55 MPa) and 7 days (23.76 MPa). No statistical difference was observed between these two groups.

Table 2
Mean bond strength values in MPa (± standard deviation) and Tukey post-hoc*.

	10 min	24 h	7 Days
Conventional glass ionomer	$4.20^{B,b}$ (± 2.01)	$7.58^{A,b}$ (± 3.34)	$9.59^{A,b}$ (± 3.28)
Resin modified glass ionomer	$19.55^{B,a}$ (± 5.35)	$29.44^{A,a}$ (± 6.64)	$23.76^{B,a}$ (± 8.49)

* Different lower case letters indicate significant difference between materials for each time elapsed after setting (vertical lines)—Tukey post-hoc test ($p<0.05$).
* Different upper case letters indicate significant difference among times elapsed after setting for each material (horizontal lines)—Tukey post-hoc test ($p<0.05$).

The resin-modified glass ionomer cement reached significantly higher bond strengths compared with the conventional cements at the same time elapsed after setting.

Optical examination of the fractured interfaces showed that all of the cylinders presented cohesive failure within glass ionomer (Mode 2).

4. Discussion

The present study evaluated the influence of different time elapsed after setting of the glass ionomer cement on the micro-shear bond strength to composite resin restorations. Different chemical compositions of ionomer cements and different storage periods were analyzed and an increase in the bond strength to conventional chemical cement was observed after 24 h and 7 days. The chemical–physical cement presented an increase in bond strength after 24 h and a decrease after 7 days. Therefore, the main hypothesis of the present study was not accepted.

Adhesive systems were introduced into dentistry to allow the development of bonded restorations, which have a number of advantages over traditional, non-adhesive methods [23–25]. Resin tags may be formed when adhesive systems are applied to enamel and dentin. The resin tag length seems to increase as the remaining dentin thickness (RDT) between the cavity floor and the pulp tissue is reduced. With further reference to resin tag formation, resin components eluted from adhesive systems may diffuse

through dentinal tubules to cause pulpal damage since these resin-based materials present highly cytotoxic effects [1,2,7,8].

The use of a glass ionomer in conjunction with a composite resin has been established as an effective means of combining the favorable properties of the two materials in a single restoration [26–29]. However, the bond between conventional GICs and resin composite is limited due to a lack of chemical bonding between the two materials, in addition to the low cohesive strength of glass ionomers [26,28,30]. It has been demonstrated that RMGIC has improved mechanical and physical properties when compared with the conventional GIC. It shows better cohesive strength and lower modulus of elasticity. The bond strength to the tooth is better than that of the conventional GIC and it also exhibits a higher bond strength to the resin composite [27,29].

The chemical setting of GICs occurs in two stages. The initial stage, which produces the clinical set, occurs within the first 10 min after mixing. The second stage, involving the release of the calcium and aluminum cations within the matrix, is a slow and long-term continuation of the acid-base reaction, and occurs for at least 24 h afterwards, and probably much longer [31]. This is probably the reason why an increase on bond strength values of the conventional cement was observed after 24 h. During the first reaction, the material is very sensitive to water uptake, whereas during the second reaction the material is very susceptible to dehydration [32]. Moisture contamination during the initial setting of GICs can cause dissolution of the weak calcium-polyacrylate chains, capable of degrading their physical properties. In order to prevent moisture contamination during the rinsing procedure, it is mandatory to allow the initial setting of the glass-ionomer to occur before the etching procedure. Moreover, it is recommended to apply dental varnish, dental adhesive or nail polish to protect the surface, particularly of conventional glass ionomer cements [26].

The setting reaction of resin-modified glass ionomer cements occurs by an acid–base mechanism and polymerization reactions, so it is plausible that each reaction depends on the other and they are influenced each by other, dependent upon reactant diffusion prior to gelation [14]. The photochemical reactions cause rapid hardening of the cement surface and reduce the early sensitivity to moisture and dehydration associated with the early stage of the acid-base setting reaction of the conventional GIC [15,33,34]. A significant increase in the bond strength to the composite resin restoration of the RMGIC groups was observed after 24 h. This cement, in the same way as the conventional GIC, undergoes an aluminum polycarboxylate reaction [31] and this is probably the reason why an increase in the bond strength values was observed. After this period a significant decrease was observed. Resin-modified GICs are very sensitive to water sorption. Samples that were kept in contact with water, either in a humid atmosphere or completely immersed, presented lower flexural strength, lower elastic modulus and softer surfaces than dry samples [15]. The ability of the resin-modified GICs to take up water is related to their chemical composition, particularly to the hydrophilic functional groups present in their network. Water has two main effects on these materials. First, water diffuses through the specimens and mainly acts as a plasticizer, reducing their flexural strength and hardness. Second, water also partly dissolves the components of the cements. Consequently, the network is altered, which presumably results in a slight but irreversible decrease of their flexural strength and hardness [15].

When analyzing the failure mode after the micro-shear bond strength test, cohesive failures of the glass ionomer cements were recorded in all of the specimens, which demonstrated that the bond between the two cements and their respective composite resin was so high that it resulted in fracture of the cements.

Restorative material bond to dental substrates is a desirable property, because it is closely related to the prevention of material dislodgement and marginal leakage [35]. As regards GICs (conventional and resin-modified) it was observed that they presented low bond strengths to dental substrates when compared with dental adhesives and composite resin restorations [36,37]. In the present study composite resin restorations made on GICs immediately after initial setting resulted in the creation of a weak mechanical bond, whereas waiting for 24 h led to a slight, but significant improvement in the bond strength. As a clinical protocol, it would seem to be financially impractical to perform a composite resin restoration using two clinical sessions, with the expectation of obtaining a slight gain in bond strength. It is possible to suggest that for the purpose of preventing the cytotoxic effects of resin-based dental adhesive, placing a thin layer of glass ionomer cement and performing the composite resin restoration in the same clinical session would be a more feasible approach considering both biological and financial aspects.

5. Conclusion

Within the limitations of this study, it was observed that the bond strength of the restorations performed on conventional chemical cement at 10 min of time elapsed after setting presented the lower mean bond strength values, which differed from the other periods analyzed. The resin-modified photo-chemical cement after 24 h presented the highest mean values and differed from the values after 10 min and 7 days. The time elapsed after setting of glass ionomer cements may interfere in the bond strength to composite restorations.

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