

Repair bond strength and degradation of glass ionomer cements after mechanical and chemical challenges

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Aim: Little is known about the reparability of glass ionomer cements (GICs) after storage in acid environments. The aim of this study was to evaluate the solubility and reparability of GICs immersed in acid solutions and subjected to brushing.

Methods: Thirty discs of each GIC (Vitremer, VitroFil LC, VitroFil, and Maxxion R) were divided into three immersion groups: distilled water, Coca-Cola, or hydrochloric acid (HCl), then subjected to brushing. The weight of discs was measured before and after the immersions to determine mass alteration. Each disc was repaired, by adding the same brand of GIC over its surface. After immersing the repaired specimens in same solutions, shear bond strengths using universal testing machine were measured. Two-way ANOVA and Tukey's test was used ($\alpha=0.05$). **Results:** Resin-modified GICs degrade after HCl immersion followed by brushing ($p<0.05$), while self-cured GICs were negatively affected by all challenges ($p<0.05$). The challenges decreased the repair strength for VitroFil LC ($p<0.05$), which had higher repair shear bond strength than the other GICs ($p<0.05$), exhibiting most cohesive failures.

Conclusion: Self-cured GICs degraded when immersed in all acid solutions with brushing while resin-modified GICs only degraded following HCl immersion with brushing. Despite exhibiting the best repair results, VitroFil LC was the only GIC that was influenced by all the acid challenges.

Keywords: Glass ionomer cement. Materials testing. Shear strength. Solubility. Surface properties.



Introduction

Glass ionomer cements (GICs) have been used in restoration of non-carious cervical lesions (NCCL)^{1,2}. NCCL can be caused by dental erosion that generates dental substrate loss by extrinsic or intrinsic acids³. Thus, the absorption of acid solutions may make a dental surface more susceptible to the effects of toothbrushing⁴.

The longevity of GIC restorations depends on its mechanical properties; however, as for any cement that contains water in its composition, GICs may exhibit weakness, which can lead to fracture and wear⁵. Further, exposure to acid solutions followed by brushing may compromise the integrity of NCCL restored with GICs. Thus, there is a clinical need to repair GICs by adding a new portion of material. The repair of resin composites has become routinely used in clinical practice⁶. There is a lack of studies evaluating the *in vitro* susceptibility of GICs to degradation when exposed to acid solutions and then subjected to brushing⁷. Additionally, little is known about the reparability of GICs after storage in acid environments.

The aim of this study was to evaluate the solubility of GICs when immersed in acid solutions and subjected to brushing. The repair strength of GICs immersed in acid solutions was also evaluated. The null hypotheses tested were: (1) immersion in acid solution and brushing will not influence the solubility of GICs and (2) repaired GICs will not be affected by acid solutions.

Materials and Methods

Thirty discs of each GIC (Table 1) were prepared (10 mm in diameter and 3 mm in thickness). The powder/liquid ratio of each material was handled according to the manufacturer's instructions, in accordance with ISO specifications (ISO 9917-1)⁸.

Table 1. Characteristics of glass ionomer cement materials analyzed in this study.

Materials	Type	Color P/L	Composition	Batch#
Vitremer 3M ESPE, St Paul, MN, USA	Resin-modified glass ionomer cement	A3 2.5:1	Powder: Fluoroaluminosilicate glass, potassium polysulfide, ascorbic acid and pigments Liquid: Modified polycarboxylic acid, methacrylate groups, water, HEMA and photo initiators	1230200140
VitroFil LC Nova DFL, Rio de Janeiro, RJ, Brasil	Resin-modified glass ionomer cement	A3 2:1	Powder: Strontium-aluminum silicate, filler particles, activators and iron oxide Liquid: 2-Hydroxyethyl methacrylate, aqueous solution of polyacrylic and tartaric acid, benzoyl peroxide and camphorquinone.	P 12030385 L 12030384
VitroFil Nova DFL, Rio de Janeiro, RJ, Brasil	Self-cured glass ionomer cement	A3 2:1	Powder: strontium aluminum silicate, dried polyacrylic acid and iron oxide. Liquid: polyacrylic acid, tartaric acid and distilled water.	120304321
Maxxion R FGM, Joinville, SC, Brasil	Self-cured glass ionomer cement	A3 3:1	Powder: micronized glass ionomer, pigments (iron oxides), fillers (silica and zirconia), fluorides (potassium fluoride) Liquid: polyacrylic and tartaric acid, deionized water	130712

A syringe (Centrix, DFL, Rio de Janeiro, RJ, Brazil) was used for insertion of the GIC to the matrix in a single increment. Self-cured GICs were covered with a polyester strip and hand pressed for 1 min. Resin-modified GICs were photocured (Ultralux, Dabi Atlanti, Ribeirão Preto, SP, Brazil, 500 mW/cm²) for 40 seconds.

Polishing procedures were performed with silicon carbide sandpaper (#360, #600). The samples were washed in an ultrasonic device and stored in 100% relative humidity at 37 °C for 24 h.

Specimens were weighed every 24 h until mass stabilization was achieved with less than 0.1 mg variation, using a precision balance (Mettler Toledo, AB204, Switzerland). Specimens were dried with absorbent paper before measurements were taken. The initial mass (M1) was registered after mass stabilization.

Ten discs of each GIC were randomly selected and individually immersed in 10 mL of distilled water for 15 days at 37 °C (control group), Coca-Cola (Coca-Cola Co., Ribeirão Preto, SP, Brazil, pH 2.5) for 15 days at 37 °C, and hydrochloric acid (0.01 M HCl, pH 1.6, Apothicário, Araçatuba, SP, Brazil)⁹ for 2 h at 37 °C.

After immersion, all discs were subjected to 10.000 brushing cycles (MEV2, Odeme Biotechnology, Joaçaba, SC, Brazil) performed with one toothbrush for each specimen (Colgate Classic Clean, Colgate, Palmolive Co., Osasco, SP, Brazil). Toothpaste (Colgate Total 12, Colgate Palmolive, Kolynos Division of Brazil Ltd., Osasco, SP, Brazil) was diluted with distilled water (ratio 1:2 by weight), and a load of 200 g was used at 250 cycles/min. Then, the samples were washed and dried with absorbent paper and reweighed until a constant weight was achieved, which was considered the final mass (M2).

The volume (V) of each specimen was calculated according to the equation: $V = \pi r^2 h$ ($\pi = 3.141$, r = radius, h = thickness). The diameter and thickness were measured using digital calipers. The solubility ($\mu\text{g}/\text{cm}^3$) was calculated as follows: $SL = M1 - M2/V$.

Replicas of specimens were made with epoxy resin and stored at 37 °C for 48 h. Next, all specimens were sputter-coated with gold–palladium and evaluated using a scanning electron microscope (Evo LS15, Carl Zeiss, Oberkochen, Germany) at 5000X magnification.

The specimens used in the solubility analysis were included in self-curing acrylic resin by leaving the entire exposed surface of the GIC exposed. A circular area of 4 mm was defined (using adhesive tape, with a central hole) and then conditioned with 37% phosphoric acid for 30 s, followed by washing and drying with absorbent paper⁷. A cylinder of the same brand of GIC was made over each sample with the aid of a plastic matrix (4 mm in diameter and 5 mm thickness). A Centrix syringe was used for insertion in the matrix in a single increment for self-cured GICs and two increments for resin-modified GICs. Each layer of the resin-modified GICs was photocured for 40 seconds.

The repaired specimens were then stored in 100% relative humidity and 37 °C for 24 h, and subsequently immersed in the solutions (water, Coca-Cola, and HCl) for the same time period as cited in the solubility test. Specimens were then submitted to a shear bond strength test using universal testing machine (0.5 mm/min, EMIC DL-1000,

EMIC, São José dos Pinhais, PR, Brazil). The shear strength value was calculated: $R = F/A$ (R = value of the shear strength, F = force applied, A = area).

The fractured specimens were examined (Olympus SZ-CTV, Olympus, Tokyo, Japan) (40×) and failure patterns were classified as adhesive along the disc surface (A), cohesive within the disc base (CB), cohesive within the repair cylinder (CR), or mixed (M). A schematic diagram is shown in Figure 1.

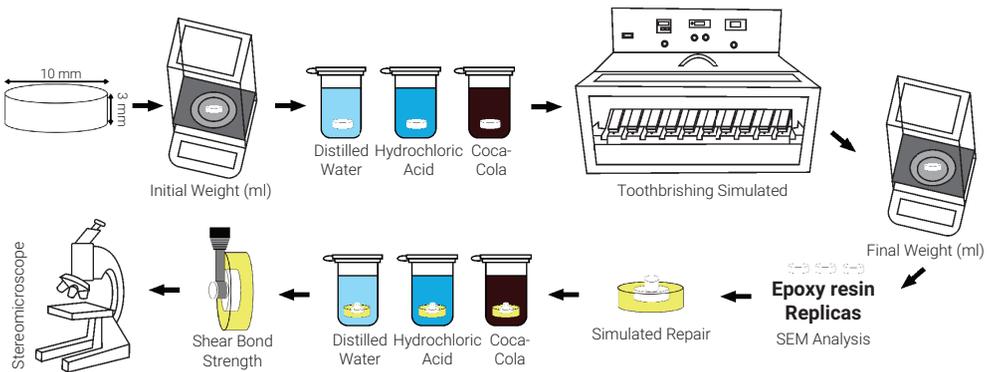


Figure 1. Schematic diagram of experimental tests.

Normal distribution of data and equality of variances were assumed after Kolmogorov–Shapiro–Wilk and Levene’s tests, respectively. ANOVA and Tukey’s post hoc test were used ($\alpha=0.05$).

Results

All data from solubility can be found in Table 2. In the water solution, no statistical differences were found among GICs for solubility ($p>0.05$). In the Coca-Cola immersed group, a statistical difference was observed only between VitroFil LC and VitroFil, with higher value for VitroFil ($p<0.05$). Following immersion in HCl, this sequence of solubility was found: Maxxion R = Vitremer > VitroFil > VitroFil LC ($p<0.05$).

Table 2. Mean (SD) values of solubility ($\mu\text{g}/\text{cm}^3$) of glass ionomer cements (GIC) after challenges.

GICs	Water	Coca-Cola	HCl
Vitremer	0.0004 (± 0.0015) ^{A,a}	0.0100 (± 0.0071) ^{AB,a}	0.0878 (± 0.0254) ^{A,b}
VitroFil LC	0.0023 (± 0.0021) ^{A,a}	0.0012 (± 0.0018) ^{B,a}	0.0330 (± 0.0085) ^{C,b}
VitroFil	-0.0003 (± 0.0134) ^{A,a}	0.0194 (± 0.0093) ^{A,b}	0.0577 (± 0.0225) ^{B,c}
Maxxion R	-0.0008 (± 0.0038) ^{A,a}	0.0142 (± 0.0190) ^{AB,b}	0.0916 (± 0.0256) ^{A,c}

Different uppercase letters in columns represent statistical significance for glass ionomer cement in the same immersion solution at $p<0.05$. Different lowercase letters in rows indicate statistically significance for immersion solution in the same glass ionomer cement at $p<0.05$.

When comparing the same GIC in the various immersion solutions, it was found that all GICs had significantly higher solubility values in the HCl solution ($p < 0.05$). Compared with water, self-cured GICs showed statistical superior solubility when immersed in Coca-Cola ($p < 0.05$), whereas resin-modified GICs did not show statistical differences ($p > 0.05$).

VitroFil LC showed the highest shear strength values with significant differences from the other GICs when immersed in water and Coca-Cola. However, the repair strength of VitroFil LC was statistically similar to those of VitroFil and Vitremer when immersed in HCl. Maxxion R had the lowest repair strength values in all solutions. VitroFil LC was the only GIC affected by acid solutions when compared with water storage, whereas the other GICs were not affected by the challenges (Table 3).

Table 3. Mean (SD) values of repair strength (MPa) of glass ionomer cements (GIC) after challenges.

GICs	Water	Coca-Cola	HCl
Vitremer	1.344 (± 0.780) ^{B,a}	1.470 (± 0.598) ^{BC,a}	2.087 (± 1.465) ^{A,a}
VitroFil LC	8.106 (± 2.117) ^{A,a}	3.746 (± 1.463) ^{A,b}	2.233 (± 0.789) ^{A,c}
VitroFil	1.642 (± 0.894) ^{B,a}	2.114 (± 0.698) ^{B,a}	1.383 (± 0.865) ^{AB,a}
Maxxion R	0.770 (± 0.240) ^{B,a}	0.684 (± 0.318) ^{C,a}	0.428 (± 0.230) ^{B,a}

Different uppercase letters in columns represent statistical significance for glass ionomer cement in the same immersion solution at $p < 0.05$. Different lowercase letters in rows indicate statistically significance for immersion solution in the same glass ionomer cement at $p < 0.05$.

For all GICs, most adhesive failures occurred after immersion in HCl, except for Maxxion R, which showed six failures as within the cylinder after immersion in Coca-Cola. Vitremer was the only GIC that presented increased numbers of adhesive failures in water (Table 4). SEM images revealed a gradual change of the GIC surface according to the aggressiveness of the acid solution (Figure 2).

Table 4. Analysis of fracture after shear bond test.

GICs	Fracture	Water	Coca-Cola	HCl
Vitremer	A	7	5	4
	CB	2	2	3
	CR	0	0	0
	M	1	3	4
VitroFil LC	A	0	5	5
	CB	9	4	0
	CR	0	0	0
	M	1	1	5
VitroFil	A	1	4	9
	CB	6	3	0
	CR	0	0	0
	M	3	3	1
Maxxion R	A	0	4	7
	CB	0	0	0
	CR	8	6	2
	M	2	0	1

A: Adhesive; CB: Cohesive base; CR: Cohesive repair; M: Mixed

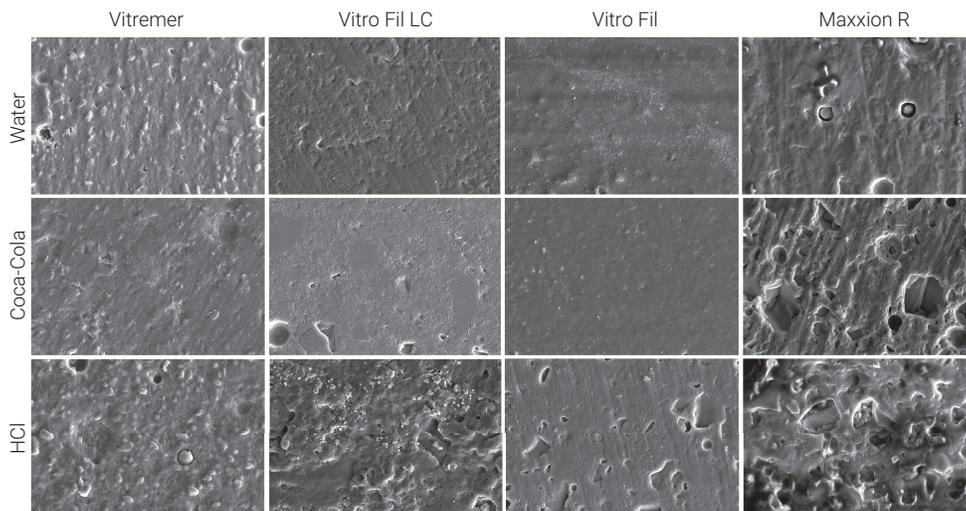


Figure 2. Representative scanning electron micrographs of GICs after tests. Original magnification 1000 kX. Greater degradation can be seen in images after immersion in HCl solution, with more rugged surfaces observed for all GICs. Similar degradation can be seen in images of self-cured GICs after immersion in Coca-Cola

Discussion

The degradative action of HCl has been previously reported⁹; in fact, HCl has been used to model cases in which patients suffering from gastroesophageal disorders and therefore secrete this acid as gastric juice⁹. Soft drinks have also been studied in this context, owing to their acidic characteristics^{9,10}. In addition, it is known that repair of degraded dental restorations is a valid alternative for improving the quality of restorations after a longer period in the mouth⁶.

The first null hypothesis was rejected because the resin-modified GICs were degraded by the action of HCl and that self-cured GICs are affected by the action of both acid solutions (Figure 2). These results corroborated those of previous studies, in which higher solubility was observed for self-cured GICs when compared with resin-modified GICs after immersion in acidic juices¹¹.

The sorption and solubility of GICs depend on their type, concentration, and particle size¹². Acid immersion degrades the particles and matrix through absorption, causing increased solubility and facilitating the detachment of particles¹². Moreover, the abrasion caused by brushing in combination with acid solutions may lead to further detachment of particles¹³, as shown in Figure 2. The majority of published solubility studies^{11,12,14}, usually dehydrate specimens in a chamber for dehydration prior to weighing; however, in the present study we opted to dry only with absorbent paper before each weighing, thus simulating clinical conditions more closely.

We did not observe a significant difference between the two types of GICs when immersed in water, consistent with previous reports in the literature¹⁴. However, the self-cured GICs had negative solubility values in water, which may be due to water absorption by these materials that increases the final weight (M2)¹⁵. The higher solu-

bility of Maxxion R could be explained by the lower powder to liquid ratio, resulting in fewer ionic bonds available for matrix formation and, consequently, in greater solubilization of the material¹⁵.

When the specimens were immersed in Coca-Cola, a difference was only observed between VitroFil LC and VitroFil. These results corroborate those of a previous study that evaluated acid solutions (acetic acid, lactic acid, and citric acid) and found that the self-cured GIC Ketac-Cem exhibited significantly higher erosion in organic acid buffer solutions when compared to resin-modified GIC Fuji Plus¹⁶. It was also observed that Coca-Cola increases the surface roughness and reduces the hardness of two types of resin-modified GICs, likely due to the presence of phosphoric acid¹⁰. However, no differences were found when immersion in water and Coca-Cola were compared for both resin-modified GICs. It is possible that the presence of phosphate ions in Coca-Cola suppress dissolution, as these ions have been shown to reduce the dissolution rate of calcium phosphate from the tooth¹⁷. It should also be noted that the damage caused by acid solutions is not simply a result of exposure to low pH, but is influenced by the overall chemical composition of the acidic beverage in question¹⁸.

In HCl solution, resin-modified GIC VitroFil LC showed the lowest solubility, with significant differences from the other GICs. This difference may not be directly correlated with incorporation of resin particles, as resin-modified GIC Vitremer showed similar solubility to self-cured GIC Maxxion R in HCl solution. High values of solubility and degradation were also found for Vitremer owing to its high hydrophilicity¹⁹. The ability of some resin-modified GICs to take up water is related to their chemical composition, particularly to hydrophilic functional monomers present in their network, resulting a softer surface²⁰.

The philosophy and principles of minimum intervention in operative dentistry are becoming widely accepted⁷, including for repair procedures⁶. In this context, resin-modified GIC VitroFil LC showed the highest resistance to repair, which rejects the second hypothesis. Therefore, for VitroFil LC, the union between the original and repaired cement was stronger than the internal strength of the material, which resulted in major cohesive fractures when immersed in water and Coca-Cola, indicating significant differences in repair strength when compared with the other GICs. Furthermore, the reparability values may be influenced by high compression and tensile strength, as resin-modified GIC VitroFil LC has shown greater mechanical resistance in comparison to self-cured GIC VitroFil^{21,22}.

Increased shear bond strength to resin for resin-modified GIC (Vitrebond) could be due to unpolymerized HEMA on the surface, which may penetrate the material and facilitate wetting of the bonding agent and composite resin during bonding²³. It has also, previously, been suggested that the availability of residual unreacted methacrylate groups on the polyacid chain within the polymerized, light-cured GIC may lead to the formation of strong covalent chemical bonds to the resin bonding agent²³. The availability of monomers on the surface of resin-modified GICs may be also associated with the best repair results observed for VitroFil LC. However, the repair of VitroFil LC was influenced by immersion in Coca-Cola and HCl solutions when compared with water immersion. In this context, Yap et al.²⁴, observed that GICs

should not be repaired beyond 3 months from damage, as all repaired GICs were affected by the action of water after this period.

The exposure of GICs to a dentifrice during toothbrushing can affect their degradation²⁵. Part of the organic matrix of these restorative materials may be removed by degradation caused during toothbrushing^{26,27}. However, the effect of toothbrushing depends on different factors, such as the type of toothpaste and brush, the water solution, and speed during simulation process, and these factors could be considered a limitation of this study, since the challenges were not tested separately.

In conclusion, self-cured GICs degraded when immersed in all acid solutions, while resin-modified GICs only suffered from the action of HCl immersion, both in combination with brushing. One type of resin-modified GICs (VitroFil LC) exhibited the best repair results, mainly when immersed in water.

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