Research Article

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Effects of a bleaching agent on properties of commercial glass-ionomer cements

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ABSTRACT

Objectives: This study evaluated the effects of a bleaching agent on the composition, mechanical properties, and surface topography of 6 conventional glass-ionomer cements (GICs) and one resin-modified GIC.
Materials and Methods: For 3 days, the specimens were subjected to three 20-minute applications of a 37% H_2O_2-based bleaching agent and evaluated for water uptake (WTK), weight loss (WL), compressive strength (CS), and Knoop hardness number (KHN). Changes in surface topography and chemical element distribution were also analyzed by energy-dispersive X-ray spectroscopy and scanning electron microscopy. For statistical evaluation, the Kruskal-Wallis and Wilcoxon paired tests (a = 0.05) were used to evaluate WTK and WL. CS specimens were subjected to 2-way analysis of variance (ANOVA) and the Tukey *post hoc* test (α = 0.05), and KH was evaluated by one-way ANOVA, the Holm-Sidak *post hoc* test (a = 0.05), and the *t*-test for independent samples (a = 0.05).
Results: The bleaching agent increased the WTK of Maxxion R, but did not affect the WL of any GICs. It had various effects on the CS, KHN, surface topography, and the chemical element distribution of the GICs.
Conclusions: The bleaching agent with 37% H_2O_2 affected the mechanical and surface properties of GICs. The extent of the changes seemed to be dependent on exposure time and cement composition.

Keywords: Dental materials; Glass-ionomer cement; Tooth whitening agents

INTRODUCTION

Glass-ionomer cement (GIC) was introduced into dentistry as a biocompatible, translucent filling material with the ability to chemically bond to the dental structure [1] and an important fluoride-releasing mechanism [2-4]. However, due to their mechanical properties, GICs as restorative materials are usually limited to primary teeth, small cavities on non-occluding surfaces in permanent teeth, or temporary restorations [5-9]. In addition,

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susceptibility to chemical degradation also limits the use of GICs as temporary restorative materials [10,11].

The demand for esthetic treatments has increased, and dental bleaching may be an important tool for achieving a pleasing smile. Although they are efficient and safe, previous studies have reported that peroxides may affect restorative materials, especially GICs [12-15], promoting negative effects such as increased surface roughness and decreased hardness of glass-ionomer materials [12,16-18]. Dissociation of the peroxides used in dental bleaching may increase the dissolution of GICs [10,12,19], since contact with peroxides can plasticize the permeable hydrogel matrix [20], allowing the bleaching agent to penetrate the material [10], which affects other properties of the cement. According to previous studies, not only contact, but also concentration and time play important roles in cement degradation, depending on the type of GIC and the bleaching agent [10,15,16].

Poorly adapted restorations may facilitate the diffusion of free radicals from bleaching agents into the tooth structure, similar to what occurs with enamel cracks [21], which may increase the toxicity of this esthetic treatment.

In this sense, the influence of bleaching treatment on current GICs must be evaluated if we are to understand its effects on the properties of these restorative materials and to determine the best clinical steps regarding the presence of or need for these materials before a bleaching procedure. Therefore, the aim of the present study was to evaluate the effects of a H_2O_2-based bleaching agent on the composition, mechanical properties, and surface topography of various GICs. The null hypothesis tested was that the effects of a bleaching agent would not vary among GICs in terms of changes in chemical composition, mechanical properties, and surface topography.

MATERIALS AND METHODS

Manipulation of cements and bleaching protocol
For all tests, the 7 GICs evaluated were manipulated using a 24F stainless steel spatula (SS White, Rio de Janeiro, RJ, Brazil) on a glass plate according to the powder/liquid ratio and time specified by each manufacturer (**Table 1**). Vitremer (3M ESPE, St. Paul, MN, USA) was light-cured with a LED source (Radii-cal, SDI, Victoria, Australia) at 1,200 mW/cm². During setting, the free surfaces of the cement were protected with a Mylar strip to avoid syneresis. After 15 minutes, the specimens were removed from the molds and stored individually in Eppendorf bottles at 100% relative humidity at 37°C for 24 hours before the bleaching protocol began.

The bleaching protocol involved the application of 37% hydrogen peroxide gel (pH = 6.5; Drogaria, Xavier, Uberaba, MG, Brazil) for 20 minutes, followed by rinsing with an air/water spray from a triple syringe for 1 minute. This process was repeated twice, for a total of 3 applications. The entire protocol was applied on 3 consecutive days, and the specimens were stored in 100% relative humidity at 37°C during the sessions.

Water uptake (WTK) and weight loss (WL)
Seventy cylindrical specimens (1 mm high and 4 mm in diameter) were prepared for the weight-loss test as previously described [22]. The specimens were dry-stored at 37°C and

Table 1. Glass-ionomer cements (GICs) evaluated in the present study

GIC/batch No.	Composition*	Manufacturer	P/L ratio	Mixing time (sec)
Ketac Molar EasyMix/56908	Powder: glass powder, polycarboxylic acid, pigments Liquid: water, tartaric acid, conservation agents	3M ESPE, St. Paul, MN, USA	1/1	60
Ketac Cem Easy Mix/56908	Powder: glass powder, polycarboxylic acid, pigments Liquid: water, tartaric acid, conservation agents	3M ESPE, St. Paul, MN, USA	1/2	60
Vitremer/544223	Powder: radiopaque fluoroaluminosilicate glass, microencapsulated potassium persulfate, ascorbic acid Liquid: aqueous solution of a polycarboxylic acid modified with pendant methacrylate groups, water, hydroxyethylmethacrylate, photoinitiators	3M ESPE, St. Paul, MN, USA	1/1	45
Vitro Fil/14111774	Powder: strontium aluminum silicate, dehydrated polyacrylic acid, iron oxide Liquid: polyacrylic acid, tartaric acid, distilled water	NOVA DFL, Rio de Janeiro, RJ, Brazil	1/1	60
Vitro Molar/15030424	Powder: barium aluminum silicate, dehydrated polyacrylic acid, iron oxide Liquid: polyacrylic acid, tartaric acid, distilled water	NOVA DFL, Rio de Janeiro, RJ, Brazil	1/1	20
Vidrion R/0321114	Powder: sodium-calcium-fluoroaluminosilicate glass, polyacrylic acid and pigments Liquid: tartaric acid, distilled water	SS White, Rio de Janeiro, RJ, Brazil	1/1	60
Maxxion R/031214	Powder: fluoroaluminosilicate glass, polycarboxylic acid, calcium fluoride, radiopacifiers Liquid: polyacrylic acid, tartaric acid, distilled water	FGM, Joinville, SC, Brazil	1/1	60

*This information was provided by the manufacturers in the Material Safety Data Sheet (MSDS) and instruction sheets.

repeatedly weighed at 24-hour intervals in an analytical balance (AY220, Marte Científica, São Paulo, SP, Brazil) until a constant mass was obtained (M1). Then, 5 specimens of each material were immersed in the bleaching gel as previously described. During the bleaching protocol, the untreated specimens were stored in distilled water under the same conditions of time and temperature. After the treatment, all specimens were immersed in distilled water for 7 days at 37°C and then gently dried with absorbent paper and weighed again (M2). The specimens were then dry-stored again at 37°C and reweighed at 24-hour intervals until a constant final dry mass was recorded (M3). WTK (M2–M3) and water solubility (M1–M3) of the specimens were calculated in micrograms (μg) based on the differences in weight gain or loss during the immersion in water and drying cycles. The median values of the groups were compared by the Kruskal-Wallis and Wilcoxon paired-samples tests ($\alpha = 0.05$).

Compressive strength (CS)
For CS testing, 112 cylindrical specimens (6 mm high and 3 mm in diameter) were prepared ($n = 8$). Vitremer specimens were light-cured for 20 seconds on the top and bottom surfaces. After storage, 56 specimens were immersed in the bleaching gel according to the previously described protocol, while the other half of the samples were stored in distilled water, as controls. CS testing was performed at 24 hours after the bleaching session in a testing machine (DL 3000, Emic Ind. Ltd., São José dos Pinhais, PR, Brazil) with a crosshead speed of 0.5 mm/minute. The values in megapascals (MPa) were analyzed using 2-way analysis of variance (ANOVA) and the Tukey *post hoc* test ($\alpha = 0.05$).

Knoop hardness
A total of 140 cylindrical specimens (2 mm high and 4 mm diameter) were prepared for Knoop hardness testing ($n = 10$). The specimens were embedded in epoxy resin, stored for 24 hours at 37°C, and polished under water cooling with #600- and #1200-grit silicon carbide papers. After specimens were polished, specimen hardness was measured with a Knoop indenter (HMV-2, Shimadzu, Tokyo, Japan) under a load of 50 g for 15 seconds. After the measurements, half of the samples were subjected to the bleaching protocol, while the other half were stored at 100% relative humidity at 37°C. The hardness measurements were repeated after the application of

each agent in all samples (bleached and non-bleached) with the same parameters, in order to evaluate how hardness changed during the course of the complete treatment. The factor of 'time' was compared within each cement by one-way ANOVA and the Holm-Sidak *post hoc* test (a = 0.05). Differences between treated and non-treated specimens at each time were compared using the *t*-test for independent samples (a = 0.05).

Characterization of the materials

For energy-dispersive X-ray spectroscopy (EDS) and scanning electron microscopy (SEM) analysis, 3 additional specimens were prepared for each material and treatment step (baseline and after each bleaching application) to evaluate the changes in chemical element percentages and surface topography. The specimens were mounted on acrylic stubs with double-face carbon tape and coated with carbon for EDS. After microanalysis, the specimens were gold-sputtered for SEM evaluation (JSM5600LV, JEOL USA Inc., Peabody, MA, USA).

RESULTS

Water uptake and weight loss

For water sorption and solubility of the cements, Vidrion R and Vitro Molar were not tested because the specimens disintegrated during the immersion period in distilled water. Only the Maxxion R cement showed increased WTK as a result of the bleaching protocol. The WTK of the other tested materials was not affected by the treatment. The bleaching protocol also did not affect the WL of any of the tested materials. The results of weight changes are shown in **Table 2**.

Compressive strength

Before the bleaching protocol was applied, Vitremer, Ketac Molar, and Ketac Cem showed similar CS values, which were significantly higher than those of the other cements (p < 0.05). Vitro Molar and Vitro Fil had intermediate values; however, Vitro Molar had a significantly higher CS than Maxxion R and Vidrion R (p < 0.05). After the bleaching protocol, Vitremer showed a significantly higher CS than the other cements (p < 0.05). No differences were found among the other cements. Vitro Fil and Vidrion R were not tested after the bleaching protocol because the specimens disintegrated. CS values are shown in **Table 3**.

Knoop hardness

In general, the GICs showed an increased Knoop hardness number (KHN) after 48 and 72 hours, especially for the groups subjected to the bleaching protocol (second and third sessions, respectively). A reduced KHN was observed for untreated specimens of Ketac Molar after 24 hours; however, this value stabilized in later periods. For untreated Vitremer, changes in KHN

Table 2. Median, interquartile range, and percentages of water uptake (WTK) and weight loss (WL)

Group	WTK (µg)				WL (µg)			
	Not treated	%	Treated	%	Not treated	%	Treated	%
Ketac Molar	1.3 (1.2–1.65)[Aab]	4.8	1.0 (1.0–1.1)[Aa]	4.3	0.9 (0.38–1.15)[Aab]	2.3	0.7 (0.45–0.83)[Aab]	2.6
Ketac Cem	0.5 (0.38–1.0)[Aab]	1.7	1.2 (1.15–1.55)[Aa]	5.2	0.8 (0.53–1.33)[Aab]	2.4	0.9 (0.18–2.85)[Aab]	5.5
Vitremer	3.6 (3.53–4.38)[Aa]	11.7	2.5 (2.38–2.6)[Aa]	11.1	0.1 (0.08–0.58)[Ab]	1.4	0.1 (0.0–0.23)[Ab]	0.5
Maxxion R	0.1 (0.1–0.85)[Bb]	2.2	4.7 (4.48–4.73)[Aa]	23.4	5.0 (2.0–5.4)[Aa]	19.3	3.0 (2.98–3.33)[Aa]	15.8
Vidrion R	0.4 (0.1–0.85)[Ab]	1.9	1.0 (1.0–1.1)[Aa]	5.2	0.8 (0.58–3.45)[Aab]	8.1	0.7 (0.45–0.83)[Aab]	3.3

WTK (M2–M3) and water solubility (M1–M3) of the specimens were calculated in micrograms (µg) from the differences in weight gain or loss during the immersion in water and drying cycles. Different uppercase superscript letters indicate a statistically significant difference within the row (p < 0.05). Different lowercase superscript letters indicate a statistically significant difference within the column (p < 0.05).

Table 3. Compressive strength (MPa) of glass-ionomer cement (GIC) restoratives used in this study

GIC	Untreated	Treated
Vitremer	113.8 ± 8.1^{Aa}	92.9 ± 15.9^{Ba}
Ketac Molar	112.6 ± 15.1^{Aa}	72.7 ± 16.7^{Bb}
Ketac Cem	112.4 ± 12.6^{Aa}	55.4 ± 15.0^{Bb}
Vitro Molar	75.0 ± 8.5^{Ab}	64.9 ± 14.3^{Ab}
Vitro Fil	66.5 ± 7.5^{bc}	-*
Maxxion R	50.9 ± 4.7^{Ac}	64.1 ± 17.5^{Ab}
Vidrion R	46.5 ± 14.9^{c}	-

Data are shown as means ± standard deviations. Different uppercase superscript letters indicate a statistically significant difference within the row ($p < 0.05$). Different lowercase superscript letters indicate a statistically significant difference within the column ($p < 0.05$).
*Vitro Fil and Vidrion R were not tested after the bleaching protocol because the specimens disintegrated.

were not observed, while the KHN of treated specimens oscillated, as previously described for most GICs. Significant differences between treatments were observed only for Vitremer and Ketac Molar ($p < 0.05$). The results of the Knoop hardness tests are shown in **Table 4**.

Characterization of materials

1. EDS analysis

Reduced carbon and oxygen counts were revealed by EDS for all GICs after the third session of the bleaching protocol. Vidrion R, Vitro Molar, Vitro Fil, and Maxxion R showed reduced fluoride counts after the first session. However, Vitremer, which presented the highest initial fluoride count of all the GICs, showed the greatest loss of this element after each session of the protocol. Data from the EDS analysis are shown in **Table 5**.

2. Surface evaluation

An increasingly erosive process was observed on the surfaces of GICs after the bleaching sessions were performed (**Figure 1**). An extensive presence of filler particles on the surfaces of all materials could be seen, especially after the second and third sessions (**Figure 1A''-1G'' and 1A'''-1G'''**, respectively). Although an increased occurrence of cracks could be seen on the GIC surfaces, they were also present on the surfaces of untreated materials (**Figure 1A-1G**), probably caused by the vacuum step during the preparation of the samples and generation of SEM images.

Table 4. Knoop hardness number of glass-ionomer cement (GIC) restoratives used in this study after different bleaching sessions

GIC	Bleaching treatment	Time (session)			
		24 hr (before the protocol)	24 hr (first session)	48 hr (second session)	72 hr (third session)
Vidrion R	−	49.8 ± 5.4^{Ba}	53.4 ± 5.2^{ABa}	57.8 ± 3.3^{Aa}	49.8 ± 5.2^{Ba}
	+	53.1 ± 4.8^{ABa}	56.0 ± 5.2^{ABa}	58.1 ± 6.6^{Aa}	50.7 ± 6.1^{Ba}
Vitremer	−	96.8 ± 11.0^{Aa}	97.6 ± 12.3^{Ab}	91.2 ± 10.0^{Aa}	106.2 ± 10.7^{Aa}
	+	100.6 ± 9.8^{Ba}	119.5 ± 20.1^{Aa}	100.9 ± 19.8^{Ba}	113.1 ± 12.1^{ABa}
Vitro Molar	−	51.1 ± 5.2^{Ca}	57.1 ± 7.0^{BCa}	60.2 ± 5.9^{Aa}	60.5 ± 9.4^{Aba}
	+	51.0 ± 5.6^{Ba}	58.1 ± 5.9^{ABa}	63.7 ± 9.5^{Aa}	53.3 ± 8.6^{Ba}
Ketac Cem	−	74.1 ± 8.8^{Ba}	83.2 ± 10.6^{ABa}	77.2 ± 8.8^{ABa}	86.7 ± 8.3^{Aa}
	+	80.7 ± 11.1^{Aa}	87.3 ± 5.9^{Aa}	80.7 ± 7.6^{Aa}	82.9 ± 5.5^{Aa}
Ketac Molar	−	143.5 ± 25.6^{Aa}	86.6 ± 17.2^{Bb}	80.8 ± 11.2^{Bb}	77.1 ± 11.1^{Bb}
	+	148.9 ± 23.9^{Aa}	120.9 ± 16.6^{Ba}	119.9 ± 26.5^{Ba}	114.1 ± 10.9^{Ba}
Vitro Fil	−	49.5 ± 5.8^{Ba}	60.0 ± 7.2^{Aa}	51.8 ± 6.3^{Ba}	42.0 ± 4.5^{Ca}
	+	48.0 ± 2.7^{Ba}	60.0 ± 7.7^{Aa}	44.8 ± 6.1^{Ba}	37.2 ± 6.5^{Ca}
Maxxion R	−	41.7 ± 7.5^{Aa}	40.2 ± 2.2^{Aa}	40.1 ± 4.5^{Aa}	40.5 ± 4.5^{Aa}
	+	48.9 ± 6.2^{Aa}	41.1 ± 9.4^{ABa}	39.1 ± 4.8^{Ba}	38.2 ± 4.7^{Ba}

Data are shown as means ± standard deviations. Different uppercase superscript letters indicate a statistically significant difference within each row, that is, within each cement separately ($p < 0.05$). Different lowercase superscript letters indicate a statistically significant difference between the presence and absence of the application of each bleaching agent ($p < 0.05$).

Table 5. Distribution of the chemical elements of the composition of the glass-ionomer cements (GICs) in relative percentage by weight (wt%).

GIC	Time (sessions)	Chemical elements (wt%)									
		C	O	F	Na	Al	Si	Ca	Nb	W	Ba-L
Vidrion R	Untreated	30.7	31.1	7.4	2.4	9.9	5.5	8.2	-	-	4.9
	24 hr	35.6	30.4	7.7	2.5	9.7	6.4	7.7	-	-	-
	48 hr	31.4	34.0	7.7	2.5	10.5	6.7	7.3	-	-	-
	72 hr	29.9	28.8	5.6	1.5	6.5	3.1	5.7	-	-	-
Vitremer	Untreated	46.5	25.2	8.9	1.4	6.6	11.5	-	26.7	-	-
	24 hr	46.7	16.1	3.3	0.9	2.6	3.7	-	26.7	-	-
	48 hr	47.2	21.1	4.3	0.7	4.0	4.2	-	18.6	-	-
	72 hr	35.0	23.7	4.7	0.8	5.3	9.0	-	21.5	-	-
Vitro Molar	Untreated	35.1	32.5	6.1	2.3	8.9	7.1	8.3	-	-	-
	24 hr	33.6	34.2	6.9	2.2	8.0	6.4	8.6	-	-	-
	48 hr	27.2	28.2	5.8	1.2	7.8	6.2	6.5	17.1	-	-
	72 hr	34.4	29.1	6.3	2.2	9.8	8.3	9.9	-	-	-
Ketac Cem	Untreated	27.6	32.4	10.3	2.7	7.9	9.1	9.9	-	-	-
	24 hr	26.3	34.3	8.9	2.3	9.2	9.7	9.2	22.3	-	-
	48 hr	29.9	32.4	8.7	2.4	9.0	10.6	6.9	18.3	-	-
	72 hr	25.3	30.1	6.0	1.4	5.4	7.7	2.4	19.1	-	-
Ketac Molar	Untreated	29.1	37.3	6.5	2.0	9.4	7.5	8.1	-	-	-
	24 hr	22.1	29.5	5.4	2.1	6.3	4.5	7.3	22.4	-	-
	48 hr	19.9	33.2	6.5	1.4	7.1	7.8	5.8	18.3	-	-
	72 hr	21.1	33.5	8.7	1.6	6.2	8.2	6.5	14.2	-	-
Vitro Fil	Untreated	28.8	25.7	4.3	1.6	5.4	7.2	1.9	12.1	11.9	-
	24 hr	20.2	22.4	5.4	1.5	6.0	5.4	1.8	17.6	14.0	-
	48 hr	43.5	18.2	3.1	1.1	3.6	4.6	-	19.8	-	-
	72 hr	22.5	23.8	6.5	1.2	8.4	6.3	-	-	33.3	-
Maxxion	Untreated	26.2	25.9	6.8	3.4	7.6	3.0	3.7	23.2	-	-
	24 hr	33.5	30.9	10.3	4.7	11.1	5.1	4.3	-	-	-
	48 hr	32.3	31.0	9.7	4.6	11.4	5.9	5.0	-	-	-
	72 hr	27.1	29.1	6.1	3.2	7.2	6.7	2.7	17.8	-	-

Although there are limitations of energy-dispersive X-ray spectroscopy in identifying and quantifying chemical elements with low atomic numbers, such as C, the relative quantities (wt%) of elements were obtained using the χ^2 test.

DISCUSSION

Bleaching agents can easily come into contact with the surfaces of permanent or temporary cervical restorations. Although permanent GIC restorations can be seen in adult patients, GICs are commonly used for the temporary replacement of unsatisfactory restorations, and even to create adequate conditions in the oral cavity before dental bleaching treatment.

Before the bleaching protocol, Vitremer, Ketac Molar, and Ketac Cem had similar CS values, significantly higher than those of the other cements; however, they were negatively affected by the bleaching agent. After the bleaching protocol, Vitremer showed higher values of CS than all conventional cements, even with the negative effect of bleaching treatment, showing that the presence of resin monomers (Bis-GMA, TEGDMA) made the material less susceptible to degradation caused by the peroxide. The better mechanical performance of Vitremer corroborates the results of previous studies [8,23-25]. Despite the fact that Vitromolar and Maxxion R were not significantly influenced by the bleaching agent, they presented lower CS values than Vitremer, Ketac Molar, and Ketac Cem. However, they apparently resisted contact with the bleaching agent, unlike Vitro Fill and Vidrion R, which disintegrated when subjected to the bleaching protocol. Although it was believed that these cements (Vitro Fill and Vidrion R) would be unlikely to disintegrate completely in the oral cavity, these results indicate possible early degradation, or at least degradation more pronounced than that of other materials, which could compromise marginal sealing of the
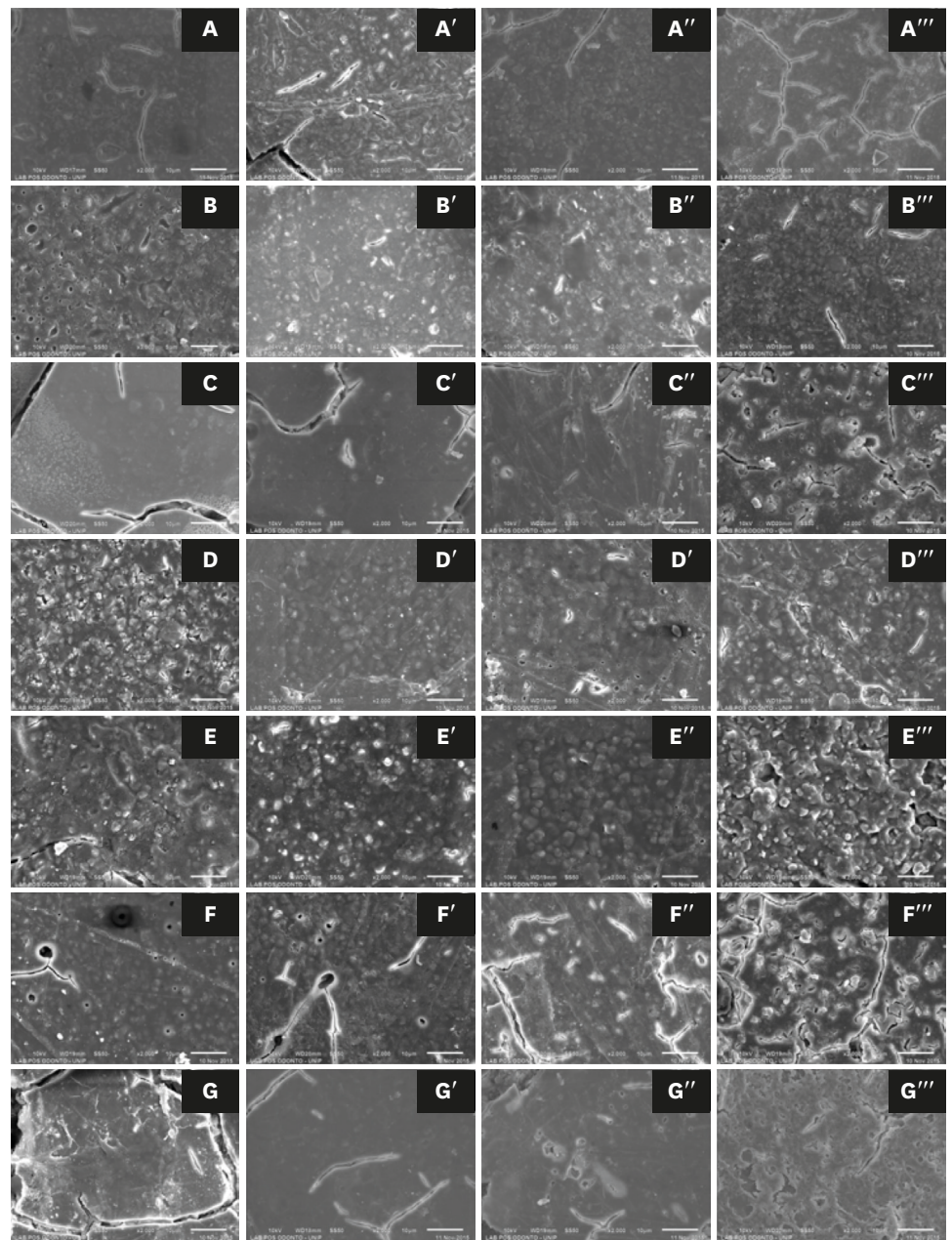
Figure 1. Scanning electron microscopic images of glass-ionomer cements (×2,000): (A) Ketac Cem, (B) Ketac Molar, (C) Maxxion R, (D) Vitremer, (E) Vitro Fil, (F) Vitro Molar, and (G) Vidrion R. In the first column are shown the untreated specimens, followed by the treated groups, including the first ('), second (''), and third (''') sessions, after 24, 48, and 72 hours, respectively.

restoration. This would increase the diffusion of free radicals from bleaching agents to the dental pulp, exacerbating the toxic effects thereof. However, clinical studies are required to confirm this hypothesis.

Although the KHN of untreated GICs presented small variations, probably because of the plasticization promoted by the humidity, treated materials had a slightly more pronounced variation. The increased KHN in some GICs, especially in the first or second session of the bleaching protocol (24 and 48 hours after manipulation), can be explained by the partial

dissolution of the ionomeric matrix on the surfaces of the cements and, consequently, the exposure of the filler particles, which are harder than the matrix. This dissolution occurs due to plasticization of the matrix after contact with the bleaching agent [10], since the ionomeric matrix is a permeable hydrogel, allowing for the passage of dissociated peroxide molecules from the bleaching gel [20]. However, the displacement and loss of these filler particles occurred at different moments, especially in the 48- and 72-hour readings. Although matrix dissolution occurred for both treated and untreated samples of cements, such as Vitro Fil and Vidrion R, the KHN of treated Maxxion R and Vitro Molar specimens was negatively affected by the bleaching agent. The reduction in the KHN of these materials could have been the result of the significant loss of filler particles, producing a porous matrix surface (**Figure 1C′′′ and 1F′′′**). The glass particles were surrounded by the silica-aluminum-calcium hydrogel produced during the acid-base reaction [8]; therefore, some level of chemical bonding was to be expected. However, the matrix of Maxxion R and Vitro Molar seemed to be more susceptible to peroxide degradation, especially when the poor performance in WL and CS tests was considered.

Despite the fact that a significant increase in KHN was seen for Vitremer at 24 hours, the behavior of treated and untreated specimens was similar. This variation in KHN was similar to that of the other cements; the presence of Bis-GMA and TEGDMA may have been reduced but did not completely prevent the matrix deterioration of the cement and the loss of filler particles. In this case, unlike the conventional GICs, cleavage of the poorly polymerized chains and oligomers in smaller and more soluble molecules is necessary to promote disintegration by ion transport outside the resin-modified material [8,26]. Compared with the results of previous studies, Vitremer could have been expected to show better performance. However, the similarity of the KHN results with those of conventional cements in the present study corroborates the results of another study that stated that the reaction between the polyacrylic acid and the glass particles occurred simultaneously with the polymerization of resin monomers, resulting in a slow reaction in an acidic environment, which could damage the final polymer, thereby promoting changes on the surface of the material (**Figure 1D**). Although these changes are less pronounced than those presented by Maxxion R and Vitro Molar, they may also have influenced the variations of the KHN during the bleaching protocol [27]. In a comparison of the cements and the different sessions, it can be seen that the influence of bleaching agent on KHN was dependent on small differences in the composition of the materials and the number of exposures, corroborating the results of previous studies [10,28].

Fluoride release from conventional GICs is commonly higher than that from resin-modified materials, because GICs are more soluble, porous, and have a slower cure reaction time [29,30], which requires protection. The bleaching protocol did not influence WL in any of the tested cements. Based on the disintegration of Vitro Fil and Vitro Molar during storage, the water seemed to be more harmful than the bleaching agent itself. It is probable that the short exposure time can cause changes on the surfaces of the cements, but cannot cause the agent to penetrate the bulk material deeply. However, different results have been shown with longer protocols or more concentrated agents. In a previous study, highly concentrated bleaching agents induced surface degradation and softening and increased fluoride release when used for between 24 and 120 hours [31]. Another protocol used in a previous study also showed increased solubility of conventional and resin-modified GICs when 38% hydrogen peroxide was applied for one week, one month, or 3 months [10]. Based on these results, the composition of the cements, concentration of the bleaching agent, and time of exposure

can be considered significant for the degradation of cements. Therefore, *in vivo* studies are necessary to confirm the capability of the materials to withstand the bleaching treatment.

A previous study has shown significant changes in the composition of GICs subjected to bleaching protocols [15]. The EDS results showed very similar elemental compositions for all cements, with small differences in concentrations. The relative counts of fundamental chemical elements such as Si, C, O, F, Na, and Al were recorded for all materials. However, other elements, such as Ca, Nb, W, Ba, and La, were seen in some of the materials in different concentrations (**Table 5**). Although EDS faces limitations in identifying and quantifying chemical elements with a low atomic number, such as C, the reductions in the fluoride and silicon counts must be highlighted. The increased release of fluoride produced by exposure to the bleaching agent may reduce the efficacy of this important mechanism, since residual amounts of fluoride will probably also be reduced. The Vidrion R, Vitro Molar, Vitro Fill, and Maxxion R cements had reduced fluoride counts after the first exposure to the bleaching agent, which might be associated with the matrix dissolution and consequent increase in the KHN after subsequent sessions. In addition, the reduced silica counts may have been primarily associated with the loss of filler particles, which might also have influenced the results for hardness and surface topography shown in SEM images (**Figure 1**).

Several studies have evaluated changes in the properties of GICs subjected to bleaching protocols [2,12,15,17,18], with a wide variation in results, hindering a consensus. Despite the careful procedures and strict criteria used in this study, it was an *in vitro* study limited by the absence of the influence of saliva and masticatory stress on restorative materials. However, most of the materials tested in the present study appear suitable for use during bleaching treatment, except for those that disintegrated during evaluation. In addition, methods to improve mechanical properties such as thermopolymerization should be developed, and clinical studies should be conducted to evaluate the durability of these materials under actual treatment conditions.

CONCLUSIONS

Based on the findings of the present study and respecting the limitations of *in vitro* methods, the null hypothesis tested was partly rejected. Although the bleaching agent did not influence the WL of the tested GICs, the effect of the agent on CS and KHN was dependent on exposure time and compositions of the cement. The bleaching agent also resulted in a reduced fluoride count, dissolution of the matrix, and exposure of filler particles of GICs during the bleaching sessions.

REFERENCES

1. Wilson AD, Kent BE. The glass-ionomer cement, a new translucent dental filling material. J Chem Technol Biotechnol 1971;21:313.
   CROSSREF
2. Baroudi K, Mahmoud RS, Tarakji B. Fluoride release of glass ionomer restorations after bleaching with two different bleaching materials. Eur J Dent 2013;7:196-200.
   PUBMED | CROSSREF
3. Markovic DL, Petrovic BB, Peric TO. Fluoride content and recharge ability of five glassionomer dental materials. BMC Oral Health 2008;8:21.
   PUBMED | CROSSREF

4.   Wiegand A, Buchalla W, Attin T. Review on fluoride-releasing restorative materials--fluoride release and uptake characteristics, antibacterial activity and influence on caries formation. Dent Mater 2007;23:343-362.
PUBMED | **CROSSREF**

5.   Arita K, Yamamoto A, Shinonaga Y, Harada K, Abe Y, Nakagawa K, Sugiyama S. Hydroxyapatite particle characteristics influence the enhancement of the mechanical and chemical properties of conventional restorative glass ionomer cement. Dent Mater J 2011;30:672-683.
PUBMED | **CROSSREF**

6.   Benetti AR, Jacobsen J, Lehnhoff B, Momsen NC, Okhrimenko DV, Telling MT, Kardjilov N, Strobl M, Seydel T, Manke I, Bordallo HN. How mobile are protons in the structure of dental glass ionomer cements? Sci Rep 2015;5:8972.
PUBMED | **CROSSREF**

7.   Bresciani E, Barata TJ, Fagundes TC, Adachi A, Terrin MM, Navarro MF. Compressive and diametral tensile strength of glass ionomer cements. J Appl Oral Sci 2004;12:344-348.
PUBMED | **CROSSREF**

8.   Carvalho FG, Sampaio CS, Fucio SB, Carlo HL, Correr-Sobrinho L, Puppin-Rontani RM. Effect of chemical and mechanical degradation on surface roughness of three glass ionomers and a nanofilled resin composite. Oper Dent 2012;37:509-517.
PUBMED | **CROSSREF**

9.   Khoroushi M, Keshani F. A review of glass-ionomers: From conventional glass-ionomer to bioactive glass-ionomer. Dent Res J (Isfahan) 2013;10:411-420.
PUBMED

10. Baroudi K, Mahmoud RS, Tarakji B, Altamimi MA. Effect of vital bleaching on disintegration tendency of glass ionomer restorations. J Clin Diagn Res 2014;8:214-217.
PUBMED | **CROSSREF**

11. de Paula AB, de Fúcio SB, Alonso RC, Ambrosano GM, Puppin-Rontani RM. Influence of chemical degradation on the surface properties of nano restorative materials. Oper Dent 2014;39:E109-E117.
PUBMED | **CROSSREF**

12. El-Murr J, Ruel D, St-Georges AJ. Effects of external bleaching on restorative materials: a review. J Can Dent Assoc 2011;77:b59.
PUBMED

13. Markovic L, Jordan RA, Glasser MC, Arnold WH, Nebel J, Tillmann W, Ostermann T, Zimmer S. Effects of bleaching agents on surface roughness of filling materials. Dent Mater J 2014;33:59-63.
PUBMED | **CROSSREF**

14. Turker SB, Biskin T. Effect of three bleaching agents on the surface properties of three different esthetic restorative materials. J Prosthet Dent 2003;89:466-473.
PUBMED | **CROSSREF**

15. Samiei M, Janani M, Vahdati A, Alemzadeh Y, Bahari M. Scanning electron microscopy and energy-dispersive X-ray microanalysis of set CEM cement after application of different bleaching agents. Iran Endod J 2017;12:191-195.
PUBMED | **CROSSREF**

16. Yu H, Li Q, Lin Y, Buchalla W, Wang Y. Influence of carbamide peroxide on the flexural strength of tooth-colored restorative materials: an *in vitro* study at different environmental temperatures. Oper Dent 2010;35:300-307.
PUBMED | **CROSSREF**

17. Yu H, Li Q, Wang YN, Cheng H. Effects of temperature and in-office bleaching agents on surface and subsurface properties of aesthetic restorative materials. J Dent 2013;41:1290-1296.
PUBMED | **CROSSREF**

18. Zavanelli AC, Mazaro VQ, Silva CR, Zavanelli RA, Mancuso DN. Surface roughness analysis of four restorative materials exposed to 10% and 15% carbamide peroxide. Int J Prosthodont 2011;24:155-157.
PUBMED

19. Crim GA. Prerestorative bleaching: effect on microleakage of Class V cavities. Quintessence Int 1992;23:823-825.
PUBMED

20. Mair L, Joiner A. The measurement of degradation and wear of three glass ionomers following peroxide bleaching. J Dent 2004;32(Supplementary 1):41-45.
PUBMED | **CROSSREF**

21. Briso AL, Lima AP, Gonçalves RS, Gallinari MO, dos Santos PH. Transenamel and transdentinal penetration of hydrogen peroxide applied to cracked or microabrasioned enamel. Oper Dent 2014;39:166-173.
PUBMED | **CROSSREF**

22. Gonçalves LS, Moraes RR, Ogliari FA, Boaro L, Braga RR, Consani S. Improved polymerization efficiency of methacrylate-based cements containing an iodonium salt. Dent Mater 2013;29:1251-1255.
PUBMED | CROSSREF

23. Fonseca RB, Branco CA, Quagliatto PS, Gonçalves LS, Soares CJ, Carlo HL, Correr-Sobrinho L. Influence of powder/liquid ratio on the radiodensity and diametral tensile strength of glass ionomer cements. J Appl Oral Sci 2010;18:577-584.
PUBMED | CROSSREF

24. Lim HN, Kim SH, Yu B, Lee YK. Influence of HEMA content on the mechanical and bonding properties of experimental HEMA-added glass ionomer cements. J Appl Oral Sci 2009;17:340-349.
PUBMED | CROSSREF

25. Topaloglu-Ak A, Cogulu D, Ersin NK, Sen BH. Microhardness and surface roughness of glass ionomer cements after APF and TiF4 applications. J Clin Pediatr Dent 2012;37:45-51.
PUBMED | CROSSREF

26. Sidhu SK, Nicholson JW. A review of glass-ionomer cements for clinical dentistry. J Funct Biomater 2016;7:16.
PUBMED | CROSSREF

27. Della Bona A, Rosa V, Cecchetti D. Influence of shade and irradiation time on the hardness of composite resins. Braz Dent J 2007;18:231-234.
PUBMED | CROSSREF

28. Cehreli ZC, Yazici R, García-Godoy F. Effect of home-use bleaching gels on fluoride releasing restorative materials. Oper Dent 2003;28:605-609.
PUBMED

29. Cacciafesta V, Sfondrini MF, Tagliani P, Klersy C. In-vitro fluoride release rates from 9 orthodontic bonding adhesives. Am J Orthod Dentofacial Orthop 2007;132:656-662.
PUBMED | CROSSREF

30. Xu X, Burgess JO. Compressive strength, fluoride release and recharge of fluoride-releasing materials. Biomaterials 2003;24:2451-2461.
PUBMED | CROSSREF

31. Lee KH, Kim HI, Kim KH, Kwon YH. Mineral loss from bovine enamel by a 30% hydrogen peroxide solution. J Oral Rehabil 2006;33:229-233.
PUBMED | CROSSREF