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Original Article

Evaluation of Fluoride Release, pH and Microhardness of Glass Ionomer Cements

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Abstract

Objective: To evaluate fluoride release, pH and microhardness of glass ionomer cements (GIC). **Material and Methods:** Four different cements: Vidrion ® R (G1), Vitro Fil ® (G2), Maxxion ® (G3) and Magic Glass ® (G4) and a composite resin (G5 - control group) comprised the sample. Thirty specimens were manufactured (10 mm x 3 mm) and stored in deionized water. Analyses were performed after 24 hours, 7 days and 28 days. The following devices were used for measurements: fluorometer, pH meter and microhardness tester. Data were analyzed by Kruskal-Wallis, Mann-Whitney, Friedman and Wilcoxon tests ($\alpha = 0.05$) using the SPSS ® software (Statistical Package for Social Sciences) version 17. **Results:** GICs were able to release fluoride; however, the amount released decreased with increasing immersion time ($p < 0.002$). The pH of all products increased over time, the lowest value was identified for Magic Glass (5.93) and the highest for Maxxion (6.94) at time of 24 h. Materials showed significant decrease in surface microhardness, especially G4 for presenting the lowest recorded values ($p < 0.05$). **Conclusion:** GICs are fluoride-releasing restorative materials with pH favorable to oral homeostasis and good mechanical behavior.

Keywords: Hardness Tests; Fluorides; Dental Materials.

Introduction

Since the early history of materials used in the human body, especially those used in the oral cavity, it is known that they should be stable and have no interactions with the surrounding environment. However, it is likely that the first sparks of producing active materials with defined interactions with the human body have originated from the fact that materials capable of releasing fluoride may exert useful effects [1]. Materials with this characteristic began to gain great prominence in dentistry due to their cariostatic properties [2].

Since its inception in 1971, Glass ionomer cement (GIC) has undergone several modifications. The initial goal was to find an ideal restorative material with physical properties similar to tooth structure with adhesion to enamel and dentin, resistance to degradation in the oral cavity and ability to release fluoride [3,4].

Conventional glass ionomer is basically a powder glass composed of fine-grained calcium fluoraluminium silicate and a viscous liquid, which is a polialcenoic acid, usually represented by polyacrylic and polymaleic acids [5]. These cements have demonstrated greater fluoride release among fluoride materials mainly due to its setting reaction (gelation) [2].

A peculiar feature is the ability of these materials to absorb fluoride from the medium for subsequent release, extending the period of time in which fluoride is available in the oral cavity [6]. Thus, glass ionomer cement acts as a rechargeable device for fluoride release [7]. This capability has already been demonstrated by previous studies indicating that the regular use of toothpaste may result in the absorption of fluoride into the glass ionomer cement and subsequent release to the tooth structure [8,9].

Thus, through the release of fluoride ions, glass ionomer cement can keep around it an environment conducive to remineralization [2], and the recurrence of caries around restorations with this material is practically nonexistent [10].

One of the advantages of glass ionomer cements as compared to other restorative materials is that they can be placed in oral cavities without any need for bonding agents and have good biocompatibility, although they also have lack of adequate strength and toughness [1].

Surface hardness tests have been used to evaluate the degradation and durability of these materials, to evaluate the effect of the storage media on the surface (since conventional ionomer glass is very sensitive to water absorption) as indicative of wear resistance and durability, and also to monitor the hardening process of cements [11-13].

Given the above and the applicability of glass ionomer cements in dental practice, the present study evaluated in vitro fluoride release, hydrogen potential (pH) and microhardness of conventional glass ionomer cements.

Material and Methods

The sample consisted of four different conventional glass ionomer cements and a micro-hybrid composite resin was used as control, as described in Table 1.

Table 1. Groups and materials tested.

Groups	Material	Manufacturer
G1	Vidrion R®	S.S. White Artigos Dentários Ltda., Rio de Janeiro, RJ, Brazil
G2	Vitro Fil®	DFL Ind. e Com. Ltda., Rio de Janeiro, RJ, Brazil
G3	Maxxion®	FGM Produtos Odontológicos Ltda., Joinville, SC, Brazil
G4	Magic Glass®	Vigodent SA Ind. Com., Rio de Janeiro, RJ, Brazil
Control	Fill Magic®	Vigodent SA Ind. Com., Rio de Janeiro, RJ, Brazil

To obtain samples of both materials (glass ionomer and composite resin), recommendations of manufacturers were followed and six samples of each product were manufactured, totaling 30 samples, which were prepared with the aid of a Teflon matrix with 10 mm in diameter and 3 mm in height. Resin photopolymarization was carried out using the Ultra-Lux device (Dabi Atlante S/A Indústrias Médico Odontológicas, Ribeirão Preto, SP, Brazil), with light intensity of approximately 400 mW / cm ². Plastic pots with lids, in which waxes # 7 were placed in the lid diameter to fix the dental floss and keep the sample suspended, carefully not to touch the container walls, were used. Samples were stored at 37 ° C throughout the study, (Nova Ética Produtos e Equipamentos Científicos Ltda., Vargem Grande Paulista, SP, Brazil). Samples were immersed in deionized water so that there was no interference from ions originating from the water, and solutions were replaced every 24 hours.

Readings were performed after 24 hours, 7 days and 28 days. The analysis of the amount of fluoride released was performed through water collected using specific electrode coupled to fluoride analyzer unit Thermo Orion 7010A+, previously calibrated every measurement with five standard sodium fluoride solutions 0.4; 0.8; 1.6; 3.2; 6.4 ppm prepared with TISAB II (Total Ionic Strength Adjustment Buffer). The device analyzed the potential difference of the solution and provided the value in millivolts, which was subsequently converted into ppm. The solution consisted of 1 mL of water from samples with the addition of 1 ml of TISAB II solution.

The pH of solutions was measured with Microprocessor pH meter HI-221 and pH meter HI 221 Microprocessor pH meter (Hanna Instruments Inc. USA), which was previously calibrated with standard pH solutions, one of pH 4.00 and another of pH 7.00. After each analysis, the electrodes were washed with distilled water and dried with paper towels.

At the end of the immersion period (28 days), microhardness analysis was performed. The samples were divided into four equal quadrants corresponding to an indentation per quadrant. The experiments were performed under a static load of 50 g for a period of 10 s (Michohardness Tester FM-700, Future-Tech Corp., Fujisaki, Kawasaki-ku, Japan).

Data were organized in SPSS software (Statistical Package for Social Sciences) version 17 and descriptively and inferentially analyzed, where Kruskal-Wallis, Mann-Whitney, Friedman and Wilcoxon tests were applied, considering α = 0.05.

Results

The amount of fluoride released from the tested materials in different time periods is shown in Table 2. At 24 hours, G2 was the material that released the greatest amount of fluoride, with significant difference (p <0.05). In 28 days, G1 released less fluoride compared to other materials, with significant difference (p <0.05).

In the analysis of the amount of fluoride released according to time (Table 2), all GIC groups decreased the amount of fluoride released over time, regardless of product, with significant difference (p <0.002). The same condition was verified for composite resin (p <0.05).

Table 2. Fluoride release values (in ppm) of groups of materials tested according to three different times of immersion in deionized water.

Groups	24 hours Mean (SD)	7 days Mean (SD)	28 days Mean (SD)	p-value
1	72.71±6.10 [aA]	20.48±5.95 [aA]	4.54±1.21 [aA]	0.002
2	124.93±9.30 [bB]	36.27±4.72 [bB]	16.41±8.57 [bB]	0.002
3	73.58±12.58 [cA]	44.31±5.14 [cC]	14.48±5.71 [cB]	0.002
4	87.45±12.37 [dA]	54.18±6.21 [dC]	26.37±9.04 [dB]	0.002
5	0.81±1.02 [eC]	0.15±0.10 [fD]	0.08±0.02 [gC]	0.030
p-value	<0.001	<0.001	<0.001	

Values expressed as mean (SD) of fluoride released (in ppm) of each tested group are analyzed according to the immersion time in rows (Friedman test, with α = 0.05). Same lowercase letters indicate that there is no significant difference in fluoride release for each group, according to immersion times (Wilcoxon test, with α = 0.05, with 3 combinations). In columns, values expressed as mean (SD) of fluoride released (in ppm) of each tested group are analyzed according to groups (Kruskal-Wallis test with α = 0.05). Same capital letters indicate that there is no significant difference between products (Mann Whitney test, with 10 combinations, α = 0.05).

It was found that over time, except for G3 group, there was a significant increase of pH values, although this group showed trend to neutrality in all time periods evaluated (Table 3).

Table 3. pH values of materials tested in three different times.

Groups	24 hours Mean (SD)	7 days Mean (SD)	28 days Mean (SD)	p-value
1	6.13±0.25	7.33±0.19	7.04±0.12	0.02
2	6.47±0.14	6.67±0.06	6.71±0.17	0.04
3	6.94±0.18	7.02±0.09	7.07±0.14	0.51
4	5.93±0.22	6.41±0.08	6.50±0.07	0.09
5	4.31±0.81	7.13±0.21	7.06±0.10	0.01

The pH values expressed as mean (SD) of each tested group are analyzed according to the time of analysis after immersion in rows (Friedman's Test Two-Way Analysis of Variance, with α = 0.05).

The microhardness of the materials analyzed at the initial (24 hours) and final period (28 days) is shown in Table 4, in which it is possible to verify that the materials tested showed significant decrease in surface microhardness, and G4 showed the lowest values (p <0.05).

Table 4. Mean microhardness values of materials tested at initial and final period.

Groups	Time 24 hours Mean (SD)	28 days Mean (SD)
G1	$20.9 \pm 5.6^{A.a}$	$12.5 \pm 2.5^{B.a}$
G2	$41.4 \pm 11.4^{A.a}$	$6.1 \pm 2.2^{B.a.c}$
G3	$17.0 \pm 3.4^{A.b}$	$6.0 \pm 2.0^{B.a.c}$
G4	$6.3 \pm 2.7^{A.b}$	$3.53 \pm 0.2^{B.b.c}$
G5	$42.0 \pm 4.7^{A.a}$	$22.8 \pm 1.6^{B.a}$

Vickers microhardness (VHN) values expressed as mean (SD) of each tested group are analyzed according to the time of analysis after immersion in rows (Wilcoxon test, with $\alpha = 0.05$). Same capital letters indicate that there is no significant difference. In columns, Vickers microhardness (VHN) values expressed as mean (SD) of each tested group are analyzed according to groups (Kruskal-Wallis and Dunn's post test, with $\alpha = 0.05$). Same lowercase letters indicate that there is no significant difference.

Discussion

Despite the limitations of in vitro studies in clinical practice, fluoride release, pH and vickers microhardness denote some of the main physicochemical characteristics of materials and their possible behavior in the oral cavity, making results useful for predicting performance of the tested materials.

It was observed that all materials released high amounts of fluoride at the initial period; however, the amount of fluoride released reduced over time ($p < 0.05$). Previous studies [14-17] obtained similar results. High initial fluoride rates released by in vivo GIC contribute to eliminate remaining microorganisms of the oral cavity and to strengthen demineralized enamel and dentin [18]. However, even with the decrease in fluoride release due to the reaction of these ions with the GIC matrix, the constant amount of fluoride released prevents the development of new caries sites near restoration and increases the concentration of ions in saliva [14].

In the evaluation of fluoride release by commercial brands according to the different periods, it was found that, within 24 hours, Vitro Fill ® showed higher fluoride release ($p < 0.05$) when compared to the other products. In contrast, at 7 and 28 days of evaluation, Magic Glass released larger amounts of fluoride but without differences between groups. In this study, Vidrion R ® released less fluoride in all periods of analysis, especially at the end of the period (28 days), with significant difference from the other groups ($p < 0.05$). Direct comparisons between the results of this study and previous studies are difficult to establish due to the presence of distinct commercial brands and to various factors that influence fluoride release such as storage medium (pH, temperature and composition), which can lead to different performances of materials [19].

All materials tested showed pH near neutrality, which favors the antimicrobial effects of cements [20] and indicates the stabilization of ions in materials at 7 and 28 days [21]. pH alkaline or close to neutrality, which was a trend observed in all materials, except for G3, may be important for the release of the growth factor of dentine, which has been implicated in signaling reparatory events necessary in response to the attack of dental caries [21].

Regarding microhardness, materials showed a decrease of this mechanical property, and the lowest values were observed for Groups 2, 3 and 4. Material can be "more" or "less" hard, depending on several factors such as chemical composition, polycarboxylic acid concentration and molecular weight, powder / liquid ratio and storage time and type [11]. Probably, a combination of these factors explains variations found in results.

Fluoride release, pH and microhardness of tested GICs varied in accordance with the commercial brand in specific immersion times, rejecting the hypothesis that fluoride release does not decrease with immersion time. However, methodologies with pH cycling can increase the performance and differences among the various available materials [22]. Thus, the use methodologies with pH cycling in studies with fluoride-releasing materials for demineralization and remineralization process are indicated.

Conclusion

Fluoride release decreased with immersion time and all products had pH near neutrality and good mechanical behavior.

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