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Fluoride release of restorative commercial conventional glass-ionomer cements

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APROVAL PAPER

DEDICATÓRIA

Dedico a concretização desse trabalho às pessoas

que dedicaram a mim a maior parte do seu tempo

e amor incondicional, meus pais.

Essa conquista também é de vocês.

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"A gratidão é o único tesouro dos humildes." Willian Shakespeare

"Conheça todas as teorias,

domine todas as técnicas,

mas ao tocar uma alma humana,

seja apenas outra alma humana."

*(*Carl G. Jung)

RESUMO

RESUMO

Liberação de flúor de diferentes marcas de ionômero de vidro convencional restaurador

Introdução: Inicialmente introduzidos por Wilson e Kent, os cimentos de ionômero de vidro (CIV) apresentam propriedades desejáveis. Desde desenvolvida em meados de 1980, a Técnica Restauradora Atraumatica (ART) é uma combinação de procedimento preventivo e restaurador introduzido para atendimento em áreas de condições precárias. A capacidade de liberação de flúor é uma das propriedade importante dos cimentos de ionômero de vidro e é fundamental para o sucesso em longo prazo das restaurações de ART. Novos CIVs tem emergido no mercado com melhores propriedades físicas, indicados pelos fabricantes para a técnica de ART. O objetivo desse estudo foi avaliar a liberação de flúor de nove cimentos de ionômero de vidro (CIV) convencionais restauradores brasileiros representando todas as marcas atualmente disponíveis no mercado. Eles foram divididos em indicados para ART [Maxxion-FGM-Brasil (MA), Ion Z-FGM-Brasil (IZ), Ionglass-Maquira-Brasil (IG), Bioglass R-Biodinâmica-Brasil (BG), Vitro Molar-Nova DFL-Brasil (VM)] e não indicados para ART [Vidrion-SSWhite-Brasil (VD), Ionomaster-Wilcos-Brasil (IM), Magic Glass-FGM-Brazil (MG), Vitro Fil-Nova DFL-Brasil (VF)]. Métodos: Espécimes (n=10) com 11.0mm de diâmetro e 3.0mm de espessura das nove marcas de CIV foram confeccionados de acordo com as instruções do fabricante em um molde de teflon. Os mesmos foram imersos em recipientes Falcon contendo 20ml de saliva artificial, fechados a 37º a as medidas de concentração de flúor foram realizadas após 1, 6, 12, 24, 72, 168, 336 horas. A concentração de flúor foi medida em duplicata, usando uma técnica micro analítica com um eletrodo sensível de íons flúor (Orion, modelo 96-09) unido a um potenciômetro (Procyon, modelo 720). Após os testes de normalidade (Shapiro-Wilk) e homogeneidade de variâncias (Levene) ($p<0.05$), os resultados foram submetidos a ANOVA e teste de Tukey para comparações múltiplas. Resultados: Em cada período avaliado foram observadas diferenças estatísticas entre os CIVs ($p<0.05$), apresentando o MA a maior quantidade de liberação de flúor para todos os períodos, com um total de 7.7 µg/mm² ($p<0.05$). O valor mínimo observado de liberação de flúor foi do VM, também em todos os períodos, totalizando em 1.14µg/mm².

Observou-se que a maioria dos CIVs apresentou o mesmo padrão de liberação de flúor, indicados ou não para ART. Houve diferenças significativas especialmente em comparação com a quantidade de liberação de F- entre os CIVs indicados e não indicados para ART. Conclusão: De acordo com os resultados obtidos, a maioria dos CIVs testados apresentaram mesmo padrão de liberação de flúor. Contrário ao esperado, no geral, os CIVs indicados para ART apresentaram quantidade maior de liberação de flúor em comparação aos não indicados para ART.

PALAVRAS-CHAVE: Cimentos de Ionômeros de Vidro; Fluoretos; Tratamento Dentário Restaurador sem Trauma.

ABSTRACT

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Fluoride release of restorative commercial glass-ionomer cements

Introduction: First discovered by Wilson and Kent, glass ionomer cements (GIC) present desirable properties. Atraumatic Restorative Technique (ART) is a combined preventive and restorative procedure developed for attendance in poor condition areas. Fluoride release (F-release) is one of the most important properties of GIC and it is essential to ART's approach high success rates. New GICs emerged in market with better physical properties and also indicated by manufacturers for the ART technique. The aim of this study was to evaluate the F- release of nine Brazilian conventional restorative GIC representing all national commercial brands available in the market. GIC were divided as indicated for ART [Maxxion-FGM (MA), Ion Z-FGM (IZ), Ionglass-Maquira (IG), Bioglass R-Biodinâmica (BG), Vitro Molar-Nova DFL (VM)] or not indicated for ART treatment [Vidrion-SSWhite (VD), Ionomaster-Wilcos (IM), Magic Glass-FGM (MG), Vitro Fil-Nova DFL (VF)]. Methods: Specimens (n=10) with 11.0mm in diameter and 3.0mm thick of the nine brands of GICs were manipulated in accordance to the manufacturer's instructions and inserted in a teflon mould. Those were immersed in falcon recipients containing 20ml of artificial saliva, closed at 37ºC in a laboratory oven and fluoride concentrations were measured after 1, 6, 12, 24, 72, 268, 336 hours. Fluoride concentration was measured in duplicate, using a micro analytical technique with an inverted fluoride ion selective electrode united at a potentiometer (Procyon, model 720). After the normality (Shapiro-Wilk) and homogeneity of variance (Levene) tests, results were submitted to ANOVA and Tukey Test for multiple comparisons ($p<0.05$). Results: In each evaluation time, significant statistical difference for amount and pattern of fluoride release was observed among the GICs ($p<0.05$), presenting Ma the highest fluoride release in all times, with a total of 7.7 µg/mm² ($p<0.05$). The lowest fluoride release was observed for VM in all times, with a total of 1.14 µg/mm². Most of GICs presented same pattern of F- release, indicated or not for ART. There was statistical significant difference especially when comparing F- release of GICs indicated for ART and those not indicated for ART ($p<0.05$). Conclusion: According to the results obtained, Brazilian commercial GICs tested presented similar pattern of F- release. Contrary to expected, most of GICs indicated for ART showed higher amount of fluoride release in comparison to those not indicated for ART.

KEY-WORDS: Glass ionomer Cements; Fluorides; Dental Atraumatic Restorative Treatment.

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LIST OF ABBREVIATIONS AND ACRONYMS

GIC - Glass Ionomer Cement

SEM – Scanning Electronic Microscope

Sps – specimens

F- – fluoride

MG – Magic Glass

BG – Bioglass

IM – Ionomaster

VF – Vitro Fill

VM – Vitro Molar

IZ – Ion Z

IG – Ionglass

MA – Maxxion

VD – Vidrion

µm/mm² - Amount of fluoride released by 1 mm² of specimen

±SD –Standard Deviation

ART – Atraumatic Restorative Technique

P/L – Powder:liquid ratio

SUMMARY

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1 INTRODUCTION

1. INTRODUCTION

Glass ionomer cements (GICs) composition involves an acid and a glass with an acid-base reaction initiating on mixing the components.[1] When first introduced in the 1970s by Wilson and Kent[6], those materials were increasingly studied for their desirable properties, as the adhesion to dental hard tissues and the anti-cariogenic effect of fluoride release.[2,4,5] Thus, the mechanical and physical properties were limited, which induced dental researchers to invest on the development of new materials, that are still improving nowadays. According to Forsten[7] (1998), if the caries activity is high and the demand is for a minimally invasive cavity, the first choice is the conventional GICs. As a combined preventive and restorative procedure, the Atraumatic Restorative Treatment (ART) is based on removing tooth's carious structure and filing it with fluoride-releasing adhesive materials, the conventional GICs.[8,9,10] Therefore, in early trials the GICs used were specifically developed for ART technique, and studies show a high quantity of failures, even in adequate operatory conditions.[10] Recently, new GICs emerged on market, with improved handling and physical properties, with smaller particle sizes, specifically for ART approach.[11] These materials fluoride release is fundamental to the success in ART, once one reason for its failure are secondary caries. [12]

The acid-base reaction by silicate glass and polyalkenoic acids set by these materials leads to the release of ionic constituents. As one of them, the fluoride release is seen as one of the most important properties of the GIC and a predominant caries-inhibiting process.[2,13] This means that, silicate and glass ionomer cements represent materials types in which fluoride is not added, but it is a part of the chemical reactions of the material.[7] The cement maturation is relatively slow, and during this reaction, the water balance with the system is maitained.[14] It occurs for conventional GICs following a higher release of fluoride over the first 24 hours, explained by the setting reaction when the glass particles react with the polyalkenoate acid with an initial burst of fluoride release. It involves a rapid dissolution from outer surface into solution.[15] The fine grain size distribution from newer reinforced GICs indicated for ART ensures an increase in physical properties.[11,16,17] Consequently, a high strength to dissolution is observed, associated to the decrease of ions released, such as fluoride.

After this initial burst, fluoride release slows down and shows a prolonged long-term fluoride release, which occurs when the acidified water from the hydrogel matrix dissolves the glass. That is a result of the sustained diffusion of ions through the bulk cement.[16] For both traditional or indicated for ART glass ionomers, it is important that both material present a strong initial release, called as "burst" effect, and a constant release, characterized by a less strong and stable release.[7] Two characteristics related to quality of ART restoration are caries development and weakness of the filling material.[10] In this case, a strong initial release will activate the expected remineralization of affected adjacent enamel while the long-term release will maintain small amounts of fluoride at the risk-areas, which leads to secondary caries resistance.[7]

Conventional glass ionomers developed for ART present lower fluoride release when compared to traditional conventional GICs.[19] Despite their relatively low fluoride release, the reinforced GICs presented some protection against demineralization of adjacent enamel.[20] Thus, the anticariogenic behavior effect of glass ionomers is related to this reduction of demineralization by changing the hydroxyapatite of teeth to fluorapatite. Also, the promotion of remineralization process induces the suspension or regression of enamel lesion. [2,20,21]

According to Cranfield[25], et al (1982), a crucial point for clinical success is the longevity of fluoride release. Low amounts (2mg/l) of F- release are able to inhibit bacterial plaque formation and encourage the hydroxyapatite formation, according to Wahab[23] ,et al(1993).

In the oral environment, many exogenous fluoride sources are frequently in contact with the dental restorations, such as dentifrices, mouth rinses and varnishes.[15] For this reason, fluoride-releasing materials may act as a reservoir for fluorides, called as "intelligent" materials, reducing even more secondary caries and neutralizing the pH, mainly in high-caries risk patients.[20,24] As shown in researches, materials with higher initial fluoride release are considered to have a higher recharge capability.[15,25] This recharge property also occurs to reinforced GICs used for ART, in proportion to its fluoride release.[26]

Many researches are published with results of fluoride release amount of different GIC formulations.[27,29,41] The elution of fluoride ions is a complex process.[28,29] A few factors can influence and affect this process, as particular materials formulations and fillers or experimental factors, as storage media, storage solution frequency of change, power-liquid ratio, several environmental conditions, composition of saliva and biofilm formation.[30,31,35] So, those factors should be well controlled and standardized to avoid inconsistent results.[7] Different studies with different methods and materials could lead to different results.[36] Basso[31], et al (2011) concluded their results were not comparable to previously published studies in literature since their study was developed in different storage media and times. It can be expected that many randomized research criteria and experimental variables may modify and/or mask the real materials results.

Many studies showed the improved success rates of ART restorations with new indicated for ART glass ionomers compared to early studies with traditional conventional GICs.[11,20] Van's Hof [46] concluded that this approach should not be executed with medium-viscosity (traditional) GICs.[49] The expansion of ART use and high cost of international brands led to an increase in the number of commercial GICs available in Brazil. Among the high number of chemically-cured conventional glass ionomer cements, many are indicated for ART by manufacturers. Tests must to be done to confirm among those materials, cheaper alternatives for ART use and materials truly suitable for this use.

There is a need for more studies about GICs recently added to market and its properties, as fluoride release. Also, is necessary to find alternative less expensive materials that may be used in large scale without distinction of social status. The aim of this study was to evaluate and compare the fluoride release of nine Brazilian commercial available restorative conventional glass ionomer cements during a 14-day period using the same methodology. The first null hypothesis (H_1) was that there would not be qualitative (pattern) difference in the fluoride release between the conventional GICs used for ART and other conventional Brazilian GICs available. The second null hypothesis (H_2) was that there would not be difference in quantitative (amount) in fluoride release among conventional GICs used for ART and other conventional GICs in this study.

2 Article

FLUORIDE RELEASE OF BRAZILIAN BRANDS GLASS-IONOMER CEMENTS RECOMMENDED TO ATRAUMATIC RESTORATIVE TECHNIQUE *

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Abstract

Introduction: The Atraumatic Restorative Treatment (ART) initiated in Tanzania in 1980s with the introduction of health oral care to prevent teeth extractions. Fluoride release (F-release) ability is important to ART's high success rates, as a caries-inhibiting element. With the diffusion of restorative treatment techniques and development of GICs specifically for ART use, manufacturers expanded the number of materials indicated for this approach. The aim of this study was to evaluate the fluoride release of nine Brazilian conventional restorative glass-Ionomer cements (GIC) representing all national commercial brands available in the market. It was divided as indicated for ART [Maxxion-FGM (MA), Ion Z-FGM (IZ), Ionglass-Maquira (IG), Bioglass R-Biodinâmica (BG), Vitro Molar-Nova DFL (VM)] or not indicated for ART treatment [Vidrion-SSWhite (VD), Ionomaster-Wilcos (IM), Magic Glass-FGM (MG), Vitro Fil-Nova DFL (VF)]. Methods: Specimens (n=10) with 11.0mm in diameter and 3.0mm thick of nine commercial Brazilian brands of GICs were made in accordance to the manufacturer's instructions in a Teflon mould. Each one was immersed in plastic recipients containing 20ml of artificial saliva, closed at 37ºC and fluoride concentrations were measured after 1, 6, 12, 24, 72, 268, 336 hours. Fluoride concentration was measured in duplicate, using a microanalytical technique with an inverted fluoride ion selective electrode united at a potentiometer (Procyon, model 720). After the normality (Shapiro-Wilk) and homogeneity of variance (Levene) tests, results were submitted to ANOVA and Tukey Test for multiple comparisons ($p<0.05$). Results: In each evaluation time significant statistical difference for amount (quantitative) and pattern (qualitative) of fluoride release was observed among the GICs ($p<0.05$), presenting MA the highest fluoride release in all times ($p<0.05$). The lowest fluoride release was observed for VM in all times. Most of GICs presented same pattern of fluoride release, indicated of not for ART. There was statistical significant difference especially in comparison with the amount of fluoride released among GICs indicated or not indicated for ART ($p<0.05$). Conclusion: According to the results obtained, all Brazilian commercial GICs presented fluoride release ability. Some of those recommended for ART showed variable profiles of fluoride release, with difference of those not indicated for ART.

KEY-WORDS: Glass ionomer Cements; Fluorides; Dental Atraumatic Restorative Treatment.

1. <u>Introduction</u>

Nowadays there are still in many developing countries areas with no access to proper dental care, where dental caries is left untreated. [1] The treatment approach called Atraumatic Restorative Treatment (ART) was initiated in Tanzania in the 1980s and introduced health oral care with the purpose to prevent tooth extractions. [3,4] The treatment is focused in partial removal of caries lesions with hand instruments and filling the cavity with a restorative material, which is usually Glass Ionomer Cement (GIC) due to its desirable properties.[2,5] Despite the good properties, some disadvantages as water sensitivity and poor strength have been limiting the success rates of ART. A more recently developed generation among conventional glass ionomer cements, introduced at 1995, called as high-strength, reinforced glass ionomers or high viscosity by manufacturers is specifically for ART use.[10] The improvement is associated to particles size reduction (about 2 µm) so, they can be added in a higher amount.[6] These new materials have showed better physical properties, including high strength to dissolution. [7,9]

Fluoride release is known as one of the most important properties of GIC[11] and ion-releasing materials may reduce the secondary caries formation, as showed in many in vitro[11,12,13,14,15] and in vivo studies.[16,17] This particular ion has been of special interest, once it is a primordial caries-inhibiting element.[20] Moreover, GICs have adhesion to tooth structure, through ionic bonds formation between the calcium ions in enamel and the polyacrylic acid in polycarboxylate cement.[19] A constant flow of fluoride by period of time[21] is required to GICs, nevertheless the amount of fluoride release within this materials is diverse. Diverse methodologies as storage media, composition, specimen size, period of aging are contributory factors, which influence and explain numerous differences. [21,24,23,24,25] These distinct experimental conditions can mask the real effects of some intrinsic variables involved on fluoride release of those materials.[25]

Materials developed for specific ART use present lowest fluoride-release compared to other materials, due to their lower solubility, as results of their better physical/mechanical properties.[7,9] Yip[26], et al (1999) observed that high viscosity GICs release similar amounts of fluoride ions to a metal-modified GIC (Ketac-Silver-ESPE GmBh, Seefeld, Germany), which is less than for the conventional restorative GICs. According to the findings of Smales and Gao (2000)[5], conventional GICs indicated for ART tested guaranteed protection against artificial caries challenge to adjacent enamel.

Although in principle the ART was created to underprivileged communities, it is reaching the public dental health services and academic areas.[28] With the development and diffusion of restorative treatment techniques, manufacturers expanded the number of GICs indicated for ART. This leads to a suspicion if the materials that are indicated for ART by manufacturers really are suitable for this use. According to Van't Hof[9], et al (2006), the medium-viscosity (traditional) conventional GIC should not be used for ART restorations. Unfortunately, most ideal materials for ART use present expensive costs to use in developing countries such as Brazil, leading to the search for alternative national materials that present suitable properties for a increased success rate.[29] Ferreira[29], et al (2006) found interesting results: a Brazilian Brand GIC presented better performance regarding cervical microleakage when compared to one imported GIC, considering it as an alternative material for the ART approach. Although, the ART indication and success rate is also related to mechanical strength, and more studies must be done to finally indicate these materials.

Because of the importance of the fluoride release ability behavior and the need of more studies focusing on alternative and cheaper materials for the ART, the aim of this study was to evaluate and compare the fluoride release profile among Brazilian brands conventional GICs indicated and not-indicated for ART for 14-day period using the same methodology. The first null hypothesis (H_1) was that there would not be qualitative (pattern) difference in the fluoride release between the conventional GICs used for ART and other conventional Brazilian GICs available. The second null hypothesis (H_2) was that there would not be quantitative (amount) difference in fluoride release among conventional GICs used for ART and other conventional GICs in this study.

2. Methods

2.1 Experimental Design

The Brazilian conventional restorative glass ionomer cements investigated were among conventional chemically-cured GICs: indicated for ART [Vitro Molar-Nova DFL-Brazil (VM), Maxxion R-FGM-Brazil (MA), Ion Z-FGM-Brazil (IZ), Ionglass-Maquira-Brazil (IG), Bioglass R-Biodinâmica-Brazil (BG)] or not-indicated for ART: [Vidrion R-SSWhite-Brazil (VD), Ionomaster-Wilcos-Brazil (IM), Magic Glass-FGM-Brazil (MG), Vitro Fil-Nova DFL-Brazil (VF)]. The formulations are present in table 1.

2.2 Specimen preparation

For each restorative material, ten disk-shaped specimens (sps) with 11.0 mm in diameter and 3.0 mm thick were made using cylindrical Teflon molds. Materials handling was carried out at controlled temperature (23º±1º) and relative humidity (50±5%). The same calibrated person manufactured all specimens. The powder:liquid (P/L) ratio used and manipulation of materials was done according to the manufacturer's instructions (Table 1) and a scale providing the accurate weight. It was made with a centrix dispenser, reducing the risk of blisters. Immediately after mixing, the cement was inserted into the mold and covered by a polyester strip at top and bottom surfaces. As well, a nylon thread piece was introduced in the specimen middle, to allow it to be suspended at the storage media. Glass plates were used as basis below and also over, tight and pressed to produce a smooth surface. After cement initial curing, 10 minutes inside the molds, the specimens were removed and excess material cleaned carefully using a scalpel blade. Then, each one was individually immersed in falcon plastic recipients. Plastic vessels were chosen because it is known that glass vessels absorb and leach fluoride, so they would interfere with the result. [30] Those falcon recipients contained 20 mL of artificial saliva, being correctly identified, hermetically closed and stored at 37^{o}C for 14 days. Fluoride concentrations were measured after 1, 6, 12, 24, 72, 168 and 336 hours. For each time measured 2 ml of solution were taken to a duplicate evaluation of results in a two different plastic tubes. The artificial saliva composition used was according to literature.[44]

2.3 F- release measurement

The fluoride ion concentration in the solutions was measured using an Orion fluoride-specific electrode (model 96-09) and a Procyon digital ion–analyzer (model SA-720). For those analysis, 1 ml of solution was removed and immediately another one for duplicate. The fluoride electrode was previously calibrated. The mV readings were transformed into µgF/mm² by the formula: (amount released*total quantity of solution)/specimen area.

2.4 Statistical analysis

The data was calculated and statistically analyzed with SPSS for Windows v.19.0 (IBM Statistics, Chicago, USA). The assumptions of normal distribution and of equality of variances were checked for all the variables using Shapiro-Wilk and Levene test, respectively. As the assumptions were satisfied, data was subjected to two-way ANOVA ($p \leq 0.05$) followed by Tukey's test ($p<0.05$) for individual comparisons.

3. **Results**

The highest amount of fluoride registered at all periods tested was for MA, with a constant and homogeneous amount of ions released ($p<0.05$) (Graphic 2 and Table 2). The lowest results obtained were for VM, with the smallest amount of fluoride ions compared to all materials and during all periods of time ($p<0.05$). Results showed differences when observed different periods of time of the same material for all of them ($p<0.05$) (Graphic 2).

Qualitative results observed was that MG, BG, IM, VF, IZ and IG showed an initial "burst" and after a decreased gradual elution. MA presented a constant and frequent fluoride release during all times. VM presented an uncommon pattern, releasing it in a "burst" of high amount of fluoride at 3 to 7 days. VD presented an initial burst at 12 hours and kept with no statistical difference up to 14 days. Graphic 1 presents the accumulated amount of fluoride ions released among time, in descending order: MA>IZ>MG>IM>IG>BG>VF>VD>VM.

4. Discussion

The first null hypothesis of this study was rejected. Different GICs indicated and not indicated for ART presented significant differences on pattern of fluoride release. The recommended materials for ART according to their manufacturers are VM, MA, IZ, BG, IG. It is expected in the first 24 hours an initial fluoride "burst" effect, which induces remineralization and contributes to reduce the viability of bacteria that may be remained in inner carious dentine in ART approach restorations.[31,32] Then, a release drop with constant levels of fluoride around a restoration's margins, is associated with secondary caries inhibition by reduce or even stop of the development of new caries sites.[31,33,34] Shiiya[35], et al (2012) observed that even after the fluoride release has diminished, dentin around fluoride-releasing restoration was protected against demineralization. Also, a recent study showed that bacterial acidogenicity, dry weight and bio-volume decreased in presence of fluoride ions.[12]

The reaction frequently happens, as confirmed by various studies, with maximum release during the first 24-48 hours.[27,31,32,33,36,37] In fact, most of glass ionomer cements in this study exhibited the initial "burst" of fluoride release at 24-48 h in accordance to the literature: MG, VF, VD (not ART indicated) and BG, IZ (indicated for ART). The setting reaction for the ART glass ionomers is basically the same typical of GICs[8], and in fact GICs indicated or not for ART present the same pattern of fluoride release. IG and IM showed basically similar behaviors, with highest fluoride release at 6-12h, and a constant release after that. IM presented the most considerable initial burst among all materials, 3.7 times higher in comparison with the register for 1h. A constant fluoride release was observed for VM that reached the highest release at 7 days and decreased at 14 storage days. Interesting results were found for MA, a GIC indicated to ART, which presented no initial "burst", with no significant difference of fluoride release for all times measured, that is, a constant amount of ions released from 1h to 14 days after manipulation.

The water absorption plays a crucial role in fluoride release, however causes a degradation of the filler particles. It leads to loss of strength and erosion of the material, indicating that a material with higher fluoride release tend to be weaker mechanically.[38,39,40] Consequently, results found that newer GICs developed for ART use release substantially less cumulative fluoride ions than any conventional or resin-modified restorative GICs.[26] The accumulation amount of fluoride ions realease along time, in descending order is: MA>IZ>MG>IM>IG>BG>VF>VD>VM. The second null hypothesis was also rejected. In terms of quantitative fluoride ions release, MA showed the highest amount during all times measured, followed by IZ. This high amount of fluoride released is proportional to the fluoride content of the material.[41] Representing materials indicated for ART by manufacturers, the results of MA and IZ were unexpected. Once the fluoride release is related to erosion and material's decrease of strength[38], it can be suggested that MA and IZ may not present the higher strength needed for the ART approach. Although, VM presented exactly the fluoride release expected. According to the manufacturer, VM is a high-viscosity conventional GIC indicated for ART use. The lowest amount of fluoride release registered for VM validates the possible low solubility and higher mechanical properties, ideal for ART restorations. De Moor[22], et al (1996) concluded that different formulations can result in similar results in fluoride releasing. In this study results of BG and VF, different commercial GICs, with different indications and composition showed no significant difference in amount of fluoride release.

Fluoride release is a complex process and depends on intrinsic and extrinsic factors. Formulations, P/L ratio, mixing time, temperature, specimen geometry, solubility or porosity of the material are some intrinsic factors that may affect the fluoride releae. [22,32,42,43] Extrinsic factors such as storage medium, experimental design, analytical methods, temperature, represent those that can be standardized in methodology.[44] Literature has revealed a limitation among a large number of studies: the unviability of comparing results with the present data due to different methods used.[21,21,27,31,34,37] Some authors observed that glass ionomer cements release significantly less fluoride in artificial saliva than they do in deionized water, being the first more appropriate to study the materials fluoride release.[23,45,46] According to Ell Mallakh and Sarker[23] (1990), deionized water does not represent the actual release that occurs in oral environment. Based on that, the present study was made using artificial saliva as the storage media. The method used to measure the results was the ion-selective electrode (ISE), an accurate and established method of determining fluoride release.[11,38] Although it is a more convenient and simple method, it is insufficiently sensitive for measurements at very low concentrations.[25] In accordance with Lee[24], et al (2000), removing and refreshing the medium every time may temporarily change the diffusion gradient and accelerate the fluoride release. Also, very small quantity of solution can cause discrepancy of the results.[42] For these reasons, it was chosen to have a 20 ml of storage media per specimen during the 14 days period of tests, into a falcon recipient at constant temperature of 37º.

Glass ionomer cements present fluoride as part of the material chemistry, instead of being an additive.[14] Analyzing two materials composition, VF and VM by the same manufacturer (Nova DFL, Rio de Janeiro, Brazil) it can be observed that powder and liquid present similar components. Besides, P/L ratio is different. It can be suggested that variable size of glass particles and P/L ratio may influence the fluoride release results. This study confirms the findings of Vermeersch[40], et al (2001), that the GICs acid base reaction and their composition (glass particles, polyacid type, and P/L ratio) should influence the fluoride release more than the material type.[40] The fluoride release discrimination among GICs occurs mainly due to the process I kinetics, during the first days, responsible for widely divergent results among investigators for cumulative amount of fluoride released.[22] In accordance, materials should be indicated by manufacturers to ART approach based on their chemistry and composition, and not just on the material type.

The present study is a preliminary investigation and limited on some distinct parameters. More complex clinical studies could provide more evidences and confirm the data showed. Furthermore, mechanical tests of the same materials are suggested, to make better conclusions about their ART indication or not.

5. Conclusion

Based in this in-vitro study and considering its limitations, it can be concluded that despite that all GICs presented fluoride release ability, the majority of them, regardless of indication, showed similar patterns of fluoride release. Contrary to expected, most of the GICs indicated for ART approach by manufacturers presented a higher quantity of fluoride released in comparison with the GICs not indicated for ART. The results suggest that despite manufacturer's indication, GICs properties should be verified by more specific tests for correct use in ART approach.

6. References

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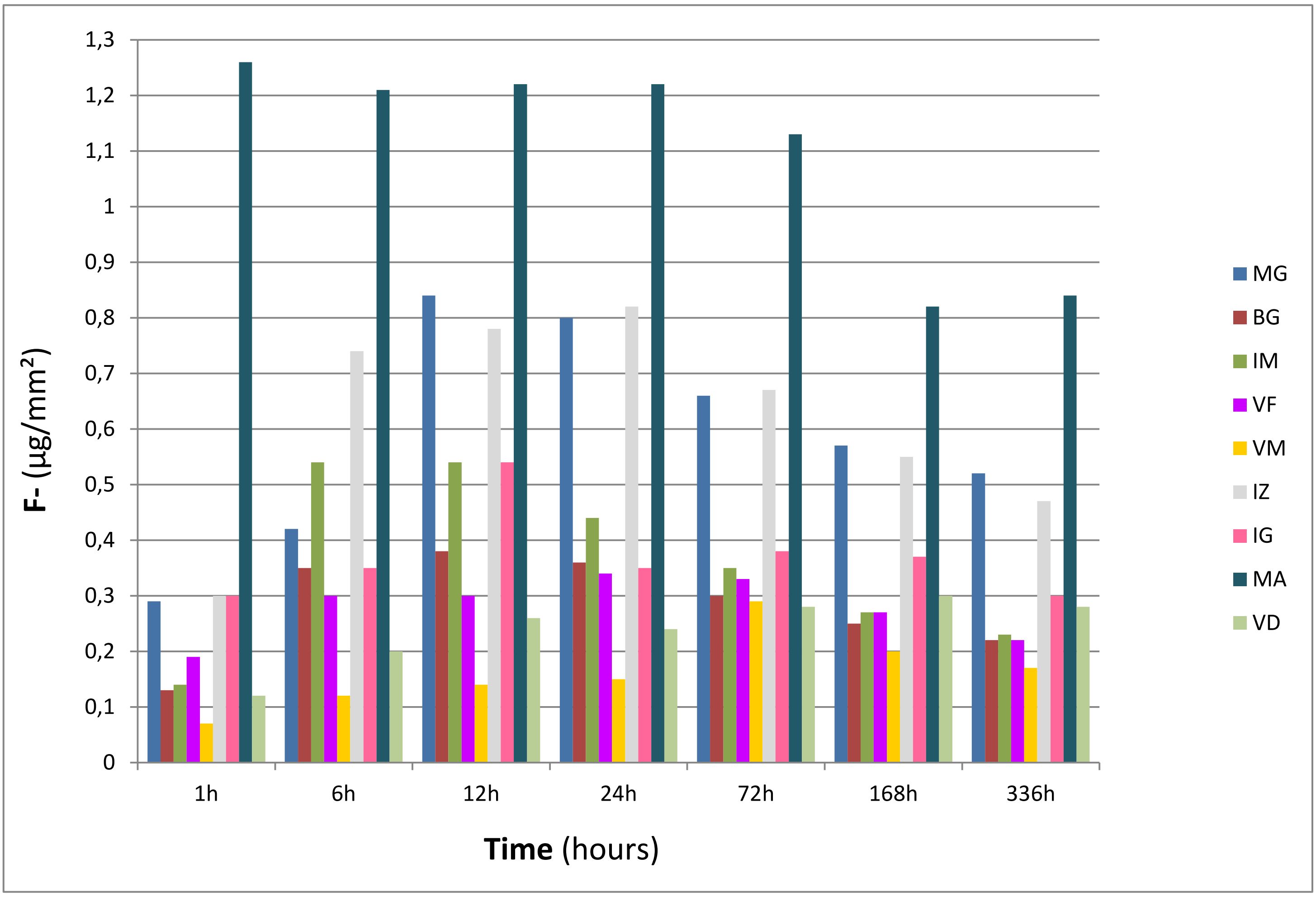
Graphic 1. Amount of fluoride of each glass ionomer cement related to period of time measured



Graphic 2. Cumulative fluoride released (µg/mm²) by all cement brands

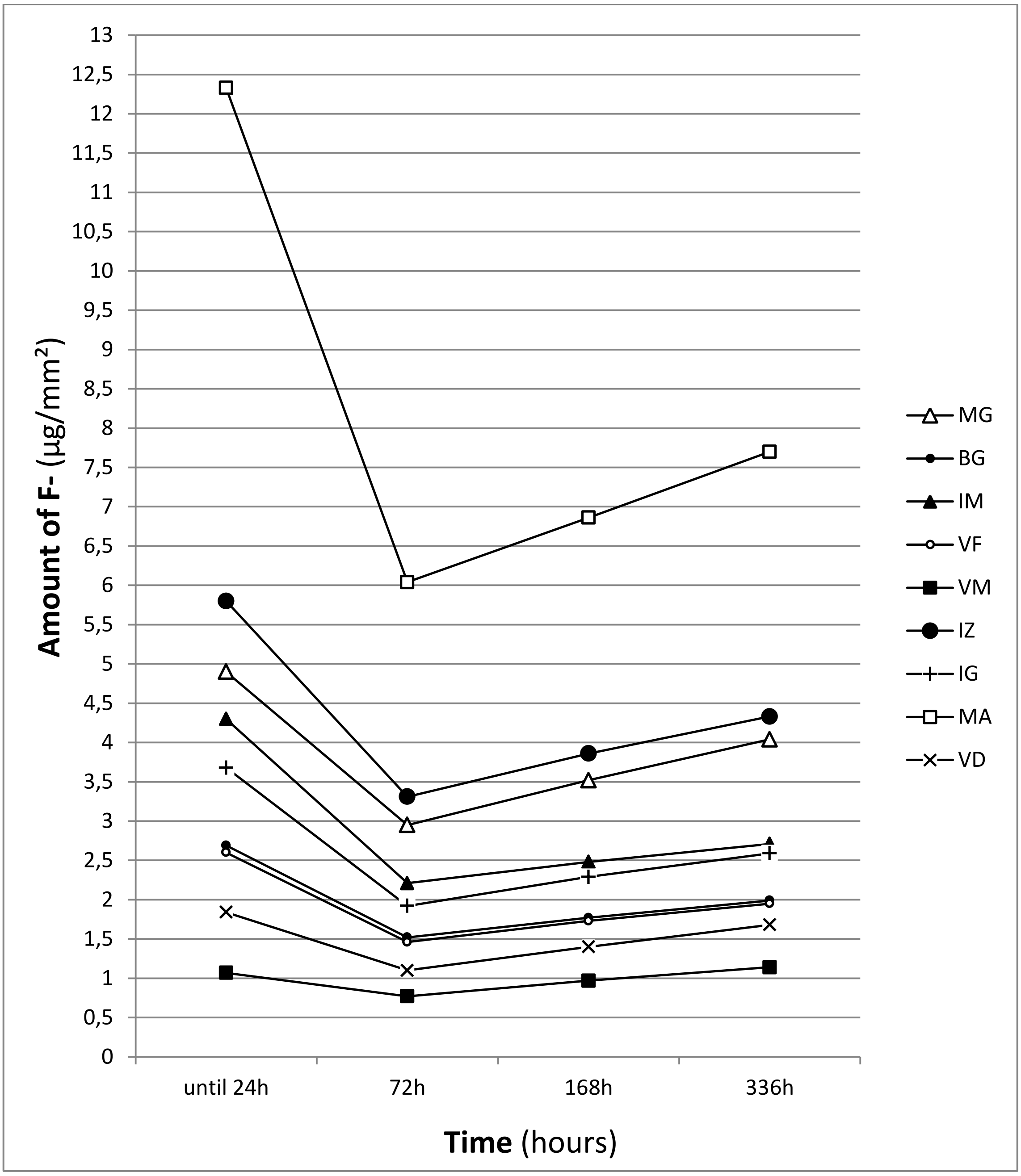


Table 1. Glass Ionomer formulations used in the present study

CAFS-glass: Calcium Alumino Fluoro Silicate glass; PAA: Polyacrylic Acid; TA: Tartaric Acid; MA: Maleic Acid; CAFS-

Material	Manufacturer	Powder	Liquid	P:L	Batch
Bioglass R (BG)	Biodinâmica, Ibiporã, Brazil	Calcium, Barium and Aluminium Fluorsilicate, PAA and inorganic fillers	PAA + TA + water	1.6 : 1	97515
Ion Z (IZ)	FGM, Joinville, Brazil	Micronized glass ionomer, pigment, CAFS-glass	TA + PCA + water	1.7 : 1	130116
Ionglass (IG)	Maquira Dental Products, Maringá, PR	PAA, Sodium fluorosilicate, calcium and aluminium	TA + water	1.5 : 1	029616
Ionomaster (IM)	Wilcos, Petrópolis, Brazil	Stroncium Aluminium Fluorosilicate	PCA + water	3 : 1	15438
Magic Glass (MG)	Vigodent, Rio de Janeiro, Brazil	Fluoroaluminosilicate, PCA and pigment	PCA + TA + MA + water	2.7 : 1	1401244
Maxxion R (MA)	FGM, Joinville, Brazil	Glass of Fluoro Aluminium Silicate, Calcium Fluoride	PAA + water	1.5 : 1	240516
Vidrion R (VD)	SS White, Rio de Janeiro, Brazil	Sodium CSFS-glass, barium sulfate, PAA, pigment	TA + water	5.8 : 1	0180616
Vitro Fil (VF)	Nova DFL, Rio de Janeiro, Brazil	Fluorine Strontium Aluminium Silicate, PAA, Iron Oxide	PAA + TA + water	2 : 1	16050647
Vitro Molar (VM)	Nova DFL, Rio de Janeiro, Brazil	Fluorine Barium Aluminium Silicate, PAA, Iron Oxide	PAA + TA + water	2.9 : 1	16030405

glass: Calcium Alumino Fluoro Silicate glass; PAA: Polyacrylic Acid; TA: Tartaric Acid; MA: Maleic Acid; PCA: Polycarboxylic Acid

Table 2. Results (mean and standard deviation, n=10) of fluoride release of nine conventional GIC for each period of time measured

	1h	6h	12h	24h	3d	7d	14d
Magic	0.29±0.07[a]	0.42±0.03 [b]	0.84±0.07 [e]	0.8±0.02 [e]	0.66±0.12[d]	0.57±0.02 [c,d]	0.52±0.05 [c]
Bioglass	0.13±0.03[a]	0.35±0.02 [e]	0.38±0.02 [e]	0.36±0.02[e]	0.3±0.03 [d]	0.25±0.01 [c]	0.22±0.01 [b]
Ionomaster	0.14±0.03 [a]	0.54±0.07 [e]	0.54±0.06 [e]	0.44±0.02 [d]	0.35±0.04 [c]	0.27±0.04 [b]	0.23±0.04 [b]
Vitro Fil	0.19±0.01 [a]	0.3±0.02 [b,c]	0.3±0.06 [b,c]	0.34±0.03 [c]	0.33±0.02 [c]	0.27±0.01 [b]	0.22±0.01 [a]
Vitro Molar	0.07±0.01 [a]	0.12±0.02 [b]	0.14±0.0 [b,c]	0.15±0.01[c]	0.29±0.02[d,e]	0.2±0.02[e]	0.17±0.02[c,d]
Ion Z	0.3±0.16 [a]	0.74±0.3 [b,c]	0.78±0.34 [c]	0.82±0.21 [c]	0;67±0.13 [b,c]	0.55±0.09 [a,b,c]	0.47±0.13 [a,b]
Ionglass	0.3±0.11 [a]	0.35±0.14 [a]	0.54±0.11 [b]	0.35±0.13 [a]	0;38±0.1 [a]	0.37±0.07 [a]	0.3±0.06 [a]
Maxxion	1.26±0.8 [a]	1.21±0.23 [a]	1.22±0.25 [a]	1.22±0.23 [a]	1.13±0.17 [a]	0.82±0.24 [a]	0.84±0.06 [a]
Vidrion	0.12±0.04 [a]	0.2±0.03 [b]	0.26±0.04 [b,c]	0.24±0.04 [b,c]	0.28±0.04 [c]	0.3±0;07 [c]	0.28±0.05 [c]

Means followed by different letters indicate statistically significant difference at 5%.

3 DISCUSSION

3 DISCUSSION

The first and second null hypotheses of the study were rejected. All materials tested, indicated and not-indicated for ART GICs, released measurable quantities of fluoride ions during 14-days period studied, there were considerable variations in the total amount and pattern of fluoride released. The results were discussed based on the follow division by conventional GICs: the traditional conventional GICs not-indicated for ART (VD, VF, IM, MG) and the conventional GICs recommended for ART by manufacturers (VM, MA, IZ, BG, IG).

Fluoride ions can replace the OH groups of hydroxyapatite crystals of enamel forming fluorapatite and because of its lower solubility and lower crystal energy, leads to a caries resistance characteristic.[2,34] Also, it is more resistant against bacterial acid attacks.[35] Kamathan and Reddy[36] (2013) reported that the fluoride release benefit from GICs was not seen just in adjacent enamel, but in areas up to three millimeters away from restorations margin. This fluoride release characteristic is also associated with the materials bioactivity. A study about the conventional CIGs, showed that a gap next to the materials was covered with crystalline deposits after 18 months storage.[13]

These desirable consequences of fluoride release are related to the release reaction of glass ionomer cements, presented by several authors as a high fluoride concentration at the first 24 hours, known as “initial burst”. [24,28,30] It is explained by the initial reaction between glass particles and the polyalkenoate acid, as a faster superficial erosion and materials dissolution, releasing its components parts.[28,29,31] This initial fluoride “burst” effect is desirable, once it induces the dentin and enamel remineralization by reducing the viability of any bacteria remaining and increases caries resistance.[4,37] Besides that, another study validated that dentin around the material was still protected from demineralization even after fluoride release was almost ended.[38] The next step of the process is more gradual, where the elution is markedly reduced and small amounts of fluoride continue to be released at the storage for long periods of time.[24,28,30] This is attributed to the diffusion of the cement through its pores and fractures. Glass Ionomer restorations may act as intraoral devices for the controlled slow release of fluoride at sites of high risk of caries activity, showing great clinical importance.[30] Once the acid-base reaction of ART glass ionomers is the same of traditional conventional GICs [16] the pattern of fluoride release is expected to be similar. Results found in this study are in accordance with the literature for most GICs with high amount of fluoride release in the first 24 hours after initiated. Instead have the same typical reaction; most ART indicated GICs presented a different pattern when compared to traditional conventional materials. The glass ionomer cements which presented the initial "burst" were MG, VF, VD, IM (not ART indicated) and BG, IZ, IG (ART indicated). IG and IM showed the highest amount of fluoride at 6-12h and a lower amount in sequence, thus with a initial burst also until 24h. The highest burst observed among all materials was of IM with a fluoride release value almost 4 times higher in comparison with the first hour. Uncommon, Ma showed a constant fluoride release, without any statistical difference among all times measured. Another unusual pattern result was VM, which revealed the highest amount of fluoride from 3 to 7 days, suggesting a different reaction process compared to other materials tested in this study or a slower reaction than others.

The amount of fluoride released is expected to be different among conventional GICs indicated or not for ART. There is a good correlation between the quantity of fluoride release and the materials strength. [42] Water absorption causes degradation of particles and consequently, erosion and lower mechanical properties.[40,41] Thus, as reinforced materials, is expected that glass ionomer cements indicated for ART present a lower release of F-.[8,19] Unusually, the highest amount of fluoride release values found in this study were for MA and IZ. As materials indicated for ART, the results of both cements do not correspond to the expected. They were expected be lower than the results found for conventional GICs not indicated for ART, not the opposite. The manufacturer recommendation for use in ART may be not truly based on materials properties. It is suggested that mechanical tests must be performed to obtain more conclusive results.

The other cements released values between 0.24 to 0.44 µg/mm² and the lower value was VM. The results of VM for this study confirm manufacturer's recommendation about the material properties. It is defined by DFL as a condensable, low-abrasive surface and high-viscosity material. Accordingly, as an ART indicated glass ionomer, it is expected that it presents lower amounts of fluoride release in comparison with the traditional conventional GICs. Findings of the present study validate this expectation, as low fluoride release means low solubility, and consequently higher mechanical properties. It is suggested that VM presents the ideal properties expected for ART restorations. The decreasing sequence for cumulative amount of fluoride elution was: MA>IZ>MG>IM>IG>BG>VF>VD>VM. The different amount of fluoride release verified in this study is probably related to the different GICs composition, particle size and porosity.[35]

Literature presents a great amount of studies on fluoride release with varied fluoride-releasing materials. Unfortunately, a huge limitation about the comparison of so many results found is the difference in study design, as some of them have already observed.[3,12,30,31,35] Different methodologies and specimen sizes, storage media, frequency of change of storage media, quantity of media used to measure fluoride level are a few factors for different results obtained.[30,35,42] For this reason, the present study was made with a standardized methodology for nine Brazilian conventional glass ionomers selected. It makes possible the comparison among different materials with safe results not only of cumulative fluoride release pattern overtime, but also the amount of fluoride release of each material.[12] Once other factors are stabilized, it is easier to understand the process involved in fluoride release.

Studies showed that glass ionomers release significantly less fluoride in artificial saliva than they do in deionized water, indicating fluoride release is induced by artificial saliva's ionic strength and composition.[32,37] Ell Mallakh and Sarker[32] (1990) concluded in their study that probably fluoride release from GICs in deionized water does not represent its actual release within the oral environment. Based on that, this study used artificial saliva as a storage media. Lee[43], et al (2000) defended in their research that removing and refreshing the medium every time may temporarily change the diffusion gradient and accelerate the fluoride release, consequently incorrect results. As known, the technique of fluoride ion-selective electrodes (ISE) is often used to measure fluoride release.[2] It is a convenient and simple method, but studies concluded that ISE is insufficiently sensitive for responsible measurement at very low concentrations.[44] The frequent solution change could lead to the register of incorrect values, due to the equipment's low sensitivity to low fluoride concentrations and to the change of the solution diffusion gradient.

Different formulations are mainly due to variable fluoride parameters and differences in the reactions kinetics, resulting in diverse quantitative and qualitative fluoride release profiles.[27] De Moor[27], et al (1996) demonstrated that differences in the amount of fluoride released among glass ionomers are due to the occurrence of two release processes with different kinetics. Two similar formulations, VM and VF, by same manufacturer, showed different results. Although the composition of both is almost the same, the powder:liquid ratio is different, and as a high-viscosity GIC, the sizes of glass particles are smaller.[17,18] According to Prentice[46], et al (2005), the lower particle size in GICs is set by viscosity of the cement and the significant glass content. Results found in this study confirm that acid-base reaction of GIC and composition (glass particles, polyacid type and P/L ratio) influence the fluoride release more than the material type.[41] Accordingly, manufacturers should be based on materials chemistry and formulation to correctly determine the indicated or not-indicated for ART.

This in vitro study has limitations on some distinct parameters. For this reason, studies involving mechanical results should be done to make better conclusions about the materials tested in this study. Controlled clinical trials in real clinical situations would also lead to more valid conclusions about Brazilian available GICs indication for ART or not.

4 FINAL CONSIDERATIONS

4. FINAL CONSIDERATIONS

Based on this in vitro study and its limitations, the conclusion was that most of conventional Brazilian restorative GICs presented same pattern of fluoride release. Contrary to expected, the majority of GICs indicated for ART showed higher amount of fluoride release compared to GICs not indicated for ART.

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