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Positive correlation between fluoride release and acid erosion of restorative glass-ionomer cements

Lígia S. Bueno[a], *Rafael M. Silva*[a], *Ana Paula R. Magalhães*[a], *Maria Fidela L. Navarro*[a], *Renata C. Pascotto*[b], *Marília A.R. Buzalaf*[a], *John W. Nicholson*[c], *Sharanbir K. Sidhu*[d], *Ana Flávia S. Borges*[a,*]

[a] *Bauru School of Dentistry, University of São Paulo, Bauru, São Paulo, Brazil*
[b] *State University of Maringá, Maringá, Paraná, Brazil*
[c] *Queen Mary University of London, Barts & The London School of Medicine and Dentistry, Institute of Dentistry,Turner Street, London and Bluefield Centre for Biomaterials, 67-68 Hatton Garden, London, UK*
[d] *Queen Mary University of London, Barts & The London School of Medicine and Dentistry, Institute of Dentistry,Turner Street, London, UK*

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ABSTRACT

Objective. The aim of this study was to determine whether there is a correlation between acid erosion and fluoride release of conventional glass ionomer cements.
Methods. Ten specimens for each material were prepared for fluoride release tests and five for acid erosion tests separately. After placed in pH cycling solution, concentration of fluoride was measured by a fluoride-ion selective electrode each day for 15 days. For the acid erosion test, specimens were immersed in a lactic acid solution and their depth measured with a spring-loaded dial gauge. The data were submitted to 3-way ANOVA, followed by Tukey's test ($p < 0.05$)
Results. All materials showed ability to elute fluoride in the 15 day period of the test, with the same pattern of high fluoride release at the first 24 h. Despite this, the amount of fluoride released was statistically different among the 18 groups, with the highest for Maxxion R and the lowest for Chemfil Rock ($p > 0.05$). The highest acid erosion values were registered for Magic Glass, Ion Z, VitroFil and Maxxion R, which exceeded the maximum stipulated by the relevant ISO test (ISO 9917-1). A positive linear correlation ($r^2 = 0.4886$) was found for both properties, i.e., higher fluoride release is related to higher acid erosion.
Significance. Acid erosion and fluoride release are related properties of GICs, though factors such as pH and P/L ratio lead to differences between actual values for individual brands of these materials.

* Corresponding author at: Alameda Dr. Otávio Pinheiro Brisola, 9-75 Vila Nova Cidade Universitária, Bauru, São Paulo, 17012-901, Brazil.
E-mail addresses: renatapascotto@gmail.com (R.C. Pascotto), john.nicholson@bluefieldcentre.co.uk (J.W. Nicholson), s.k.sidhu@qmul.ac.uk (S.K. Sidhu), afborges@fob.usp.br (A.F.S. Borges).
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1. Introduction

Dental caries is a dynamic process [1] and recently better understanding of the disease has led to minimal invasive management instead the traditional operative approach [2]. As a suitable material and the first to represent bioactive restorative materials, glass ionomer cements are the materials of choice when caries activity is high [3].

Bioactive materials can be defined as ‘materials that elicit a specific biological response at the interface between tissues and the material’ [4]. Glass ionomer cements chemically bond to tooth structure [5] and their fluoride release and recharge abilities are important for their clinical application. Although the preventive effect of GICs on caries progression is widely discussed in the literature, it is not well understood [6]. However, a recent in vitro study suggests the anti-cariogenic biofilm activity of GICs is associated with their fluoride release rate [7]. In vivo studies show that fluoride is taken up by saliva and the surrounding enamel and that fluoride release can continue for at least 1 year [8]. Besides that, fluoride is known to destabilize the glass network, which facilitates bulk partial loss of fluoride directly from the glass [9].

Clinical limitations of glass ionomer cements are mainly associated with poor mechanical properties and low wear resistance. Some studies have correlated high fluoride release with poor mechanical properties [10,11] but more recent glass ionomer formulations show improved mechanical properties and longevity compared to dental amalgam in Atraumatic Restorative Treatment (ART) as well as sustained fluoride release [12]. Because of the need to consider the place of glass ionomer cements in future restorative dentistry, particularly from the perspective of developments in minimally invasive dentistry [13], commercial restorative conventional glass ionomer cements need to be studied. In particular, it is important to understand the relationship between their acid erosion and fluoride release, both of which are essential factors for durable long term restoration.

The aim of this study was to determine fluoride release and acid erosion of selected conventional restorative glass ionomer cements with standardized methodology and to determine whether these properties are correlated. The null hypotheses were: (1) there is no difference among the 18 glass ionomer cements studied in the pattern (quality) and amount (quantity) of fluoride released; (2) there is no difference among these glass ionomer cements for acid erosion rate; and (3) there is no correlation between fluoride release and acid erosion rate.

2. Materials and methods

The brands and composition of restorative conventional glass ionomers tested are described in Table 1.

2.1. Fluoride release test

Materials handling was carried out at controlled temperature (23° ± 1°) and relative humidity (50% ± 5%). The same operator prepared all specimens. The powder-to-liquid (P/L) ratio used and materials were manipulated according to the manufacturers’ instructions. Powders and liquids were weighed accurately using an analytical balance, and the resulting mixture was dispensed using a Centrix syringe (Centrix, Shelton, CT, USA). For each restorative material, ten disk-shaped specimens (sps) 11.0 mm in diameter and 1.5 mm thick were made using cylindrical Teflon molds. Immediately after mixing, the cement was inserted into the mold and covered by a polyester strip at top and bottom surfaces. Also, a nylon thread piece was introduced to the specimen’s middle to suspend it at the storage media. Glass plates were used as a base below and above, tightened and pressed to produce a smooth surface. After the cement cured initially for 10 min inside the molds, the specimens were removed and excess material was cleaned carefully using a scalpel blade. Then, each specimen was individually immersed in Falcon plastic containers with 4 mL of solution, hermetically closed and stored at 37 °C for 15 days. During this period, the specimens were periodically transferred to new experimental solutions. The solutions represented a pH-cycling system, where the specimens were immersed for 6 h in a demineralizing solution (calcium 2.0 mmol L^{-1}, phosphate 2.0 mmol L^{-1} and acetate buffer 75 mmol L^{-1} pH 4.3) and for 18 h, in a remineralizing solution (calcium 1.5 mmol L^{-1}, phosphate 0.9 mmol L^{-1}, potassium chloride 150 mmol L^{-1} and Tris buffer 20 mmol L^{-1} pH 7.0). This system was designed to simulate a situation of high caries challenge, with rapid changes in pH. Fluoride ion concentrations were determined in duplicate 1 mL volumes of solution removed from the individual sample containers with the use of total ionic strength adjustment buffer II (TISAB II). Fluoride ion concentration was measured using an Orion fluoride-specific electrode (model 96-09) and a Procyon digital ion analyzer (model SA-720) that had been previously calibrated. The mV readings were transformed into $\mu gF/mm^2$ by the formula (amount released*total quantity of solution)/specimen area.

2.2. Acid erosion test

For all materials, five specimens were made with a mold (30 mm × 30 mm × 5 mm) with a hole of 5 mm diameter and 2 mm deep. The whole assembly was placed in a cabinet maintained at (37 °C ± 2 °C) and a relative humidity of 90%. After 24 h, all specimens were smoothed with abrasive papers and continuous water irrigation until flat. Specimens with flaws or air inclusions were rejected. Five points of the specimen mold were measured with a spring-loaded dial gauge (Digimess, São Paulo, SP, Brazil) to calculate the cements’ initial depth. It was spaced at 90° intervals of 0.5 mm–1.0 mm from the specimen. Each specimen was immersed horizontally in an individual container with 30 ml of the eroding solution. The eroding solution was made with lactic acid and lactate, both previously calculated and dissolved in water. The solution’s pH was 2.74. Afterward, it was stored for about 24 h in the cabinet at (37 °C ± 1 °C). After a 24 h of immersion, the specimens were removed and rinsed with water, as defined in ISO 3696:1987. Measurements and calculations were made in five points, as described before the erosion challenge.

Table 1 – Brands and composition of glass ionomer cements tested.

Brand name of GICs	Manufacturer	Groups' names	Powder	Liquid	Powder/ liquid ratio	Batch number
Bioglass R	Biodinâmica, Ibiporã, Brazil	BG	Calcium, barium and aluminum fluoride + PAA + inorganic fillers	PAA + TA + water	1.6:1	97515
ChemFil Rock	Dentsply Caulk, Milford, USA	CR	Calcium-aluminum-zinc-fluoro-phosphor-silicate glass + PA + pigments + TA + water		capsule	1511000720
Equia Forte	GC Corporation, Tokyo, Japan	EF	Strontium fluoro-alumino-silicate + PAA + water		capsule	1611221
Fuji II	GC Corporation, Tokyo, Japan	FII	Fluoroaluminumsilicate + PAA	PAA + water	2.7:1	1601121
Fuji IX	GC Corporation, Tokyo, Japan	FIX	Fluoroaluminumsilicate + PAA	PAA + other ingredients	3.6:1	1506011
GlasIonomer Type II	Shofu Inc. Kyoto, Japan	GI	Alumino fluoro silicate glass	Copolymer of acrylicacid and tricarboxylic acid + TA	2.5:1	051501
Ion Z	FGM, Joinville, Brazil	IZ	Pigment + calcium aluminum zinc fluoride silicate glass + Micronized glass powder	PCA + TA	1.7:1	130116
Ionglass	Maquira Dental Products, Maringá, Brazil	IG	PAA + sodium fluorosilicate, calcium, aluminum	TA + water	1.5:1	075216
IonoFil Plus	Voco GmbH, Cuxhaven, Germany	IP	Strontium aluminium fluorosilicate glass	PAA + tartaric acid + water	4.7-5.6:1	1514030
Ionomaster	Wilcos, Petrópolis, Brazil	IM	Strontium Fluoroaluminosilicate + pigments	PAA + water + pigments	3:1	15438
IonoStar Molar	Voco GmbH, Cuxhaven, Germany	IS	Strontium aluminum fluorosilicate + PAA + TA + water		capsule	1618227
Ketac Molar Easymix	3M ESPE, Seefeld, Germany	KM	Strontium Fluorosilicate + Aluminum + lanthanum + pigments	PCA + TA + water	4.5:1	628875
Magic Glass	Vigodent, Rio de Janeiro, Brazil	MG	Fluoroaluminosilicate + PAA + pigments	PAA + TA + MA + water	2.7:1	1503044
Maxxion R	FGM, Joinville, Brazil	MA	Fluoroaluminum silicate + Calcium fluoride	PCA + TA + water	1.5:1	240516
Riva Self Cure (SC)	SDI, Victoria, Australia	RV	Fluorine Silicate Aluminum + PAA	PAA + TA + water	3.03:1	73072V
Vidrion R	SS White, Rio de Janeiro, Brazil	VD	Sodium fluorosilicate calcium and aluminum + barium sulphate + PAA + pigments	TA + water	5.8:1	0210716
Vitro Fil	Nova DFL, Rio de Janeiro, Brazil	VF	Fluorine Strontium Aluminum Silicate + ‘PAA + Iron Oxide	PAA + TA + water	2:1	16050647
Vitro Molar	Nova DFL, Rio de Janeiro, Brazil	VM	Fluorine Barium Aluminum Silicate + PAA + Iron Oxide	PAA + TA + water	2.9:1	16030405

TA: tartaric acid/ PAA: polyacrilic acid/ MA: maleic acid/ PCA: polycarboxylic acid.

Table 2 – Mean (μg/mm²) and standard deviation (SD) of fluoride release for restorative glass ionomer cements.

Material	1. Mean	Day SD	7. Mean	Day SD	15. Mean	Day SD
Chemfil	$0,28^{a}$	0,023	$0,06^{a}$	0,008	$0,05^{a}$	0,011
Ketac M	$0,4^{a,b}$	0,067	$0,04^{a}$	0,023	$0,02^{a}$	0,017
Fuji II	$0,49^{a,b}$	0,043	$0,05^{a}$	0,073	$0,02^{a}$	0,002
Riva	$0,55^{b}$	0,039	$0,049^{a}$	0,006	$0,03^{a}$	0,004
Vidrion	$0,85^{c}$	0,085	$0,07^{a,b}$	0,015	$0,04^{a}$	0,014
Fuji IX	$0,86^{c}$	0,1	$0,05^{a}$	0,01	$0,03^{a}$	0,006
Ionostar	$1,07^{c,d}$	0,151	$0,21^{c,d,e}$	0,051	$0,1^{a,b}$	0,03
Vitro Molar	$1,11^{d,e}$	0,097	$0,18^{b,c,d}$	0,038	$0,18^{b}$	0,027
Bioglass	$1,14^{d,e,f}$	0,13	$0,44^{g}$	0,12	$0,71^{f}$	0,152
Ionofil	$1,32^{e,f,g}$	0,08	$0,23^{e,f}$	0,036	$0,17^{b}$	0,024
Maxxion R	$1,36^{f,g,h}$	0,12	$1,01^{i}$	0,1	$0,94^{g}$	0,1
Equia	$1,38^{g,h}$	0,189	$0,11^{a,b,c}$	0,031	$0,05^{a}$	0,011
Ionomaster	$1,42^{g,h}$	0,217	$0,44^{g}$	0,021	$0,29^{c}$	0,035
Magic	$1,55^{h,i}$	0,153	$0,72^{h}$	0,1	$0,81^{f}$	0,077
GlassIonomer TII	$1,55^{h,i}$	0,186	$0,23^{d,e}$	0,125	$0,1^{a,b}$	0,058
Ion Z	$1,7^{i,j}$	0,148	$0,37^{f,g}$	0,048	$0,57^{e}$	0,112
Ionglass	$1,92^{j}$	0,25	$0,23^{d,e}$	0,033	$0,41^{d}$	0,044
VitroFil	$2,19^{l}$	0,23	$0,76^{h}$	0,132	$0,79^{f}$	0,1

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

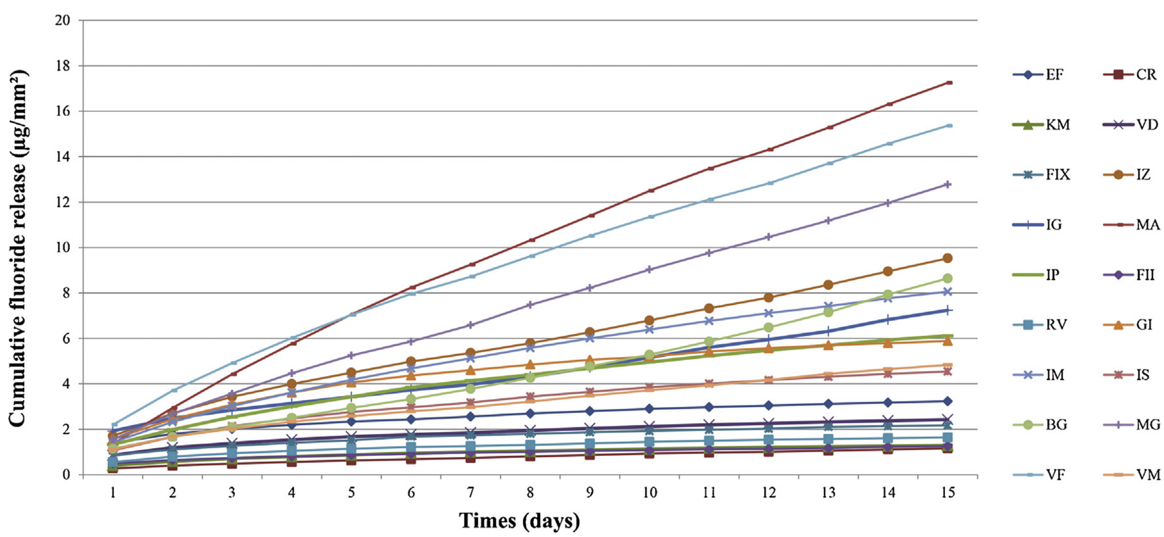

Fig. 1 – Cumulative fluoride release (μg/mm²) of 18 commercial glass ionomer cements in 15 days.

2.3. *Statistical analysis*

The data were statistically analyzed with SPSS for Windows v.19.0 (IBM Statistics, Chicago, USA). The normal distribution and equality of variances assumptions were checked for all variables using the Shapiro–Wilk test and the Levene test, respectively. As the assumptions were satisfied, data were subjected to 3-way ANOVA ($p \leq 0.05$), followed by Tukey's test ($p < 0.05$), for individual comparisons.

3. Results

All conventional restorative glass ionomer cements showed capacity of releasing fluoride during the 15 day storage period. The amount of fluoride released varied considerably between materials, as shown in Table 2 and Fig. 1. However, the pattern of fluoride release was the same for all glass ionomer cements, with a high initial amount in the first 24 h and a lower sustained amount for the remaining 15 days. The total amount of F^- ions released for the 15 days is, in descending order: Maxxion R> Vitrofil> Magic Glass > Ion Z > Bioglass > Ionomaster > Ionglass > Ionofil Plus > Glass Ionomer Type II > Vitromolar > Ionostar > Equia Forte > Vidrion> Fuji IX > Riva > Ketac Molar > Fuji II >Chemfil Rock. Fig. 2 shows the cumulative quantity of fluoride ion released from each material, according to the pH of the used solutions.

The highest acid erosion values were registered for Magic Glass, Ion Z, Vitrofil and Maxxion R with no statistical difference between them. The brands Ionomaster, Ketac Molar, Vidrion, Ionofil Plus, Fuji IX, Chemfil Rock, Glass Ionomer Type II, Riva, Fuji II ($p < 0.05$) showed the lowest acid erosion with no significant differences between them ($p > 0.05$). The results for acid erosion are presented in Table 3 and Fig. 3. The other groups, Vitromolar, Equia Forte, Ionostar and Ionglass showed

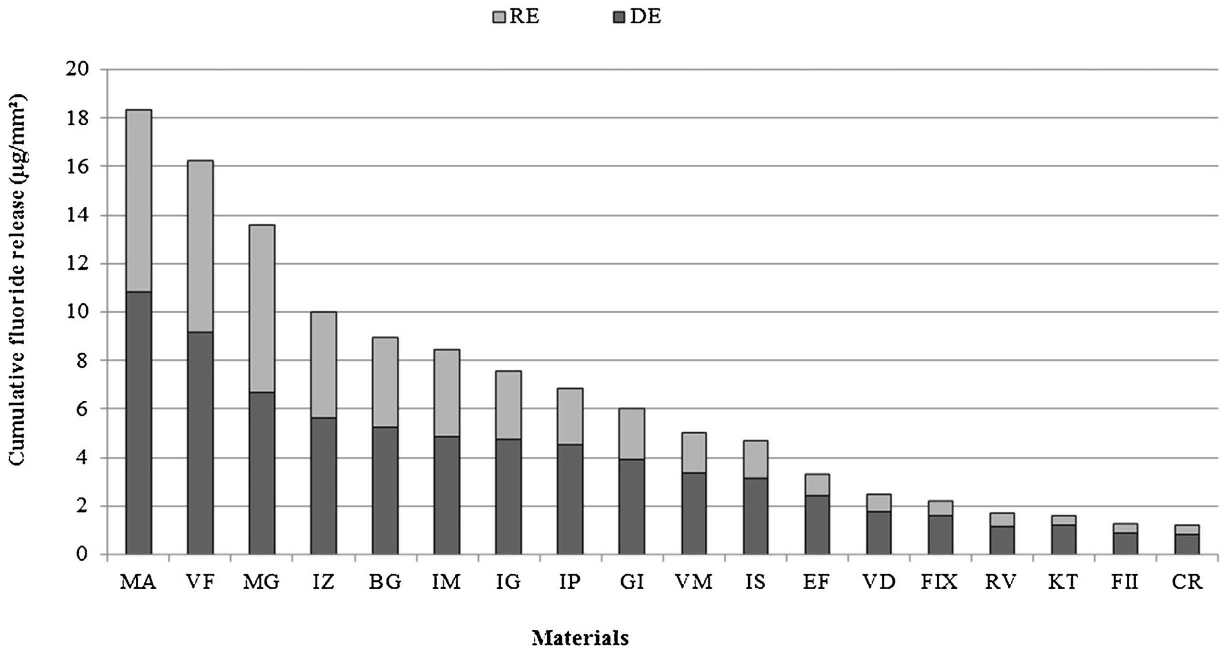

Fig. 2 – Cumulative fluoride release (µg/mm^2) of restorative glass ionomer cements considering pH cycling.

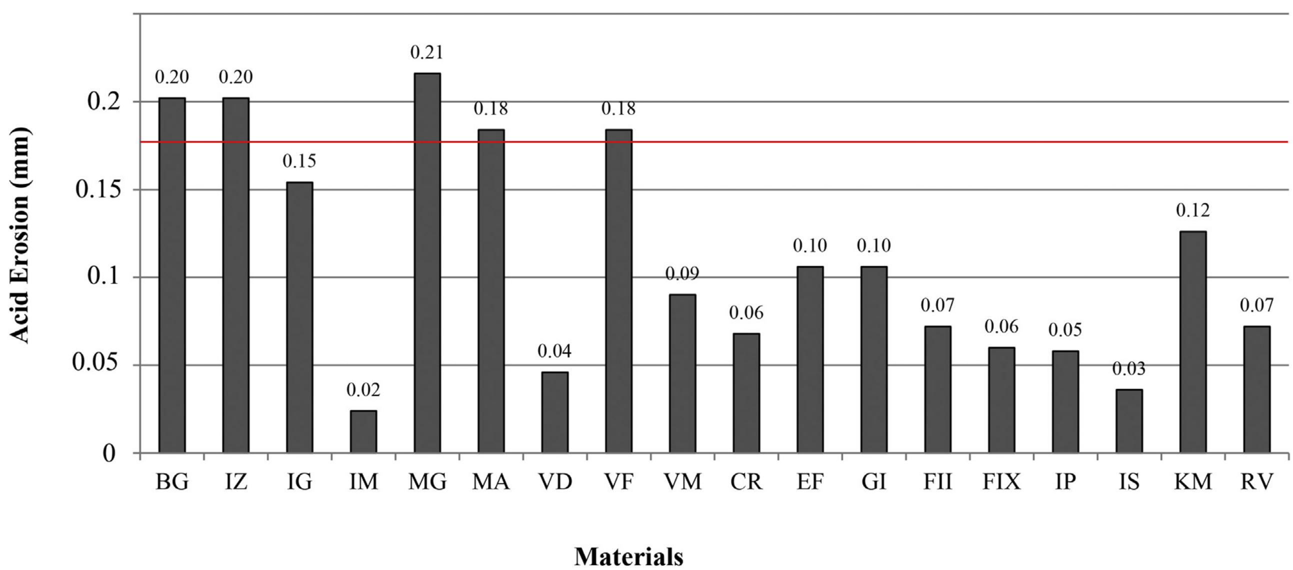

Fig. 3 – Acid erosion (mm) of restorative glass ionomer cements. The red line means the maximum limit stipulated by ISO 9917-1. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

intermediate values. Surface images after the acid erosion test are presented in Fig. 4.

A linear correlation was found to exist between fluoride release and acid erosion ($r^2 = 0.4886$) as seen in Fig. 5. In Figs. 6 and 7, the influence of high or low P/L ratio on the fluoride release of the materials is strongly seen.

4. Discussion

This is the first study to determine the relationship between acid erosion and fluoride release for restorative conventional glass ionomer cements. Results show great scatter in both properties by the eighteen commercial glass ionomer cements, which allows comparison among materials. One previous study used fifteen materials in a test of fluoride release, but materials were of different types, and only three of them were conventional glass ionomer cements. [10]

Unfortunately, for most of materials tested in this study, there is no data in the literature about their fluoride release or their acid erosion. Anyway, it can be difficult to compare fluoride release results in the literature because of the different methodologies used to determine fluoride release [14,15].

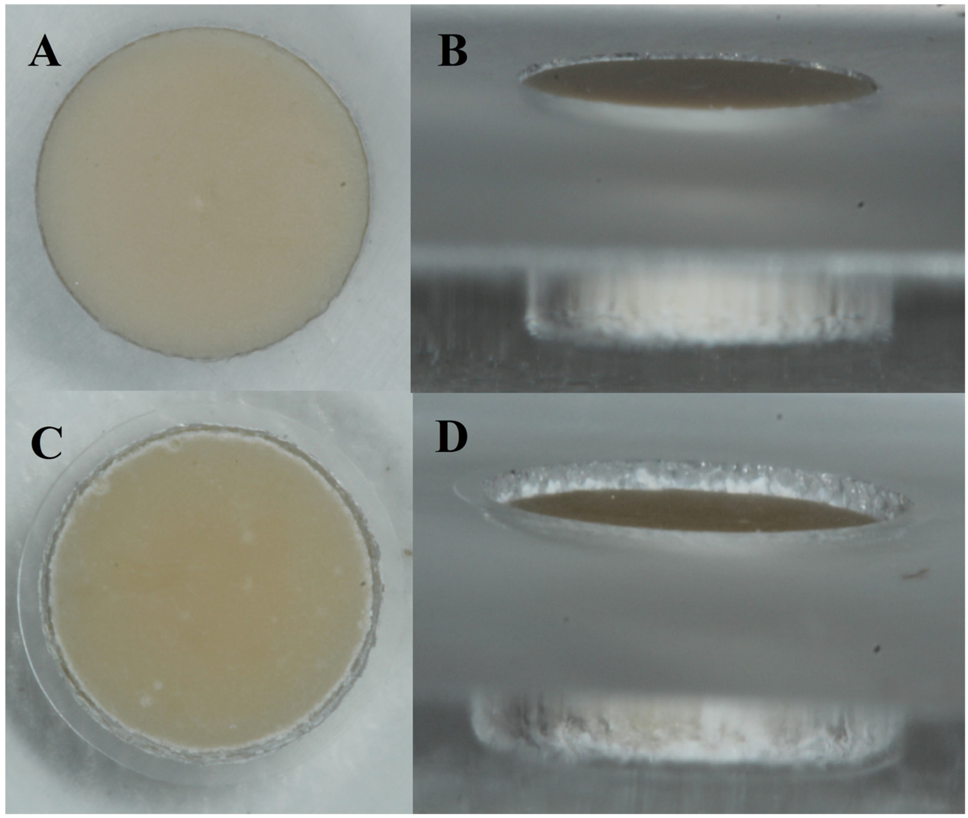

Fig. 4 – Surface images of front and side views of two materials tested with different values after acid erosion test. (A,B) Ketac Molar. (C,D) Ionglass.

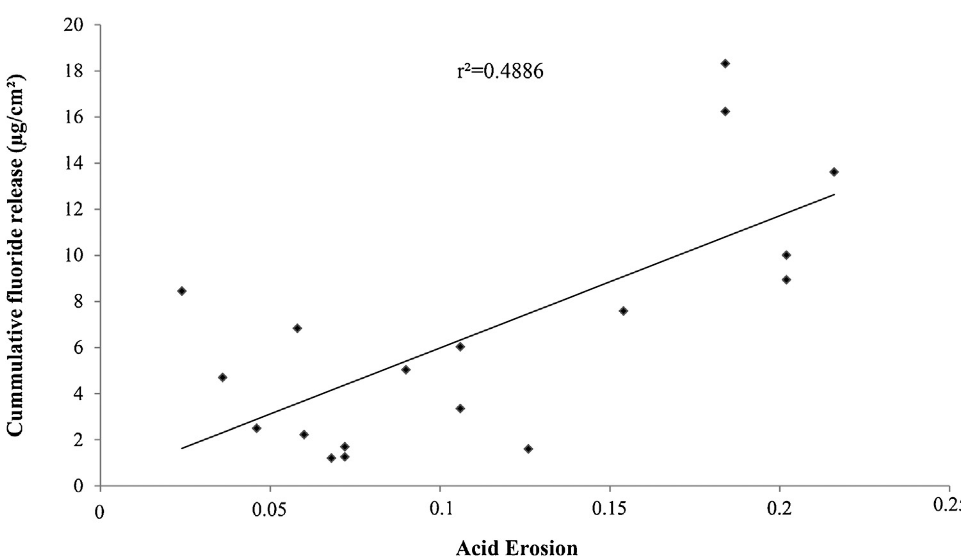

Fig. 5 – Correlation between acid erosion and fluoride release.

The first null hypothesis of the present study was partially accepted, as the findings confirm those of several previous studies in terms of the pattern of fluoride release [14,16,17]. However acceptance was only partial, as the amount of fluoride released by each of the 18 glass ionomer cements were significantly different. The pattern is of a two-part release profile, with an "initial burst" lasting around 24 h (process I), and a longer term sustained release of smaller amounts of fluoride which continued up to the end of the 15 day period of the study (process II) [6].

The materials Glass Ionomer Type II and Equia Forte had different fluoride-release behavior from the other materials studied. At the 24 h period, they showed the fourth and seventh highest values, respectively, but by 15 days, they were

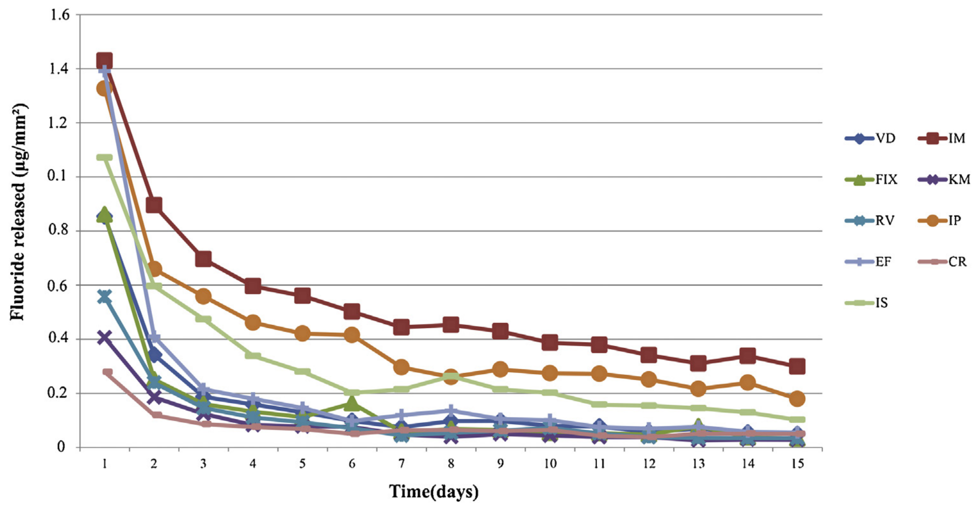

Fig. 6 – Medium or high P/L ratio glass ionomer cements fluoride released (µg/mm^2).

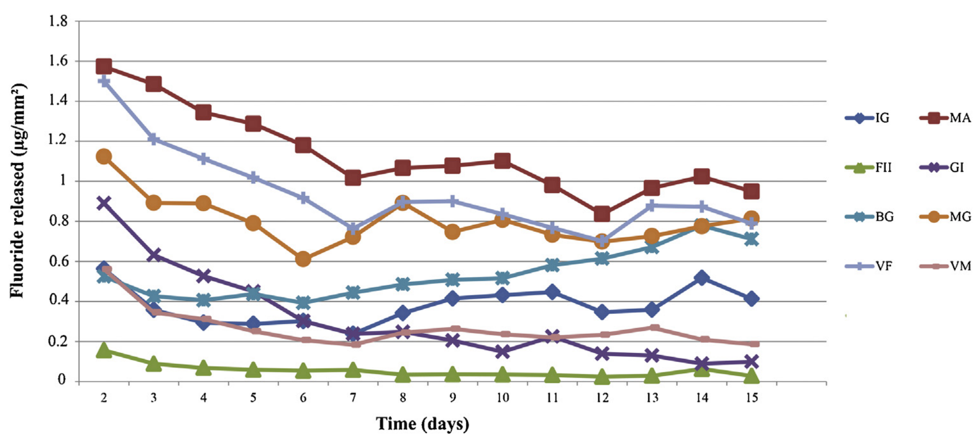

Fig. 7 – Low P/L ratio glass ionomer cements fluoride released (µg/mm^2).

Table 3 – Mean and standard deviation of acid erosion for restorative glass ionomer cements.

Material	Acid erosion (mm)
Ionomaster	0.0250 (0.0153)^{a}
Ketac Molar	0.0350 (0.0101)^{a,b}
Vidrion R	0.0455 (0.0261)^{a,b,c}
Ionofil Plus	0.0570 (0.0231)^{a,b,c}
GC Gold Label 9	0.0580 (0.0148)^{a,b,c}
Chemfil Rock	0.0665 (0.0205)^{a,b,c,d}
Glass Ionomer Type II	0.0675 (0.0098)^{a,b,c,d}
Riva	0.0705 (0.0194)^{a,b,c,d}
GC Gold Label 2	0.0720 (0.0131)^{a,b,c,d}
Vitro Molar	0.0870 (0.0196)^{b,c,d}
Equia Forte	0.1035 (0.0385)^{c,d,e}
Ionostar	0.1255 (0.0249)^{d,e,f}
Ionglass	0.1510 (0.0706)^{e,f,g}
Maxxion	0.1810 (0.0154)^{f,g,h}
Vitro Fil	0.1825 (0.0280)^{f,g,h}
Bioglass	0.2000 (0.0088)^{g,h}
Ion Z	0.2015 (0.0211)^{g,h}
Magic Glass	0.2150 (0.0263)^{h}

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

in the group with the lowest values, and showed no statistical difference from other materials that had released only low amounts of fluoride at each time period ($p < 0.05$). These results suggest that the two release processes are not related to each other, and that the occurrence of large amounts of loosely bound fluoride that is easily released in process I does not imply that there are also large amounts of fluoride available for release in process II. The longer term release (process II) occurs by diffusion of fluoride through a developing matrix which is undergoing gradually increasing crosslink. The amount of fluoride released in this second process depends on the nature of the matrix formed. The fact that these materials showed drastically decreased fluoride release after the first 24 h confirms that fluoride diffusion is impeded as the matrix forms [18].

Although many studies have been made of fluoride release, most are empirical and not concerned with the mechanism of release [19]. Studies on older materials suggested that total amount of fluoride release is less than 1% of fluoride content available in the cement [20,21]. Thus, fluoride release is not simply a function of the fluoride content. The composition of the cement and the kinetics of its setting reaction are also factors in determining the amount of fluoride released [22]. Even with high amounts of total fluoride in the cement, fluoride release may not be high, because fluoride may become trapped within the matrix and able to diffuse out only slowly. The ease of diffusion may vary depending on material composition and extent of reaction, and this will determine the amount of ions released [9].

Results in the present study showed that pH influenced fluoride release from glass ionomer cements, confirming previous literature reports [11]. The results showed that there was higher fluoride release with lower pH for all glass ionomer cements tested (pH cycling with remineralization/demineralization), as seen in Fig. 2. This is a general observation for all ions, since release of ions such as sodium, phosphate and silicate have also been found to increase at lower pH values [23]. Lower pH solutions contain higher amounts of H^+ ions [24] and these attack the cement matrix, causing release of ions and consequently dissolution [25,26]. This is clinically significant as plaque pH after a sucrose rinse can decrease to a pH of 4–4.5 [11], which is a level at which active caries occurs. The fact that such pH values also promote high fluoride release may be beneficial in preventing damage due to caries, given fluoride's well-established role in promoting remineralization of teeth.

The second null hypothesis was rejected, since acid erosion rates among the eighteen brands were significantly different. The test was performed with lactic acid, as specified in ISO 9917-1 for water-based cements and, using this acid, five commercial brands showed acid erosion values above the maximum of 0.17 mm stipulated by ISO 9917-1 (Fig. 3). This raises concerns about the suitability of these brands for use in clinical practice.

The factor powder/liquid (P/L) ratio interfered drastically in erosion and fluoride release of glass ionomer cements results in this study, as reported by other studies [18,27]. All materials with P/L equal or below 2:1 considered low viscosity (used usually for luting cements), Bioglass, Ion Z, Ionglass, Maxxion R and Vitrofil, are 5 among 6 of the highest values of acid erosion. Also, the same materials presented great amount of fluoride release compared to the others. None of those were encapsulated. Accordingly, amount of elution may be inversely proportional to the P/L ratio [28]. As the P/L ratio is reduced, the amount of water inside the cement matrix is higher [27], since the liquid used is an aqueous solution of acid. This type of formulation is associated with mechanically weak cements [27]. This is evidence that the matrix is different from that formed from higher P/L ratio formulations, and is more susceptible to acid erosion as well as more permeable to diffusing fluoride ions. The plot shown in Fig. 4 demonstrates the relationship between fluoride release and acid erosion, with higher fluoride release being positively correlated with high levels of acid erosion and the controlling factor being the P/L ratio.

As a result of this finding, the third null hypothesis was also rejected. The correlation was demonstrated between the mean of fluoride-release and acid erosion methods, submitted to different challenges. In the fluoride-release test, the specimens were kept for 6 h in a demineralizing solution (pH 4.3) and in the acid erosion challenge, specimens were immersed in a solution with lower pH (2.74) for 24 h, both in the first 24 h. Despite the differences between the methods, a positive linear correlation was found between values obtained ($r^2 = 0.4886$). The five glass ionomer cements in this study with the highest acid erosion also had the highest cumulative values for fluoride release after the 15 days of test. The materials are Bioglass, Magic Glass, Ion Z, Maxxion R and Vitrofil. It may be expected that erosion and ions release are related, as obviously greater amounts of dissolution result in higher amounts of ion release [10].

The reason for the association of the characteristics of fluoride release and acid erosion is not clear. Previous studies of the kinetics of fluoride release show that there are significant differences in release under acidic and neutral conditions [22], with the release being described by different kinetic equations in each case. Despite this, our results clearly show that fluoride release and acid erosion are positively correlated, so it follows that there is an association between the two phenomena. High fluoride release must occur because of relatively large amounts of fluoride being transferred from the glass into the matrix during the setting reaction. From the matrix, it can then be eluted by a combination of early wash-out and sustained diffusion-controlled release. The high-fluoride matrix itself may be more susceptible to acid attack than a low-fluoride matrix. If this were the case, it would explain why these two processes are associated. Further work is needed to determine whether these phenomena are linked in the way suggested.

5. Conclusion

The fluoride release profile was the same for all restorative commercial brands tested, with a high early washout in the first 24 h followed by a lower release rate up to 15 days. However, amounts of fluoride released were significatively different. Acidic conditions were shown to increase fluoride release in all cases. P/L ratio was found to influence both fluoride release and acid erosion in all materials tested. Acid erosion and fluoride release have been found to be positively correlated, showing that they are related properties.

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