



Fluoride release of two glass-ionomer cements: *in vitro* study

Liberación de fluoruro de dos cementos de ionómero de vidrio: estudio in vitro

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ABSTRACT

Objective: The aim of the present study was to compare the amount of fluoride released by two glass-ionomer cements: Ketac Molar Easymix® (3M ESPE), which has recently appeared in the market and offers the additional advantage of easy mixing, and FUJI II® (GC Dental Industrial Corporation), which has been for a longer time available in the market. **Methods:** Both materials were handled according to the manufacturer's instructions. 15 samples were made of each glass ionomer cement using sample molds which measured 4 mm diameter x 6 mm height. Glass ionomer cement was placed into the molds and then pressed. Samples were introduced in an oven (37 ± 1 °C at 90% relative humidity). After one hour had elapsed, samples were withdrawn from the oven and then individually immersed in 1 mL de-ionized water in a hermetically sealed plastic container, to be later stored in a chamber at 37 ± 1 °C. After 24 hours, samples were removed from the chamber and withdrawn from the storing solution, they were then rinsed with deionized water and excess water was removed with blotting paper. Samples were then transferred to another container adding 1 mL deionized water in order to be placed in the oven under the same conditions. Total ionic strength adjustment buffer solution was incorporated into the storing solution for it to be read with the help of a potentiometer (pH-meter) and a fluoride selective electrode for fluoride under magnetic agitation. Prior to conducting fluoride measurements, solutions were prepared in different concentrations in order to establish the calibration curve. The same procedure was performed in the aforementioned manner during the 36 days which the study lasted. **Results:** Both cements released fluoride. For both cements, highest release was observed during the first 24 hours. Release declined during the second day and kept gradually decreasing with time. Results were statistically analyzed using the Mann-Whitney test. No statistically significant differences were observed. **Conclusions:** Both glass ionomer cements exhibited similar fluoride release. Glass ionomer cement are ideal for use in preventive dentistry as well as in atraumatic restorative technique.

Key words: Glass ionomer cement, fluoride release.

Palabras clave: Cemento de ionómero de vidrio, liberación de fluoruro.

RESUMEN

Objetivo: El propósito de este estudio fue comparar la cantidad de fluoruro liberada por dos cementos de ionómero de vidrio: Ketac Molar Easymix® (3M ESPE), el cual es de reciente aparición en el mercado y ofrece la ventaja de mezclarse fácilmente, y FUJI II® (GC Dental Industrial Corporation), el cual ha estado a la venta por más tiempo. **Métodos:** Ambos materiales se manipularon de acuerdo con las indicaciones del fabricante. Se realizaron 15 muestras de cada cemento de ionómero de vidrio, utilizando conformadores de muestras de 4 mm de diámetro por 6 mm de altura. Se colocó el cemento de ionómero de vidrio en los conformadores y se prensó. Las muestras fueron introducidas en una estufa (37 ± 1 °C a 90% de humedad relativa). Una hora después las muestras se retiraron de la estufa y fueron inmersas individualmente en 1 mL de agua desionizada, en un recipiente de plástico sellado herméticamente. Posteriormente se almacenaron en una cámara a 37 ± 1 °C. A las 24 horas se retiraron las muestras de la cámara y de la solución de almacenaje, se enjuagaron con agua desionizada, se eliminó el exceso de agua con papel secante y se transfirieron a otro recipiente, añadiendo 1 mL de agua desionizada para colocar las muestras en la estufa bajo las mismas condiciones. A la solución de almacenaje se le agregó la solución tampón de ajuste de la fuerza iónica total para ser leída utilizando un potenciómetro y un electrodo selectivo para fluoruros bajo agitación magnética. Para llevar a cabo la determinación de fluoruro, previamente se prepararon soluciones del mismo a diferentes concentraciones para establecer la curva de calibración. El mismo procedimiento se realizó de la manera antes descrita los 36 días que duró el estudio. **Resultados:** Ambos cementos liberaron fluoruro. La más alta liberación se presentó durante las primeras 24 horas para ambos cementos, declinó en el segundo día y gradualmente disminuyó con el tiempo. Los resultados fueron analizados estadísticamente utilizando la prueba Mann-Whitney, en la cual no se encontraron diferencias significativas. **Conclusiones:** Ambos cementos de ionómero de vidrio presentan una liberación similar de fluoruro. Los cementos de ionómero de vidrio son ideales en odontología preventiva y en la técnica restaurativa atraumática.

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INTRODUCTION

Clinical applications of glass-ionomer cements (GIC) in the practice of dentistry have greatly increased since they were first introduced by Wilson and Kent in 1971. Although possessing low resistance to occlusal loads and being opaque, GIC exhibit great advantages over other restorative materials.¹

GIC cements are ideal for use in primary teeth, especially when adhering to the ART² (atraumatic restorative technique), since they chemically bond to dental structures, are biocompatible³ and release fluoride during prolonged periods: five years according to Croll and his team⁴ and eight years according to Forsten.⁵

GIC fluoride release is beneficial for patients since it prevents onset of carious lesions as well as secondary caries. This is due to the fact that fluoride elicits remineralization of early carious lesions.^{6,7} Therefore, many authors suggest that fluoride in low concentrations is necessary in oral fluids in order to decrease caries incidence.⁸⁻¹⁰ Caries incidence decrease is due to a reduction in enamel solubility to oral acids, as well as inhibition of bacterial enzymes elicited by the fluoride.^{11,12}

Upon contacting dentin and enamel, the GIC fluoride undertakes ion exchange with the tooth's hydroxyapatite, thus forming fluorapatite. Fluoroapatite is harder and less soluble in acids; this characteristic favors also its use as pits and fissure sealant.¹³

Certain intrinsic variables are involved in the fluoride release process. They are mainly determined by the way the cement is manufactured: the composition of the aluminum-silicate glass and polyalkenoic acid, the size of the powder particle, the relative proportion of components (glass/polyacid/tartaric acid/water) in the mixed cement, and finally, the mixing process.^{14,15}

When the components of the glass ionomer are mixed, they experience a reaction involving neutralization of acid groups elicited by the solid base of the glass powder. Important amounts of fluoride are released during the mixing process and after the reaction: this release is higher during the first days.^{16,17}

Dentin irritation could be expected with the use of GIC, since they present acidity values very similar to those of zinc phosphate. Nevertheless, in glass-ionomer cements, the molecular size of the polyacid prevents its penetration into the dentin tubules, and therefore, its irritant effect is decreased. Notwithstanding this fact, it is advisable to use a calcium hydroxide lining in those cavities that are either very deep or recently carved, where cement is to be placed.¹⁸ Additionally, glass-ionomer cements are susceptible to dissolution or desiccation while the

hardening chemical reaction takes place, and they tend to fracture if during this period they are in contact with humidity. It is therefore recommended to protect them with varnish during the initial hours of setting.¹⁹

It is worth mentioning that, due to the polyacid high viscosity, GIC are difficult to mix.²⁰ The glass ionomer Ketac Molar Easymix[®] (3M ESPE) is not so viscous, and thus easier to manipulate.²¹ This characteristic is useful for pedodontists since, due to its easy mixing, the clinical procedure results simplified.

The aim of the present study was to compare the amount of fluoride released by CIV Ketac Molar Easy Mix[®], which exhibits the property of easy mixing, with CIV Fuji II cement[®] which is an already reputed glass ionomer cement.

MATERIALS AND METHODS

Two glass-ionomer cements used for restoration were selected : Ketac Molar Easymix[®] (3M ESPE) and Fuji II[®] (GC Dental Industrial Corp). 15 samples were manufactured of both GIC (n = 30). Both cements were mixed according to the manufacturers' indications. The mixes were placed in 4 mm diameter x 6 mm height sample formers, which were in turn placed in presses. After this procedure, the samples were taken to a Hanau[®] oven (37 ± 1 °C and 90% RH). Samples were withdrawn after 60 minutes, and then were individually submerged in 1 mL deionized water in a plastic container, and stored in a chamber at 37 ± 1 °C (Felisa[®]).

24 hours later, samples were withdrawn from the storing solution, and rinsed with 1 mL deionized water. Excess water was removed with blotting paper. Samples were then once more placed in a container, 1 mL deionized water was incorporated, and samples were then stored under the same conditions (37 ± 1 °C). 1 mL of the TISAB solution was incorporated to the solution where samples had been stored for 24 hours. This was performed in order to carry out fluoride determination with a potentiometer (Oakton[®] pH/Con 510) and a fluoride-selective electrode (Orion[®] 9609BN) under magnetic agitation. In order to carry out fluoride determination, fluoride solutions at different concentrations were prepared so as to establish a calibration curve. The aforementioned procedure was implemented during all 36 days of the study. Results were statistically analyzed using the Mann-Whitney test.

RESULTS

It was ascertained that both cement brands released fluoride. The amounts of released fluoride were

greater during the first 24 hours, to then decline on the second day; it then gradually decreased along the time span of the study. This behavior was observed in both glass-ionomer cements. It was also observed that the profiles of released fluoride were almost parallel, especially during the first two days of the experiment (Figure 1).

During the course of the study, it was observed that GIC Fuji II® released greater amounts of fluoride. Likewise, this cement exhibited greater peaks in the graph, whereas GIC Ketac Molar Easy Mix® exhibited a more homogeneous release (Figure 1).

Table 1 depicts released fluoride average results (ppm) of both cements, as well as their standard deviation.

Results were statistically analyzed with the Mann-Whitney test, in order to compare amounts of fluoride released by both cements. No statistically significant differences were found.

DISCUSSION

The present study established the fact that both GIC released fluoride. It was likewise established that the amount of released fluoride was greater during the first 24 hours (burst effect), to then decline on the second day, and then gradually decrease with the passing of time. The results were in concordance with results obtained by Dr De Shepper,²² Dr Wilson,²³ Dr Perrin C,²⁴ Dr Globber and their teams,²⁵ Dr Bala O²⁶ among others.

According to studies conducted by Dr Forsten,⁵ fluoride is the most effective caries-prevention agent. The metabolism of the caries-causing bacteria is inhibited and dentin and enamel resistance are increased. Porous enamel and softened dentin can be remineralized when subjected to the presence of fluoride.

The «burst» or jet effect, which takes place during the first 24 hours, refers to the massive fluoride release.²⁷ This effect is of paramount importance, since at this point lies the greater bacteriostatic and bactericidal effect of the GIC.^{28,29}

From there derives the fact that GIC are the choice materials for caries prevention. On the other hand, *in vitro* studies indicate that, although compomers did not exhibit a «burst» (jet) effect, they exhibited a long-term release which was close to the release elicited by glass-ionomer cements.^{30,31} Even these low, but constant, fluoride release levels decreased bacterial growth, dental plaque and acid production.²⁷

According to Dr Swift and his group,³² caries which persists after placing a GIC restoration is due to spaces between the restoration and the dental wall. It is therefore of paramount importance to verify that restorations be in close contact with the dental structure. It is equally vital to use a varnish on the restoration: this will prevent microfiltrations and decrease metabolic activity of acidogenic and aciduric bacteria, such as *Streptococcus mutans*, which are important agents in caries development.^{33,34}

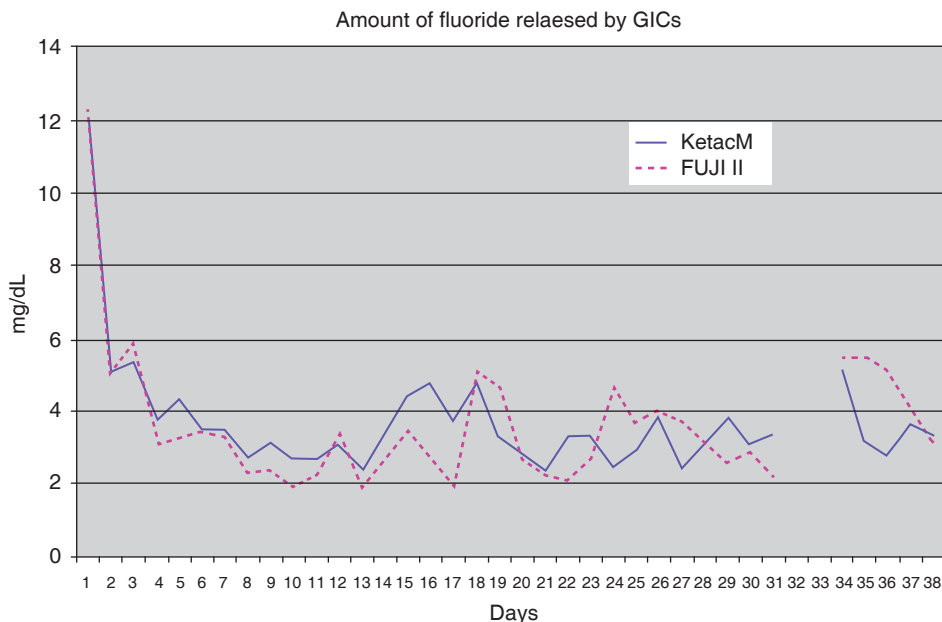


Figure 1.

The behavior of both cements during the 36 days of study can be observed. Greatest fluoride release took place during the first 24 hours, and decreased with time. This indicated the greatest peak in the graph (jet or burst effect). On day 34, an increase in fluoride could be observed, since no measurements were taken on days 32 and 34, which caused fluoride accumulation.

The present study mentions decrease in caries risk due to fluoride released from a temporary restoration with GIC. Other authors mention the fact that, when GIC is placed as a base under another restoration, a greater *S mutans* inhibition is recorded, since GIC are in contact with the carious lesion and do not experience the disadvantage of suffering the constant flow of saliva which dilutes fluoride concentration.^{28,34} Nevertheless, in the latter case, long term, constant release of fluoride into the mouth as such would not take place.

On the other hand, Forsten²⁸ and Swift³² mention that, in the presence of acid oral pH due to deficient oral hygiene there is no guarantee that fluoride-releasing agents might prevent carious lesions, recurrent caries and proximal surface caries which are most frequent in children.

It is noteworthy to mention the fact that, due to their poor physical properties, glass-ionomer materials must not be considered a universal restoration to be subjected to tensions and loads.³² GIC are ideal for primary teeth, for temporary restorations of permanent teeth, for restorations in permanent teeth which are not subjected to occlusal loads, as well as in the ART technique. This technique has gained interest for patient populations which have no access to modern dentistry.⁴

The amount of fluoride required to prevent and treat caries has not been documented. It is assumed that fluoride content must be as high as possible without incurring in adverse effects on the material's physical properties.⁵

In the present study, amounts of released fluoride were similar for both GIC. Nevertheless, there could be variations, as mentioned by Roeland and his team.¹⁶ These variations could be related to the GIC brand, matrix, filling, amount of incorporated fluoride, liquid/powder ratio, as well as mixing procedures.

Table I shows the fact that on day 34, in both glass-ionomer cements, there was an increase in fluoride amount. This could be due to the fact that on days 32 and 33 of the study no measurements were performed; therefore, the fluoride was accumulated.

Figure 1 depicts fluoride release of the GIC Fuji II®. More peaks can be observed in this graph; this could be due to small variations among liquid and powder proportions, as has been pointed out by Wiegand and his group.²⁷

CONCLUSIONS

Both glass-ionomer cements released fluoride. The most important fluoride release took place during the first 24 hours. It then gradually decreased and became constant during the following days of the study.

Table I. Average results and standard deviation of released fluoride are presented (ppm) for both cements during the whole study. For both glass ionomer cements average as well as standard deviation are very similar.

CIV	Days																																					
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	34	35	36	37	38		
Average	12.0	5.2	5.4	3.8	4.3	3.5	3.5	2.7	3.2	2.8	2.7	3.1	2.4	3.5	4.4	4.8	3.7	4.8	3.3	2.8	2.4	3.3	3.4	2.5	2.9	3.9	2.4	3.2	3.8	3.1	3.3	5.1	3.2	2.8	3.7	3.3		
σ ±	2.4	1.6	3.6	2.2	2.5	1.6	2.1	1.0	1.0	0.9	0.8	1.4	0.8	3.1	1.6	2.5	2.4	3.1	2.0	1.9	1.1	2.1	1.5	1.2	1.3	1.5	0.9	1.5	2.6	1.5	1.6	2.2	0.6	0.6	1.8	1.3		
Average	12.4	5.0	6.0	3.0	3.3	3.5	3.3	2.3	2.4	1.9	2.2	3.4	1.8	2.6	3.5	2.8	1.9	5.1	4.7	2.7	2.3	2.1	2.7	4.6	3.6	4.1	3.7	3.1	2.6	2.8	2.2	5.5	5.5	5.1	4.0	3.1		
σ ±	2.6	2.2	1.7	1.1	1.0	1.5	1.4	0.8	0.6	0.7	0.7	1.3	1.1	1.0	2.1	1.2	1.4	2.6	2.4	1.2	0.9	0.9	1.2	2.3	1.2	1.3	1.2	0.9	0.9	0.8	0.7	1.8	1.5	1.3	1.1	0.8		

More studies are required on GIC fluoride release, since these materials represent yet another alternative for pedodontists in the prevention of caries during childhood as well as ART technique.

REFERENCES

1. Verbeeck RMH, De Moor RJG, Van Even DFJ, Martens LC. The short-term fluoride release of a hand-mixed versus capsulated system of a restorative glass-ionomer cement. *J Dent Res.* 1993; 72 (3): 577-581.
2. Botelho M. Inhibitory effects on selected oral bacteria of antibacterial agents incorporated in a glass ionomer cement. *Car Res.* 2003; 37: 108-114.
3. Dhondt CL, De Maeyer EAP, Verbeeck RMH. Fluoride release from glass ionomer activated with fluoride solutions. *J Dent Res.* 2001; 80 (5): 1402-1406.
4. Croll P, Nicholson JW. Glass ionomer cements in pediatric dentistry: review of the literature. *Pediatr Dent.* 2002; 24: 423-429.
5. Forsten L. Fluoride release and uptake by glass-ionomers and related materials and its clinical effect. *Biomaterials.* 1998; 19: 503-508.
6. Tam L, Chan G, Yim D. *In vitro* caries inhibition effects by conventional and resin-modified glass-ionomer restorations. *Oper Dent.* 1997; 22: 4-15.
7. Benelli E, Serra M, Rodriguez Jr A, Cury J. *In situ* anticariogenic potential of glass ionomer cement. *Car Res.* 1993; 27: 280-284.
8. Fahinur E, Rengin E, Cemal E. A comparative study of plaque mutans streptococci levels in children receiving glass ionomer cement and amalgam restorations. *J Dent Child.* 2003; 70: 10-14.
9. Seppä L, Torppa E, Luoma H. Effect of different glass ionomers on the acid production and electrolyte metabolism of *Streptococcus mutans* in vitro. *Car Res.* 1992; 26: 434-438.
10. Duckworth RM, Morgan SN. Oral fluoride retention after use of fluoride dentifrices. *Car Res.* 1991; 25: 123-129.
11. Fross H, Näse L, Seppä L. Fluoride concentration, mutans streptococci and lactobacilli in plaque from old glass ionomer fillings. *Car Res.* 1995; 29: 50-53.
12. Rippa LW. Dental materials related to prevention-fluoride incorporation into dental materials: reaction paper. *Adv Dent Res.* 1991; 5: 56-59.
13. Graham JM. *Atlas práctico de cementos de ionómero de vidrio.* Barcelona: Ed Salvat; 1990. pp. 120-123.
14. Roeland JG, De Moor, Verbeeck RMH, Erna AP, De Maeyer. Fluoride release profiles of restorative glass ionomer formulations. *Dent Mater.* 1996; 12: 88-95.
15. Weidlich P, Miranda LA, Maltz M, Samuel SMW. Fluoride release and uptake from glass ionomer cements and composite resins. *Braz Dent J.* 2000; 11 (2): 89-96.
16. Mc Lean JW. Clinical applications of glass ionomer cements. *Op Dent.* 1992; 5: 184-190.
17. Barceló FH, Palma JM. *Materiales dentales.* México: Ed. Trillas; 2004. pp. 97-102.
18. Johnson GH, Powell LV, De Rouen TA. Post-cementation pulpal sensitivity: zinc phosphate and glass ionomer luting cements. *J Am Dent Assoc.* 1993; 124 (11): 38-46.
19. Castro GW, Gray SE, Buikema DJ, Regan SE. The effect of various surface coatings on fluoride release from glass ionomer cement. *Oper Dent.* 1994; 19 (5): 194-198.
20. Scholtanus JD, Huysmans MC. Clinical failure of class II restorations of a highly viscous glass-ionomer material over a 6-year period: a retrospective study. *J Dent.* 2007; 32 (2): 156-162.
21. Instructivo anexo del CIV Ketac Molar Easymix® (3M ESPE).
22. De Shepper EJ, Berr EA 3rd, Cailleteau JG, Tate WH. A comparative study of fluoride release from glass-ionomer cements. *Rev Belge Med Dent.* 1996; 51 (1): 22-35.
23. Wilson AD, Groffman DM, Kuhn AT. The release of fluoride and other chemical species from a glass ionomer cement. *Biomaterials.* 1985; 6: 431-433.
24. Perrin C, Persin M, Sarrazin J. A comparison of fluoride release from four glass-ionomer cements. *J Nihon Univ Sch Dent.* 1997; 39 (3): 123-127.
25. Gloor SR, Rossouw RJ, Van Wyk-Kotze TJ. A comparison of fluoride release from various dental materials. *J Dent.* 1998; 26 (3): 259-265.
26. Bala O, Uctasil M, Can H, Türköz E, Cam M. Fluoride release from various restorative materials. *J Dent.* 1998; 26 (4): 355-359.
27. Wiegand A, Wolfgang B, 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.
28. Forsten L. Fluoride release of glass ionomers. *Journal of Esthetic Dentistry.* 1994; 6: 217-222.
29. Forss H, Jokinen J, Spets-Happonen S, Seppä L, Louma H. Fluoride and mutans streptococci in plaque grown on glass ionomer and composite. *Car Res.* 1999; 25: 454-458.
30. Shaw AJ, Carrick T, McCabe JF. Fluoride release from glass-ionomer and compomer restorative materials: 6 month data. *J Dent.* 1998; 26 (4): 344-359.
31. Ausmussen E, Peutzfeldt A. Long-term fluoride release from glass ionomer cement, a compomer, and from experimental resin composites. *J Prosthet Dent.* 1998; 80 (4): 474-478.
32. Swift E. Effects of glass ionomers on recurrent caries. *Oper Dent.* 1989; 14: 40-43.
33. Loyola J, García F, Lindquist R. Growth inhibition of glass ionomer cements on mutans streptococci. *Pediatr Dent.* 1994; 16 (5): 346-349.
34. Forss H, Näse L, Seppä L. Fluoride concentration, mutans streptococci and lactobacilli in plaque from old glass ionomer fillings. *Car Res.* 1995; 29: 50-53.

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