

Fluoride Release from Glass Ionomer Cements and Compomers *in vitro*

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Abstract

Objective: To measure the fluoride released from a group of glass ionomer cements and compomers, *in vitro* using the fluoride ion specific electrode over a period of four months.

Materials and Methods: The daily fluoride release of three conventional glass-ionomer cements (Ketac-Fil, Fuji IX GP and Fuji VII), two light cured glass-ionomer cements (Fuji II LC and Vitremer) and two compomers (Dyract AP and F-2000) was measured over a period of four months. Ten cylindrical specimens (surface area= 1.996 cm²) were made of each of the materials, 10 mm in diameter and 1.5mm in thickness using the same mould. Measurements of the fluoride release in 5 ml of deionized water (25°C) were performed at intervals of one day, weekly for the rest of the first month, and monthly for the remaining three months.

Results: A burst effect of fluoride release was observed initially with at least a ten-fold decrease occurring for all the materials by the end of the first week. Compomers were found to release significantly less fluoride than the glass-ionomer cements during the first four months. The (mean ± SD) daily fluoride release for the materials at four months was: Fuji VII (0.31± 0.06 ppmF), Ketac-Fil (0.31± 0.08 ppmF), Vitremer (0.27± 0.05 ppmF), Fuji II (0.12± 0.03 ppmF), FujiIX (0.09± 0.05 ppmF), F2000 (0.07± 0.05 ppmF) and Dyract (0.06± 0.01 ppmF).

Conclusions: The most fluoride was released by Fuji VII and Ketac-Fil. Dyract released the least fluoride of all the materials.

Keywords: Fluoride, glass ionomer cements, compomers, *in vitro*, ion specific electrode, four months.

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Introduction

Glass-ionomer cements, the modern version of silicate, are perhaps the best known fluoride-releasing restorative materials and they have been shown to have anticariogenic properties due to

their significant release of fluoride,^{1,2} the uptake of fluoride (and aluminum) in cavity walls,³ enamel and plaque² and the enhanced reprecipitation of calcium and phosphate³ promoted by the fluoride release.

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The introduction of the first glass-ionomer cement by Wilson and Kent in (1972),⁴ brought a new era in the field of the dental materials, and initiated a whole series of research on the several properties and possible applications of the new material. Glass-ionomer cements had two main advantages; chemical adhesion to dental structures and possible cariostatic effects through their fluoride release.

Unfortunately the first glass-ionomer cements had rather poor properties and limitations in their clinical use. During the last twenty years however, an amazing progress has taken place, and an improved range of glass-ionomer cements is now available for use. As a result, glass-ionomer cement systems have become important dental restorative and luting materials for use in preschoolers, children and teenagers. These materials form a chemical bond to tooth structure, are biocompatible, release fluoride ions for uptake by enamel and dentine, and are able to take up fluoride ions from dentifrices, mouthwashes and topically applied solutions.⁵ Glass-ionomer cements widely used as restorative materials for restorations of Class I and II cavities in primary teeth,⁶ Class V erosive lesions, and Class III cavities in anterior permanent teeth. Moreover, glass-ionomer cements as liners, bases, luting cements, fissure sealants and metal-reinforced core build-up materials are also available.⁷ Glass-ionomer cements have also been associated with reduced secondary caries development.^{8, 9} Fluoride-releasing materials, predominantly glass-ionomers and compomers, did show cariostatic properties and may affect bacterial metabolism under simulated cariogenic conditions *in vitro*. This favourable result has been attributed to the release of a high concentration of fluoride ions from the cement and an initially low material pH.¹⁰⁻¹³

The aim of this study is to measure the fluoride released from a group of glass-ionomer cements and compomers, *in vitro* using fluoride ion-specific electrode.

Materials and methods

Seven different dental restorative materials from the following three groups comprised the study materials under investigation (Table 1; Fig1): Group A comprised of 2 compomer restorative materials, group B comprised 2 light-cured glass-ionomer restorative materials, and group comprised 3 conventional glass-ionomer cements. The daily fluoride release of the three groups of materials was measured over a period of four months. 10 cylindrical specimens (surface area: 1.996 cm²) were made for each of the materials measuring 10 mm in diameter and 1.5 mm in thickness using the same mould. They were suspended in 5 ml of deionized water and transferred to fresh aliquots of deionized water (Fig 2). Measurements of the fluoride release in 5 ml of deionized water (25°C) were performed at intervals of one day, weekly for the rest of the first month, and monthly for the remaining three months. All the materials were prepared as described by the manufacturers. The fluoride release in ppm was determined using an ion-specific fluoride electrode (Orion 920A Ion analyzer). At the time of every measurement, each specimen was removed from its test tube, and was transferred to a new tube containing a fresh aliquot of 5 ml of deionized water. The amount of fluoride ions released from each sample was determined by the use of a fluoride ion-specific electrode, which was standardized before each reading according to the manufacturer's instructions. Calibration of the fluoride ion-specific electrode was accomplished by the use of standardized solutions with known fluoride concentrations, prior to every measurement. The following standardized solutions; 100 ppmF, 10 ppmF, 1 ppmF, 0.1 ppmF, 0.05 ppmF, and 0.01 ppmF were used and included in the process in order to more accurately reproduce the non-linear part of the calibration curve. The mV readings were subsequently transferred to a spreadsheet software computer application, by the use of which the fluoride concentration in parts per million (ppm) was estimated according to the calibration values priorly obtained to every measurement (Microsoft® Excel®, version 7.0).



Figure 1: The seven restorative materials included in the study.



Figure 2: Study materials in 5 ml aliquots of distilled water.

Results

Initial daily fluoride release in deionized water

The main finding was that compomers released considerably less fluoride in deionized water than glass-ionomer cements throughout the four months. The compomers with the most fluoride release was F 2000, and the glass-ionomer cement with the best performance was Fuji VII. The mean and the Standard Deviation (SD) of daily fluoride release for the study materials throughout the duration of the study are shown in (Table 2; Fig. 3).

The mean \pm (SD) of fluoride release for each of the materials at 24 hours, one week, two weeks, three weeks, four weeks two months, three months, four months were calculated and are presented in Table (2). A one way Analysis Of Variance (one way ANOVA) combined with a Tukey's pairwise comparison was used in order to identify possible differences of the fluoride

release among glass-ionomer cements over a period of four months.

Fluoride measurements took place after 24 hours of immersion of the specimens in deionized water. According to the Tukey's pairwise comparisons, fluoride release displayed the highest release among all the glass-ionomer cements during the first 24 hours. The mean \pm SD for Fuji VII of fluoride release was the highest (22.39 ± 15.9 ppm F) and the Ketac-Fil displayed the second highest fluoride release after 24 hours (16.12 ± 4.41 ppm F). After one week however, performance of the Fuji VII could not be statistically differentiated from Ketac-Fil and Vitremer. On the other hand, Fuji IX GP displayed the least fluoride release after 24 hours among the conventional glass-ionomer cements, however, although no statistical difference could be identified between Fuji IX GP and Vitremer at 24 hours.

The two light-cured glass-ionomer cements; Vitremer and Fuji II LC released significantly more fluoride than compomers during the 24 hours, with Vitremer released significantly more fluoride than Fuji II LC. The two compomers released less fluoride than conventional glass-ionomer cements and light-cured glass-ionomer cements. Dyract AP displayed the least fluoride release among all the experimental materials at 24 hours.

After the first one week, fluoride measurements took place every week for the following three weeks and finally every month for the rest of the four months period. In order to obtain the average daily fluoride release of the study materials, the weekly and monthly amounts of fluoride were divided by 7 and 30, respectively. A burst effect of fluoride release was observed initially with at least a ten-fold decrease occurring for all the materials by the end of the first week. A slow decrease in fluoride release, however, was evident during the rest of the 1st month. Finally, during the last two months fluoride release dropped to a very low level for all the materials except for Fuji VII and Ketac-Fil.

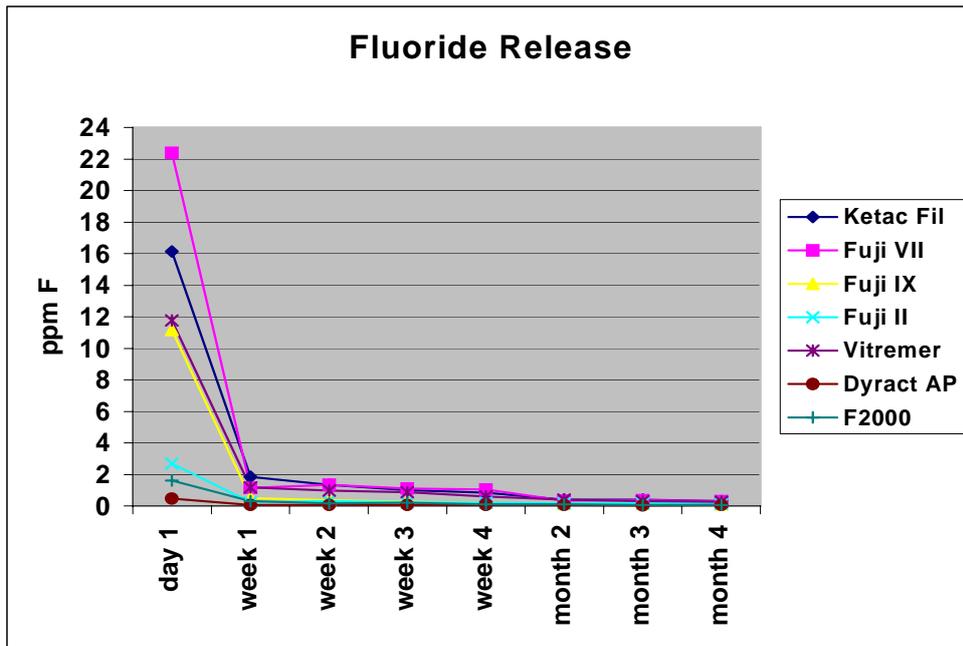


Figure 3: Daily Fluoride release of study materials over 4 month duration.

Table (1): Experimental groups that participated in the study.

Study Materials	Dental Restorative Materials
Group A, Compomers	Dyract® AP (Dentsply-DeTrey GmbH) F-2000 (3M ESPE Dental Products)
Group B, Light-cured glass-ionomer cements	Vitremer (3M ESPE Dental Products) GC Fuji II LC (GC Corporation)
Group C, Conventional glass-ionomer cements	Ketac™ Fil Plus Aplicap (3M ESPE Dental Products) GC Fuji IX GP FAST (GC Corporation) GC Fuji VII Capsule (GC Corporation)

Table (2): Mean (\pm SD) of daily fluoride release for study materials over 4 month's duration.

Group	Material	Day 1	Week 1	Week 2	Week 3	Week 4	Month 2	Month 3	Month 4
A	Dyract AP	0.47	0.07	0.06	0.07	0.06	0.07	0.05	0.06
	(SD)	(0.16)	(0.02)	(0.01)	(0.01)	(0.01)	(0.04)	(0.01)	(0.01)
A	F2000	1.62	0.3	0.18	0.19	0.11	0.1	0.08	0.07
	(SD)	(0.86)	(0.19)	(0.14)	(0.13)	(0.06)	(0.05)	(0.05)	(0.05)
B	Fuji II LC	2.69	0.25	0.3	0.24	0.2	0.17	0.16	0.12
	(SD)	(0.84)	(0.09)	(0.1)	(0.09)	(0.06)	(0.03)	(0.04)	(0.03)
B	Vitremer	11.75	1.18	0.97	0.87	0.6	0.42	0.3	0.27
	(SD)	(6.96)	(0.27)	(0.23)	(0.21)	(0.15)	(0.1)	(0.05)	(0.05)
C	Ketac-Fil	16.12	1.86	1.36	1.01	0.85	0.39	0.41	0.31
	(SD)	(4.41)	(0.33)	(0.35)	(0.29)	(0.23)	(0.09)	(0.1)	(0.08)
C	Fuji IX GP	11.17	0.51	0.37	0.28	0.21	0.13	0.15	0.09
	(SD)	(2.92)	(0.27)	(0.22)	(0.24)	(0.18)	(0.05)	(0.06)	(0.05)
C	Fuji VII	22.39	1.15	1.35	1.1	1.05	0.34	0.36	0.31
	(SD)	(15.9)	(0.41)	(0.36)	(0.34)	(0.29)	(0.05)	(0.05)	(0.06)

Discussion

Measurement of fluoride using ion-specific electrode

Several authors in previous studies have been changing the immersing solution every 24 hours, regardless of the measurement intervals.¹⁴⁻¹⁸ McCabe, (1998)¹⁷ suggested that daily changes of the immersing solution could minimise or even prevent the inhibition of fluoride release by the equilibration of the solution.

Many other authors, however, have used a different protocol changing the immersing solution either at the time of the estimation of the fluoride concentration of the solution¹⁹⁻²² or 24 hours before.^{23,24} Their argument has been that when small specimens are used, 24 hours concentrations of fluoride are too low to be effectively measured.²¹ In the present study, it would be ideal if the distilled water contained in the test tubes could be renewed on a 24 hours basis during the whole experimental period. Something like that though, would be extremely time consuming. Therefore, it was decided not to change the immersing solution prior to weekly or monthly measurement, but to measure the cumulative amount of fluoride release during the respective weekly or monthly period. A continuous-flow fluoride-measuring system that monitors the amount of fluoride released over time was used to determine the release of fluoride from a resin-modified glass-ionomer cement. The results show that the release rate began with a fast burst of fluoride which quickly diminished to low levels in 3 days.²⁵

It is clear that a long-term measurable release of fluoride can be observed from certain restorative materials, *in vitro*, particularly glass ionomer cement, resin modified glass ionomer cement, fluoridated cements, fluoridated dental amalgam and certain fissure sealants. In general, the rate of fluoride release is not constant but exhibits a relatively rapid initial rate, which decreases with time.

However, the fluoride release profiles may be dependent on specific formulation and on experimental design and sampling methods.²⁶

Initial daily fluoride release

Compomers in this study were found to release significantly less fluoride than glass-ionomer cements. This finding is in agreement with previous studies.^{16, 18, 24, 27, 28} This difference between the two types of materials may be attributed to two main factors; the chemical composition and the setting reaction of the materials.

First of all, the chemical composition of compomers is different to that of glass-ionomer cements. Glass-ionomer cements consist of an ion-leachable aluminosilicate glass and an acidic polymer. Compomers, on the other hand, consist of an ion-leachable glass which is different to the one used in glass-ionomer cements and a modified monomer. The different composition of the glass components between the two materials results in its fluoride content being much lower in compomers than in glass-ionomer cements. The fluoride content of the glass can be up to 2½ times higher in glass-ionomer cements than in compomers.^{24, 29} Despite the fact that not all this fluoride is available for release from the materials, the fluoride content obviously affects the reported difference in fluoride release between compomers and glass-ionomer cements. In glass-ionomer cements the acid-base setting reaction releases fluoride ions from the glass particles in the polysalt matrix, thus increasing the fraction of fluoride available for release from the material.³⁰ In compomers, however, no acid-base reaction takes place initially but they set through a polymerization reaction. A limited acid-base reaction can eventually occur only after the slow penetration of the material by water.²⁷ This means that the only initial source for the fluoride release in compomers is the surface of the exposed glass particles, while in glass-ionomer cements additional fluoride can be released by the silica-gel phase surrounding the glass particles and the polysalt matrix.

As a result, fluoride release from glass-ionomer cements is higher.

Conclusion

Compomers released significantly less fluoride than the glass-ionomer cements. All of the compomer materials that participated in the study released similar amounts of fluoride, with F 2000 displaying the highest and Dyract AP the lowest fluoride release.

The most fluoride was released by Fuji VII and Ketac-Fil. Dyract released the least fluoride of all the materials.

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قياس مادة الفلوريد المتحررة من مجموعة من الحشوات السنية التي تم تحضيرها في المختبر

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الملخص:

يهدف البحث إلى قياس مادة الفلوريد المتحررة من مجموعة من الحشوات السنية التي تم تحضيرها في المختبر خلال فترة زمنية محددة.

تم قياس مادة الفلوريد المتحررة من استخدام ثلاث مجموعات من الحشوات السنية المكونة من ثلاث مواد من:

conventional glass- ionomer cements (Ketac-Fil, Fuji IX GP and Fuji VII),

ومادتين من: light cured glass-ionomer cements (Fuji II LC and Vitremer)

و مادتين من: compomers (Dyract AP and F-2000)

وقد تم قياس مادة الفلوريد المتحررة من هذه المواد خلال أربعة شهور على فترات زمنية متتالية، وهي أول 24 ساعة وكل أسبوع لمدة شهر، وكل شهر حتى نهاية الفترة الزمنية المحددة، بعد تحضير عينات من هذه المواد داخل المختبر حسب التعليمات اللازمة لتحضيرها باتباع الإرشادات المصنعية لكل مادة. وقد تم حساب مادة الفلوريد المتحررة من استخدام مجموعات من الحشوات السنية عن طريق استخدام جهاز الايونات الكهربائي وهو جهاز حساس يستخدم لقياس مادة الفلوريد المتحررة من المواد.

لقد وجد في الدراسة أن أكبر كمية لتحرير الفلوريد تمت خلال 24 ساعة من تحضير المادة، ثم يتناقص تحرير الفلوريد تدريجياً لجميع العينات في نهاية الأسبوع الأول، وقد تم حساب الوسط الحسابي والانحراف المعياري لكل مادة خلال فترة الدراسة ووجد كالتالي:

Ketac-Fil (0.31 ± 0.08 ppmF), Fuji VII (0.31 ± 0.06 ppmF), Fuji IX (0.09 ± 0.05 ppmF), Fuji II (0.12 ± 0.03 ppmF), Vitremer (0.27 ± 0.05 ppmF), Dyract (0.06 ± 0.01 ppmF) and F2000 (0.07 ± 0.05 ppmF).

وتم استنتاج أن مادة (Fuji VII) ومادة (Ketac-Fil) من أكثر المواد التي لها القدرة على تحرير الفلوريد، وان مادة (Dyract) اقل المواد قدرة على تحرير الفلوريد من بين المواد التي تمت دراستها، وان معظم المواد السنية التي تم استخدامها في الدراسة لها القدرة على تحرير الفلوريد أكثر من أربعة شهور.

الكلمات الدالة:

الفلوريد، compomers، glass- ionomer cements، المختبر، جهاز الايونات الكهربائي، أربعة شهور.