

ASSESSING THE RELEASE OF THE FLUORIDE ION IN FOUR RESTORATIVE GLASS IONOMER CEMENTS

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ABSTRACT: Fluoride is considered to be an important caries-preventive agent as it decreases the solubility of tooth minerals. The aim of the present study was to investigate the amount of fluoride ion released, over a 9 day period, from four restorative glass ionomer cements (GIC). Elemental analysis, for O, F, Na, Mg, Al, Si, P, S, Ca, Sr, Zn, and Zr, was performed on three conventional glass ionomers [ChemFil ROCK (Dentsply), Fuji IX (GC), and Riva self-cure (SDI)] and one resin-modified glass ionomer, Ketac Nano (3M ESPE) using Energy Dispersive Spectrometry (EDS). A digital caliper was used to measure the length and diameter of each sample. The fluoride release, in $\mu\text{g}/\text{cm}^2$, from the samples in deionized water was measured for 9 days using a fluoride ion selective electrode and analyzed using the SPSS. The release of fluoride on day 1 from Fuji IX was significantly higher ($6.19 \pm 1.04 \mu\text{g}/\text{cm}^2$) than that from Ketac Nano ($3.88 \pm 0.13 \mu\text{g}/\text{cm}^2$), Riva self-cure ($3.19 \pm 0.66 \mu\text{g}/\text{cm}^2$), and ChemFil ROCK ($1.68 \pm 0.08 \mu\text{g}/\text{cm}^2$). The value found for Fuji IX was comparable to that found in various other experimental settings. The difference in fluoride release on day 1 between Ketac Nano and Riva self-cure was not significant. The fluoride release from day 5 to day 9 stabilized at a lower level for all the ionomers and no statistically significant difference in the level of release was present between them during that period. I concluded that there is a wide range in the release of the fluoride ion among restorative glass ionomer cements with the highest level of release on day 1 being observed in Fuji IX, followed by Ketac Nano, Riva self-cure, and ChemFil ROCK.

Keywords: ChemFil ROCK; Ketac Nano; Resin Modified Glass Ionomer; Restorative Glass Ionomer Cements; Riva self-cure.

INTRODUCTION

The release of fluoride from conventional glass ionomer cements (CGI) gives them anti-cariogenic and anti-microbial properties.¹ The quest for developing the ideal restorative material has led to the development of newer materials and the modification of previous ones. The range of these materials includes glass ionomer cements, giomers, resin-modified glass ionomers, polyacid modified resins, and resin composites.² Their characteristics for the release and uptake of fluoride differ according to the dental materials they contain. These materials can serve as a fluoride reservoir that enhances the fluoride level in saliva, plaque, and hard dental tissues to either decrease or prevent secondary caries.³

Fluoride reduces and prevents dental caries in the oral cavity by forming fluorapatite and fluorohydroxyapatite which are resistant to acid dissolution.⁴ Glass ionomer cements contain fluoride, polyalkeonoic acid, and silicate glass. It is known that there is increase in the release of fluoride from glass ionomers in the initial 24 hours, when the acid and base react together.⁵ This reaction results in the bursting of

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fluoride from the glass particles as it reacts with the polyaklenoic acid.⁶ The release of fluoride continues for a long time because the glass gets dissolved in the acidified water of a hydroxyl matrix, although there is a reduction in the amount of fluoride release after a specific amount has been released.

Originally, the components of the cement comprise a powder of alumino-calcium silicate glass, a fluoride flux, and a liquid, which is mainly modified polyacrylic acid.⁷ Essentially, the liquid is an aqueous solution of tartaric and polyacrylic acid. According to Kiri and Boyd,⁸ the release of fluoride from the resin-modified glass ionomer cement depends on the amount of resin used for the photochemical polymerization. The acid tends to react with the glass powder and form an ionic bond with the fluoride, aluminum, and calcium ions when the liquid is mixed with the powder. The fluoride, aluminum, and calcium ions interact with the polyacrylic acid around the glass particles within the matrix. The cement gets attached to the tooth structure via ionic bonding at the interface due to the chelation of carboxyl groups present within the acid and calcium ions in the apatite of the dentin and enamel.⁸

Conventional glass ionomer cements have weak mechanical properties in contrast to the newer products which show an increase in the compressive strength.⁹ The presence of fluoride in high concentration makes the conventional glass ionomer cements opaque but with the use of more translucent glasses and a decreased fluoride content they are more translucent.¹⁰ The GIC in the wet oral cavity are able to resist the parafunctional and masticatory stresses. Moreover, the polycarboxylate and glass ionomer cements possess good wettability and bonding properties. The GIC need to have good compressive and tensile strength to remain intact for a long time period in the oral cavity.¹¹

The process of caries and tooth cavitation occurs when there is demineralization and destruction of the organic substance and inorganic portion of the hard-dental tissue. The release of the fluoride ion from GIC depends on various factors like the composition of the material, the powder-liquid ratio, the setting mechanism, the fluoride content, and the nature of the fluoride.¹² The release of fluoride ions from the restorative materials, including resin-modified glass ionomer and glass ionomer cement, prevents the occurrence of dental caries by decreasing the microbial metabolism and the solubility of the mineral tooth structure.¹³ Recently, a variety of options have been used for restoring primary teeth, among which are some tooth-colored materials. The attention of the majority of clinicians and researchers was drawn towards this issue after fluoride was added to the dental materials. Kishore et al. found that the amount of fluoride released from the restorative materials was significantly affected by the routine use of fluoride containing dentrifices.¹⁴

The pattern of fluoride release is similar to sustained release, although there are differences between several restorative glass ionomer cements. The aim of the present study was to investigate the pattern of fluoride release, over a 9 day period, from four restorative glass ionomer cements.

MATERIAL AND METHODS

The study examined three conventional restorative glass ionomers [ChemFil ROCK (Dentsply), Fuji IX (GC), and Riva self-cure (SDI)] and a resin modified glass ionomer [Ketac Nano]. Their composition is described in Table 1.

Table 1. Composition of restorative materials used in the study

Sample no.	Material	Composition*
1	GC Fuji IX GP Capsule (radiopaque posterior glass ionomer restorative cement in capsules).	Powder: alumino silicate glass 95%, polyacrylic powder 5% (by volume) Liquid: polyacrylic acid powder 5-10% (by volume)
2	SDI Riva self-cure capsule (glass ionomer restorative material).	Compartment 1: polyacrylic acid 20-30% and tartaric acid 10-15 % (by weight) Compartment 2: fluoro aluminosilicate glass 90-95% and polyacrylic acid 5-10% (by weight)
3	ChemFil ROCK (advanced glass ionomer restorative).	1: calcium-aluminum-zinc-fluoro-phosphor-silicate glass 2: polycarboxylic acid 3: iron oxide pigments 4: tartaric acid 5: water
4	3M ESPE Ketac Nano (light-curing glass ionomer restorative and primer).	1: silane-treated glass 40-55% (by weight) 2: silane-treated zirconia 20-30% (by weight) 3: polyethylene glycol dimethacrylate (PEGDMA) 5-15% (by weight) 4: silane treated silica 5-15% (by weight) 5: 2-hydroxyethyl methacrylate (HEMA) 1-15% (by weight) 6: glass powder <5% (by weight) 7: bisphenol a diglycidyl ether dimethacrylate (BISGMA) <5% (by weight) 8: triethylene glycol dimethacrylate (TEGDMA) <1% (by weight)

*The compositions are from the manufacturer provided information

Four-disc samples measuring 6 mm (diameter) × 3 mm (height) were prepared in a customized Teflon mold. The samples were prepared with a split Teflon mold and then pressed between two microscopic glass slides. The diameter and length of each specimen were measured, using an electronic digital caliper, after the material had completely set. The specimens were then transferred into a plastic tube that contained 15 mL of deionized water for the ionic release test.

Each specimen was removed, dried, and returned into a new tube containing 15 mL of deionized water after 24 hours equilibration. A clean sealed tube was used to store a 10 mL sample of the equilibrated solution for the ion concentration test. A fluoride ion selective electrode (Fisher Scientific Accumet 13-620-629) connected to a pH/

ionic meter was used to measure the daily release of fluoride in the deionized water for 9 days in the deionized water (Figure 1). Before measuring the fluoride concentration in the samples, the ionic meter was calibrated with three different fluoride concentrations, of 1, 2, and 10 ppm, for the high concentration range and with concentrations of 0.1, 0.2, 0.5, and 1 ppm for the low concentration range. The samples and the standard solution were mixed in a ratio of 1:1. Before taking measurements, 10 mL of the solution was pipetted into a clean plastic tube to which was then added 10 mL of TISAB (Total Ionic Strength Adjustment Buffer) II solution (Thermo Scientific) with the addition of CDTA (1,2-cyclohexylenedinitrotetraacetic acid). This provided a constant background ionic strength, decomplexed the fluoride, and adjusted the solution pH. The concentration of fluoride was displayed on the instrument in ppm and converted into $\mu\text{g}/\text{cm}^2$ using equation 1:

$$\text{Fluoride released } (\mu\text{g}/\text{cm}^2) = \frac{\text{Fluoride concentration (ppm)} \times \text{Volume (mL)}}{\text{Specimen surface area (cm}^2\text{)}} \dots \text{Equation 1}$$

The restorative material compositions were examined under an electron microscope using Energy Dispersive Spectrum (EDS) at 15kV to identify the presence and relative proportions of the elements oxygen, fluorine, sodium, magnesium aluminum, silicon, phosphorus, sulphur, calcium, strontium, zinc, and zirconium (O, F, Na, Mg, Al, Si, P, S, Ca, Sr, Zn, and Zr, respectively). The SEM/EDS system was well calibrated to detect elements such as silicon and calcium with a decreased error ratio. Before measurement,

The obtained data obtained were analyzed using the Statistical Package of Social Sciences (SPSS) version 20.0.



Figure 1. Fluoride ion selective electrode used for testing the concentration of fluoride.

RESULTS

The restorative glass ionomers tested showed some differences in the release of fluoride over the 9 day period. The high level of fluoride release on day 1 was followed by a significant drop on the second day. The results showed a significant difference in fluoride release in all the materials except for Ketac Nano and Riva self-cure on day 1. The highest release of fluoride on day 1 was observed in Fuji IX ($6.19 \pm 1.04 \mu\text{g}/\text{cm}^2$), followed by Ketac Nano ($3.88 \pm 0.13 \mu\text{g}/\text{cm}^2$), Riva self-cure ($3.19 \pm 0.66 \mu\text{g}/\text{cm}^2$), and ChemFil ROCK ($1.68 \pm 0.08 \mu\text{g}/\text{cm}^2$). Figure 2 and Table 2 show differences on day 2 between the higher fluoride release materials (Fuji IX and Ketac Nano) and the lower fluoride release materials (Riva self-cure and ChemFil ROCK). After day 5, the differences became less and stabilized at a low level. ChemFil ROCK accounted for the lowest fluoride release and Fuji IX had the highest fluoride release. On day 1, there was a higher level of fluoride release which reduced significantly on day 2. Within the same material, there was no statistically significant difference in the level of fluoride release from day 5 to day 9, indicating that the release of fluoride had stabilized.

As the fluoride release was significantly influenced by the chemical composition of the GIC, the size of the glass particles, and the porosity of glass ionomer, the material content was examined using elemental microanalysis performed with microscopic Energy Dispersive Spectrometry (EDS). The filler of Fuji IX had a higher content of fluorine (10.70 ± 0.56 weight %) followed by Riva self-cure (8.33 ± 3.38 weight %), Ketac Nano (6.91 ± 0.06 weight %), and ChemFil ROCK (3.14 ± 1.09 weight %). Riva self-cure had more aluminum (18.88 ± 2.72 weight %) and less sodium (0.97 ± 0.36 weight %) compared to Ketac Nano (aluminum 8.24 ± 0.87 weight % and sodium 1.61 ± 0.60 weight %). The difference for Ketac Nano in the ratio of aluminum release to sodium release can be attributed to a lower ionization free energy in an aqueous solution so that ions are more easily dissolved.

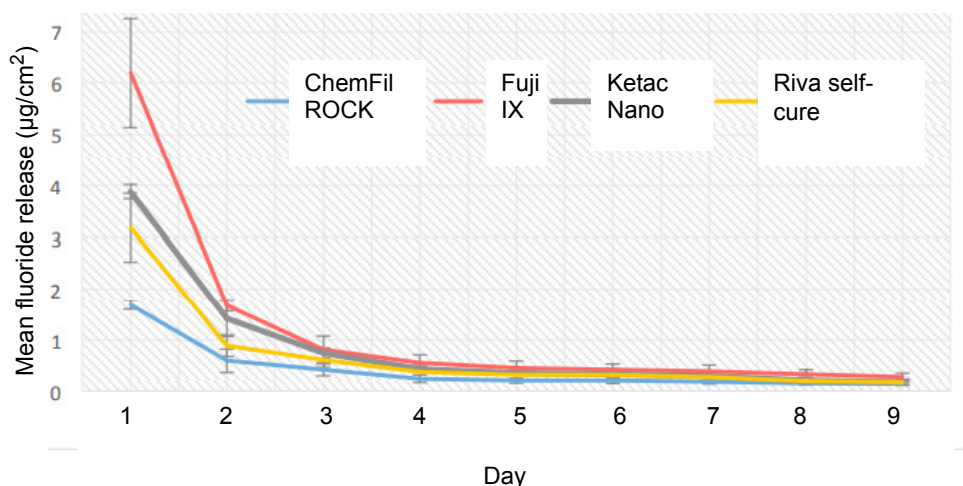


Figure 2. Release of fluoride ($\mu\text{g}/\text{cm}^2$), measured with a fluoride ion selective electrode, from day 1 to day 9, from the glass ionomer cements Fuji IX, Ketac Nano, Riva self-cure, and ChemFil ROCK. The highest release of fluoride on day 1 was observed in Fuji IX ($6.19 \pm 1.04 \mu\text{g}/\text{cm}^2$), followed by Ketac Nano ($3.88 \pm 0.13 \mu\text{g}/\text{cm}^2$), Riva self-cure ($3.19 \pm 0.66 \mu\text{g}/\text{cm}^2$), and ChemFil ROCK ($1.68 \pm 0.08 \mu\text{g}/\text{cm}^2$).

Table 2. Elemental analysis. Values are mean±standard deviation (weight %) (GIC=glass ionomer cement, O=oxygen, F=fluorine, Na=sodium, Mg=magnesium, Al=aluminum, Si=silicon, P=phosphorus, S=sulphur, Ca=calcium, Sr=strontium, Zn=zinc, Zr=zirconium)

GIC	Element (mean±SD, weight %)									
	O	F	Na	Mg	Al	Si	P	S	Ca	Sr
Reva self-cure	41.1	8.33	0.97	0.28	18.88	13.13	1.81	0.67	2.59	1.24
	± 5.94	± 3.38	± 0.36	± 0.04	± 2.72	± 1.91	± 0.47	± 0.40	± 1.18	± 4.62
Ketac Nano	O	F	Na	Al	Si	P	Ca	Zn	Sr	Zr
	37.5	6.91	1.61	8.24	18.01	0.99	0.87	4.71	10.36	15.74
	± 5.49	± 0.60	± 0.33	± 0.87	± 2.2	± 0.21	± 0.64	± 0.86	± 3.39	± 4.45
Chem Fil ROCK	O	F	Na	Al	Si	P	Ca	Zn	Sr	
	36.32	3.14	1.54	18.11	10.52	7.12	5.7	8.15	12.03	
	± 9.76	± 1.09	± 0.196	± 5.36	± 2.75	± 2.71	± 2.09	± 2.25	± 4.22	
Fuji IX	O	F	Na	Al	Si	P	Ca	Sr		
	32.85	10.70	0.75	16.23	14.96	2.43	0.33	21.79		
	± 2.22	± 0.56	± 0.07	± 0.71	± 0.51	± 0.16	± 0.15	± 1.41		

DISCUSSION

The present study showed that the release of fluoride was higher in Fuji IX, as compared to Ketac Nano, Riva self-cure, and ChemFil ROCK. Our results are not completely consistent with the findings of Bansal and Bansal⁴ who reported that conventional glass ionomers had an increased release of fluoride as compared to resin-modified glass ionomers. We found that although the conventional glass ionomer of Fuji IX had a higher fluoride release on day 1 compared to the resin modified glass ionomer Ketac Nano, Ketac Nano had a higher release than the CGI ChemFil ROCK. No significant difference was present between the fluoride release from Ketac Nano and the CGI Riva self-cure on day 1. The difference we found may be related to the presence of nanoparticles in both Fuji IX and Ketac Nano but not in Riva self-cure or ChemFil ROCK. The presence of an active filler with nanoparticles increases the fluoride release. The nanoparticles possess an increased surface area that increases the amount of fluoride released as compared to materials with a crystalline filler.

The release of fluoride reached a plateau after day 5, which remained stable at a low level for some time. These results clearly show that there was a significant depletion in the release of fluoride after day 5. These findings were consistent with the results reported by Cabral et al.¹³ that showed a high level of fluoride release on day 1 which then decreased and reached a plateau, with no statistically significant differences, after day 7. Kunzelmann et al.¹⁵ found that Fuji IX possessed the lowest material loss, as compared to other materials such as Ketac Molar and Ketac Silver. However, the level of material loss in the present study and the study conducted by Kunzelmann et al.¹⁵ was not comparable because of the use by Kunzelmann et al. of a different antagonist (aluminum), load, and velocity.

Our results are also consistent with the study by Preston et al.¹⁶ which found a greater release of fluoride from Ketac Nano than from ChemFil ROCK. These variations in fluoride release among the different glass ionomer cements cannot be attributed to differences in the methodologies used, such as the media used and the sample sizes. Xu and Burgess found Fuji IX was in the highest fluoride release group of glass ionomer cements.¹⁷ Similar results were obtained by Cabral et al.¹³

The efficacy of fluoride-releasing restorative materials can be evaluated on the basis of the important property of the capability to recharge with fluoride. Through our EDS microanalysis of the glass ionomer fillers, we found that fluoroaluminosilicate glass (F-Al-Si) was the major component of the filler and the main source of fluoride in all the fluoride-releasing materials. Some of the fluoride-releasing materials also contain a small amount of sodium fluoride (NaF) and strontium fluoride (SrF₂).

In the present study, the findings for the amounts of fluoride released by the four glass ionomer cements were supported by the elemental microanalysis. The results showed that the content of fluorine was highest in the filler of Fuji IX (10.70±0.56 weight %), and was followed by Riva self-cure (8.33±3.38 weight %), Ketac Nano (6.91±0.06 weight %), and ChemFil ROCK (3.14±1.09 weight %). In a similar context, Riva self-cure possessed an increased level of aluminum (18.88±2.72 weight %) and less sodium (0.97±0.36 weight %) as compared to Ketac Nano with aluminum 8.24±0.87 weight % and sodium 1.61±0.60 weight %.

The results of the present study show that the high level of fluoride release on day 1 dropped significantly on day 2. This result is consistent with the results presented by Bahammam.¹⁸ Furthermore, the release of fluoride reached a plateau after day 5 that stayed relatively consistent for several days. These results are consistent with the previous studies which investigated the mean compressive and diametral tensile strength values of the four materials within a similar range of values.¹⁷⁻¹⁹ The methods used in these studies were considered to be standard for the preparation of the samples and the measurement of loads using a universal testing machine. Bresciani et al.¹⁹ compared the materials after 24 hr and 7 days and demonstrated that all the tested materials showed higher compressive and diametral tensile strengths.

Mahvi et al.²⁰ have examined the fluoride content of different commercial tea brands supplied in Iran and compared it with the release of fluoride using an infusion. Based on their findings, Mahvi et al.²⁰ concluded that, in Iran, there is a substantial contribution to the daily dietary intake of fluoride from drinking black tea. Dobaradaran et al.²¹ determined and compared the fluoride concentrations of 17

brands of bottled drinking water sold in Iran with the labeled values. The study found the mean fluoride content of the samples was 0.3 mg/L with a range of 0.00 to 0.59 mg/L. A similarly wide range was found by Dobaradaran et al.²² in the total fluoride content in particulate matter in the air at 20 sampling sites near the largest aluminum production plant in Iran at Arak city. The study measured the total fluoride particulate content in triplicate every 10 days at each site for a 3-hr period and found a mean fluoride content of 73.43 $\mu\text{g}/\text{m}^3$ with a range of 3.2–390.1 $\mu\text{g}/\text{m}^3$.

Yousefi et al.²³ studied fluoride exposure in children and teenagers in different regions of Poldasht city, Iran, and found that the level of fluoride in drinking water exceeded a predefined limit in 57% of the samples. Similarly, Mohammadi et al.²⁴ found that a high percentage of a population in rural areas of West Azerbaijan had symptoms of skeletal fluorosis. Rahmani et al.²⁵, found that the fluoride level in water was negatively associated to the pH and positively associated with the total dissolved solids (TDS), the Mg^{2+} level, and the Ca^{2+} level. Electrocoagulation, using iron and aluminum electrodes, has been used to remove fluoride from water.²⁶

The present study did not evaluate the diametral tensile strength and the compressive strength of the materials but it is known that the strength of these materials increases over time. The core focus of the study was the chemical properties and the release of fluoride ion of the restorative glass ionomer cements and the ignoring of the mechanical properties was a limitation of the study.

CONCLUSIONS

We found that conventional glass ionomer materials possessed a wide range of fluoride release values. The highest release of fluoride was observed in Fuji IX, followed by Ketac Nano, Riva self-cure, and ChemFil ROCK. For all of these materials, there was a significant difference in the release of fluoride except for Ketac Nano and Riva self-cure on day 1. The release of fluoride was high on day 1 and then decreased on day 2 ($p < 0.05$). For all the materials, the fluoride release on day 2 was higher in Fuji IX and Ketac Nano and lower in Riva self-cure and ChemFil ROCK. The results also showed that the release of fluoride decreased and stabilized at a low level after day 5. Fuji IX had the highest level of fluoride release of fluoride and ChemFil ROCK the lowest.

Considering the limitations of the study, future studies need to assess the compressive strength and the diametral tensile strength of the materials over a particular time period. The strength of materials is the core factor for determining the diametral tensile strength with respect to fluoride release value over a particular time period. Future studies of glass ionomer materials will need to evaluate the mechanical properties together with the release of the fluoride ion over a specific time period. Moreover, there is also need to assess the anticariogenic potential of glass ionomer dental restorative materials by measuring the amounts of fluoride and aluminum released. The results of the present study can not be generalized to the clinical scenario and future studies are required to substantiate our findings and to evaluate the fluoride recharging characteristics of restorative glass ionomer cements.

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