



## COMPARATIVE EVALUATION OF FLUORIDE RELEASE AND RECHARGING ABILITY OF GLASS IONOMERS UNDER DIFFERENT TEMPERATURE CONDITIONS – AN IN-VITRO STUDY

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Article Info	ABSTRACT
<p>Received 26/08/2015 Revised 17/09/2015 Accepted 28/09/2015</p> <p><b>Key words:</b> Glass ionomer, Fluoride release, Recharging.</p>	<p>The present study aimed to evaluate comparatively fluoride release and recharging ability of various glass ionomer cements under differing temperature conditions. In phase I of the study, fifteen discs (15 mm x 1 mm) each of Glass ionomer cements Fuji I™ luting cement (Group ‘A’), Fuji IX™ high strength posterior (Group ‘B’), Chemflex™ restorative (Group ‘C’) and Fuji II LC™ resin – modified (Group ‘D’) (N=10 TOTAL) were prepared and stored in double distilled water at 4°C, 37°C and 55°C. The fluoride released in solution was measured on 1<sup>st</sup>, 2<sup>nd</sup>, 5<sup>th</sup>, 7<sup>th</sup> and 14<sup>th</sup> day, using an Ion selective electrode. For phase II, an additional nine specimens of the same materials were prepared and stored in double distilled water at 37°C for 30 days, followed by exposure to fluoride solution at same temperature using 200 ppm sodium fluoride solution (Fluoritop™) for five minutes. The fluoride hence re-released was measured after storage in fluoride solution. Statistical analysis showed that at all temperatures, the Group A showed greatest fluoride release at all measurement points, followed by Group D, and Group B and C respectively. Temperature strongly influenced the fluoride releasing profile of glass ionomers.</p>

### INTRODUCTION

Among several dental restorative materials containing fluoride [1], the glass ionomer cements has been most successful in terms of its anticariogenic potential [2]. Ever since the introduction of glass ionomer cement by Wilson and Kent in 1969 [3], its inherent properties of true chemical adhesion to tooth structure, biocompatibility, aesthetics and additional benefit of

continuing fluoride release has intrigued researchers and clinicians alike [4, 5].

From clinical point of view, the extent and longevity of fluoride release from GICs is important for its sustained cariostatic activity. In addition, it has been noted that decreased physical properties are associated with increased fluoride release, so research into the development of fluoride containing materials is ongoing



with the hope of maintaining the physical properties of these materials and providing long term fluoride release [6]. Initial rapid fluoride release that lasts only a fraction of expected life time of restoration would not be of much significance. Tooth pastes with fluoride, topical fluoride solutions and fluoride rinses have potential to recharge fluoride depleted from these restorative materials [7].

For the long-term anticariogenic activity of GICs efficacy of the glass ionomers, fluoride recharging ability and re-release is essential [8], since there is rapid reduction in amount of fluoride released after an initial high release rate [9].

Several studies have addressed different aspects of fluoride releasing properties of GICs as function of the type of cement used, pH of storage solution, type of storage medium, surface treatment of the specimens and durations of study period. However, the effect of temperature of the oral cavity on fluoride release and recharging ability of GICs is still unclear. Only a few studies have been conducted to understand the effect of this parameter on both fluoride release and recharging ability of GICs [10].

Keeping in mind the broad temperature fluctuations taking place in the oral environment, the purpose of the present study was to evaluate comparatively the effect of temperature on the pattern of fluoride release and recharging ability of glass ionomer cements.

## MATERIALS AND METHODS

The study included following restorative materials: conventional luting glass ionomer (Fuji I, GC Corporation, Tokyo, Japan); a high strength posterior restorative glass ionomer (Fuji IX GC Corporation, Tokyo, Japan); a conventional restorative glass ionomer (Chemflex<sup>TM</sup>, DENTSPLY) and a light cured resin modified glass ionomer (Fuji II LC, GC Corporation, Tokyo, Japan). They were divided in to four groups (Table 1).

A Teflon mold (15 mm in diameter x 1 mm thick) was used to prepare disc specimens of each material. Each material was handled as per the manufacturer's instructions at room temperature ( $23 \pm 1^\circ\text{C}$ ), by the single operator, so as to reduce the variables induced. The mixed material was packed into the Teflon mold placed on the glass slab and was covered with mylar strip under pressure to expel excess material from the mould. The auto-cured glass ionomers were allowed to set before their removal from the mold whilst Fuji II LC samples were light cured for 20 seconds using a light-curing device with a visible light intensity of 500 mW/cm<sup>2</sup> (Dabi Atlante, Ribeirão Preto, SP, Brazil).

A pilot study was done, taking significance level to be 0.05 and power of the study was found to be 80%, the sample size for four comparative groups in the study came out to be 54, thereby tentative sample of 60 was taken for the study. Fifteen disc specimens of each material were prepared for phase I of the study, which aimed to measure the fluoride

release from various GICs under different temperature conditions. The specimens thus prepared were stored at 37<sup>0</sup>C for 24 hours in 100% relative humidity. After this they were finished and polished to create a smooth surface. All the materials were checked for their dimensions to ensure uniformity of sample size using Vernier's calipers and digital weighing balance.

Following this each disc specimen was stored in 5 ml of double distilled water in plastic test tubes. The water was changed daily in the first week and every third day thereafter. Each group was further divided into three subgroups with five specimens each (n=5) to be stored at 4<sup>0</sup>C, 37<sup>0</sup>C and 55<sup>0</sup>C. These temperature conditions were achieved through the use of a refrigerator, water bath and hot air oven respectively. These equipments had a digital temperature read outs which were checked prior to collection of sample for each measurement.

Samples from the storage solution were collected on the 1<sup>st</sup>, 2<sup>nd</sup>, 5<sup>th</sup>, 7<sup>th</sup> and 14<sup>th</sup> day of the study. After each measurement of fluoride concentration, the solution was discarded and 5 ml of fresh double distilled water was placed into the storage container.

The fluoride release was measured with combination fluoride electrode (Orion Research, Lumberton NJ, USA). The fluoride electrode is an ion-selective sensor. The key element in the fluoride electrode is the laser-type doped lanthanum fluoride crystal across which a potential is established by fluoride solutions of different concentrations. The crystal contacts the sample solution on one side and an internal reference solution on the other. A potential is established by the presence of fluoride ions across the crystal, which is measured with a device called ion meter or with any modem having an expanded millivolt scale. Fluoride activity depends on the total ionic strength of the sample.

Calibration of the fluoride electrode is determined before each measurement session using standard fluoride solutions (Orion Research Inc.) containing 0.1, 0.5, 1, 5, and 10 ppm fluoride.

Exactly 5 ml of storage solution, used for immersion of individual disk specimens, was dispensed into a beaker. An equal amount of TISAB (total ionic strength acetate buffer) solution was added to stabilize the pH. The total volume was sufficient to immerse the electrode and permit the operation of stirring bar.

Electrodes were immersed in the sample solution and solution was stirred with magnetic stirrer. Stirring before immersion of electrodes should be avoided because entrapped air around the crystal can produce erroneous reading. Electrodes were left in the solution (average of 3 min) until reading was constant before taking the final reading. Electrodes were withdrawn, rinsed with distilled water, and blotted dry between every reading. For all groups, fluoride ion concentration was calculated in parts/million/microgram of fluoride /sq cm.

Phase II of the study; aimed to assess the fluoride recharging ability and re-release after immersing the



specimens in 200 ppm sodium fluoride solution (Fluoritop mouth rinse). Another 15 disc specimens (15 mm x 1mm) of the same materials used in phase I were prepared and were stored at 37°C for 30 days in double distilled water. The double distilled water was changed daily in first week and every three days thereafter.

Next, three subgroups of each materials (n = 5) were stored at 40C, 370C and 550C in 200 ppm sodium fluoride solution for 5 minutes. Fluoride thus re-released was measured two days prior and two days after the exposure to the solution using ion selective electrode as described above (FLOWCHART 1).

**FLOWCHART 1**

SIXTY (N=15 ) DISC- SHAPED SPECIMENS OF SAME MATERIALS USED IN PHASE I OF THE STUDY WERE MADE



ALL WERE STORED IN DOUBLE DISTILLED WATER AT 37 DEGREES CELSIUS FOR 30 DAYS.



THE FLUORIDE RELEASE IN THE SOLUTION WAS MEASURED AT DAY 30<sup>th</sup>



AFTER THIS (N=5) OF EACH GROUP WERE EXPOSED TO FLUORIDE SOLUTION (FLUORITOP 200PPM) AT 4, 37, 55 DEGREES CELSIUS FOR 5 minutes.



FLUORIDE RE-RELEASED TWO AFTER RECHARGING WITH FLUORIDE SOLUTION AT THREE DIFFERENT TEMPERATURE CONDITIONS WAS MEASURED AND COMPARED.

**Statistical methods used**

To see the intergroup differences Analysis of Variance (one way-ANOVA) and post-hoc test (Tukey HSD) was carried out. This test has been proven to be specifically critical and hence reliable in the samples and

methodology used in similar clinical research works documented as per literature. The confidence limit of the study was kept at 95%, hence a “p” value below 0.05 indicated a statistically significant difference All the statistical tests were done through SPSS 16.0 (SPSS Inc, Chicago II) for Windows analytical software (Microsoft, Inc. Redmond, WA, USA).

**RESULTS**

For phase I of the study, all materials showed a cumulative fluoride release, which was maximum at higher temperature ranges *i.e.* at 55°C, as compared to release at 37°C and 4°C. The release was lowest at 4°C. The fluoride release on day 1 was ranked as (‘p’ <0.05)

**Fuji I > Fuji II LC > Fuji IX and Chemflex™ (Fig. 1)**

On day 2 the amount of fluoride released was less than that released on day 1. The order, however remained the same.

**Fuji I > Fuji II LC > Fuji IX > Chemflex™ (Fig. 2)**

Similarly on day 5, 7 and 14 of the study, the amount of fluoride decreased gradually for all the materials tested. The order of fluoride released on day 5, 7 and 14. was –

**Chemflex™ < Fuji IX < Fuji II LC = Fuji I (Fig.3, 4, 5)**

Hence luting glass ionomer (Fuji I) showed greatest fluoride release among all the materials, irrespective of storage temperature (‘p’ <0.05). The fluoride release of resin modified glass ionomers, (Fuji II LC) was significantly higher than that of higher viscosity glass ionomers (Fuji IX and Chemflex™) (‘p’ <0.05). TABLE 2

It was found that the fluoride re-released after recharging at 55°C was significantly greater than for 4°C and 37°C for all materials tested (‘p’<0.05). TABLE 3 Statistically significant differences were observed for the higher viscosity glass ionomers *i.e.* Fuji IX and Chemflex™ and resin modified glass ionomers (Fuji II LC) and not for luting glass ionomers (Fuji I) (‘p’ <0.05).

**Fuji IX <Chemflex™ < Fuji II LC = Fuji I (Fig.6)**

**Table 1. Number and Type of Materials Selected for Study**

Group	No. of Samples	Types	Manufacturer
A	15	Fuji I Luting GIC (Luting Glass Ionomer)	GC Corporation, Tokyo, Japan
B	15	Fuji IX High Strength Posterior restorative GIC	GC corporation, Tokyo, Japan
C	15	Chemflex™ – Conventional Restorative Glass Ionomer	Dentsply
D	15	Fuji II LC–Resin Modified Light Cured GIC	GC Corporation, Tokyo, Japan

**Table 2. Mean fluoride released from different groups at differing temperature conditions on day 1, 2, 5, 7 and 14**

Groups	Day 1			Day2			Day 5			Day 7			Day 14		
	4°C	37°C	55°C	4°C	37°C	55°C	4°C	37°C	55°C	4°C	37°C	55°C	4°C	37°C	55°C
A (Fuji I)	1.460	4.583	8.897	0.780	2.443	6.073	0.73	2.13	4.40	0.68	1.66	4.14	0.673	1.727	4.213
I)	0.820	3.270	5.627	0.740	1.383	2.877	3	3	7	7	7	7	0.637	1.330	4.340

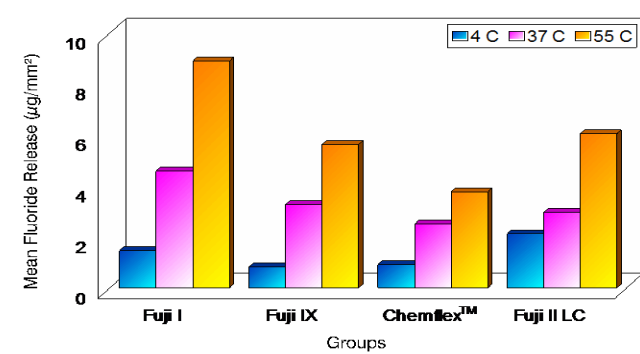


B (Fuji IX)	0.913	2.500	3.767	0.720	1.450	1.977	0.66	1.51	4.29	0.63	1.31	4.02	0.673	1.343	1.417
C(chemflex)	2.123	2.963	6.047	1.047	2.880	5.860	0	0	7	7	0	7	1.267	1.753	4.407
D(Fuji II LC)							0.65	1.32	1.45	0.61	1.33	1.20			
							0	7	0	3	3	3			
							0.99	2.40	4.52	1.13	1.13	4.18			
							0	0	7	3	3	7			

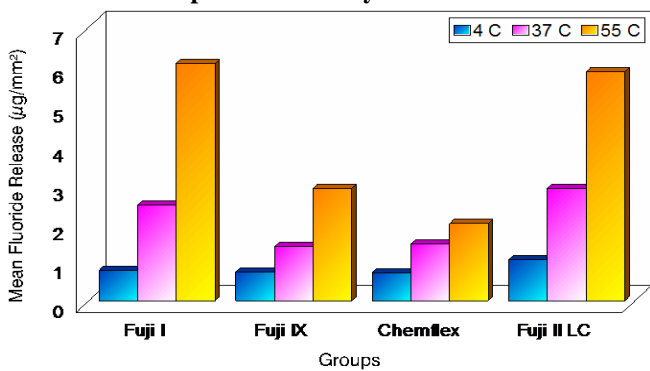
**Table 3. Mean fluoride released from different groups, 2 days after exposure to Fluoritop solution at differing temperature conditions**

Groups	Fluoride Released 2 Days After Exposure to Fluoride Solution		
	4°C	37°C	55°C
A (Fuji I)	4.313	5.513	6.510
B (Fuji IX)	1.367	2.217	3.863
C(chemflex)	1.977	3.867	4.060
D(Fuji II LC)	3.207	4.893	6.467

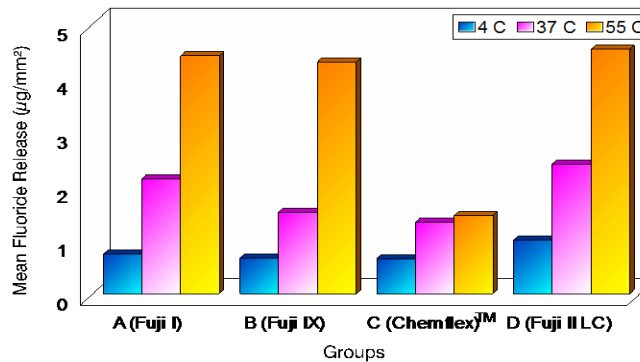
**Fig 1. Intergroup Comparison of Mean fluoride release at different temperatures at day 1**



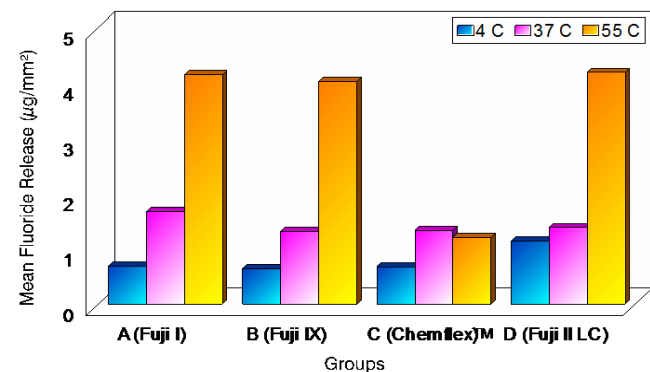
**Fig 2. Intergroup Comparison of Mean fluoride release at different temperatures at day2**



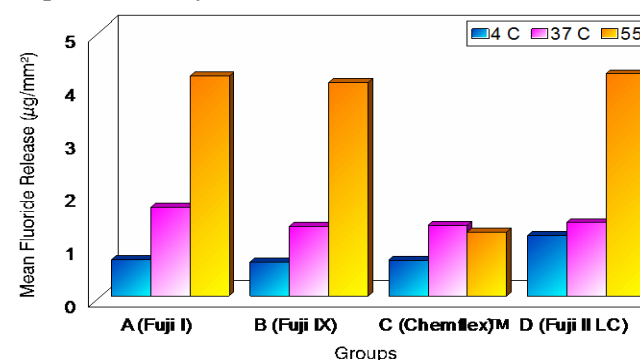
**Fig 3. Intergroup Comparison of Mean fluoride release at different temperatures at day5**



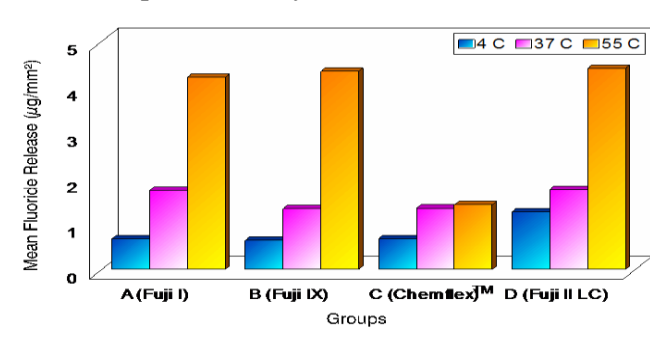
**Fig 4. Intergroup Comparison of Mean fluoride release at different temperatures at day 7**



**Fig 5. Intergroup Comparison of Mean fluoride release at different temperature sat day14**



**Fig 6. Inter group Comparison of Mean fluoride release at different temperature sat day14**



## DISCUSSION

The study simultaneously measured the fluoride release and uptake property of various glass ionomers, in an attempt to simulate oral dynamics more accurately, because GIC restorations release and uptake fluoride simultaneously. The use of standardized disc specimens of the materials permitted comparison of fluoride released to be expressed as fluoride release per millimeter square of the cement, suggesting that fluoride release occurred from whole body of the specimen and not only from the surface.

The fluoride release and recharging ability of glass ionomer cements used in the study varies according to the storage temperature. The amounts released at 40C, 37C and 55C were significantly different from each other ( $p < 0.05$ ). It has been suggested by [11] that the leached fluoride is derived from the unreacted glass particles and from fluoroaluminophosphates in the matrix, and is released without affecting the physical properties of the cement. The release occurs by three distinct mechanisms namely; surface wash off, diffusion through pores and cracks, and bulk diffusion. Factors effecting the concentration of fluoride release include [12] temperature, time of immersion into aqueous media, pH, mixing time, composition of aluminosilicate glass and polyalkenoic acid used and solubility of cement. Water diffusion through the matrix drives fluoride ions to surface, where they can be released into the storage media [13, 14]. Also the fluoride release of glass ionomers was linearly proportional to  $t^{1/2}$  within a relatively short time (a few days) after setting [15]. The researchers agree that the greater quantities of fluoride are released during first few days after that it reaches a constant level [16].

The present study suggests that fluoride release from glass ionomers under different temperatures is a diffusion controlled process. The fluoride release significantly increased with increase in temperature. It can be supported by Arrhenius equation, which implies that rate constant increases as the temperature is raised. The temperature ranges were selected keeping in mind the

fluctuations taking place in the oral cavity during consumption of hot and cold food stuffs.

Among all the materials tested in the study luting glass ionomers showed greatest fluoride release and recharging ability at all temperature ranges, owing to their ability to support high water diffusion in the matrix [17]. The fluoride release could be ranked as luting > resin modified > high viscosity glass ionomers. The fluoride release for all materials was highest at 55C and lowest at 40C. The fluoride release of all glass ionomers was considerably reduced after 30 days of storage before recharging. Also the fluoride re-release was highest for the specimens recharged at 55C. The amount of fluoride re-released 2 days after recharging was lowest for high viscosity glass ionomers.

The lower values of fluoride release from light cured glass ionomer could be attributed to occurrence of photo chemical reactions, which reduces the early sensitivity to the moisture. The resin matrix could reduce the diffusion of water into the cement; thus reducing the elution of unbound fluoride in the material matrix [18, 19]. The type and amount of resin employed for light curing may affect the fluoride release.

The ultimate goal of correlating fluoride release with actual caries reduction is an objective that can only be achieved by completing controlled clinical trials on materials with well characterized kinetics of fluoride release. The results of the present study are quite clear and this aspect may be important in developing regimes for improving the delivery of topical fluoride products.

## CONCLUSIONS

1. An increase environmental temperature increased both release and recharging ability of glass ionomers.
2. A low environmental temperature should be avoided during topical Fluoride application.
3. Both fluoride release and recharging ability of luting glass ionomers is highest among all materials tested at all temperature ranges.

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