

# Comparative Evaluation of Fluoride Release and Recharge of Zirconia-reinforced, Resin-modified, and Conventional Glass Ionomer Cements

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## ABSTRACT

**Aim and objective:** To quantitatively assess and compare the fluoride release and recharge of zirconia-reinforced, resin-modified, and conventional glass ionomer cement.

**Materials and methods:** Fifteen disk-shaped pellets having dimension 5 × 3 mm were made in zirconia-reinforced (Zirconomer improved, Shofu), resin-modified (GC Gold label LC), and conventional glass ionomer (GC Gold label) cements concurring to the manufacturer's instruction. Each pellet was individually dipped in 10 mL deionized water in an air-tight container for 24 hours. After 24 hours, the specimens were removed and the elutes were collected. This procedure was repeated daily. The quantity of fluoride ions released in the solution was analyzed after 24 hours, 7th day, and 15th day. After 15 days, all samples from each group were recharged with 1.23% APF gel for 4 minutes and were reimmersed in 10 mL of fresh deionized water. Fluoride analysis was carried out on 16th, 22nd, and 30th day by a digital ion analyzer having a specific fluoride ion electrode.

**Results:** The amount of fluoride released was highest for zirconia-reinforced GIC in comparison to conventional GIC and RMGIC. There was also a statistically significant difference in fluoride release after recharge for zirconia-reinforced GIC when compared with conventional GIC and RMGIC.

**Conclusion:** Zirconia-reinforced GIC has added fluoride release and recharging property than conventional GIC and RMGIC.

**Clinical significance:** Zirconia-reinforced GIC having superior compressive strength and fluoride release is an assuring material for restoration holding anticariogenic property.

**Keywords:** Anticariogenic, Fluoride, Glass ionomer, Release and recharge, Zirconia.

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## INTRODUCTION

Restorative materials that release fluoride play an imperative role in the diminution of caries. Fluoride ion interferes with sugar metabolism by bacteria and hence reduces acid production that results in demineralization and caries formation. These restorative materials act as fluoride reservoirs hence reduce secondary caries and have a protective role in high caries risk individuals.<sup>1</sup> However, the fluoride release and uptake characteristics among these materials fluctuate significantly. Over time, these materials exhibit a decline in fluoride release which negatively affects the shielding characteristics against secondary caries.<sup>2</sup> The ability of these materials to adsorb fluoride from the contiguous milieu, also known as recharging provides for long-term caries inhibitory effect and also adds to the total fluoride released from the material. Among the restoratives, glass ionomer cement is acclaimed to have a remarkable fluoride recharging ability.<sup>3</sup>

Conventional glass ionomers and resin-modified glass ionomers are among the most widely used fluoride-releasing materials. Though the major shortcomings of GIC are its inferior mechanical properties like brittleness, toughness, and strength.<sup>4</sup> Consequently, it cannot be used in load-bearing posterior areas. Composites on the contrary have good mechanical properties but meager fluoride release.<sup>5</sup>

In an attempt to increase the mechanical properties, silver-tin alloy, gold, or stainless steel were added to GIC. Although none had better fluoride release and recharging property than conventional GIC.<sup>3</sup>

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Currently, zirconia-reinforced GIC is evidenced to have strength consistent with amalgam along with protective attributes of GIC. The exclusive characteristic of zirconia termed transformation toughening gives it greater strength, toughness, and hardness.<sup>6</sup> Zirconia-reinforced glass ionomer having superior mechanical properties and fluoride release and recharge could be used beneficially in caries-prone individuals. Consequently, the objective

of this study was to evaluate the fluoride release and recharge potential of zirconia-reinforced, resin-modified, and conventional glass ionomer cement.

## MATERIALS AND METHODS

### Study Design

This *in vitro* study was conducted at the Geological Survey of India, Thiruvananthapuram, Kerala. A total of 45 samples were fabricated, with 15 each in three groups.

### Specimen Preparation

Disk-shaped pellets, 15 each were made from the three restorative materials, zirconia-reinforced (Zirconomer improved, Shofu dental, Asia Pacific Ltd., Kyoto, Japan), resin-modified (GC Gold Label 2 LC, GC Corporation, Tokyo, Japan), and conventional glass ionomer cement (GC Gold Label Universal Restorative, GC Corporation, Tokyo, Japan). An adequate amount of material was placed into a disk-shaped mold (custom-made Teflon mold) with a specification of 5 × 3 mm which ensured standardization of the shape and size of each pellet. Initially, a layer of material was placed inside the mold and a small part of 15 mm floss (Colgate) was incorporated into it, then the remaining volume of material was added over it and allowed to set. RMGIC pellets were light-cured after mixing, with LED curing light (Woodpecker, LED curing light) from both sides of the specimens for 20 seconds. The chemically activated materials were allowed to be set for 20 minutes at room temperature before removing them from the mold. The specimens were then ground with a dry 800-grit silicon carbide paper and their diameter and thickness were measured. Each of the pellets was separately immersed in 10 mL of deionized water, in an air-tight container, and kept in an incubator (Salec) at 37°C until analysis was completed.

### Fluoride Analysis

Subsequently, 24 hours later, the containers were thoroughly agitated and the samples were removed, dried, and then returned into a fresh plastic container containing 10 mL of deionized water and the elutes were collected. This process was done constantly every day. The quantity of fluoride ions released in the solutions was analyzed after 24 hours, 7th day, and 15th day.

### Fluoride Recharge

Samples were exposed on the 15th day to 1.23% APF gel for 4 minutes and washed copiously with deionized water for 10 seconds and dried on an absorbent paper. Each sample after fluoride application was suspended in plastic containers of 10 mL of deionized water and incubated at 37°C for 24 hours.

### Fluoride Re-release

After 24 hours, the samples were removed from the container, washed with 1 mL of distilled water by a syringe. The pellets were dried with absorbent paper and then put back in 10 mL of fresh deionized water. Deionized water thus collected was then assessed for fluoride release on 16th, 22nd, and 30th day, respectively.

### Measurement of Fluoride Release

For the evaluation of fluoride released from samples in deionized water, an ion-selective electrode (ISE) connected to an ion meter (Orion, USA) was used. A TISAB III (total ionic strength adjustment buffer) solution was added to the solution to control pH and avoid the formation of fluoride complexes. The TISAB III solution frees

fluoride ions bound to hydrogen and also eliminates hydroxyl ion interference, and ensures exact measurement of the total fluoride content.

### Statistical Analysis

Statistical analysis was done using analysis of variance (ANOVA) for multiple groups and Tukey's multiple *post hoc* procedure (T HSD) for pairwise comparison of two groups. Repeated measure ANOVA was used to compare fluoride release on the 24th hour, 7th day, 15th day, 16th day, 22nd day, and 30th day.

## RESULTS

The mean *F* release values (ppm) of tested materials after 24 hours, 7th day, 15th day, and release after recharge on 16th day, 22nd day, and 30th day are represented in Table 1. All the materials demonstrated greater fluoride release in the initial stage and then decreased abruptly. The mean fluoride release of Zirconomer at 24th hour, 7th day, 15th day, 16th day, 22nd day, and 30th day as 28.25, 3.8753, 2.3480, 7.6427, 2.1420, and 1.4653, respectively. Mean fluoride release of conventional GIC at 24th hour, 7th day, 15th day, 16th day, 22nd day, and 30th day is 4.6533, 0.5490, 0.3203, 2.0393, 0.3151, and 0.2749, respectively. Mean fluoride release of RMGIC at 24th hour, 7th day, 15th day, 16th day, 22nd day, and 30th day is 4.3715, 0.6254, 0.4581, 2.6967, 0.4949, and 0.3835, respectively. *p* value was significant in all cases (<0.05).

Zirconia-reinforced GIC released the greatest amount of fluoride followed by RMGIC and GIC. Higher fluoride release was observed during the first day, decreasing in the subsequent days. Re-release after recharge was also highest for zirconia-reinforced GIC when compared with the other two materials.

Table 2 presents a pairwise comparison of fluoride release among three materials at various time periods. It was observed that zirconia-reinforced glass ionomer exhibited higher fluoride release compared with the other two materials at all time periods (Fig. 1).

## DISCUSSION

Evaluation of initial fluoride release among the three materials indicated that zirconia-reinforced GIC had more fluoride release than RMGIC and GIC at the 24th hour, 7th day, and 15th day. The fraction of fluoride in restorative materials should be high without affecting the mechanical or physical properties of the material. The fluoride release pattern established in the present study was in concurrence to Tiwari et al.'s study where zirconomer revealed greater fluoride release at 1st, 7th, 14th, and 21st days compared with GIC II, GIC IX, and compomer. According to Tiwari's study on first-day fluoride release of zirconomer was (33.33), day 7 (40.3), day 14 (29.69), and day 21 (15.43) ppm, respectively, whereas GC II it was 16.7, 19.56, 10.46, and 5.51 ppm, for GC IX it was 16.96, 19.42, 10.27, and 5.44 ppm, and for compomer 2.11, 3.13, 2.00, and 1.07 ppm, respectively, on 1st, 7th, 14th, and 21st days.<sup>7</sup>

Fluoride release from glass ionomer cements can be explained by three mechanisms: surface loss, diffusion through pores and cracks, and bulk diffusion.<sup>8</sup> The highest fluoride release from the restorative materials was observed on the first day, i.e., "burst effect" and declined thereafter.<sup>8</sup> In this particular study, with time the rate of fluoride release lessened, which is in accordance with previous studies by Neelakantan et al., Nicholson, and Cardoso et al.<sup>9-11</sup> The present study revealed mean fluoride release of zirconomer on day 1 as the highest (28.25), day 7 (3.87), and day 15 (2.34) ppm after

**Table 1:** Mean fluoride release

Time period	Variable	N	Mean	Std. deviation	Std. error	p value
24 hours	Z-group	15	28.2527	3.30459	0.85324	0.001*
	G-group	15	4.6533	0.40181	0.10375	
	R-group	15	4.3715	0.47638	0.12300	
Day 7	Z-group	15	3.8753	0.76027	0.19630	0.001*
	G-group	15	0.5490	0.10961	0.02830	
	R-group	15	0.6254	0.04005	0.01034	
Day 15	Z-group	15	2.3480	0.51551	0.13310	0.001*
	G-group	15	0.3203	0.04486	0.01158	
	R-group	15	0.4581	0.03203	0.03157	
Day 16	Z-group	15	7.6427	1.28192	0.33099	0.001*
	G-group	15	2.0393	0.23708	0.06121	
	R-group	15	2.6967	0.23916	0.06175	
Day 22	Z-group	15	2.1420	0.58612	0.15133	0.001*
	G-group	15	0.3151	0.07325	0.01891	
	R-group	15	0.4949	0.01312	0.00339	
Day 30	Z-group	15	1.4653	0.28598	0.07384	0.001*
	G-group	15	0.2749	0.07003	0.01808	
	R-group	15	0.3835	0.01875	0.00484	

p value < 0.05 is considered statistically significant

\*Significant at the 0.05 level

**Table 2:** Pairwise comparison of the three groups among the time periods

Time period	(I) var	(J) var	Mean difference (I-J)	Sig.
24 hours	Z-group	G-group	23.59933*	0.001*
		R-group	23.88120*	0.001*
Day 7	G-group	R-group	0.28187	0.917
	Z-group	G-group	3.32633*	0.001*
Day 15		R-group	3.24993*	0.001*
	G-group	R-group	-0.07640	0.885
Day 16	Z-group	G-group	2.02773*	0.001*
		R-group	1.92040*	0.001*
Day 22	G-group	R-group	-0.10733	0.607
	Z-group	G-group	5.60333*	0.001*
Day 30		R-group	4.94600*	0.001*
	G-group	R-group	-0.65733	0.059
Day 30	Z-group	G-group	1.82693*	0.001*
		R-group	1.64707*	0.001*
Day 30	G-group	R-group	-0.17987	0.328
	Z-group	G-group	1.19040*	0.001*
Day 30		R-group	1.08187*	0.001*
	G-group	R-group	-0.10853	0.201

p value < 0.05 is considered statistically significant

\*Significant at the 0.05 level

the initial burst. After fluoride recharge on day 16, it was (7.64), day 22 (2.14), and day 30 (1.4) ppm, respectively. On the other hand for GIC fluoride release and re-release was, respectively, day 1 (4.6), day 7 (0.5), day 15 (0.32), day 16 (2.03), day 22 (0.31), and day 30 (0.27) ppm, respectively. RMGIC fluoride release and recharge was correspondingly as, day 1 (4.37), day 7 (0.62), day 15 (0.45), day 16 (2.69), day 22 (0.49) and day 30 (0.38) ppm.

The initial high level of fluoride release might be due to erosion from the surface, while the fairly constant fluoride release during the following days might be due to the diffusibility of fluoride through cement pores and fractures.<sup>12</sup> Bulk fluoride diffusion ensues during the maturation period as a result of contact of the material with the storage medium.

Virmani et al. showed fluoride release of zirconomer was constant from 14 hours to 10 days, and then decreased.<sup>13</sup> The rapid elution pattern of fluoride by zirconomer may be ascribed to finely controlled micronization of the glass ionomer particles. The fact that smaller glass particles provide a larger surface area hence greater reactive interface was reported in numerous studies. Hence, acid-base reactivity is accelerated leading to the enhanced release of fluoride from these materials.<sup>11,14,15</sup>

After fluoride recharge on the 15th day by APF gel, fluoride release from zirconia-reinforced GIC did not attain the initial level but was superior in comparison to the initial fluoride levels of GIC and RMGIC. The fluoride released was greater than GIC and RMGIC on the 16th day suggesting superior recharging of zirconia-reinforced GIC. Fluoride re-release was highest for zirconia-reinforced GIC compared with the other two materials on 22nd and 30th days also. On reviewing the literature, studies that investigated the fluoride recharge of zirconia-reinforced GIC in comparison to conventional GIC and RMGIC were not available. According to Paul et al., improved zirconomer showed higher release and re-release compared to cention.<sup>16</sup>

The porosity of the materials might have a great influence on the amount of fluoride released before and after recharge. Higher porosity allows deeper diffusion of the recharge agent. But this is detrimental to the mechanical properties. But, zirconia-reinforced glass ionomer shows both higher strength and better fluoride-releasing properties.<sup>7,17</sup> This material reinforced with nano-zirconia fillers is responsible for the improved mechanical properties which make it suitable in posterior load-bearing areas as per various studies.<sup>17,18</sup> The zirconia fillers have a property of transformation

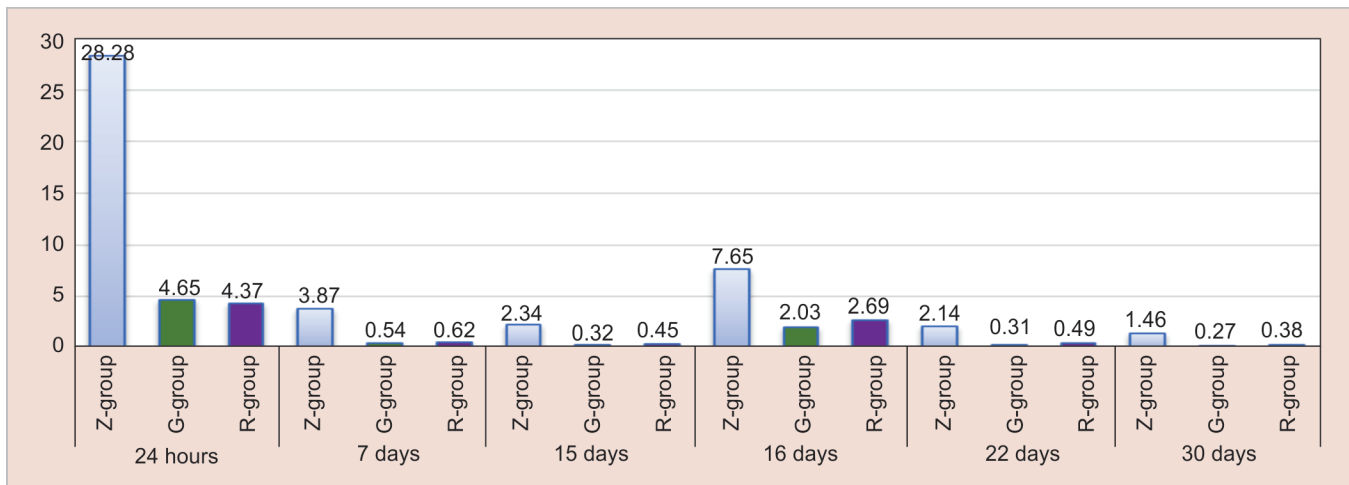


Fig. 1: Mean daily fluoride ions released from each material for 30 days

toughening by which it can stop the growth of cracks. The stress due to crack propagation causes the transformation of zirconia in the tetragonal phase to a stable monoclinic phase, and a slight increase in volume during this transformation also enhances compressive strength.<sup>19</sup> The combination of alumina with yttrium stabilized zirconia provides the material a higher elastic modulus and greater toughness.<sup>19–23</sup>

## LIMITATIONS

This is an *in vitro* study that may not exactly simulate the oral conditions. The oral environment is dynamic and different from *in vitro* conditions. Therefore, results may not exactly extrapolate the clinical scenario. It is also a short-term study and the exact mechanism of fluoride recharge need to be further explored.

## CONCLUSION

The present study found that zirconia-reinforced glass ionomer cement has better fluoride release and recharging properties compared to glass ionomer and RMGIC. Fluoride release from restorative materials is imperative in clinical dentistry. Further *in vivo* studies should be done to evaluate fluoride release and recharge in real environmental conditions.

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