ORIGINAL RESEARCH

Comparative Assessment of Fluoride Release of Two Different Bioactive Composites with a Conventional Glass Ionomer Cement: An *In Vitro* Study

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Aims and background: Dental caries prevention and eradication pose significant challenges for dentists globally. By combining with inorganic tooth structure to generate the caries-resistant complex fluorapatite, fluorides serve a critical role in preventing dental cavities. Consequently, materials used for dental restoration containing fluoride can act as reservoirs of fluoride, increasing levels of fluoride in saliva, plaque, and dental tissues, thereby aiding in caries prevention and reducing the risk of secondary caries. So, this study aimed to evaluate and compare the fluoride release patterns of three different dental restorative materials.

Materials and methods: A total of 15 disc-shaped specimens were prepared from three different restorative materials using Teflon molds and plates. Five samples of group I—GC Gold Label 2, group II—Activa™ BioACTIVE, and group III—Activa™ Pronto were suspended in 20 mL of deionized water. The experiment spanned 28 days, with fluoride release measurements taken on the 7th, 14th, 21st, and 28th days using a digital ion analyzer.

Results: Significantly more fluoride was released by GC Gold Label 2 (conventional glass ionomer cement), than by the bioactive composites Activa™ BioACTIVE and Activa™ Pronto.

Conclusion: Fluoride ions were released by all the three dental restorative materials. Conventional glass ionomer cements had shown superior fluoride release in comparison with bioactive composites.

Keywords: Dental caries, Fluorides, Secondary caries.

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Introduction

In addition to restoring dental cavities, the main goal of dental care should be to lower the risk of developing new cavities.[1] Fluoride has a well-established role in preventing tooth cavities. Fluorides are known to have anticariogenic effects through two mechanisms: (1) Reducing demineralization and enhancing remineralization by strengthening enamel's resistance to acid attack by converting hydroxyapatite to fluorapatite crystals; and (2) inhibiting the enolase enzyme, which stops microbial metabolism and replication.[2] Therefore, fluoride-containing dental restorative materials serve as fluoride reservoirs, raising fluoride levels in saliva, plaque, and hard tooth tissues to help prevent or lessen cavities. Additionally, it aids in preventing secondary caries in teeth that have already been repaired.[3]

Particularly noteworthy is glass ionomer cements that has exceptional capacity to release fluoride, which aids in halting enamel demineralization, encouraging remineralization, lowering plaque development, and ultimately preventing dental cavities. They do not have good mechanical qualities, though.[4] So, in this regard, there was evolution of novel hybrid restorative materials such as resin-modified glass ionomer cement, compomers, and giomers that combine properties of resin composite (mechanical strength, esthetics, and high bond strength) and glass ionomer cement (self-adhesive properties, moisture tolerance, and ion release). They had better mechanical properties but their fluoride-releasing capacity was lower than that of conventional glass ionomer cements.[5]

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Due to these limitations, bioactive composites were introduced. The development of bioactive composites has significantly advanced the field of restorative dentistry, offering materials that not only restore the structural integrity of teeth but also contribute to the remineralization process. Unlike conventional composites, they can release fluoride and other beneficial ions, which helps to remineralize tooth structure and protect against secondary caries.[5] This study aims to provide a comparative assessment of fluoride release from two distinct bioactive composites, examining their potential to maintain long-term fluoride release, which is essential for sustained anticariogenic effects.

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Materials and Methods

The reason and intention of the work were explained and ethical clearance was obtained with institutional review board (no: VDCW/IEC/351/2023).

This study consisted a sum of 15 samples categorized in three groups concerned to three dental restorative materials. Materials used were conventional glass ionomer cement—group I: GC Gold Label 2, bioactive composites—group II: Activa™ BioACTIVE, and group III: Activa™ Pronto.

A total of 15 disc-shaped specimens (n = 5 per group) were fabricated using Teflon molds measuring 20 mm in diameter and 1.5 mm in thickness, according to ISO specification #7489 (Fig. 1). Powder and liquid of glass ionomer cement were hand-mixed on a mixing pad placed on glass slab with a plastic spatula, according to the manufacturer's recommended ratio (mixing time: 30–40 seconds) until a glossy consistency was achieved. The mix was inserted into the molds, covered with Mylar strips and a Teflon plate to expel excess material. After initial set (2 minutes), specimens were coated with a thin layer of petroleum jelly to prevent dehydration. Bioactive composites were dispensed into the molds and covered with a Mylar strip and Teflon plate. Specimens were polymerized for 20 seconds using an light emitting diode (LED) curing unit (SS WHITE) with wavelength 470 nm. The curing tip was positioned perpendicular to the specimen surface at a distance of 2 mm.

During fabrication, unwaxed dental floss was embedded into each specimen to enable suspension in the storage medium.

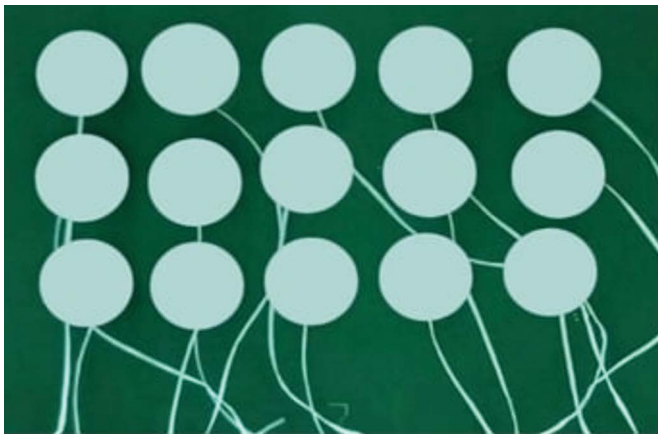

Fig. 1: Specimens prepared

Specimens were stored in individual plastic vials containing 20 mL of deionized water (pH 7.0) (Fig. 2) and incubated at 37°C for 24 hours. After 24 hours, the samples were removed from the vials, and the medium was checked for fluoride ions using a digital ion analyzer (ORION Star A214) with a fluoride-specific electrode. Specimens were then rinsed, dried, and immersed in a new vial containing fresh solution of 20 mL deionized water and placed in the incubator until the next reading. Similarly, incremental fluoride measurements were done at 7th, 14th, 21st, and 28th day. Cumulative fluoride measurements were done by adding the incremental values obtained from 1st day to 28th day (Figs 3 and 4).

Fluoride release was calculated using the formula:

$$Fluoride\,release = \frac{F(\mu g/mL) \times volume\,of\,solution(L)}{Area\,of\,sample\,in\,cm^2}$$

Statistical Analysis

IBM SPSS Statistics for Windows, version 26.0, Armonk, New York: IBM Corp., Released 2019, was used to analyze the data. The mean and standard deviation were used to express the descriptive statistics. Kolmogorov–Smirnov and Shapiro–Wilk normality tests were done. They showed that the data were normally distributed. Intergroup comparison was done by using one-way analysis of variance (ANOVA), and intragroup comparison was done by repeated-measures ANOVA followed by *post hoc* analysis. The fixed significance level is 5% (α = 0.05). A p-value of less than 0.05 is regarded as statistically significant.

Results

Table 1 presents the intergroup comparison of the three restorative materials using a one-way ANOVA, revealing a highly significant difference across all five intervals. Table 2 indicates highly significant differences at all five intervals between GC Gold Label 2 and Activa™ Pronto, GC Gold Label 2 and Activa™ BioACTIVE, and Activa™ Pronto and Activa™ BioACTIVE. Table 3 shows that repeated-measures ANOVA detected highly significant differences across the five intervals within each of the three groups. Table 4 demonstrates statistically significant differences ($p < 0.05$) between all intervals in all three groups, except for Activa™ BioACTIVE, where no significant difference ($p > 0.05$) was observed between the 14th and 21st days. It reveals that at different intervals, GC Gold Label 2 released greater fluoride ions, followed by Activa™ Pronto and Activa™ BioACTIVE.

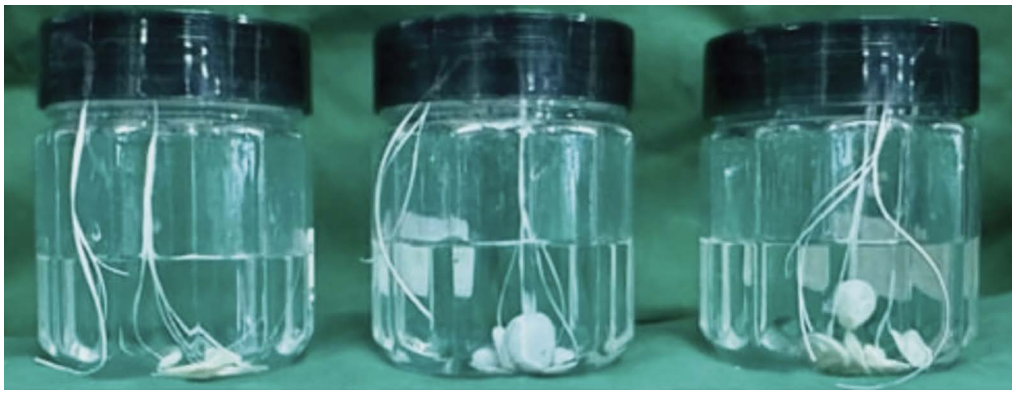

Fig. 2: Specimens kept in plastic vials

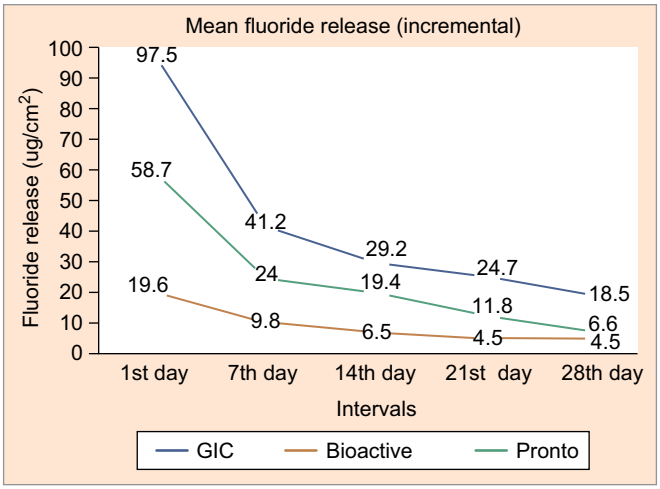

Fig. 3: Incremental fluoride release of three materials at different intervals

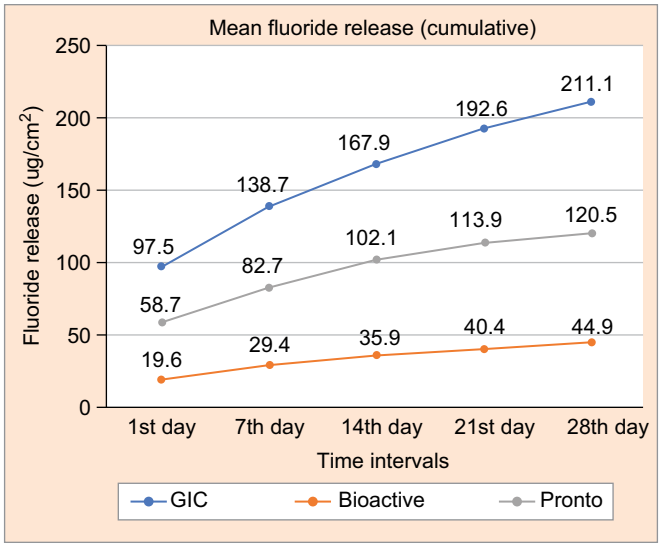

Fig. 4: Cumulative fluoride release of three materials at different intervals

Table 1: Intergroup comparison between three groups at various intervals

Intervals and groups		*Mean ± standard deviation*	*df*	*F*	*p-value*
1st day	GC Gold Label 2	97.5 ± 1.7	23	1568.715	0.001**
	Activa™ BioACTIVE	19.6 ± 0.5			
	Activa™ Pronto	58.7 ± 0.2			
7th day	GC Gold Label 2	41.2 ± 0.5	22	1414.403	0.001**
	Activa™ BioACTIVE	9.8 ± 0.3			
	Activa™ Pronto	24.0 ± 0.5			
14th day	GC Gold Label 2	29.2 ± 0.9	22	371.997	0.001**
	Activa™ BioACTIVE	6.5 ± 0.3			
	Activa™ Pronto	19.4 ± 0.4			
21st day	GIC	24.7 ± 0.6	22	461.666	0.001**
	Activa™ BioACTIVE	4.5 ± 0.4			
	Activa™ Pronto	11.8 ± 0.4			
28th day	GC Gold Label 2	18.5 ± 0.3	22	514.909	0.001**
	Activa™ BioACTIVE	4.5 ± 0.3			
	Activa™ Pronto	6.6 ± 0.3			

**$p < 0.005$ highly statistically significant; test used: One-way ANOVA; fluoride measurement unit: µg/cm^2

Discussion

In addition to stabilizing mineral apatite and promoting enamel remineralization, which improves the dental surface's acid resistance and prevents cavities, fluoride is regarded as the gold standard among the various remineralization regimens. To lower the incidence of dental cavities, fluoride has been added to several restorative materials, which, according to the World Health Organization, affects 60–90% of schoolchildren and approximately 36% of global population suffer from this disease in permanent teeth.[6]

Glass ionomer cement is a member of the acid-base cement material class. Because they discharge biologically active ions (fluoride, sodium, phosphate, and silicate) into the surrounding aqueous media at amounts that are advantageous to biology, these are inherently bioactive. These ions are emitted in greater amounts in acidic environments compared to neutral ones.[6] Bioactive composites offer antimicrobial and remineralizing properties. They have demonstrated encouraging results in restoring lost minerals, preventing recurrent cavities, neutralizing acids, repelling proteins, reducing acid generation and biofilms, lowering cytotoxicity, preserving tooth pulp, and encouraging the creation of tertiary dentin.[7]

A number of intricate processes, including the diffusion of water into the material, the dissolution of fluoride in the solid, and the diffusion of fluoride ions from the substance into the solution, contribute to the release of fluoride from restorative materials. A number of variables, including temperature, the area in contact with the storage medium, and the media in which the samples are kept, can influence how quickly fluoride is released from a substance in the present study, keeping the samples in an incubator at 37°C maintained the temperature at or near the average body temperature. In addition, to prevent the samples from adhering to the polypropylene vial walls and allowing for consistent sample soaking by the storage media, dental floss was used for aiding the suspension of samples into the medium.

Table 2: Multiple pairwise comparisons between groups

					95% Confidence interval	
Interval	*Group (I)*	*Group (J)*	*Mean difference (I – J)*	*p-value*	*Lower bound*	*Upper bound*
1st day	GC Gold Label 2	Activa™ BioACTIVE	77.941	0.001**	74.0694	81.0769
		Activa™ Pronto	38.802	0.001**	34.9306	41.9381
	Activa™ BioACTIVE	Activa™ Pronto	39.13	0.001**	–42.6425	–35.6350
7th day	GC Gold Label 2	Activa™ BioACTIVE	31.329	0.001**	29.4947	32.6128
		Activa™ Pronto	17.181	0.001**	15.3460	18.4640
	Activa™ BioACTIVE	Activa™ Pronto	–14.149	0.001**	–15.7078	–12.5897
14th day	GC Gold Label 2	Activa™ BioACTIVE	22.632	0.001**	20.6883	24.8204
		Activa™ Pronto	9.769	0.001**	7.8258	11.9579
	Activa™ BioACTIVE	Activa™ Pronto	12.863	0.001**	–14.9286	–10.7964
21st day	GC Gold Label 2	Activa™ BioACTIVE	20.274	0.001**	17.5608	21.7617
		Activa™ Pronto	12.954	0.001**	10.2414	14.4423
	Activa™ BioACTIVE	Activa™ Pronto	–7.319	0.001**	–9.4198	–5.2189
28th day	GC Gold Label 2	Activa™ BioACTIVE	14.009	0.001**	12.5950	15.0288
		Activa™ Pronto	11.896	0.001**	10.4818	12.9157
	Activa™ BioACTIVE	Activa™ Pronto	–02.113	0.001**	0.8962	3.3300

**$p < 0.005$ highly statistically significant; test used: Bonferroni test; fluoride measurement unit: µg/cm^2

Table 3: Intragroup comparison among intervals

Groups	*df*	*F*	*p-value*
GC Gold Label 2	8	1033.996	0.001**
Activa™ BioACTIVE	8	288.671	0.001**
Activa™ Pronto	8	2537.669	0.001**

**$p < 0.005$ highly statistically significant; test used: Repeated-measures ANOVA; fluoride measurement unit: µg/cm^2

Table 4: Multiple pairwise comparison between intervals

Intervals	*GC Gold Label 2*	*Activa™ BioACTIVE*	*Activa™ Pronto*
1st–7th day	0.000**	0.000**	0.000**
1st–14th day	0.000**	0.000**	0.000**
1st–21st day	0.000**	0.000**	0.000**
1st–28th day	0.000**	0.000**	0.000**
7th–14th day	0.000**	0.003*	0.004*
7th–21st day	0.000**	0.000**	0.000**
7th–28th day	0.000**	0.000**	0.000**
14th–21st day	0.003*	0.054	0.000**
14th–28th day	0.000**	0.046*	0.000**
21st–28th day	0.001**	1.000	0.000**

*$p < 0.05$ statistically significant; **$p < 0.005$ highly statistically significant; $p > 0.05$ statistically nonsignificant; test used: Bonferroni test; fluoride measurement unit: µg/cm^2

Deionized water was chosen as it provided the baseline of fluoride release potential in unstimulated conditions. This is in accordance with previous study done by Nigam et al. in 2009. In this study, fluoride released from the restorative materials was evaluated at 1st, 7th, 14th, 21st, and 28th days. During the first 24 hours, all the three restorative materials had high release of fluoride which then decreased with time. However, they pursued to release fluoride until the end of the study. There are two distinct processes involved in fluoride elution. The first burst of fluoride release from the surface, followed by a dramatic reduction in elution, is what defines the first step. A second bulk diffusion process follows, during which a tiny quantity of fluoride is continuously released into the media around it for a minimum of 2–2.5 years.[2]

Conventional glass ionomer cements are recognized for their ability to release fluoride, contributing to their anticariogenic properties. Our study confirmed that GC Gold Label 2 exhibited a substantial initial fluoride release, which gradually decreased over time. This is in accordance with the studies conducted by Nigam et al. and Jingarwar et al.[2,3] This burst release is beneficial as it provides immediate protection against caries right after the restoration is placed. Following the initial release, a lower but steady rate of fluoride release was observed, which is advantageous for providing prolonged protection and enhancing the longevity of the restoration.[8] These findings align with previous research, underscoring the effectiveness of conventional glass ionomer cements in offering a dual-phase fluoride release profile.

Bioactive composites are designed to combine the esthetic and mechanical benefits of composite resins with the bioactivity similar to that of glass ionomer cements. The study revealed that bioactive composites release fluoride, but the release pattern is different from conventional glass ionomer cements. The initial fluoride release from composites was lower compared to GC Gold Label 2, indicating a less pronounced burst release. However, they demonstrated a more gradual and sustained release over time. This continuous release is beneficial for long-term caries prevention and enamel remineralization.[9] The extended-release profile suggests that bioactive composites can provide ongoing fluoride availability, which is crucial for maintaining dental health, especially in patients with a higher risk of caries.[10]

On comparing the fluoride release profiles of the three materials, distinct differences were evident. GC Gold Label 2 demonstrated the highest initial fluoride release. This may be because of its component filler and material porosity. Fluoroaluminosilicate glass is the leading filler in glass ionomer cements; it is more dissoluble than barium and strontium existing in composites, thus making it to release more fluoride. Also, glass ionomer cement has lesser filler particles making it more porous when compared to that of composites. Therefore, fluoride was able to diffuse through the pores and showed more release. However, its fluoride release diminished more rapidly over time compared to the bioactive composites. In contrast, both Activa™ BioACTIVE and Activa™ Pronto exhibited a more controlled and prolonged fluoride release. This sustained release is advantageous for long-term dental health by providing continuous fluoride exposure.[11,12]

The cumulative fluoride release results suggest that conventional glass ionomer cements are effective for immediate fluoride release, whereas bioactive composites offer a more sustained release, which is beneficial for ongoing caries prevention and tooth health maintenance. The differences in fluoride release patterns indicate that each material has unique benefits and can be selected based on specific clinical requirements and patient needs. The results of this study have essential clinical implications. The choice of restorative material can significantly influence the fluoride release profile and, consequently, the preventive benefits for patients. Conventional glass ionomer cements, such as GC Gold Label 2, are effective for immediate fluoride release, making them suitable for high-caries-risk situations, where rapid fluoride availability is necessary. On the contrary, bioactive composites, such as Activa™ BioACTIVE composite and Activa™ Pronto, provide sustained fluoride release, which is beneficial for long-term caries prevention and enamel remineralization. In clinical practice, understanding the fluoride release profiles of different restorative materials can help dentists select the most appropriate material for each patient. For instance, in patients with a history of recurrent caries, materials with sustained fluoride release may be more beneficial. Additionally, the esthetic and mechanical properties of bioactive composites make them suitable for restorations in visible areas where both function and appearance are crucial.

Certain limitations pertaining to the study were small sample size, lack of total ionic strength adjustment buffer (TISAB), or pH control, which may have influenced the accuracy and reproducibility of ion activity measurements and the use of deionized water rather than saliva or a pH-cycling model, which does not adequately replicate the complex oral environment.

Conclusion

Conventional glass ionomer cements release a higher amount of fluoride initially, while bioactive composites such as Activa™ BioACTIVE and Activa™ Pronto provide a more sustained release. These findings suggest that bioactive composites may offer extended protective benefits against caries. Further clinical studies are necessary to validate these *in vitro* results and to explore the long-term effects of these materials in various clinical scenarios. The selection of restorative materials should be based on a comprehensive understanding of their fluoride release profiles.

Author Contributions

M Manoharan: Concepts, design, definition of intellectual content, literature search, data analysis, statistical analysis, manuscript preparation, manuscript editing, manuscript review, guarantor.
Prakash Sriharitha: Concepts, design, definition of intellectual content, literature search, data acquisition, data analysis, statistical analysis, manuscript preparation, manuscript editing, manuscript review, guarantor.
Gawthaman M: Concepts, design, definition of intellectual content, literature search, data analysis, statistical analysis, manuscript preparation, manuscript editing, manuscript review, guarantor.
Vinodh S: Concepts, design, definition of intellectual content, literature search, data analysis, statistical analysis, manuscript preparation, manuscript editing, manuscript review, guarantor.
M Kamatchi: Concepts, design, definition of ntellectual content, literature search, data analysis, statistical analysis, manuscript preparation, manuscript editing, manuscript review, guarantor.
Kruthika Murali: Concepts, design, definition of intellectual content, literature search data analysis, statistical analysis, manuscript preparation, manuscript editing, manuscript review, guarantor.

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I, Sriharitha P attest that all individuals who contributed to the manuscript have been appropriately acknowledged, and that all contributors who are not authors have agreed in writing to be named in the Acknowledgement section of the article.

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