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Fluoride Release and Recharge in Glass Ionomer Restoratives: A Comparative Study

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ABSTRACT

Purpose: This in vitro study aimed to evaluate the fluoride release and recharge properties of three distinct fluoride-containing glass ionomer restorative materials, both before and after fluoride varnish application. It was hypothesized that there would be significant differences in the initial fluoride release and recharge capacities among the tested materials.

Methods: Three glass ionomer restorative materials—Riva Self Cure HV (Southern Dental Industries, Victoria, Australia), EQUIA (GC Corporation, Tokyo, Japan), and EQUIA Forte (GC Corporation, Tokyo, Japan)—were tested using disc-shaped specimens. Fluoride release was measured daily during the first week and cumulatively up to seven weeks using a fluoride ion-selective electrode. Following this period, fluoride varnish (MI Varnish, containing 22,600 ppm sodium fluoride and CPP-ACP; GC Corporation, Tokyo, Japan) was applied, and cumulative fluoride release was reassessed at 1 and 3 weeks.

Results: All materials released the most fluoride within the first two days. EQUIA exhibited the highest fluoride release in the early period, while EQUIA Forte demonstrated superior fluoride recharge after varnish application (p -value<0.05).

Conclusions: The results confirmed the initial hypothesis. EQUIA may be more beneficial for early caries prevention due to higher initial fluoride release, whereas EQUIA Forte shows better long-term fluoride recharge potential. These findings have important implications for selecting restorative materials in patients requiring enhanced caries prevention.

Keywords: Dental materials; topical fluorides; ion-selective electrodes; in vitro techniques; tooth remineralization

INTRODUCTION

Fluoride is a naturally occurring mineral known for its crucial role in dental caries prevention. It strengthens enamel by promoting remineralization and inhibiting demineralization processes, thereby increasing tooth resistance to acid attacks. In addition to its localized action within the oral cavity, systemic fluoride intake during tooth development contributes to the formation of stronger enamel. Fluoride's anticariogenic effects have made it a cornerstone of public health initiatives, such as water fluoridation, topical applications, and incorporation into dental materials. Fluoride-releasing restorative materials play a crucial role in modern dentistry by continuously releasing fluoride, which helps prevent secondary caries and supports the remineralization of tooth structures. [1, 2]. Glass ionomer cements (GICs) are well known for their natural ability to release and recharge fluoride, making them especially beneficial for

patients at high risk of developing caries [3,4]. Fluoride ions released from these materials can inhibit demineralization, enhance remineralization, and exert antimicrobial effects, thereby contributing to the overall longevity of restorations [5].

The fluoride release and recharge capability of glass ionomer restoratives are affected by various factors, including their composition, setting process, and interaction with external fluoride sources like fluoride toothpaste or varnish[6]. Studies have demonstrated that glass ionomers undergo an initial burst of fluoride release during the first 24–48 hours, followed by a more gradual, sustained release over time [7]. However, the long-term efficacy of fluoride-releasing restoratives depends on their ability to recharge fluoride after exposure to fluoride-containing products, which can enhance their protective effects in the oral environment [8].

Recent advancements in high-viscosity glass ionomer cements (HV-GICs), such as the development of surface-treated glass particles and hybrid formulations, have aimed to improve fluoride release, mechanical properties, and recharge capacity [9]. Among these, EQUIA (GC Corporation, Tokyo, Japan), EQUIA Forte (GC Corporation, Tokyo, Japan), and Riva Self Cure HV (Southern Dental Industries, Victoria, Australia) are widely used for their fluoride-releasing properties and clinical durability. However, there is still limited research comparing their fluoride release patterns before and after fluoride varnish application.

The objective of this in vitro study was to assess and compare the fluoride release patterns of three fluoride-releasing glass ionomer restorative materials (EQUIA, EQUIA Forte, and Riva Self Cure HV) both before and after the application of fluoride varnish. The study aimed to assess the early fluoride release of each material within the first week. Analyze cumulative fluoride release over a seven-week period. Evaluate the ability of each material to absorb and re-release fluoride after the application of fluoride varnish.

MATERIAL AND METHODS

Study Design and Sample Preparation

This laboratory study assessed the fluoride release and recharge capability of three restorative glass ionomers: Riva Self Cure HV (SDI Limited, Victoria, Australia), EQUIA (GC Corporation, Tokyo, Japan), and EQUIA Forte (GC Corporation, Tokyo, Japan) (Table 1).

A total of 30 disc-shaped specimens (n=10 per material) were fabricated according to manufacturers' guidelines using customized cylindrical Teflon molds (8 mm diameter × 2 mm height). The materials were prepared under controlled conditions at room temperature. Each sample was allowed to set for 15 minutes before being removed from the molds and then stored in distilled water at 37°C within a sealed container to mimic oral conditions.

Fluoride Release Measurement

Fluoride release from the samples was assessed at different time intervals using a fluoride ion-selective electrode connected to an ion analyzer (Mettler TOLEDO Seven Compact pH/Ion S220 Meter, Switzerland). Each specimen was individually placed in 5 mL of distilled water inside a tightly sealed polypropylene tube and maintained at 37°C. The fluoride concentration in the solution was measured at specific time points [10]: (Daily for the first week (Days 1–7), Weekly up to 7 weeks (Weeks 1, 3, 5, 6, and 7). To ensure measurement accuracy, the storage medium was replaced with fresh distilled water after each reading. Fluoride concentrations were expressed as µg/dL/mm².

Fluoride Recharge and Fluoride Varnish Application

To evaluate the fluoride recharge capability, after completing the 7-week fluoride release assessment, the specimens were dried and coated with fluoride varnish (MI Varnish, GC Corporation, Tokyo, Japan), which contains 22,600 ppm fluoride (5% sodium fluoride) along with CPP-ACP (casein phosphopeptide-amorphous calcium phosphate complexes) (Table 1). After varnish application, the specimens were stored in distilled water, and cumulative fluoride release was reassessed at 1 and 3 weeks' post-application using the same measurement method.

Statistical Analysis

The data were processed using IBM SPSS Statistics (version 20.0, IBM Corp.). Prior to the application of ANOVA, the data were tested for normality using the Shapiro-Wilk test and for homogeneity of variances using Levene's test. The assumptions were met. One-way ANOVA was employed to compare fluoride release values across the three materials at each time interval. To assess significant differences in cumulative fluoride release among the groups, Welch ANOVA and Bonferroni post hoc tests were utilized. Pairwise comparisons for each restorative material were conducted using Welch ANOVA with a significance level of α = 0.05. All statistical analyses were performed at a 95% confidence level (p-value < 0.05).

Table 1. Restorative glass ionomers and fluoride varnish tested in the study

	Materials	Compositions
Restorative glass ionomers	Riva Self Cure HV (SDI Limited, Victoria, AUS)	Powder Content: Fluoro aluminosilicate glass, Polyacrylic acid Liquid Content: Polyacrylic acid, Tartaric acid
	EQUIA Fil (GC Corporation, Tokyo, Japan)	Powder Content: Fluoro aluminosilicate glass, polybasic carboxylic acid Liquid Content: distilled water, polyacrylic acid
	EQUIA Forte Fil (GC Corporation, Tokyo, Japan)	Powder Content: Fluoroaluminosilicate glass, polyacrylic acid, powder, surface-treated glass Liquid Content: polyacrylic acid
Fluoride varnish	MI Varnish (GC Corporation, Tokyo, Japan)	22,600 ppm F ⁻ 10% w/v CPP-ACP, 5% sodium fluoride (NaF)

CPP-ACP, casein phosphopeptide-stabilized amorphous calcium phosphate complexes

Table 2. Means and standard deviations of fluoride release for each restorative glass ionomer during the first week ($\mu\text{g}/\text{dl}/\text{mm}^2$)

	Riva Self Cure HV	EQUIA	EQUIA Forte
Day 1	3.81 (0.72) ^a	10.18 (3.02) ^b	5.19 (0.27) ^a
Day 2	1.26 (0.13) ^a	3.63 (1.40) ^b	2.00 (0.19) ^a
Day 3	1.01 (0.09) ^a	2.70 (0.96) ^b	1.76 (0.15) ^c
Day 4	0.88 (0.07) ^a	2.10 (0.52) ^b	1.61 (0.13) ^c
Day 5	0.75 (0.07) ^a	1.48 (0.22) ^b	1.44 (0.10) ^b
Day 6	0.73 (0.09) ^a	1.74 (0.42) ^b	1.34 (0.13) ^b
Day 7	0.70 (0.12) ^a	1.99 (0.70) ^b	1.24 (0.16) ^b

Different letters (for each day) indicate significantly different groups (p -value<0.05).

RESULTS

The findings of this study reveal that all evaluated glass ionomer restorative materials exhibited the highest fluoride release within the first two days, followed by a gradual decline over time. Among the materials tested, EQUIA demonstrated the greatest fluoride release during the initial four days (p -value<0.05) (Table 2), while EQUIA Forte displayed a similar fluoride release pattern to EQUIA for the remainder of the first week (p -value>0.05). Conversely, Riva Self Cure HV showed the lowest fluoride release throughout the entire study period (Table 2).

Initial Fluoride Release

Throughout the first week, fluoride release was monitored daily. EQUIA demonstrated significantly higher fluoride release than EQUIA Forte and Riva Self Cure HV (p -value<0.05) (Table 2). The fluoride release pattern exhibited an initial rapid burst within the first two days, followed by a more gradual and sustained release over the following days. Among the three

materials, Riva Self Cure HV consistently displayed the lowest fluoride release (Table 2).

Cumulative Fluoride Release

Prior to the application of fluoride varnish, cumulative fluoride release was assessed at 1, 3, 5, 6, and 7 weeks. The findings indicated that EQUIA exhibited the highest cumulative fluoride release, followed by EQUIA Forte, whereas Riva Self Cure HV had the lowest cumulative release values (p -value<0.05) (Table 3).

Fluoride Recharge After Varnish Application

Following the application of fluoride varnish, all tested glass ionomers demonstrated an increase in fluoride release, confirming their ability to uptake and re-release fluoride. The fluoride release was measured again at 1 and 3 weeks' post-application. Among the materials, EQUIA Forte exhibited the highest cumulative fluoride release after varnish application, surpassing both EQUIA and Riva Self Cure HV at both time points (p -value<0.05) (Table 3, Figure 1).

Table 3. Means and standard deviations of initial cumulative fluoride release before (1-, 3-, 5-, 6- and 7-week) and after (1- and 3-week) fluoride varnish application for each restorative glass ionomers tested ($\mu\text{g}/\text{dl}/\text{mm}^2$)

Evaluation periods		Riva Self Cure HV	EQUIA	EQUIA Forte
Before fluoride varnish application	1 week	9.16 (1.13) ^a	23.84 (6.89) ^b	14.60 (1.01) ^c
	3 weeks	12.90 (1.02) ^a	30.55 (5.23) ^b	21.35 (0.95) ^c
	5 weeks	16.65 (1.30) ^a	37.26 (8.27) ^b	28.01 (1.80) ^c
	6 weeks	18.30 (1.22) ^a	41.22 (6.27) ^b	32.23 (1.93) ^c
	7 weeks	20.40 (1.56) ^a	44.08 (9.92) ^b	34.48 (2.39) ^c
After fluoride varnish application	1 week	3.28 (0.66) ^a	4.55 (0.70) ^b	6.61 (0.75) ^c
	3 weeks	4.58 (1.50) ^a	5.76 (1.55) ^a	9.71 (2.60) ^b

The different superscript letters indicate significant differences among restorative glass ionomers in same evaluation periods. Welch ANOVA test indicates significant differences in fluoride release among restorative glass ionomers (p -value<0.05).

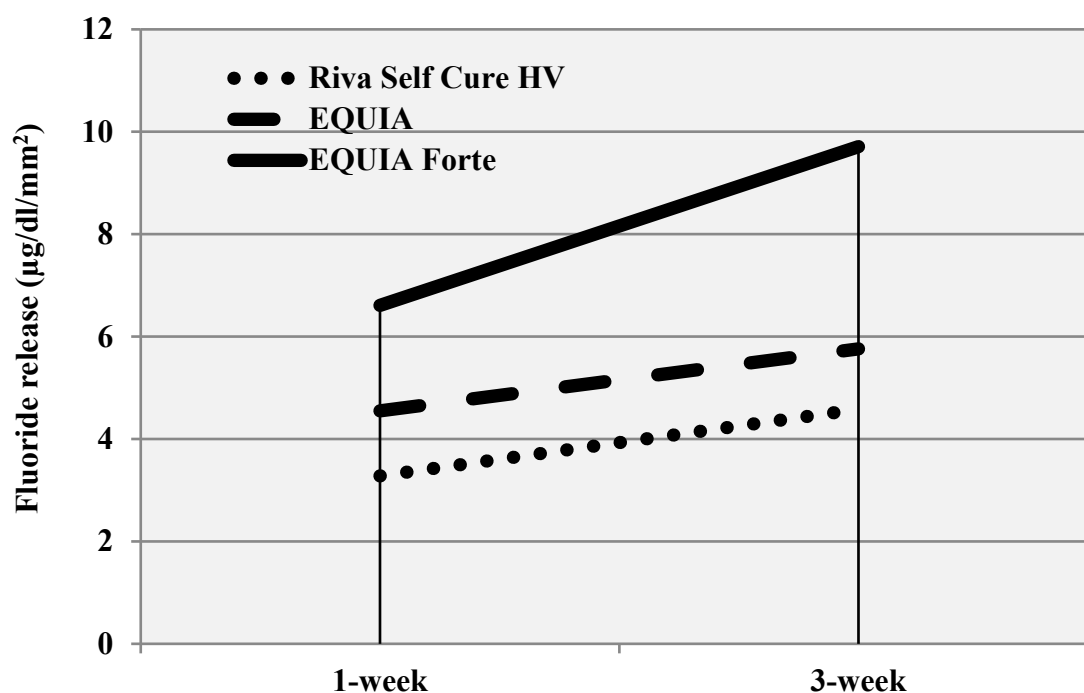


Figure 1. Pattern of cumulative fluoride release 1 and 3 weeks after fluoride varnish application (x-axis: Evaluation periods in weeks; y-axis: Fluoride release in $\mu\text{g/dL/mm}^2$)

DISCUSSION

The present study demonstrated that all three tested glass ionomer restorative materials exhibited an initial burst of fluoride release during the first 48 hours, followed by a gradual decline. This release pattern is in agreement with previous research indicating that the early phase of fluoride emission is primarily due to the rapid dissolution of loosely bound fluoride ions at the surface of the material upon exposure to moisture [6,11].

Among the materials tested, EQUIA exhibited the highest initial fluoride release during the first four days. However, from the fifth day onward, EQUIA Forte displayed a fluoride release pattern similar to EQUIA, suggesting more sustained performance. In contrast, Riva Self Cure HV consistently released the lowest levels of fluoride throughout the study period. These differences are likely attributable to variations in material composition. For example, the presence of surface-treated fluoroaluminosilicate glass particles in EQUIA Forte may enhance its long-term fluoride releasing capacity [3,12]. Ariffin et al. [13] similarly reported that modifications in the glass matrix composition can significantly influence the fluoride release profile of GICs.

The cumulative fluoride release analysis supported these observations, with EQUIA maintaining the highest fluoride release across all evaluation periods, followed by EQUIA Forte and Riva Self Cure HV. The sustained fluoride release of EQUIA may be linked to its

matrix formulation, allowing for prolonged ion diffusion [14,15]. Such characteristics are essential in clinical scenarios where long-term caries protection is needed.

Following the application of fluoride varnish, all materials exhibited enhanced fluoride release, confirming their ability to absorb and re-release fluoride—commonly referred to as the “fluoride recharge” phenomenon. This is a crucial factor for restorative longevity in high-caries-risk patients receiving periodic fluoride therapies [16]. Notably, EQUIA Forte demonstrated the highest fluoride recharge at both 1 and 3 weeks’ post-application, surpassing both EQUIA and Riva Self Cure HV. This is consistent with studies indicating that surface-treated glass ionomers offer greater fluoride uptake capacity compared to conventional GICs [17,18].

The clinical implications of these results are significant. While EQUIA may be preferred for its high initial fluoride release, EQUIA Forte appears to be more beneficial for patients requiring long-term fluoride exposure and caries prevention, especially when used alongside periodic fluoride varnish applications. Riva Self Cure HV, although showing the lowest performance in both release and recharge, may still be clinically relevant when combined with external fluoride sources [19].

Comparison with previous studies further validates our findings. Bae et al. [22] reported similar trends, showing that high-viscosity GICs containing surface-

treated glass demonstrated better fluoride recharge ability. In contrast, Cabral et al. [23] observed limited recharge potential in conventional GICs compared to resin-modified alternatives, underscoring the importance of material selection based on clinical context.

One of the primary limitations of this study lies in its *in vitro* design, which, by nature, cannot fully replicate the multifactorial complexities of the oral environment, including dynamic salivary flow, pH fluctuations, enzymatic activity, and microbial presence. These variables can significantly influence the behavior of restorative materials *in vivo*. Consequently, further *in vivo* studies are necessary to assess the long-term clinical performance of fluoride-releasing materials and to validate their extended release and recharge potential under actual oral conditions.

Another important limitation is the absence of structural and surface characterization analyses of the restorative materials and their interactions with fluoride varnish. Critical factors such as surface topography, morphological characteristics, mechanical resistance, and the nature of fluoride bonding to tooth structures were not investigated. The application of advanced instrumental techniques—including atomic force microscopy (AFM), X-ray fluorescence (XRF), X-ray diffraction (XRD), and high-resolution transmission electron microscopy (HRTEM)—could provide valuable insights into the material–varnish interface, elucidating potential mechanisms such as crystallization, complex formation, and surface reactivity that may influence fluoride release kinetics. Incorporating these methods in future studies would deepen the understanding of material behavior and support the development of optimized fluoride delivery systems.

In addition to these structural considerations, the biological impact of the released fluoride was not addressed in the present investigation. While fluoride is well recognized for its cariostatic benefits, its excessive or uncontrolled release may pose cytotoxic risks to adjacent oral tissues. Therefore, future research should include biocompatibility assessments, such as *in vitro* cytotoxicity evaluations using pulpal or gingival cell lines, to establish safe and effective thresholds for fluoride exposure. These studies will be instrumental in bridging the gap between laboratory findings and clinical application, ensuring both efficacy and biological safety in fluoride-releasing restorative materials.

CONCLUSIONS

This *in vitro* study demonstrated that fluoride-releasing glass ionomer restorative materials exhibit distinct patterns in both initial release and recharge capabilities. Among the tested materials, EQUIA showed the highest initial fluoride release, with 10.18

µg/dL/mm² on Day 1 and a cumulative release of 44.08 µg/dL/mm² at Week 7. In comparison, EQUIA Forte exhibited superior fluoride recharge, releasing 6.61 µg/dL/mm² and 9.71 µg/dL/mm² at 1 and 3 weeks' post-fluoride varnish application, respectively. Riva Self Cure HV consistently demonstrated the lowest fluoride release and recharge values but may still be suitable in cases where external fluoride sources (e.g., toothpaste, mouth rinses) are routinely used.

These findings emphasize the importance of material selection based on patient-specific needs. For immediate caries prevention, materials like EQUIA may be preferable due to their high initial fluoride release. In contrast, for patients requiring long-term protection, EQUIA Forte presents a promising option due to its superior fluoride recharge potential.

Although these results are promising, further *in vivo* studies are warranted to evaluate their performance under clinical conditions and to confirm the long-term effectiveness of fluoride recharge mechanisms in the oral environment.

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Not applicable.

CONFLICT OF INTERESTS

None.

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