

Variation and contact time of protective surface coating on fluoride release and recharge from glass ionomer restoration

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ABSTRACT

Background: Glass ionomer cement (GIC) is an effective restorative material known for its ability to release and recharge fluoride over an extended period. The application of fluoride-containing varnish and acidulated phosphate fluoride (APF) gel can substantially enhance the fluoride release and recharge properties of GIC. These additional treatments provide an extra source of fluoride, contributing to the long-term protection and strengthening of teeth. **Purpose:** This study aims to investigate the fluoride release and rechargeability of a tooth-colored restorative material (glass ionomer) over different time periods following the application of fluoride varnish (F varnish) and APF gel. **Methods:** Seventy-two specimens were prepared and divided into three groups, each further categorized into four subgroups based on the type and duration of fluoride application. Fluoride release was measured at 24 hours, 1 week, and 1 month, while fluoride recharge was evaluated after the application of F varnish and APF gel. **Results:** There was no significant difference in fluoride release between 24 hours and 1 week, but a significant decrease was observed after 1 month. The highest fluoride release occurred at 24 hours, while the lowest was recorded at 1 month. After fluoride recharge, there was a significant increase in fluoride release. **Conclusion:** Fluoride release from glass ionomer restorations was highest at 24 hours, and the rechargeability was greater with a 24-hour F varnish application compared with 12 hours. APF gel demonstrated higher fluoride release than F varnish, and prolonged contact time between the restorative material and the recharging agent resulted in greater fluoride uptake.

Keywords: fluoride; glass ionomer restoration; medicine; release; recharge

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INTRODUCTION

Dental caries is a prevalent non-communicable disease with a multifactorial etiology. Studies show that it can be prevented and reversed in its early stages.¹ Early detection of dental disease is important to avoid complications from advanced caries.² Prevention is crucial in intercepting and managing dental caries and avoiding relapses by modifying pathological and protective factors.³

Fluoride has a long-standing relationship with dental caries prevention and plays a key role in preventive dentistry. There is a strong link between fluoride and carious activity.⁴ Insufficient fluoride exposure is a causative factor in caries development, along with dietary habits, bacterial activity, low salivary flow, and poor oral hygiene. The anti-cariogenic property of fluoride is related to its ability to inhibit bacterial

metabolism and growth, prevent demineralization, and accelerate the remineralization process. For this reason, fluoride ion release and recharging capability are important properties of restorative materials.⁵

One of the fluoride-releasing materials used for caries treatment is glass ionomer cement (GIC). It is an appropriate restorative material, particularly for primary teeth. The purpose of this study is to examine fluoride release from restorations, which is beneficial in inhibiting secondary marginal caries and providing additional protection during fluoride release. GIC is a medical product in powder form containing fluoride ions, aluminum oxide, and silicate. When combined with a mild acidic liquid, it can be used as a tooth-colored restoration.⁶

Fluoride varnish (F varnish) and acidulated phosphate fluoride (APF) gel are widely used materials for delivering

fluoride benefits, especially in pediatric patients. F varnish (NaF 5%) is used for caries prevention and the treatment of dentin hypersensitivity. It increases the amount of time fluoride remains in contact with teeth.⁷ F varnish reacts with the enamel surface to form fluorapatite, making the enamel more resistant to demineralization and damage.⁸ APF gel (1.23%) is the most widely used fluoride agent due to its stability and commercial availability. Regular use of fluoride gel has a beneficial effect primarily for individuals at high caries risk, particularly those who do not live in fluoridated water areas or do not use fluoride toothpaste.⁹

Limited studies have addressed the fluoride release ability of restorative materials, particularly in pediatric patients. This study aims to investigate the fluoride release and recharge properties of a tooth-colored restoration (glass ionomer) over different periods and after the application of F varnish and APF gel.

MATERIALS AND METHODS

The current experimental in-vitro study was carried out on 72 specimens, which were fabricated and randomly assigned in April 2023 in the central lab of the College of Agriculture and Forestry, Mosul University. Materials: Glass ionomer restoration (Medifil, Promedica, Germany), F varnish NaF (Proshield Varnish, Germany), Fluoride gel APF (Germiphene Corporation, Canada), Deionized water (Ibn-Sina General Hospital-Kidney Dialysis, Mosul, Iraq), Distilled water (College of Agriculture and Forestry, Mosul, Iraq), Fluoride ion solution (College of Agriculture and Forestry, Mosul, Iraq). Instrument: Polyethylene Mold (Insulin syringe, Abu Dhabi Medical Devices L.L.C, United Arab Emirates), Engine (Saeshin, model Strong 204, TAEGU, Korea), Mandrel and Disk, Polyethylene Container, Spatula, Mixing Pads, Glass Microscopic Slide (Citoplus, China), Mass (50 g), Disposable Syringe (Med Dawliaico, Egypt), Balance (Adam Equipment, UK), Fluoride ion-selective electrode (FISE; EUTECH Instruments, Singapore), Disposable Gloves (Kay La, China), Phone Camera (iPhone 11, USA). Grouping of the samples: The total sample was composed of 72 specimens divided into three groups.¹⁰ Group 1: (24 specimens were fabricated) after 24 hours. Fluoride release from GIC was checked, then GIC was recharged with F varnish (six specimens for 12 hours and six specimens for 24 hours) and also recharged with APF gel (six specimens for 1 minute and six specimens for 4 minutes). Group 2: (24 specimens were fabricated) after 1 week. Fluoride release from GIC was checked, then GIC was recharged with F varnish (six specimens for 12 hours and six specimens for 24 hours) and also recharged with APF gel (six specimens for 1 minute and six specimens for 4 minutes). Group 3: (24 specimens were fabricated) after 1 month. Fluoride release from GIC was checked, then GIC was recharged with F varnish (six specimens for 12 hours and six specimens for 24 hours) and

also recharged with APF gel (six specimens for 1 minute and six specimens for 4 minutes).

Specimen Preparation: 24 disk specimens were prepared for each group, with dimensions of 2 mm in height and 5 mm in diameter.¹¹ The weight of each specimen was 0.1 g, as confirmed using a balance. Specimens were prepared using a custom-made polyethylene mold (5 mm diameter, 2 mm depth), filled with glass ionomer restoration (Medifil, Promedica, Germany) (powder/liquid ratio 1:1, mixing time 30–40 seconds). According to the manufacturer's instructions, mixing was performed on a paper mixing pad. A glass microscopic slide was placed on both surfaces of the specimen, pressure was applied using a 50 g mass to extrude excess material, and the sample was allowed to set inside the mold for 6 minutes through chemical curing.¹² Specimens were removed from the mold and transferred into polyethylene containers containing 5 mL of deionized water at 37°C. Fluoride release was measured using a FISE (EUTECH Instruments, Singapore), and all data were recorded in parts per million (ppm) at time intervals of 1 day, 7 days, and 1 month.¹³ For each group, specimens were recharged with F varnish (Proshield Varnish, Germany) for either 12 or 24 hours and APF gel (Germiphene Corporation, Canada) for either 1 or 4 minutes. Fluoride recharge was also measured using a FISE, and all data were recorded in ppm.

Method of measuring fluoride release from GIC and fluoride recharge with F varnish and APF Gel at different durations: The group was divided into subgroups, each containing six specimens, according to expert instructions. The head of the FISE was initially cleaned with sandpaper, then washed with distilled water and dried with tissue paper. The concentration of fluoride in distilled water was measured by placing the electrode head in a container of distilled water, followed by measuring the standard concentration of fluoride in the same manner. The amount of fluoride released from each specimen after 24 hours was measured. The electrode head was washed with distilled water and dried after each reading. Upon completion of the reading for each subgroup of six specimens, the electrode head was cleaned with sandpaper, washed, and dried. Fluoride release was calculated in millivolts. After checking the release of all specimens, the first six specimens were recharged with F varnish for 24 hours, the second six specimens were recharged with F varnish for 12 hours, the third six specimens were recharged with APF gel for 1 minute, and the last six specimens were recharged with APF gel for 4 minutes. The F varnish was applied using a brush, coating all surfaces of the specimens for specific durations (12 and 24 hours), then washed with 2 mL of deionized water and placed in a new container containing 5 mL of deionized water for 24 hours to check the amount of fluoride release after recharging. Other specimens were recharged with APF gel by immersing them in the gel (six specimens for 1 minute and six specimens for 4 minutes), then washed with 2 mL of deionized water and placed in a new container containing 5 mL of deionized water for 24 hours to check

the amount of fluoride release after recharging.¹⁴ A FISE was used to measure the amount of fluoride released from GIC after recharging with F varnish and APF gel. The FISE provided results in millivolts, which were converted to ppm using an equation in Excel. The same procedure was repeated for the 1-week and 1-month groups. Before being measured at 24 hours, the specimens were transferred to a new container containing 5 mL of fresh deionized water, and then fluoride release was measured.¹⁵

Statistical Analysis: The data were analyzed using the Statistical Package for the Social Sciences program, version 26 (IBM, USA), which included the following: Descriptive Statistics—mean, number of specimens, standard deviation (SD), amount of fluoride release from GIC, and amount of fluoride after recharging with F varnish and APF gel in ppm. One-way analysis of variance (ANOVA): The data obtained from previous measurements were initially analyzed using the one-way ANOVA test. Duncan's multiple range test: The data were further analyzed using Duncan's multiple range test to determine significant differences among the groups. Results were considered significant at $P \leq 0.05$.

RESULTS

The 24 specimens present in the exposed data yielded the following results. A test of normality using Kolmogorov–Smirnov and Shapiro–Wilk tests was performed on the data. The results were significant ($P \geq 0.05$), indicating that the data were normally distributed. Table 1 demonstrates the analysis of variance, which compares fluoride release before and after recharging the specimens with F varnish and APF gel. There was no significant difference in fluoride release from GIC, but a highly statistically significant difference was observed after recharging ($P \geq 0.000$).

Table 2 displays the mean and SD of fluoride release and recharge from glass ionomer with F varnish and APF gel according to Duncan's multiple range test. No difference was observed in fluoride release, which ranged between 8.088 and 8.132 ppm. However, differences in the recharging abilities of GIC restoration were noted. The highest fluoride release was recorded after recharging with APF gel for 4 minutes (9.124 ppm), followed by 1 minute of APF gel (8.732 ppm). Recharging with F varnish for 24

Table 1. ANOVA of fluoride release and recharge values (ppm) of the specimens after 24 hours

	Groups	Sum of squares	df	Mean square	F	Sig.
Release	Between Groups	0.007	3	0.002	1.516	0.241
	Within Groups	0.030	20	0.002		
	Total	0.037	23			
Recharge	Between Groups	7.863	3	2.621	146.527	0.000**
	Within Groups	0.358	20	0.018		
	Total	8.221	23			

** Highly statistically significant

Table 2. Duncan's multiple range test (mean and standard deviation of fluoride release and recharge) after 24 hours

Type of F products		Release	Recharge
F varnish (12 hours)	Mean	8.094 a	7.680 d
	N	6	6
	Std. Deviation	0.034	0.040
F varnish (24 hours)	Mean	8.088 a	8.001 c
	N	6	6
	Std. Deviation	0.047	0.028
APF gel (1 minute)	Mean	8.107 a	8.732 b
	N	6	6
	Std. Deviation	0.038	0.206
APF gel (4 minutes)	Mean	8.132 a	9.124 a
	N	6	6
	Std. Deviation	0.033	0.162

a, b, c, d: To determine significant differences between means.

It involves comparing pairs of means and calculating the shortest significant range.

Table 3. ANOVA of fluoride release and recharge values (ppm) of the specimens after 1 week

	Groups	Sum of squares	df	Mean square	F	Sig.
Release	Between Groups	0.009	3	0.003	1.521	0.240
	Within Groups	0.039	20	0.002		
	Total	0.048	23			
Recharge	Between Groups	8.279	3	2.760	163.845	0.000**
	Within Groups	0.337	20	0.017		
	Total	8.616	23			

**Highly statistically significant

hours resulted in a release of 8.001 ppm, while the lowest fluoride release was observed after recharging with F varnish for 12 hours (7.680 ppm).

Table 3 presents the analysis of variance comparing fluoride release before and after recharging the specimens with F varnish and APF gel after 1 week. There was no significant difference in fluoride release from GIC restoration, but a highly statistically significant difference was observed after recharging ($P \geq 0.000$). Table 4 displays the mean and SD of fluoride release and recharge from glass ionomer restoration with F varnish and APF gel after 1 week. According to Duncan's multiple range test, no difference was observed in fluoride release, which ranged between 7.833 and 7.883 ppm. However, for fluoride recharge after 1 week, differences were noted in the recharging ability of the fluoride gel. No difference was observed between the samples recharged with APF gel for 1 minute or 4 minutes, both of which exhibited the highest release compared with F varnish. For specimens recharged with F varnish, significant differences were observed,

with those recharged for 24 hours showing higher fluoride release than those recharged for 12 hours.

Table 5 demonstrates the analysis of variance, which compares the fluoride release before and after recharging the specimens with F varnish and APF gel after 1 month. There was a highly statistically significant difference in fluoride release before and after recharging with F varnish and APF gel. Table 6 displays the mean and SD of fluoride release and recharge from glass ionomer with F varnish and APF gel after 1 month, where the release values ranged between 7.686 and 7.861 ppm. According to Duncan's multiple range test, there was little difference in the amount of fluoride release, but a difference in the recharging abilities of GIC restoration was observed. The highest fluoride release was recorded after recharging with APF gel for 1 minute and 4 minutes, with values of 8.994 and 9.075 ppm, respectively, while the lowest fluoride release was observed after recharging with F varnish for 12 hours and 24 hours, with values ranging from 7.602 to 7.716 ppm.

Table 4. Duncan's multiple range test (mean and standard deviation of fluoride release and recharge) after 1 week

Type of F Products		Release	Recharge
F varnish (12 hours)	Mean	7.843 a	7.605 c
	N	6	6
	Std. Deviation	0.028	0.033
F varnish (24 hours)	Mean	7.883 a	7.843 b
	N	6	6
	Std. Deviation	0.075	0.024
APF gel (1 minute)	Mean	7.833 a	8.957 a
	N	6	6
	Std. Deviation	0.029	0.194
APF gel (4 minutes)	Mean	7.848 a	8.807 a
	N	6	6
	Std. Deviation	0.019	0.166

a, b, c: To determine significant differences between means.

It involves comparing pairs of means and calculating the shortest significant range.

Table 5. ANOVA of fluoride release and recharge values (ppm) of the specimens after 1 month

	Groups	Sum of squares	df	Mean square	F	Sig.
Release	Between Groups	0.110	3	0.037	52.127	0.000**
	Within Groups	0.014	20	0.001		
	Total	0.124	23			
Recharge	Between Groups	11.409	3	3.803	294.840	0.000**
	Within Groups	0.258	20	0.013		
	Total	11.667	23			

**Highly statistically significant

Table 6. Duncan's multiple range test (mean and standard deviation of fluoride release and recharge) after 1 month

Type of F Products		Release	Recharge
F varnish (12 hours)	Mean	7.748 b	7.602 b
	N	6	6
	Std. Deviation	0.022	0.114
F varnish (24 hours)	Mean	7.686 c	7.716 b
	N	6	6
	Std. Deviation	0.019	0.029
APF gel (1 minute)	Mean	7.707 c	8.994 a
	N	6	6
	Std. Deviation	0.033	0.179
APF gel (4 minutes)	Mean	7.861 a	9.075 a
	N	6	6
	Std. Deviation	0.028	0.073

a, b, c: To determine significant differences between means.

It involves comparing pairs of means and calculating the shortest significant range.

DISCUSSION

Dental caries remains a substantial issue for many children and adolescents despite a gradual decrease in prevalence in some countries. If left untreated, it can lead to severe discomfort, pain, and a detrimental impact on oral health-related quality of life.¹⁶ Fluoride is the most beneficial topical agent for inhibiting microbial growth and metabolism, as well as promoting remineralization to prevent caries.¹⁷ F varnish offers several advantages over other topical fluorides, including rapid setting on enamel, prolonged surface adherence, and slow fluoride release, allowing for a higher fluoride concentration to reach the teeth. Additionally, it is easy to apply, quick, and does not require complete drying of the teeth before application.¹⁸

APF gel 1.23% is another widely used fluoride application agent due to its stability and commercial availability. It replaces hydroxyapatite mineral with fluorapatite, which is more robust and acid-resistant. However, it has an acidic taste and requires longer chair time.^{19,20} An ideal restorative material should possess good color stability, biocompatibility, a thermal expansion coefficient similar to natural teeth, excellent marginal seal, and the ability to chemically adhere to enamel and dentin.²¹ GICs meet these criteria, making them excellent dental restorative materials for pediatric patients. GICs provide a slow release of fluoride for caries prevention, chemically bind to enamel and dentin, reducing the need for extensive cavity preparation, and are biocompatible with pulpal tissue.

This study evaluated the fluoride release from GIC restorative material over different periods (1 day, 1 week, and 1 month) and its recharging capability with F varnish and APF gel at varying durations. The study did not utilize adhesives or moisture-contamination-preventing agents on the specimens. Various techniques, such as spectrophotometry, ion chromatography, FISE, and capillary electrophoresis, were employed to measure fluoride release.²²

This study utilized a FISE due to its simplicity, cost-effectiveness, and lack of need for complex laboratory equipment. Furthermore, it provides a direct and precise measurement of the free fluoride quantity in the solution. The initial burst effect, observed within the first 24 hours during the early setting phase of GIC, is a phenomenon in which the material releases the highest amount of fluoride.¹⁷ This burst effect may be attributed to initial surface rinsing, while the subsequent decrease in fluoride release after the first day could be due to fluoride ion diffusion through cement pores and fracture lines. Generally, the quantity of fluoride released is directly proportional to the fluoride content in the cement. Various storage media, such as deionized water, artificial saliva, and lactic acid, can influence the amount of fluoride released from a material.

In this investigation, a slight variance in fluoride release was noted between 1 day and 1 month, with the difference not exceeding 0.5 ppm. Notably, the burst effect was not

prominently evident, as fluoride release did not surpass 8.132 ppm. Furthermore, minimal differences were observed in fluoride release before and after recharging with F varnish over 1 day, 1 week, and 1 month. Conversely, the disparity before and after recharging with APF gel was approximately 1 ppm across the same time intervals. The recharging of GIC with fluoride is influenced by several factors, including the type of material, fluoride concentration in the recharging agent, and frequency of exposure. It has been previously demonstrated that higher medium concentrations lead to increased GIC recharging.^{17,23}

Deionized water was chosen as the storage medium in this study due to its lack of ions, allowing for precise assessments of fluoride release. This choice aligns with previous research.²⁴ Additionally, using saliva as a storage medium could potentially impact the study's findings due to the presence of minerals or chemical compounds, which is consistent with other studies.²⁵

Fluoride release was assessed after recharging GIC restorations with F varnish for 12 to 24 hours and APF gel for 1 to 4 minutes over 1 day, 1 week, and 1 month. No similar studies were found to compare fluoride release from GIC after recharging with F varnish and APF gel at different durations. This study observed a decline in fluoride release from GIC over time. Initially, a substantial amount of fluoride was released within the first day, but this quantity gradually decreased.

When GIC was recharged with F varnish for 12 and 24 hours, fluoride release after 24 hours was higher than after 12 hours, indicating that longer recharging durations may lead to increased fluoride release. The duration of recharging with APF gel also influenced fluoride release from GIC. After recharging with APF gel for 4 minutes, a higher amount of fluoride was released compared with 1 minute. However, after 1 week, there was little difference in fluoride release between the two durations. After 1 month, fluoride release from GIC after 4 minutes of recharging remained higher than after 1 minute.

In deionized water, approximately 1 ppm of fluoride was released from GIC after recharging with APF gel for both 1-minute and 4-minute durations. These findings highlight the importance of considering the duration of fluoride recharging when using GIC in dental care. Prolonged contact durations can lead to increased fluoride uptake, which may be beneficial for patients requiring higher fluoride levels for preventive purposes. However, it is important to note that overall fluoride release from GIC remains stable over time, even after recharging with different materials.

The study's results contrast with those of another study²⁵, which concluded that GIC initially produced high amounts of fluoride ions that quickly decreased but continued to release low amounts of fluoride ions throughout the study period. Fluoride exposure from other sources, such as mouthwash or varnish, can recharge glass ionomer materials. The study's results showed a sustained,

slow release of fluoride after varnish application, with a slightly greater release when the varnish remained in contact with the GIC restoration for 24 hours. Therefore, GIC restorations can serve as intraoral devices for slow fluoride release.

The pH, dose, concentration, duration, and frequency of treatment all affect the amount of fluoride released after topical application. This study contradicts the findings of Takahashi et al.²⁶, which reported that fluoride release is proportional to fluoride concentration. According to Nagi et al.²⁷, their study revealed that on the first day after recharging with APF gel, fluoride release exhibited statistically significant mean values, followed by a sharp decline. This might be attributed to the short fluoride recharge time (4 minutes) applied to the specimens in this investigation, which likely recharged only the outermost layer of the samples.

These findings suggest that the choice of recharging material and duration can impact the fluoride release of GIC. Overall, this study supports the idea that fluoride release varies depending on recharging materials and durations. Dentists should consider these factors when using GIC in dental treatments. By selecting the appropriate recharging material and duration, they can optimize fluoride release from GIC and provide effective dental care for their patients.

Within the limitations of the present research, both fluoride gel and varnish proved to be suitable for recharging GIC restorations for the two durations at different time intervals. The study recommends considering the age of the patient and their ability to swallow when selecting a recharging material. For children over 6 years old, recharging with APF gel may provide higher fluoride release. This is important because fluoride has well-documented benefits for dental health, such as preventing tooth decay. However, F varnish may be a safer option for younger children who have difficulty swallowing.

This study found that fluoride release from glass ionomer restorations was highest after 24 hours, compared with 1 week and 1 month. Additionally, the rechargeability of the restorative material after F varnish application for 24 hours was higher than after 12 hours, and the rechargeability after APF gel application for 4 minutes was higher than after 2 minutes. APF gel also released more fluoride than F varnish. Furthermore, longer contact times between the restorative material and the recharging material resulted in greater fluoride uptake compared with shorter durations.

Other types of topical fluoride, such as dentifrice, should be studied to determine their recharging abilities for GIC restorations. Additionally, future studies could use extracted teeth instead of polyethylene molds and artificial saliva instead of deionized water as a storage medium. It is also recommended that fluoride release and recharge be evaluated over extended periods, such as 3 months, 6 months, and 1 year, and the fluoride release and recharging characteristics of GIC restoratives at different pH levels be investigated.

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