Assessment of Fluoride Release and Recharge of Giomer After Recharge With Two Different Topical Fluoride Materials and Two Durations: An *In Vitro* Study

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Abstract

Aims: The objectives of this study were to determine the fluoride release from a giomer after 24 h, 1 week, and 1 month) and to examine the recharge ability of the giomer after recharge with two different topical fluoride materials and two different durations. **Materials and Methods:** This is an *in vitro* study that involved the use of 72 specimens of the tested material; the specimens were divided randomly into three main groups (24 h., 1 week, and 1 month group). Each group consisted of 24 specimens, out of which 12 specimens were recharged with fluoride varnish (six specimens for 12 h and six specimens for 24 h.) and 12 specimens were recharged with APF gel (six specimens for 1 min and six specimens for 4 min). First fluoride release was measured from the tested specimens, and then all of the tested specimens were recharged with different topical fluoride materials with different durations. One-way analysis of variance (ANOVA) and Duncan's multiple range test were used to determine the significant differences among the groups. The statistical results were considered significant at $P \leq 0.05$. **Results:** The statistical ANOVAs showed there were no statistical significant variations in the mean values of release of all specimens in the 24-h and 1-week group, but there was a highly statistically significance difference in the mean values of release of all specimens in the 1-month group, and after recharge with FV (12 h; 24 h) and APF gel (1 min; 4 min), there was a highly statistically significance difference in the mean values of fluoride release after recharge in the groups (24 h, 1 week, and 1 month). **Conclusions:** The amount of fluoride released from the giomer was more in the 24-h. group than in other groups. Recharge with APF gel released more fluoride than fluoride varnish. Increasing the time of giomer contact with the recharging material resulted in a greater uptake of fluoride.

Keywords: APF Gel, Demineralization, Fluoride Varnish, Giomer, Remineralization

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Introduction

Dental caries is an infectious illness induced by a variety of circumstances causing the gradual loss of dental hard tissue components, ultimately destroying the tooth. Tooth decay is caused by changes in the balance of remineralization and demineralization. Minerals are lost from tooth structure during demineralization, whereas they are re-precipitated inside the tooth structure during remineralization.[1]

Dental caries can be avoided by avoiding conditions that promote the demineralization of tooth hard tissues in the oral environment. Fluoride is the most helpful topical drug, exerting an anti-caries action via multiple mechanisms, including promoting remineralization and reducing microbial growth and metabolism.[2]

Fluoride has been well-documented as a key contributor to the decrease in the frequency and severity of dental caries, and it plays an important role in its prevention. Fluoride varnishes (FVs) are professionally administered fluoride treatments that are applied twice or four times per year based on a specific risk assessment of caries in children. This varnish may adhere

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to the tooth surfaces for a reasonably long period, releasing fluoride in an efficient and effective manner.[3]

For more than three decades, topically applied fluoride gels have been widely employed in school-based programs as a caries-prevention strategy. Depending on the kid's age, fluoride gel is often applied by a dental professional or applied by the child themselves under supervision one or more times a year. The child or young people must hold the gel in their teeth and bite into it for around 4 min after it is typically placed on a plate.[4]

Giomer has thoroughly proven that the presence of fluoride ions at low, sustained concentrations over an extended length of time reduces enamel demineralization and promotes remineralization. However, the number of fluoride ions leaching out of the fluoride-containing materials decline over time; hence, "recharging" these fluoride-containing materials must maintain a continually increasing level of fluoride release.[5]

Giomer is a relatively recent revolutionary resin composite filler technology utilized as an attractive direct restorative material for anterior and posterior tooth repair,[6] and it may be used in both primary and permanent teeth.

Aims of the study

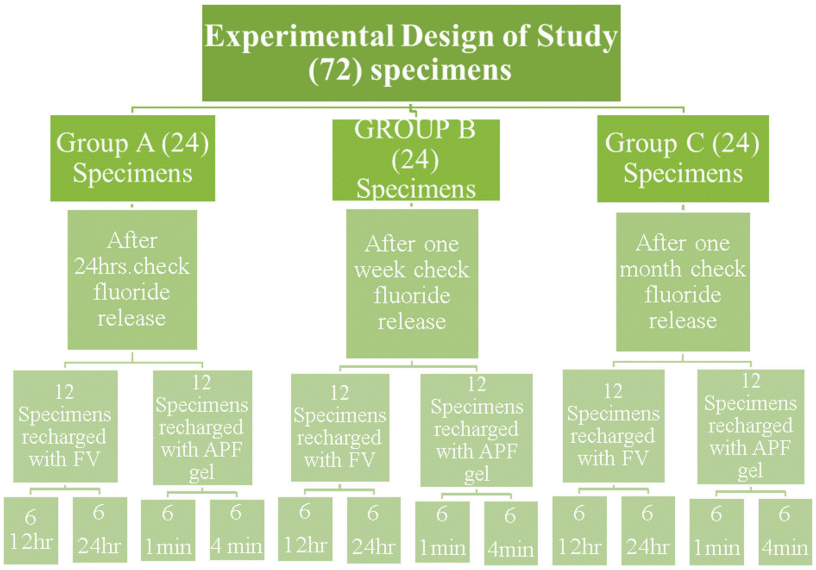
This study aimed to ascertain fluoride release from tooth-colored restorations (giomer) after 24 h, 1 week, and 1 month, as well as to assess the ability of the restorative material (giomer) to recharge after applying fluoride varnish at different time points (12- and 24-h application) and the ability of the giomer to recharge after applying the APF gel at different time points (1- and 4- min application).

The null hypothesis of this study was that all the tested specimens would release the same amount of fluoride ions during the 1-month period before the fluoride varnish and gel treatment and that no difference was observed in the fluoride recharging ability of the dental material tested and the duration of the fluoride varnish and gel application.

Materials and Methods

This *in vitro* study was undertaken at the University of Mosul, College of Dentistry from February to April 2023 (Ethical approval no. UoM. Den). The material used for this study was giomer (Shofu Inc. Japan). A total of 72 specimens were created and randomly distributed among three groups:

Group A: (24 specimens) fluoride release from specimens was measured after the first 24 h. A total of 12 specimens were randomly assigned to the varnish group, and then the giomer was recharged with fluoride varnish (six specimens were coated for 12 h. and six specimens for 24 h). Additionally, 12 specimens were randomly assigned to the fluoride gel group, and the giomer was recharged with APF gel (six specimens for 1 min and six specimens for 4 min).
Group B: (24 specimens) fluoride release from giomer was measured after 1 week. A total of 12 specimens were randomly assigned to the varnish group, and then the giomer was recharged with fluoride varnish (six specimens were coated for 12 h. and six specimens for 24 h). Additionally, 12 specimens were randomly assigned to the fluoride gel group, and then the giomer was recharged with APF gel (six specimens for 1 min and six specimens for 4 min).
Group C: (24 specimens) fluoride release from giomer was measured after 1 month. A total of 12 specimens were randomly assigned to the varnish group, and then the giomer was recharged with fluoride varnish (six specimens were coated for12 h. and six specimens for 24 h). Additionally, 12 specimens were randomly assigned to the fluoride gel group, and then the giomer was recharged with the APF gel (six specimens for 1 min; six specimens for 4 min).



Specimen preparation

Each specimen measured 2 mm in height and 5 mm in diameter.[7] The weight of each specimen was measured by accurate balance to be 0.1 mg.

Methods

Polyethylene molds were prepared by cutting an insulin syringe with a disk fixed to an engine with a mandrill; the dimensions of each mold were as follows: 2 mm in height and 5 mm in diameter. Then, specimens were prepared by filling the polyethylene mold with giomer between two glass slabs, and pressure was applied by using a 50-g weight to eliminate any excessive material.[8]

After the weight was removed, a light cure tip was used to cure the material (wavelength 385–515 nm; light intensity 1200 mW/cm^2). On both surfaces, pass through the glass slides for 40 s. A light cure tip was applied directly on the glass slide.

Specimens were removed from the polyethylene mold by using a tweezer and kept at 37°C in polyethylene containers containing 5 mL of deionized water.[9] Using a radiometer, each of the five cured specimens (Foshan Stardent Equipment Co.) was assessed to check the intensity of the light which was constant.

A fluoride ion-selective electrode was used to assess fluoride release (Eutech Instruments, Singapore). All data were recorded in ppm at 1-day, 1-week, and 1-month intervals.[10]

Procedure

Before measuring fluoride release, the electrode was cleaned with sand paper, washed with distilled water, and dried with tissue, and then standard solutions of sodium fluoride (NaF) were measured with concentration (0.25%, 0.5%, 0.75%, 1%, 1.5%, 2%, 30%, and 40%.[11]The specimens' fluoride release was then assessed for each group. Each set of specimens was sorted into subgroups (six specimens) for recharge with F.V (Proshield, Germany; 12 h; 24 h) and APF gel (Germiphene Corporation, Canada; 1 min; 4 min).

After measuring the release of specimens, six specimens were recharged with F.V (Proshield) for 12 h.[12] and six specimens for 24 h.[13] Each specimen was brushed with F.V., and then after the specific duration, the specimens were cleaned with 2 mL of deionized water and then transferred to a fresh polyethylene container containing 5 mL of deionized water.[14] Additionally, six specimens were recharged with APF gel (Germiphene Corporation) for 1 min, and six specimens were recharged with APF gel for 4 min by immersing the specimens in APF gel, washing with 2 mL of deionized water, and placing in new polyethylene containers containing 5 mL of deionized water. The next day, fluoride release of the specimens was quantified using a fluoride ion-selective electrode; this was done for recharge after 1 day. For the other two groups the specimens were kept in deionized water 24 h. prior to the measurement of fluoride release. After 1 day, the specimens were transferred to a fresh polyethylene container containing 5 mL of deionized water, and the measurement was performed[14] for both 1-week and 1-month groups.

Statistical analysis

Statistical Package for the Social Sciences version 19 (IBM Corp., Chicago, IL, USA), was used for data input and analysis, which included the following steps:

1- A normality test was done to ensure the data's validity.
2- Descriptive statistics which include mean and standard deviation for each individual variable.
3- Duncan's multiple range test combined with one-way analysis of variance (ANOVA): These tests were run to find any significant group differences. At $P < 0.05$, the findings were considered statistically significant.

Results

The Shapiro–Wilk and Kolmogorov–Smirnov tests were used to determine if the collected data were normal. All of the data were found to be regularly distributed, as $P \geq 0.05$.

Table 1 displays one-way ANOVA which compares the fluoride release (ppm) of the specimens after 24 h. and fluoride release after recharge with different materials. There was no statistically significant difference in the mean values of fluoride release of all specimens before recharge, while there was a highly statistically significant difference between groups in the mean values of fluoride release after recharge.

Table 2 demonstrates Duncan's multiple range tests of fluoride release and recharge with fluoride varnish (12 h; 24 h) and APF gel (1 min; 4 min) after 1 day, in which release of fluoride was nearly the same in all groups, regardless of the type of fluoride application and the time of application. Release of fluoride after recharge of giomer with APF gel was slightly greater in the 4-min group than in the 1-min group with no significant difference and release of fluoride after recharge of giomer with fluoride

Table 1: ANOVA results of fluoride release values (ppm) of specimens and fluoride release after recharge after 24 h (1-day group)

		Sum of squares	df	Mean square	*F*	Sig.
Release	Between groups	0.001	3	0.000	2.454	0.093
	Within groups	0.004	20	0.000		
	Total	0.005	23			
Recharge	Between groups	1.057	3	0.352	635.075	0.000*
	Within groups	0.011	20	0.001		
	Total	1.068	23			

df = degree of freedom.
*Highly statistically significant difference at $P \leq 0.01$

varnish was slightly greater in the 24-h group than in the 12-h group and exceeded the amount of fluoride released in the beginning for the APF gel group, while it was slightly less in the varnish group.

Table 3 depicts one-way ANOVA test which compares the fluoride release (ppm) of the specimens after 1 week and fluoride release after recharge with the two different materials. There was no statistically significant difference in the mean values of release of all specimens, while there was a highly statistically significant difference between groups in the mean values of fluoride release after recharge.

Table 4 shows Duncan's multiple range tests of fluoride release and recharge with fluoride varnish (12 h; 24 h) and APF gel (1 min; 4 min) after 1 week, in which release of fluoride was nearly the same with slight differences in the fluoride varnish group (12 h) and ranged between 0.841 and 0.886 ppm. Release of fluoride after recharge of giomer with APF gel was greater in the 4-min group (1.309 ppm) than in the 1-min group, and also differences were observed as the 24-h duration varnish group was better than the 12-h. duration varnish group, which showed the least recharging ability.

Table 5 displays the one-way ANOVA test which compares the fluoride release (ppm) of the specimens in the 1-month group and fluoride release after recharge with two different topical fluorides. There was a highly statistical significance difference in the mean values of release of all specimens, and there was a highly statistical significance difference between groups in the mean values of fluoride release after recharge.

Table 6 demonstrate Duncan's multiple range tests of fluoride release and recharge with fluoride varnish (12 h; 24 h) and APF gel (1 min; 4 min) after 1 month, in which more release of fluoride was observed in the APF gel 4-min group, followed by the APF 1-min group, with no significant difference, and FV 24-h group and then the FV 12-h group (although the difference in the release was slight but statistically it was evident). Release of fluoride after recharge of giomer with APF gel was greater in general when compared with the varnish group, and the values of the fluoride release from the varnish were higher in the 24-h. group in comparison with the 12-h. group.

Discussion

Dental caries, which remains a major public health problem, is the most frequent chronic illness in both children and adults.[15] Enamel, dentine, and cementum

Table 2: Duncan's multiple range test of mean values of fluoride release and recharge with fluoride varnish (12 h; 24 h) and APF gel (1 min; 4 min) for the 1-day group (24-h group)

		Release	Recharge
Fluoride varnish 12 h	Mean	0.924 b	0.849 b
	N	6	6
	Standard deviation	0.014	0.031
Fluoride varnish 24 h	Mean	0.930 ab	0.885 b
	N	6	6
	Standard deviation	0.011	0.029
APF gel 1 min	Mean	0.938 ab	1.283 a
	N	6	6
	Standard deviation	0.015	0.010
APF gel 4 min	Mean	0.944 a	1.289 a
	N	6	6
	Standard deviation	0.013	0.015

Means with different small letters are significant (Vertically)

Table 4: Duncan's multiple range tests of fluoride release and recharge with fluoride varnish (12 h; 24 h) and APF gel (1 min; 4 min) for the 1-Week Group

		Release	Recharge
Fluoride varnish 12 h.	Mean	0.841 b	0.991 d
	N	6	6
	Standard deviation	0.003	0.019
Fluoride varnish 24 h.	Mean	0.886 a	1.035 c
	N	6	6
	Standard deviation	0.048	0.041
APF gel 1 min	Mean	0.870 ab	1.247 b
	N	6	6
	Standard deviation	0.008	0.040
APF gel 4 min	Mean	0.881a	1.309 a
	N	6	6
	Standard deviation	0.008	0.008

Means with different small letters are significant (vertically)

Table 3: ANOVA results of fluoride release values (ppm) of specimens and fluoride release after recharge (1-week group)

		Sum of squares	df	Mean square	F	Sig.
Release	Between groups	0.007	3	0.002	3.880	0.025
	Within groups	0.012	20	0.001		
	Total	0.020	23			
Recharge	Between groups	0.439	3	0.146	154.523	0.000*
	Within groups	0.019	20	0.001		
	Total	0.458	23			

df = degree of freedom.

*Highly statistically significant difference at $P \leq 0.01$

Table 5: ANOVA results of Fluoride Release Values (ppm) of Specimens and Fluoride Release after Recharge. (1-Month Group)

		Sum of Squares	Df	Mean Square	F	Sig.
Release	Between groups	0.175	3	0.058	191.391	0.000*
	Within groups	0.006	20	0.000		
	Total	0.181	23			
Recharge	Between groups	0.833	3	0.278	381.078	0.000*
	Within groups	0.015	20	0.001		
	Total	0.847	23			

df = degree of freedom.

*Highly statistically significant difference at $P \leq 0.01$

Table 6: Duncan's multiple range tests of fluoride release and recharge with fluoride varnish (12 h; 24 h) and fluoride gel (1 min; 4 min) for the 1-month group

		Release	Recharge
Fluoride varnish 12 h	Mean	0.905 b	0.887 c
	N	6	6
	Standard deviation	0.017	0.031
Fluoride varnish 24 h	Mean	0.723 c	0.955 b
	N	6	6
	Standard deviation	0.016	0.028
APF gel 1 min	Mean	0.901 b	1.295 a
	N	6	6
	Standard deviation	0.021	0.007
APF gel 4 min	Mean	0.944 a	1.286 a
	N	6	6
	Standard deviation	0.013	0.032

Means with different small letters are statistically significant (vertically)

of deciduous and permanent teeth are demineralized as a result of dental caries.[16] Secondary (recurrent) caries is described as caries seen around the edges of an existing restoration. In both permanent and primary dentitions, secondary caries is the main cause for restorative failure.[17] One of the most recent developments in fluoride-releasing dental materials is the use of giomer, which combines strength, mechanical resistance, and a polished surface along with a visually appealing appearance.[18]

Fluorides are used therapeutically to inactivate developing carious lesions and are crucial in the prevention of dental cavities. The effects of fluoride are mostly realized when administered topically, and they are further boosted when combined with appropriate oral hygiene.[19]

Due to their exceptional esthetics, perfect color matching, smooth surface finish, little microleakage, and clinical stability, giomers are a great solution for treating non-carious cervical lesions such as abrasion or erosion and class V cavities in primary or permanent teeth.[20]

Additionally, the hydrogel around the inner glass of S-PRG particles includes acidified water, which accelerates the release of fluoride due to the ongoing disintegration of the fluoride-containing glass core.[21]

The present investigation showed that in comparison to the 1-week and 1-month groups, the fluoride release was somewhat higher in the first 24 h. Later on, the release of fluoride was slow, which may be explained by the fact that fluoride diffuses via cement cracks and fissures as glass dissolves in storage media and leaches out of the substance, as noticed by.[22]

The current study showed no burst effect; this result is in line with the results of other studies.[23,24] This explains why, throughout the course of the current experiment, the emission of fluoride from giomer steadily decreased in the current investigation in which release in the 24-h. group was 0.944 ppm, and at the end of study (after 1 month), the release of fluoride was 0.723 ppm.

According to the present study, giomer releases most fluoride after 24 h and that the quantity of fluoride release stays largely stable for 1 week; this is in line with.[12] A steady reduction in fluoride release was noticeable after 1 month. However, until the 30th day, fluoride release resumed a more or less steady pattern. This result was consistent with.[25]

The present study's findings conflict with[26]; they evaluated the quantity of fluoride release from giomer and two varieties of composite after 1 day, 7 days, and 1 month. They found that Giomer had a significant drop in fluoride release from day 1 to day 30.

The current investigation showed that the average fluoride release levels before and after FV replenishment (12 h.; 24 h.) and APF gel (1 min.; 4 min.), in which maximum release was obtained in the first 24-h. group and release of fluoride after recharge was higher in the APF gel (4 min more than 1 min) and fluoride varnish (24 h more than 12 h). The more the contact time of the fluoride product with the giomer, the larger is the absorption of fluoride.

When comparing results after recharge with two different materials with two different durations, it has been found that there are no similar studies to compare the results of this study.

Acidulated phosphate fluoride (APF) gel 1.23% is the most used fluoride application agent due to its stability and widespread commercial availability. The tooth enamel is etched by the acid phosphate in the gel; the mineral

hydroxyapatite is subsequently replaced by fluoride ions to produce fluoroapatite, which is more durable and acid-resistant.[27]

In pediatric dentistry, fluoride varnish (NaF 5%), which is professionally administered, is frequently used as a caries-prevention strategy. The evaluated restorative materials were recharged using it in a manner reminiscent of a clinical setting. The amount, frequency, concentration, and time of application for a topical fluoride agent determine its capacity to recharge a substance.[23]

The specimens that were recharged with APF gel released more fluoride than the specimens which were recharged with FV, despite the time of application of rechargeable materials; these findings were consistent with those of the research done by Gao.[28] The most likely reason for this was the low pH of the APF gel, which degrades the surfaces of many restorative materials and increases the release of fluoride and other ions. Hydrofluoric and phosphoric acids are found in the APF gel. Also, the materials' permeability would have a major impact on the fluoride recharging process.

The ability of a topical fluoride agent to replenish a material is contingent upon several factors, including the dose, frequency, concentration, and duration of application. The topical fluoride's temporary effect causes the fluoride re-release that occurs immediately after recharging; nevertheless, the ability of fluoride to infiltrate into the materials' pores and be stored for re-release is responsible for the release that occurs over the next several days.[21]

The current study found that recharging the giomer with APF gel works better in the APF gel 4-min group than in the 1-min group. Moreover, there were variations in the amount of fluoride released from the tested material over time and in the giomer's capacity to recharge itself after applying fluoride varnish or gel for varying lengths of time.

In summary, and within the parameters of the ongoing *in vitro* investigation, giomer restoration can be used as an option in treatment of caries in childhood. There was a stable and sustained release of fluoride for the entire experiment time (1 month). This release of fluoride into the surrounding environment may slow or stop demineralization, which means that there will be a lower chance of recurrent caries than with other non-fluoride-containing composites. It may also have therapeutic implications because it will weaken any bacteria that may have survived in the inner dentine of a carious tooth. Acidic, demineralizing, and plaque fluids with fluoride concentrations as low as 1 ppm can halt the demineralization process and promote the dentin and enamel's remineralization. Because fluoride released from the restoration material (giomer) reduces enamel and dentin demineralization around restorations, albeit to varying degrees depending on their fluoride release ability, fluoride-releasing restorative materials are therefore preferred for use in children and elderly patients in order to prevent formation of secondary caries.

Limitations of the study

The study had certain limitations, during the procedure, for example, the preparation of polyethylene molds during cutting by an engine to have an accurate mold of 2 mm height. Future research should concentrate on performing *in vivo* investigations to assess the clinical behavior and fluoride release of the tested material, as the current work was conducted in an *in vitro* setting.

Conclusions

The following conclusions were drawn from the research:

1. The 24-hour group released more fluoride from the studied materials (giomer) than the 1-week and 1-month groups.
2. . The 24 h group released more fluoride from the tested specimen (giomer) than the 12 h group after recharging with fluoride varnish.
3. The 4 min group released more fluoride from the tested specimen (giomer) than the 1 min group after recharging with APF gel.
4. Recharge with APF gel released more fluoride than recharge with fluoride varnish.
5. Increasing the contact time of giomer with the recharging material resulted in significant recharge of fluoride compared with the short duration for which the fluoride comes in contact with giomer.

The results of the current investigation showed changes in the fluoride released from the substance under examination with time (Giomer) and in the material's capacity to recharge when fluoride varnish or gel is applied for different periods of time, so that the null hypothesis was refused and the alternative hypothesis was accepted because there were variations in the fluoride released from giomer throughout the duration and the recharging ability of giomer when professional fluoride was applied for different time points and different forms (varnish and gel).

The suggestions that can be improved in the future are as follows:

1. To determine the release of fluoride and the recharging characteristics of giomer restoration at different pH values.
2. To calculate the quantity of fluoride released and recharged over longer time periods, such as 3 months, 6 months, and 1 year.

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Author contributions
All researchers participated in designing the research. The first researcher carried out the practical work. The second researchers completed the task of statistical analysis, devising tables, and writing.

Ethical policy and institutional review board statement
An *in vitro* study.

Patient declaration of consent
Not applicable.

Data availability statement
Not applicable.

List of Abbreviations
ANOVA Analysis of variance
APF Acidulated phosphate fluoride
df Degree of freedom
F.G. Fluoride gel
F.V. Fluoride varnish
HA Hydroxyapatite
mg Milligram
N Number
Naf Sodium fluoride
pH Potential of hydrogen

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