Original Article

Fluoride Release and Recharge in Conventional Varnishes, Compared to a Giomer and a Resin Modified Glass Ionomer

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Introduction: Fluoride varnishes are used for caries prevention and treatment of dentin hypersensitivity, and its main purpose is to prolong the contact time between fluoride and tooth. The present study aimed to compare the amount of fluoride released and recharge from three conventional varnishes with resin-modified glass ionomer (RMGI). **Methods:** This experimental *in vitro* study was carried on blocks of human teeth extracted for orthodontic reasons. Three commercially available fluoride varnishes (Fluor protector (FP), Duraphat (DP), Clinpro White Varnish (CWV)) a Giomer (PRG-Barrier Coat), and an RMGI (Clinpro XT) were applied in these blocks, divided into five groups (eight samples each one). The readings were carried out using an ion-selective electrode and a potentiometer. After 30 days of study, the recharge capacity of these materials was evaluated immersing the samples in 20,000 parts per million (ppm) sodium fluoride gel. **Results:** Significant differences were found when comparing FP with the rest of the materials analyzed in this study since it released the lowest amount of fluoride with 1.01 ppm. The Giomer released 1.90 ppm, whereas CWV and DP released the highest amount of fluoride with 5.41 ppm and 4.76 ppm, respectively. The RMGI was more constant during the first five days and demonstrated a greater recharge capacity. **Conclusion:** All varnishes demonstrated the greatest fluoride release during the first 24 h, and a marked decrease was observed after this period. The RMGI presented a considerable amount of fluoride and the best capacity for recharge.

Keywords: Fluoride release, Giomer, Glass Ionomer, Recharge, Varnishes

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Introduction

Fluoride varnishes are used worldwide for caries prevention and treatment of dentin hypersensitivity. These materials were introduced as an alternative method of topical application (1). Their effectiveness, safety, and easy application have made them a perfect candidate for dental prevention protocols (2). Studies have mentioned that the use of a topical fluoride could be the most effective protocol in the prevention and control of early childhood caries (3). The risk of developing cervical dentin hypersensitivity increases in treatments of caries and periodontal disease, and fluoride varnishes are a useful option for the treatment of this pathology (4). Dentin hypersensitivity is one of the most common symptomatic conditions affecting oral comfort and can be defined as a short sharp pain arising from exposed dentin in response to stimuli that cannot be ascribed to any other dental pathology (5).

This pathology, which may affect 8%-57% of the adult population, is associated with exposed dentin, gingival recession, abrasion, attrition, erosion, abfraction, and improper brushing habits (6). The prescription of fluorine-containing products has become very popular, since the application of fluoride salts causes calcium fluoride precipitation inside the tubules through the interaction between sodium fluoride and calcium contained in dentin hydroxyapatite crystals (7). The highest and constant fluoride release in dental materials is indispensable to prevent caries and treat hypersensitivity.

New fluoride varnishes are designed to release fluoride slowly over a prolonged period and are supposed to have the ability of recharging. Therefore, it is necessary to identify the varnish which has the highest and more stable amount of fluoride and is capable of recharging. The present study aimed to compare the amount of fluoride released and the recharge capacity from three conventional sodium fluoride varnishes with a Coat Giomer and Resin modified glass ionomer (RMGI).

Materials and Methods

Sample preparation

The materials used in this study were three fluoride-releasing varnishes (Duraphat “DP”, Clinpro White Varnish “CWV”, Fluor protector “FP”), a Giomer (PRG Barrier Coat “BC”) and one Resin modified glass ionomer (Clinpro XT Varnish “CXT”) divided in 5 groups: with eight samples of each group. The materials used were manipulated in accordance with the instructions of the respective manufacturers. (Table I).

Table I. Materials used in this study, instructions and detail compositions

Varnish	Compositions	Manufacturer	Instructions
Duraphat	5% Sodium fluoride, olophony, ethyl alcohol, shellac, mastic, saccharin, aroma, white beeswax 2.26% 22,600 ppm	Colgate Palmolive, New York, USA	After clean the tooth and dry field with cotton and air syringe, apply a thin layer to tooth surface using a cotton applicator.
Clinpro White Varnish	5% Sodium fluoride in an alcohol-based solution of modified resin 2.26% 22,600 ppm	3M ESPE, Minnesota, USA	After clean the tooth and dry field with cotton and air syringe, apply the entire contents of the individual container to the dark inner circle on the foil wrap of the dosing guide. Carefully mix the varnish with the brush, keeping the material inside the inner circle. Evenly disperse the varnish.
Fluor protector	0.9% difluorsilane in a polyurethane varnish base with ethyl acetate and isoamylpropionate solvents. This corresponds to 1 mg fluoride 0.1% 1000 ppm	Ivoclar-Vivadent, Schaan, Liechtenstein	After clean the tooth and dry field with cotton and air syringe, apply a thin layer to tooth surface using a suitable single-use applicator. Evenly disperse and dry the varnish with an air syringe.
PRG-Barrier Coat	fluoroboroaluminosilicate glass and a polyacrylic acid aqueous solution Base: glass powder, purified water, methacrylate monomer, S-PRG filler, phosphonic acid monomer Activator: methacrylate acid monomer, bis-MPEPP, carboxylic acid, TEGDMA, catalyzer.	Shofu Inc, Kyoto, Japan	Put one drop of activator into base and mix together. Apply thin layer of the mixture, Light-cure for 10 s.
Clinpro XT Varnish	Liquid: HEMA, water, camphorquinone, calcium glycerophosphate and polyalkenoic acid Paste: HEMA, Bis-GMA, water, initiators and fluoroaluminosilicate glass.	3M ESPE, Minnesota, USA	Apply acid etchant for 15s with 35% phosphoric acid. Rinse with water. Apply air for 5s. Mix paste/liquid components together rapidly for 15s (2.5 min working time). Apply thin layer to tooth surface. Light cure for 20s.

Forty rectangular blocks were obtained from human premolars and molars extracted for orthodontic reasons. The teeth received prophylaxis with low speed handpiece, later were cleaned with deionized water to remove any surface debris. The samples were sectioned using a carbide disc under irrigation, with dimensions (8mm x 4mm x 2mm). The teeth blocks were washed with deionized water and dried for 20 seconds. All light-curing materials were polymerized using a led device (Elipar Light Curing Unit 3M ESPE) verifying with a radiometer that the intensity of light emitted by the led lamp had a minimum value of 400 mW/cm^2. The samples were placed at room temperature for 10 minutes, after were stored in plastic bottles with 5 ml of deionized water, the samples were conserved at 37º C for 30 days, and measured in days 1, 2, 5, 15 and 30.

To determinate the amount of fluoride in varnishes, an ion selective electrode for sodium fluoride (model 1011, Hanna Instruments, USA) and a potentiometer (model HI 3222, Hanna Instruments) were necessary. The total ionic strength adjustment buffer (TISAB) solution was used to keep the pH stable and to prevent that fluoride ion produce complexes with different cations (8-10).

The fluoride solutions used in this study were prepared in concentrations of (1ppm, 2ppm, 10ppm, 100ppm, and 1000ppm). TISAB was used to get calibration slope with fluoride solutions; in equal volumes (25ml of each fluoride solution and 25ml of TISAB) were placed and mixed in a plastic bottle of 100ml; the device was calibrated until the readings were reached.

Fluoride determination

At the end of each period, the samples were removed from their respective plastic bottle and each sample was washed with 1ml of deionized water inside the bottle that originally contained it, in order to preserve the total amount of fluoride released by each sample. After we proceed to introduce the sample in a new 5ml plastic bottle with deionized water. Five ml of solution used to store the sample, and 1ml used to wash the sample gave a total of 6ml that were mixed with 6 ml of TISAB, because this solution works in a proportion 1:1.

The readings were performed with the electrode immersed at least 3 centimeters in the solution where was previously the sample, under magnetic stirring with a time interval of 3 minutes for each measurement. The values of the readings were expressed in parts per million.

The data were analyzed with analysis of variance (ANOVA) one way and for repeated measures using the statistical program SPSS Statistics (IBM New York, USA).

Fluoride recharge

After thirty days of study, the samples were immersed in 1 ml of 2% (20,000ppm) sodium fluoride gel (Ionite Borgatta, Mexico) for 4 minutes and subsequently rinsed with a sterile solution. The fluoride released after the recharge was determined at 24, 48 and 72 hours through the procedure previously described.

Results

The highest fluoride release of each varnish occurred on the first day. From the second day, a slow steady decline began in fluoride release and continued until day 30, except for Fluor protector (FP), demonstrating a marked decline since the first day. Figure 1 clearly displays the pattern of fluoride released for each varnish during study time. The amount of fluoride release according to the time intervals is represented in TableII, and Duraphat (DP) and ClinPro White Varnish (CWV) showed a greater concentration of fluoride during the first day

Table II. Fluoride release and recharge in varnishes under study

Group	1 day	2 days	5 days	15 days	30 days	1dayAR	2daysAR	3daysAR
DP	4.76±1.43	1.75±0.62	1.00±0.0	1.00±0.0	1.00±0.0	1.00±0.0	1.00±0.0	1.00±0.0
CWV	5.41±1.07	1.70±0.73	1.05±0.08	1.00±0.01	1.00±0.0	1.02±0.01	1.00±0.0	1.00±0.0
FP	1.01±0.01	1.00±0.0	1.00±0.0	1.00±0.0	1.00±0.0	1.00±0.0	1.00±0.0	1.00±0.0
BC	1.90±0.53	1.05±0.07	1.01±0.01	1.00±0.0	1.00±0.0	1.57±0.46	1.06±0.05	1.00±0.0
CXT	4.31±1.70	2.21±0.84	1.47±0.21	1.13±0.06	1.02±0.02	1.89±0.55	1.09±0.07	1.01±0.01

Fluoride release from each varnish under study represented in ppm (parts per million), analyzed in different periods and three days after a recharge with a sodium fluoride gel. (AR: After Recharge)

FIGURE

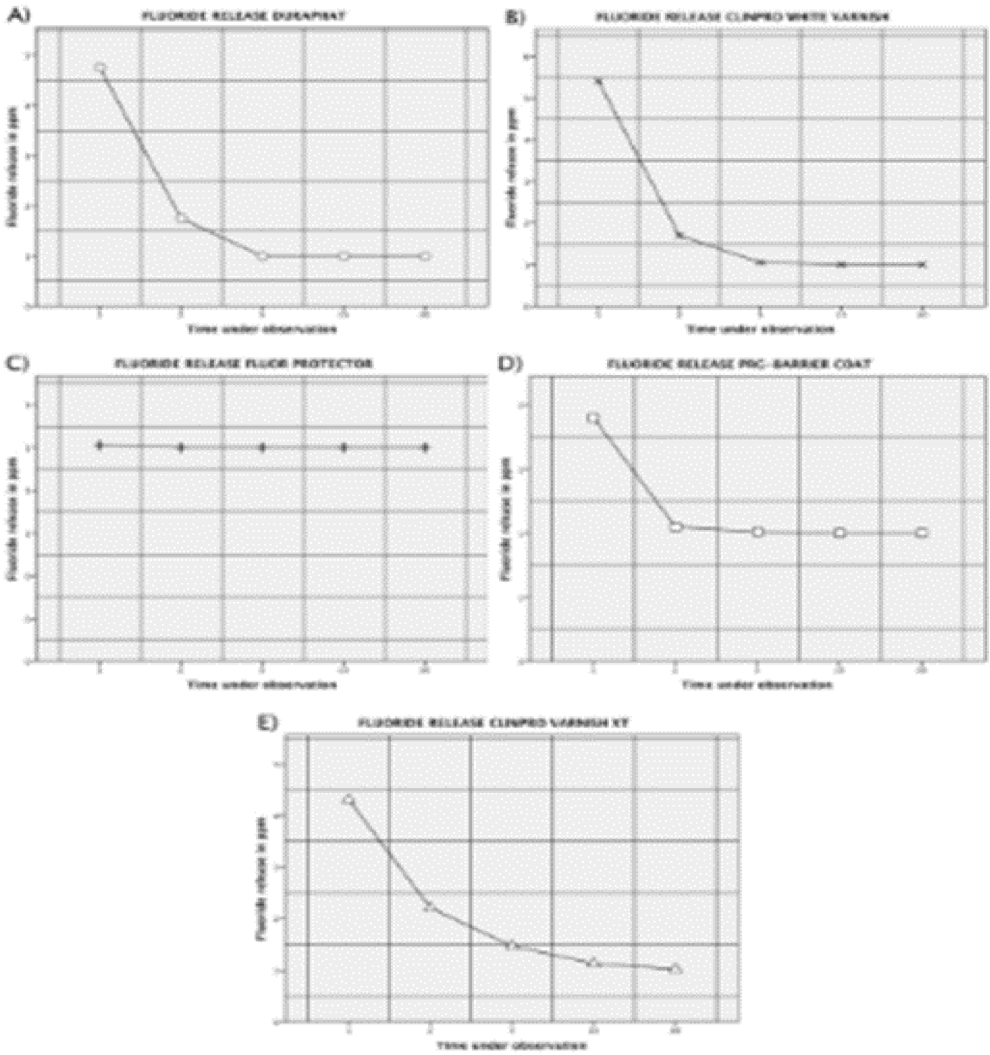

Figure 1. Fluoride released from varnishes during 30 days.

The analysis of variance (Table III) illustrated significant differences during the first day when DP, CXT, and CWV were compared with FP and BC since these varnishes released a higher concentration of fluoride. On the second day, only CXT displayed significant differences from BC and FP since this varnish showed a lower decrease. From the fifth day, CXT showed a more constant fluoride release since significant differences were observed compared to other varnishes. The behavior of materials in the established periods of the study was determined using the repeated measures ANOVA after Mauchly's test of sphericity (Table IV). Differences were observed in terms of time, and FP was the only material that did not display any significant differences when the first day was compared with other periods of study since it showed the lowest values from the first day. The DP displayed significant differences between days one and two, signifying a marked decrease of fluoride in this varnish after the second day.

The CWV showed significant differences between day one and the other established periods. This varnish did not demonstrate any difference between days 5 and 15, as well as days 15 and 30, since fluoride levels were minimal. In the BC group, marked differences were observed only between the first day and the rest of the periods, and this material presented the most significant decrease on the second day. The CXT presented a more constant fluoride release, and this could be the reason why differences were observed between all of time intervals. The first day showed significant differences with the other periods, and day two demonstrated a difference when compared with day 30. Day 5 was different from days 15 and 30, and significant differences were observed between day 30 and the rest of the time periods.

The concentration of fluoride released once the samples were recharged with the fluoride gel is presented in Table 1. The highest concentration of fluoride mainly occurred in groups BC and CXT 24 h after the application of the gel. For group CWV, the amount of fluoride was minimum, whereas DP and FP did not show any release. After 48 h of recharge, the amount of fluoride decreased to minimum levels in BC and CXT groups. After 72 h, the fluoride released in all groups was practically null.

Table III. One-way ANOVA test. Significative differences between the groups at different periods.

Varnish	Comparision	Day 1	Day 2	Day 5	Day 15	Day 30
PRG-Barrier Coat	Clinpro White Varnish	.000	.186	.953	.992	.999
	Duraphat	.000	.130	1.000	1.000	.999
	Fluor protector	.522	1.000	1.000	1.000	.999
	Clinpro XT	.001	.002	.000	.000	.001
Clinpro White Varnish	PRG-Barrier Coat	.000	.186	.953	.992	.999
	Duraphat	.780	1.000	.895	.992	.984
	Fluor protector	.000	.130	.895	.992	.984
	Clinpro XT	.316	.397	.000	.000	.002
Duraphat	PRG-Barrier Coat	.000	.130	1.000	1.000	.999
	Clinpro White Varnish	.780	1.000	.895	.992	.984
	Fluor protector	.000	.088	1.000	1.000	1.000
	Clinpro XT	.931	.507	.000	.000	.000
Fluor protector	PRG-Barrier Coat	.522	1.000	1.000	1.000	.999
	Clinpro White Varnish	.000	.130	.895	.992	.984
	Duraphat	.000	.088	1.000	1.000	1.000
	Clinpro XT	.000	.001	.000	.000	.000

Clinpro XT	PRG-Barrier Coat	.001	.002	.000	.000	.001
	Clinpro White Varnish	.316	.397	.000	.000	.002
	Duraphat	.931	.507	.000	.000	.000
	Fluor protector	.000	.001	.000	.000	.000

Table IV. ANOVA test for repeated measures

Fluoride release-effect of time in each group		**PRG-BC**	**Clinpro WV**	**Duraphat**	**Fluor Protector**	**Clinpro XT**
Day 1	Day 2	.013	.000	.001	1.000	.008
	Day 5	.018	.000	.001	.796	.011
	Day 15	.020	.000	.001	.950	.012
	Day 30	.020	.000	.001	.923	.009
Day 2	Day 1	.013	.000	.001	1.000	.008
	Day 5	.672	.325	.106	1.000	.220
	Day 15	.787	.313	.107	.796	.089
	Day 30	.732	.309	.108	1.000	.050
Day 5	Day 1	.018	.000	.001	.796	.011
	Day 2	.672	.325	.106	1.000	.220
	Day 15	1.000	1.000	1.000	1.000	.035
	Day 30	1.000	1.000	1.000	1.000	.006
Day 15	Day 1	.020	.000	.001	.950	.012
	Day 2	.787	.313	.107	.796	.089
	Day 5	1.000	1.000	1.000	1.000	.035
	Day 30	1.000	1.000	1.000	1.000	.043
Day 30	Day 1	.020	.000	.001	.923	.009
	Day 2	.732	.309	.108	1.000	.050
	Day 5	1.000	1.000	1.000	1.000	.006
	Day 15	1.000	1.000	1.000	1.000	.043

Discussion

The minimum amount of fluoride that must be released to inhibit demineralization and promote remineralization has not been precisely recognized (11). In the case of fluoride toothpaste, some authors indicated that this value would be between 0.05% (225 ppm) and 0.2% (900 ppm). They pointed out that concentrations of 0.2% have a significant effect on caries prevention in adults, and lower concentrations are recommended for children (12). Comar et al (13). suggested that a low salivary fluoride level, around 0.04, has been correlated with a significant protective effect on dental caries. Nonetheless, the use of dental materials with the highest and prolonged fluoride release is advantageous since the enamel solubility is low when the fluoride ion is present in the saliva and biofilm (14-15), and fluoride-releasing restorative materials are able to reduce the occurrence of secondary caries (16).

Based on the results of the current study, it can be argued that varnishes release between 1.01 and 5.41 ppm during the first 24 h. From the second to fifth day, an average of 1.00-2.21 ppm was observed, while all materials released only small amounts of fluoride within day 5-30. In the present study, the CWV was the varnish with the initial highest fluoride release. This result is consistent with those reported by Bolis et al (17). Who compared CWV with DP and reported low fluoride release for the latter.

Hazelrigg et al (18). determined the remineralization ability in three fluoride varnishes and they found no significant differences in terms of the amount of remineralization between the varnishes. These authors concluded that all varnishes were able to remineralize the incipient carious lesions. The BC showed low levels of fluoride release, and released 1.90 ppm on the first day and did not demonstrate any amounts greater than 1.05 ppm from the second day. Some studies have recognized its ability to remineralize enamel probably due to the fact that in addition to fluoride, Surface Pre-reacted Glass-ionomer (S-PRG) technology release other ions that could induce remineralization (19).

The DP was one of the varnishes that released a high amount of fluoride, releasing 4.76 ppm, compared to other varnishes, such as FP that only released 1.01 ppm. These differences can be attributed to the amount of sodium fluoride in each one (Table 1). In agreement with the results of the present study, Virupaxi et al (20). reported that varnish XT release consistently more fluoride than FP over an extended period of time, and FP had the lowest rate of fluoride release among all the varnishes that were compared. On the contrary, other studies evaluated the antibacterial effect of FP which had the greatest inhibitory effect although it contained a lower fluoride concentration (21). In another study, the demineralization inhibitory effect of FP and the other two varnishes was evaluated, and the first obtained the least values for calcium and phosphorus dissolution (4).

Castillo et al (22). reported a higher release when the varnish was applied three times a week (9.56±1.70 μmol), as compared to being applied only once a week (5.73±1.47 μmol). In the current study, only one application of varnish was placed, and the values of fluoride released were similar to those reported by the authors, with values of 5.41, 4.76, and 4.31 ppm for CWV, DP, and Clinpro Varnish XT, respectively. Godoi et al. (23) compared the total fluoride concentration of CWV and DP, concluding that the latter showed higher fluoride content. Nevertheless, the authors reported that both materials presented a total fluoride content lower than what was declared by manufacturers. Mainly CWV with only 7,336.27 ppm, according to the manufacturers, CWV and DP have a concentration of 22,600 ppm.

Some studies evaluated fluoride release in hours and reported that out of the four varnishes evaluated in their study, three varnishes presented the maximum release during the first 4 h, whereas the other varnish presented the highest fluoride release in the first 8 h (24). Along the same lines, another study indicated that the peak of fluoride release occurred in the first 3 h (2). Piesiak-Pańczyszyn and Kaczmarek (25) evaluated the cumulative fluoride release of DP during 168 h (7 days), concluding that this material released 5.72 ppm in this period, and the highest fluoride release was observed in the first hours after application. Nevertheless, in the current study, after five days, a cumulative fluoride release of 7.51 ppm was detected, representing similar values. Moreover, the highest fluoride release of this material was observed during the first 24 h. Although the present study did not assess the fluoride release in hours, the obtained results are in line with the findings of the aforementioned study since the highest amounts of fluoride were released in the first 24 h.

Regarding the recharge, an increase was observed in fluoride concentration mainly in groups BC (Giomer) and CXT (RMGI), and concentration decreased after 48 h, while it completely disappeared after 72 h. It can be observed that RMGI showed the greater capacity of recharge, which is in line with a study conducted by Nassar et al. (26) who reported that RMGI released a considerable amount of fluoride, being surpassed only by conventional glass ionomer cement, and they showed a greater capacity when they were subjected to a recharge. There is sufficient evidence on the effectiveness of varnishes as a preventive strategy, accompanied by correct tooth brushing and an adequate diet. Oral health programs should include varnishes as complementary aid preventive. Therefore, it is suggested to develop materials that can maintain a greater and more constant fluoride release to improve preventive treatments.

Conclusion

All materials analyzed in the present study demonstrate the greatest fluoride release in the first 24 h, followed by a marked decrease. After five days of analysis, all varnishes showed minimum levels of fluoride. Clinpro Varnish XT presented the highest and more stable fluoride release, as well as the best capacity of recharge. The fluoride release *in vitro* from FP and the Giomer was significantly lower on the first day of the study, as compared to the other varnishes.

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Conflicts of interest

The authors declare that they have no conflict of interest regarding the publication of the present study.

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