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Fluoride release and recharge of enhanced resin modified glass ionomer at different time intervals

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ABSTRACT

Aim: To investigate the fluoride release and recharge from two fluoride releasing materials at different time intervals.

Materials and methods: Sixty specimens were prepared then equally divided into 12 groups (n = 5/group), representing materials used; one resin modified glass ionomer restoration (RMGI); Fuji II LC and one enhanced RMGI; ACTIVA Bioactive-Restorative. Each material was evaluated for its fluoride release before and after fluoride recharge using a topical fluoride recharging gel at 1, 2, 7, 14, 21 and 28 days.

Results: There was a statistical significant difference between different tested time intervals regarding fluoride release before and after fluoride recharge for both tested materials, where ($p \leq 0.001$). The highest mean value of fluoride release was in (Day-1), and the least mean value of fluoride release was in (Day-28). There was no statistical significant difference between both materials at each time interval regarding fluoride release before recharge. While after fluoride recharge; RMGI (Fuji II LC) showed higher fluoride release mean values compared to the enhanced RMGI (ACTIVA) at each time interval (1, 2, 7, 14, 21, 28 days).

Conclusion: RMGI and enhanced RMGI, showed the same pattern of fluoride release in deionized water, but RMGI was more successful to be recharged.

1. Introduction

Recurrent caries is recorded to be the common cause of restorations failure in dental clinics. Efforts in industrialized dental materials are continuing to formulate their compositions to decrease recurrent caries formation. Fluoride containing restorative materials gained great attention over the last two decades. Fluoride decreases caries activity by being a biocide and by decreasing the solubility of enamel and dentin through its integration into tooth tissue to form fluoroapatite. Moreover, it has been revealed that fluoride aids to remineralize impaired tooth tissue after demineralization [1,2].

All fluoridated dental materials demonstrated varying degrees of fluoride reduction over time [3]. Fluoride is generally integrated into these materials in the form of either NaF, CaF_2, SnF_2, KPF_6, YbF_3, or fluoro-alumino-silicate glass. Due to their different solubility and the amount of fluoride release for each substance is dissimilar. The rate and pattern of release of fluoride ions from restorative materials depends on many elements such as; structure of the materials, temperature, mixing technique, powder liquid ratio, pH and media of the surrounding environment, and the exposed part to the oral environment [4]. These materials frequently act as fluoride reservoirs and can also be recharged from a topical source [5]. The main problem, is when such salts dissolve to release fluoride, they leave voids in the matrix which may affect the materials properties [6].

Glass-ionomers (GIC) are considered to be the conventional restorative materials that are bioactive with heaps of uses in all restorative techniques. But their sensitivity to moistness is one of their main drawbacks. Changes in their structures have been made to overcome this defect. The resin modified glass ionomer (RMGI) materials appear to have considerable benefits, keeping the benefits of fluoride release and adhesion [6].

Till now, conventional GICs and RMGICs are still considered the exclusive materials with higher fluoride release ability and could be clinically specified to repair decayed non-biting areas in high caries risk patients [6,7].

Activa Bioactive-Restoratives are lately introduced enhanced RMGIs, which their manufacturer clams to possess the general properties of a RMGI with modified resin matrix with enhanced resilience and physical properties. Thus, an enhanced RMGI was an interest to be evaluated with respect to its fluoride release and recharge properties.

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The aim beyond this study was to compare the fluoride release and recharge properties of Activa-enhanced RMGIs to conventional RMGIs at different time intervals.

2. Materials and method

2.1. Study design and specimen grouping

Sixty cylindrical-shaped specimens were prepared and divided equally into 12 groups (n = 5/group), representing the two fluoride-releasing restorative materials used in the study [RMGI; Fuji II LC (GC Corporation, Tokyo, Japan) and [an enhanced RMGI; Activa Bioactive-Restorative (Pulpdent Corporation, Oakland Street, Watertown, MA, USA)]. Each material was evaluated for its fluoride release before and after fluoride recharge using a topical fluoride recharging gel (1.23% Alpha-PRO®APF, Dental Technology, Lincolnwood, Illinois, USA) at 1, 2, 7, 14, 21 and 28 days.

2.2. Specimens' preparation

Split Teflon molds were fabricated to standardize the dimensions of each tested specimen. The molds dimensions were 3 mm thickness and 6 mm in diameter [8,9]. The split Teflon mold was encircled with a copper ring to stabilize the mold during specimen preparation. Each mold was placed on the top of a microscope glass slide and a Mylar strip, each mold was then filled in two increments with either of the tested materials. Each increment of the inserted material was photo-polymerized for 20 s according to manufactures recommendations using LED light-curing device (Elipar S10, 3M ESPE; USA). Then the second increment of the material was introduced into the mold and a second Mylar strip was used to cover the top side of the mold in order to prevent formation of oxygen inhibited layer. Another microscope glass slide and 1 kg load were applied over the second Mylar strip for 30 s to guarantee a reliable packing of the prepared specimens [10].

Applied load and microscope slide were removed and the top surfaces of the second increments of each specimen were photo-polymerized for 20 s according to the manufacturers' directions using LED Elipar S10 light curing unit. The guiding tip of the light curing unit was held centered in direct contact with the second Mylar strip. The light output intensity of the LED light curing device was ≥800 mW/cm^2. A portable radiometer (Curing Radiometer, Demetron, Danbury, CT, USA) was equipped to monitor the power intensity of the light curing device throughout the study. After photo-polymerization, the cylindrical-shaped specimens were removed from their molds and flashes were gently removed manually using 600-grit SiC paper [11], rinsed continuously with tap water [12] for 1 min and then rechecked for their diameter and thickness using a digital caliper.

Each specimen was then stored in 5 ml of deionized water at 37 °C in a tightly sealed polyethylene test tube. Each specimen was moved to a new polyethylene test tube with 5 ml of fresh deionized water that was replaced every 24 h. The previous procedure was repeated for each specimen for 28 days. The fluoride ion release measurement was tested using ion chromatography analyzing device at day 1, 2, 7, 14, 21 and 28 respectively.

2.3. Fluoride recharge

After fluoride release measurement at the 28th day, each one of the specimen was carefully rinsed with deionized water, then recharged with fluoride by being completely dipped in a plastic well filled with Alpha-PRO®APF gel to make sure that it was applied to all surfaces of the specimen and kept in position for 4 min [13]. Rinsing of the specimens done using deionized water for 1 min to remove any gel residues. Then each recharged specimen was stored in deionized water, in tightly sealed polyethylene test tubes at 37 °C. Each specimen was removed again to a new polyethylene test tube that contained 5 ml of deionized water which was replaced every 24 h. The previous procedure was repeated for each specimen for another 28 days. The release of fluoride ion measurement after recharge with 1.23% Alpha-PRO®APF gel was done at the same release days as before; at 1, 2, 7, 14, 21, 28 days respectively.

Table 1
Three-way ANOVA for the effect of different variables on fluoride release.

Source of variation	Type III Sum of Squares	df	Mean Square	*F*-value	*P-value*
Before and After recharge	1344.929	1	1344.929	2773.797	< 0.001*
Material type	28.304	1	28.304	58.375	< 0.001*
Time interval	706.874	5	141.375	291.573	< 0.001*
Before and after recharge x Material type x Time interval interaction	39.988	5	7.998	16.494	< 0.001*

df: degrees of freedom = (n-1), * Significant at P ≤ 0.05.

2.4. Fluoride release and recharge measurements

From each container; 5 ml of the deionized water of the test days were obtained and 0.5 ml of TISAB (total ionic strength adjustment buffer solution, Germany) was further added to it. The concentration of the fluoride ion was measured after equilibration of the solution in duplicate by a fluoride-specific ion electrode (96-09-00 Orion Research Inc., Cambridge, MA, USA), and then it was calibrated with multiple standard solutions of 0.1, 1, 10, 50 and 100 ppm fluoride.

Every 10 measurements, recalibrations were performed using standard solutions of 1 and 10 ppm fluoride [14,15]. The fluoride ion release measurement was done before and after recharge with Alpha-PRO®APF gel at 1, 2, 7, 14, 21, 28 days.

2.5. Statistical analysis

Calculation of the mean and standard deviation (SD) values were done for each group. Kolmogorov-Smirnov and Shapiro-Wilk tests were used to explore data for normality. Parametric (normal) distribution was shown. To compare between two groups the independent sample-t test was used. Repeated measure ANOVA test was employed to compare between more than two groups. The significance level was set at P ≤ 0.05. Statistical analysis was completed using IBM® SPSS® Statistics Version 20 for Windows.

Table 2
Fluoride release before recharge of different groups.

Variables	Fluoride release before recharge		*P-value*
	RMGI (Fuji II LC) Mean ± SD	Enhanced RMGI (Activa) Mean ± SD	
Day 1	17.05 ± 2.31^{a}	14.47 ± 0.81^{a}	0.143ns
Day 2	14.36 ± 0.28^{a}	13.49 ± 0.98^{ab}	0.219ns
Day 7	11.20 ± 0.45^{b}	12.17 ± 1.09^{b}	0.230ns
Day 14	10.16 ± 0.69^{bc}	10.83 ± 0.55^{bc}	0.262ns
Day 21	9.52 ± 0.71^{bc}	9.54 ± 0.05^{cd}	0.976ns
Day 28	8.16 ± 0.47^{c}	8.10 ± 0.82^{d}	0.909ns
P-value	≤0.001*	≤0.001*	

Mean with different letters in the same column indicate statistically significance difference *; significant (p < 0.05) ns; non-significant (p > 0.05).

Table 3
The mean, standard deviation (SD) values of Fluoride release after recharge of different groups.

Variables	Fluoride release after recharge		*P-value*
	RMGI Fuji II LC Mean ± SD	Enhanced RMGI (Activa) Mean ± SD	
Day 1	11.39 ± 0.52[a]	9.72 ± 0.28[a]	0.009*
Day 2	9.21 ± 0.46[b]	0.64 ± 0.02[b]	0.001*
Day 7	2.95 ± 0.59[c]	0.19 ± 0.01[c]	0.001*
Day 14	0.25 ± 0.02[d]	0.18 ± 0.009[c]	0.015*
Day 21	0.24 ± 0.01[d]	0.17 ± 0.008[c]	0.003*
Day 28	0.22 ± 0.02[d]	0.15 ± 0.008[c]	0.005*
P-value	≤0.001*	≤0.001*	

Mean with different letters in the same column indicate statistically significance difference *; significant ($p < 0.05$) ns; non-significant ($p > 0.05$).

3. Results

Three-way ANOVA analysis for the effect of different tested variables on fluoride release was shown in Table 1. The results revealed that fluoride recharge had a statistical significant effect on mean fluoride release at F-value 2773.797 and P-value < 0.001. Material type had statistically significant effect at F-value 58.375 and P-value < 0.001. Time interval had statistically significant effect at F-value 291.573 and P-value < 0.001. The interaction between the three variables had a statistically significant effect on fluoride release.

Mean and SD values of fluoride release before recharge of the tested materials at different tested periods were shown in Table 2. For each of RMGI (Fuji II LC) and enhanced RMGI (Activa Bioactive-restorative) groups; there was a statistically significant difference between different tested times at (1, 2, 7, 14, 21, 28 days) where ($p \le 0.001$). The highest mean value of fluoride release was in (Day 1), (17.05 ± 2.31) and (14.47 ± 0.81) for RMGI (Fuji II LC) and enhanced RMGI (Activia Bioactive-restorative) respectively. The least mean value of fluoride release was in (Day 28), (8.16 ± 0.47) and (8.10 ± 0.82) for RMGI (Fuji II LC) and enhanced RMGI (Activia Bioactive-restorative) respectively. Regarding comparing both tested materials at each time interval; results revealed no statistical significant difference between both materials at each time interval (1, 2, 7, 14, 21, 28 days) at p-values (0.143, 0.219, 0.230, 0.262, 0.976, and 0.909) respectively.

Mean and SD values of fluoride release after recharge of different groups were shown in Table 3. For each of RMGI (Fuji II LC) and enhanced RMGI (Activa Bioactive-restorative) groups; there was a statistically significant difference between different tested times (1, 2, 7, 14, 21, 28 days) where ($p \le 0.001$). The highest mean value of fluoride release after recharge was in (Day 1), (11.39 ± 0.52) and (9.72 ± 0.28) for RMGI (Fuji II LC) and enhanced RMGI (Activa Bioactive-restorative) respectively and the least mean value of fluoride release after recharge was in (Day 28), (0.22 ± 0.02) and (0.15 ± 0.008) for RMGI (Fuji II LC) and enhanced RMGI (Activa Bioactive-restorative) respectively.

On the other hand; there was a statistically significant difference between the fluoride release mean values after fluoride recharge between the tested materials at each tested time interval. Where RMGI (Fuji II LC) showed higher fluoride release mean values after recharge compared to the enhanced RMGI (Activa Bioactive-restorative) at each time interval (1, 2, 7, 14, 21, 28 days) at p-values (0.009, 0.001, 0.001, 0.015, 0.003, and 0.005) respectively.

4. Discussion

Release of fluoride from the restorative materials undergoes several phases. First water diffuses into the material, followed by dissolution and diffusion of fluoride ions out of the materials [16].

This study was carried out to compare the amount and pattern of fluoride release form a RMGI (Fuji II LC) to an enhanced RMGI (Activa Bioactive-Restorative). Resin modified glass ionomers undergo both light activated polymerization followed by the acid-base reaction that arises from water sorption. The type and amount of resin used for the photochemical polymerization reaction plus the formation of complex fluoride compounds and their interactions are all factors that affect the fluoride release potentiality from resin modified glass ionomers [17].

On the other hand, the enhanced RMGI (Activa Bioactive-Restorative) tested in this study involves three hardening mechanisms: which are the acid/base hardening reaction of all glass-ionomer systems due to its glass particles and polyacid components, besides both light and chemical cure ability due to their “bioactive ionic resin matrix” component [18].

Several factors affect the rate of fluoride release from the dental materials, such as; the composition of the material, the storage media of the specimens, temperature, and the contact area with the storage medium [19].

In this study specimens were stored in deionized water, as it provides a baseline of fluoride release potential in un-stimulated environments. Deionized water is a medium with no minerals or organic molecules that might influence the results [19–21].

Our results revealed that there was no statistically significant difference between both materials regarding fluoride release at each time intervals. Both materials had a significant release of fluoride ions, especially in the 1st and 2nd days of evaluation. This was in agreement with a number of *in vitro* studies that have also shown higher fluoride release in the first two days [22,23]. This high amount of fluoride released in the first two days is named “The initial Burst Effect”. As the fluoride release from glass ionomer is dependent on its concentration and diffusion limitation in both the matrix and the particles. A large amount of fluoride becomes part of reaction product matrix, following the initial acid dissolution of powder particle surfaces. This fluoride diffuses rapidly from the matrix uncovered on the surface of the material and is slowly substituted by fluoride diffusing from the matrix beneath the surface [21,22].

Results revealed that there was decline in the fluoride release during the subsequent days. This was related to the slower dissolution of glass particles through the pores of the restorations with time. During maturation period, bulk fluoride release occurs as a consequence of contact between the materials with the storage medium. This was in agreement with other studies [22–24].

Restorative materials to perform as a fluoride reservoir are largely dependent on the type and permeability of the material, plus their ability to retain fluoride [21,22]. In addition, the rate of fluoride exposure and the type and concentration of the fluoridating agent had also great influence [25].

After the application of the Alpha-PRO®APF for fluoride recharge; there was a statistically significant difference between the fluoride release mean values, where RMGI (Fuji II LC) showed higher fluoride release after fluoride recharge compared to the enhanced RMGI (Activa Bioactive-restorative) at each time interval (1, 2, 7, 14, 21, 28 days). Activa Bioactive-Restorative, contain a patented, resilient resin matrix with energy-absorbing elastomeric components (a blend of diurethane and methacrylates with modified polyacrylic acid and polybutadiene modified diurethane dimethacrylate) [18]. This patented resin matrix might affect the permeability of this enhanced RMGI, leading to lower ability to be recharged and acting like fluoride reservoir.

Also results of our study revealed that the highest statistical significant mean values of fluoride release were at the first day after recharge, then declines rapidly for both tested materials.

This indicates that only superficial part of the specimens has been recharged due to the short fluoride recharge time (4 min) that was applied once to the specimens in this study.

5. Conclusions

Under the limitation of this *in vitro* study; it could be concluded that the tested conventional RMGI and enhanced RMGI, presented the same pattern of fluoride release in deionized water. On the other hand; they have different capability for fluoride recharge, where conventional RMGI was more successful to be recharged.

Conflicts of interest

The authors have no conflict of interest to disclose.

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