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FLUORIDE RELEASE AND RECHARGE BEHAVIOR OF BIOACTIVE GLASS IONOMER CEMENTS USING ION CHROMATOGRAPHY

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ABSTRACT

Objective: To evaluate fluoride release and uptake behaviors of the new bioactive resin modified glass ionomer cement (RMGIC) (activa bioactive glass ionomer) compared to conventional glass ionomer cement(CGIC) (Fuji IX GP).

Material and Methods: Forty specimens were prepared (10 mm x 2 mm) from Fuji IX GP glass ionomer and Activa Bioactive RMGI according to manufacturer's instructions. The specimens stored in deionized water and fluoride release were assessed daily in the first week then weekly till the third week using ion chromatography. The specimens of each material were divided into two equal subgroups according to the recharging agents used; either 0.05% mouth wash (226 ppm sodium fluoride, G.U.M, Sunstar, Germany, GmbH) or 5% sodium fluoride varnish (22.600 ppm fluoride, enamel pro varnish Premier Dental, Plymouth Meeting, PA,19462,USA). Fluoride release after recharge was measured and recorded daily for a total of 5 days. Data were analyzed by Two way ANOVA and Duncans test ($\alpha = 0.05$) using the SPSS software program.

Results: CGIC release higher amount of fluoride than RMGIC. Professionally applied fluoride varnish showed better recharging ability than home care fluoride mouth wash and CGIC had greater recharge ability than new RMGIC.

KEYWORDS: Fluoride, resin-modified glass ionomer, glass ionomer and recharge

INTRODUCTION

A bioactive material defined by Hench In 1969 as one that produce a biological response at the interface of the material which resulting in formation of a bond between the tissues and the material, (1) so bioactive materials have the ability to interact with living tissues or system. Depending on

previous definition; glass ionomer cements(GICs) are considered bioactive dental materials due to their dynamic release of fluoride, their mineral-based poly-salt matrix composition that contribute to the ability to remineralize tooth structure, their bonding ability to tooth structure and proved biocompatibility.⁽²⁾

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GICs materials were invented by Wilson and Kent in 1969. (3) These materials are water-based, known as polyalkenoate cements. It composed mainly of fluoro-alumino-silicate glass and polyalkenoic acid. The setting reaction is an acid/base reaction. GICs materials combine the strength, rigidity, and fluoride release properties of silicate glass powder with the biocompatibility and adhesive characteristics of polyacrylic acid liquid. (4) Despite the proved advantages of GICs (as ion exchange adhesion with tooth structure, (5) fluoride release(6), coefficient of thermal expansion and contraction close to tooth structure^(7,8) and biocompatibility⁽⁹⁾, they have many disadvantages as moisture sensitivity and lack of sufficient strength. (10) Several modifications of GICs have been done in an effort to overcome these disadvantages.

One of these modifications is the RMGIC which was introduced in the late 1980s to overcome moisture sensitivity, low physical properties (early mechanical strength), and short working time of CGICs. RMGIC are glass-ionomer cements with small quantity of monomers and initiators so the acid-base reaction is supplemented by a second polymerization reaction.(11) Activa Bioactive-Restoratives are recently introduced as the first bioactive composite with an ionic resin matrix, resin component and fillers which simulates properties of natural teeth. It is a RMGIC that has the ability to releases and recharges different ions such as calcium, phosphate and fluoride ions. The manufacturer claimed that the hydrophilic resin may provide better fluoride release and uptake than conventional glass ionomer.(12)

Progression or control of dental caries depends on the balance between caries process and protective factors. The best method for caries management is remineralization. (13, 14) Fluoride is an anti-cariogenic agent, as it is well documented to decrease the tooth demineralization, increase the remineralization, interfere with pellicle and plaque formation, and

inhibit microbial growth and metabolism. Fluoride also forms the more acid-resistant fluorapatite instead of carbonated hydroxyapatite. (15) Several clinical studies demonstrated the effects of fluoridated restorative materials on management of dental caries and they reported that fluoride with levels of 0.095 to 0.190 ppm in the saliva for long time may be sufficient for cariostasis. (16-18)

To achieve the goal of caries prevention, there must be a constant supply of low levels of intraoral fluoride. Maintenance of Saliva fluoride level from 0.001 to 0.005–0.010 mmol/l, may be efficient for caries control. Fluoride leached out of fluoride containing dental materials decreased over time so fluoride charging is used to sustain constant levels of fluoride. The ability of a restorative material to take and release fluoride is dependent on intrinsic factors (type and permeability of filling material, fluoride content, and nature of fluoride incorporated) and environmental factors (frequency of fluoride exposure and the kind and concentration of the fluoridating agent). (20,21)

GIC and their derivatives not only release fluoride but also act as a fluoride reservoir. (22) The recharging power of glass ionomer cements was first detected by Walls. (23) Many theories explained the recharge mechanism. Dewitte et al. postulated two different mechanisms of recharge; the first is short term release, where fluoride ions simply diffused out through the cement matrix. The second is long term release, where fluoride ions reacting with the intrinsic ions in the matrix, re-released again decomplexation. (24) Diaz-Arnold and co-workers described recharging as surface phenomenon, where the adsorbed fluoride ions to the surface of the restoration get washed off. (25) Recharging agents include; fluoridated dentifrices, mouth rinses, fluoride gels and fluoride varnishes.

Fluoride mouth rinse remains the most widely recommended routine home care oral hygiene due to its anticariogenic properties and ease of use. Fluoride mouth rinses are typically based on neutral sodium fluoride solutions ranging from 0.05 to 0.2% (225–1,000 ppm). Amine and stannous fluoride formulations are also available.⁽²⁶⁾ Fluoride varnishes are composed of a natural resin, fluoride ions, an organo-phosphoric acid and solvent such as ethanol, also may include proprietary additives in their formulations such as tri-calciumphosphate (TCP), amorphous calcium phosphate (ACP), and xylitol-coated calcium phosphate (CXP). Enamel Pro® Varnish is 5% sodium fluoride varnish that additionally contains amorphous calcium phosphate (ACP) formula.⁽²⁷⁾

So the aim of this study is to compare the fluoride release and recharge from new bioactive resin modified glass ionomer compared to conventional glass ionomer cement. The null hypothesis of the study is that there is no significant difference between conventional and resin-modified GICs regarding the fluoride release and recharge.

MATERIALS AND METHODS

Two glass-ionomer cements were selected (table 1): one CGIC (Fuji IX GP) and a bioactive RMGI (Activa Bioactive). Twenty disc specimens (10 mm diameter x 2 mm height) of each material were prepared using a split teflon mould according to manufacturer's instructions. After placement of the material in the mould, the surface of the

restorative materials was covered with a polyester strip and a glass slab under pressure to expel excess material from the mould. A piece of nylon thread was incorporated into the cement during setting to suspend the samples in the test medium. RMGI, the polymerization procedure was done through the polyester strip from top and bottom following the manufacturers' instruction, using a light-curing device with light intensity of 500 mw/ cm² (DabiAtlante, RibeirãoPreto, SP, Brazil). The light intensity was controlled at 500 mw/cm² by measuring with a curing radiometer. All samples were removed from the moulds after 10 min. Specimens were allowed to set for an additional 24h in a humid atmosphere at 37°C±1°C. All specimens were stored at 37°C in individually capped, 15-ml polystyrene tubes containing 4 ml deionized water.

Samples were removed from the storing solution, and rinsed with 1 mL deionized water. Blotting paper was used to remove excess water. Samples were then once more placed in a container containing 4 mL deionized water, then stored under the same conditions $(37 \pm 1^{\circ}\text{C})$. This procedures were repeated every day for one week and then after 15 and 21 days. Daily fluoride release was measured in ppm with ion chromatography. Cumulative fluoride release was calculated for each sample as the total of the amounts of fluoride released during the consecutive elutions. A cumulative release curve against time was then plotted for each group.

TABLE (1) Materials used in the study.

Product	Composition	Manufacturer	Lot number
Fuji IX GP	Powder : 95% by weight fluoro- aluminum silicate glass; 5% by weight	GC Corporation,	1401131
(CGIC)	polyacrylic acid powder	Japan.	
	Liquid: 40% polyacrylic acid; 50% distilled water		
	10% polybasic carboxylic acid		
Activa Bioactive	Blend of diurethane and other methacrylates with modified polyacrylic	Pulpdent Corporation,	160314
(RMGI)	acid (44.6%)	Watertown, MA US	
	Amorphous silica (6.7%)		
	Sodium fluoride (0.75%		

After 21 days of first fluoride release, Samples in each group were divided into two equal subgroups (n=10). The samples of subgroup A; were immersed in 0.05% mouth wash (226 ppm sodium fluoride) for 5 min. Then specimens were washed with 5 mL of deionized water, air-dried for 1 min, and immersed in individual plastic tubes containing 4mL of deionized water at 37°C. for 5 days, fluoride rerelease measurements were done at 24-h intervals. The samples of subgroup B; were coated with a thin layer of enamel pro varnish (5%Na F varnish, 22.600ppm F) for 2 min. Each sample was returned to a new container filled with 4mL fresh deionized water. Fluoride release after recharge was measured and recorded daily.

The fluoride ion was measured using an ion chromatograph (DX 100; Dionex, Camberley, Surrey, UK). Measuring of fluoride ions was made using 0.5 ml of each storage solution which was injected into the injection loop of the instrument. Each solution was examined three times and fluoride concentration measured.

The data was collected, tabulated and statistically

analyzed by Two way ANOVA and Duncans test $(\alpha = 0.05)$ using the SPSS software program (SPSS version 22, IBM, Chicago, IL, USA).

RESULTS

Fluoride release

The results from (Table 2) and (Figure 1) revealed means of daily fluoride release per each material. There was a significant difference of Fluoride-release between CGIC and RMGIC (p< 0.05). Also, it was clear that daily release of fluoride was at maximum level at 1st day and decrease till day seven. That decrease was significant for 1st four days and become less manifested till the end of the 7th day.

From the cumulative fluoride release results (Table 3) and (Figure 2), It was clear that the cumulative fluoride release of CGIC is higher than that of RMGIC. For CGIC, increase in fluoride release was significant between 1st, 7th and 15th days, while it became not significant between 15th and 21th day. On the other hand for RMGIC, there was significant increase only between 1st and 7th days.

TABLE (2) Means and standard deviation of each group in ppm and results of Duncans test

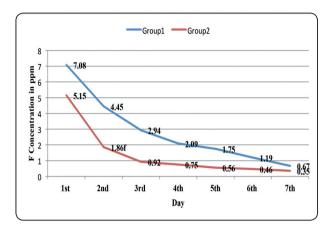
Group/ Days	1 st	2 nd	3 rd	4 th	5 th	6 th	7 th
CGIC	7.08 ± 0.25^{a}	4.45±0.33 ^b	2.94±0.23°	2.09±0.32 ^d	1.75±0.27 ^{de}	1.19±0.17 ^{ef}	0.67±0.13 ^f
RMGI	5.15±0.29a	1.86±0.18 ^b	0.92±0.16°	0.75±0.19 ^d	0.56±0.10 ^{de}	0.46±006 ^{ef}	0.35±0.13 ^f

Means with the same superscripted letters have no significant difference in the same rows

Table 3: Means and standard deviation of cumulative fluoride release in ppm and results of Duncans test.

Group/Day	1 st	7 th	15 th	21 th	
CGIC	7.084±0.25°	20.178±2.12 ^b	22.90±1.02a	24.78±1.03 ^a	
RMGIC	5.15±0.29°	10.04±1.03b	11.76±1.01 ^{ab}	12.84±1.68 ^a	

Means with the same superscripted letters in the same rows had no significant difference



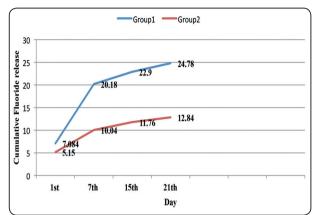


Fig. (1) Daily fluoride release per each material.

Fig. (2) Cumulative Fluoride release for each material.

Table 4 Means of fluoride re-release after recharge in ppm.

Group/	Subgroup A				Subgroup B					
Day	1 st	2 nd	3 rd	4 th	5 th	1 st	2 nd	3 rd	4 th	5 th
CGIC	3.16	0.95	0.85	0.69	0.54	7.39	4.30	3.58	1.00	0.94
RMGI	1.87	0.68	0.60	0.54	0.35	5.42	2.64	1.90	1.58	0.63

Fluoride Re-release after recharge

Table 4 and figure 3 show Means of fluoride re-release after recharge in ppm. Fluoride release from restorative materials after recharging with 5%NaF varnish (enamel pro varnish) was higher than fluoridated mouth wash (0.05% NaF). Also, it was clear that CGIC had a greater recharge potential than RMGIC.

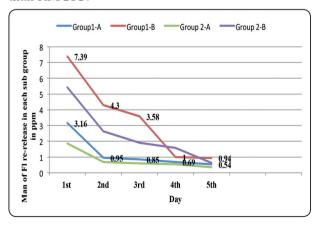


Fig. (3) means of Fluoride re-release after recharge in each subgroup.

DISCUSSION

Several studies have been done with different results of fluoride release using different materials. (2,28,29) The difference between results may be due to different methods and specimen size, storage media (type and frequency of change), quantity of media used to measure fluoride level. (30) The ideal fluoride-release material should release high fluoride without changing its properties or filling degradation. An initial high level of fluoride release in the majority of restoration will inhibit caries by the bacteriostatic effect of fluoride; more over it induce remineralization of tooth structure. (31)

The rate of fluoride release from dental materials can influenced by multiple factors including; storage media, temperature, sample size and powder liquid ratio of the material.⁽³²⁾ The temperature was kept at 37°C by placing the samples in an incubator. In addition, the samples were suspended in the storage media by non-fluoridated dental floss allowing uniform wetting of the samples without clinging to the walls of the storage vials.⁽³³⁾

Samples were immersed in deionized water to avoid any influence of minerals or organic molecules which might be presented into de/re solutions or saliva. (34) The use of saliva as a storage medium may affect negatively on the results of Ion chromatography . (35) Storage medium was changed every 24 hours as fluoride release is based on a concentration gradient mechanism and when equilibrium is achieved fluoride release is stopped . (36)

Ion chromatograph (IC) used in this study for fluoride detection as it offers lower detection limits (ultra trace levels) and better accuracy. (37-39) IC also allows the detection of free fluoride ions which are taken up into the tooth. This free fluoride transform hydroxyapatite into fluoroapatite enhancing the resistance of tooth substance to secondary caries. (40)

Studied materials released high amount of Fl ions on the first day then the released Fl ions declined sharply but continued to release low amounts of Fl ions throughout the study period. This release pattern is in agreement with previous studies. (41, 42) The high release on the 1st day is induced by the burst of fluoride released from the glass particles when reacting with the polyalkenoate acid during the setting reaction (initial burst) and during the subsequent days release is declined and this is attributed to the dissolution of the glass in the acidified water of the hydrogel matrix and its ability to diffuse through cement pores and fractures (bulk diffusion). (43, 44)

Our hypothesis is rejected as there was significant difference between CGIC and RMGIC regarding Fl release and recharge. Regarding initial Fl release; CGIC gave higher results than RMGIC which may be attributed to the amount of matrix. (36) For CGIC, the ion released by acid attack at the surface. (22) The acid base reaction is more in CGIC and resulting in thicker matrix in these materials. (36) Initial setting of RMGIC is polymerization reaction followed by an acid base reaction arises from water sorption. (30) The

amount of Fl released from RMGIC is affected by amount and type of resin within the material.

Fluoride exposure from external source like varnish or mouth wash can recharge Glass ionomer materials. (22, 25, 45)So, Glass ionomer restoration can act as an intra-oral device for fluoride slow release. Release of fluoride after topical application depends on the pH, dose, concentration, duration and frequency of application. (46) This study agrees with the results of Takahashi et al., they found that amount of fluoride release proportion with fluoride concentration. (47)

Regardless the time and the recharging agent used, CGIC gave higher fluoride release than RMGIC. This may be due to that CGIC has greater glass ionomer matrix. The matrix phase not only promotes fluoride release but also recharging. (36) Xu and Burgess suggested that high fluoride release material has a higher fluoride recharging ability. (30)

Regardless the type of recharging agent, on the 1st day, both restorative materials had an increased amount of fluoride re-release. The fluoride release decrease rapidly suggesting that fluoride release after topical fluoride application represents a washout of ions adsorbed to the surface, rather than an actual diffusion into the matrix. (48)

CONCLUSION

The initial Fluoride release with CGIC was higher than RMGIC. Fluoride varnish (5% NaF) has higher recharging ability than mouth rinse (0.05% NaF). CGIC can be recharged better than RMGIC.

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