

ORIGINAL ARTICLE

FLUORIDE RELEASE FROM GLASS IONOMER CEMENT CONTAINING FLUOROAPATITE AND HYDROXYAPATITE

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Background: Many investigators reported the amount of fluoride release from glass ionomer cement. However, the work on fluoride release from GIC containing fluoroapatite and hydroxyapatite is scarce. Therefore, this study was conducted to find out the amount of fluoride release from Glass ionomer cement containing fluoroapatite and hydroxyapatite. **Methods:** The study was conducted in the Department of Materials, Queen Mary University of London. A total of 108 samples equally divided in to three groups namely fluoroapatite added GIC, Hydroxyapatite added GIC as an experimental group and unmodified GIC as a control group. The specimens were prepared by mixing powder and liquid in the ratio of 1:1. Amount of fluoride released was measured by Ion electrode method at 1, 3, 7, 14, 21 and 28 days. **Results:** On day 1, the combination of FA +GIC showed the highest amount of fluoride release followed by the control group (GIC) whereas the combination of HA+GIC released the least amount of fluoride. On day 7, the amount of fluoride release started declining in all three groups. The amount of fluoride release continued decreasing on day 21 in which combination of FA +GIC and the control group are shown to release equal amount of fluoride whereas the combination of HA+GIC gave the least activity the amount of fluoride release fall to a minimum level in all three group by day 28. **Conclusion:** It is concluded that addition of fluoroapatite into GIC has significant effect on the amount of fluoride release as compared to GIC alone; however, addition of hydroxyapatite into GIC has no additive effect on the amount of fluoride release.

Keyword: Fluoroapatite, Hydroxyapatite, Glass ionomer cement

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INTRODUCTION

Fluoride releasing property of glass ionomer cement has been known for a long time.¹ The pattern of fluoride release from glass ionomer cements is characterized by an initial rapid release, followed by a gradual reduction in the rate of release of fluoride after short time.^{2,3} Studies have shown that fluoride is released for up to 18 months in ever diminishing amount and some of it is available for leaching.⁴ Fluoride release from GIC has many effects on tooth structure like it increases acid resistance to tooth structure and prevent secondary caries by inhibition of bacterial growth.⁵

Since hydroxyapatite (HA) has excellent biocompatible properties and a crystalline structure similar to apatite in the human dental hard tissues and skeletal system, a number of studies have tried to evaluate the effect of the addition of fluoroapatite (FA) and Hydroxyapatite (HA) powders to restorative dental materials such as Glass ionomer cement (GIC).^{6,7} They found that the incorporation of HA and FA into GIC may not only improve the biocompatibility of GIC, but also have the potential of enhancing the mechanical properties (e.g. compressive, diametral tensile and biaxial flexural strength). Moreover, such modified GIC make strong bond to tooth structure due to its inherent similarity to the enamel and dentine structure and composition. ChiuS-Y *et al*⁸ reported that addition of

hydroxyapatite in to GIC did not prevent its fluoride release but improved its fracture toughness. Moshaverinia A *et al*⁹ also reported enhanced mechanical properties of GIC containing 4 wt.% HA as compared to commercial GICs.

Several studies conducted to evaluate the amount of fluoride release at different temperature, PH and with different modification in glass ionomer cement.^{10,11} However, the amount of fluoride release after addition of Fluoroapatite and Hydroxyapatite in GIC has not been reported previously. Therefore, it is worthwhile to conduct this experimental study in order to evaluate the amount of fluoride release from fluoroapatite and hydroxyapatite containing GIC.

MATERIAL AND METHODS

The materials used in this experiment study were Fluoroapatite + GIC, Hydroxyapatite + GIC as an experimental group whereas GIC (Fuji IX, GC) was used as a control group.

Preparation of Experimental Groups

a. Preparation of Fluoroapatite + GIC

0.5 gm of fluoroapatite (FA) and 10 gm of GIC were accurately weighed on a weighing scale then mixed well and stored in a dry container.

b. Preparation of Hydroxyapatite + GIC

0.5 gm of hydroxyapatite (HA) and 10 gm of GIC were accurately weighed on weighing scale. Gently mixed the two powders together and stored in a dry container.

Total sample sizes of 108 specimens were equally divided into two (02) experimental groups and one control group (i) FA+ GIC (ii) HA+ GIC (iii) GIC; each group comprising of 36 samples. The test specimens were prepared by mixing the respective powder and liquid in 1:1 ratio according to manufacturer instructions on a cool glass slab for 2–3 minutes using a metal spatula. The mixed cement was then poured into cylindrical polyethylene mould of 4mm diameter and 2.5 mm height (Figure-1). To remove the excess material, the moulds were covered with transparent matrix and subsequently pressed between two glass plates. After 30 minutes of initial setting, the samples were gently de-moulded.

After 24 hours, when the setting reaction for the cements has been completed, the samples were placed into a centrifuge tube containing 15 ml of distilled water and stored in an oven at 37 °C at neutral pH. The temperature was maintained at 37 °C for 28 days. The solutions were repetitively changed at day 1, 3, 7, 14, 21 and 28 to detect the amount of fluoride released using Ion Electrode method.

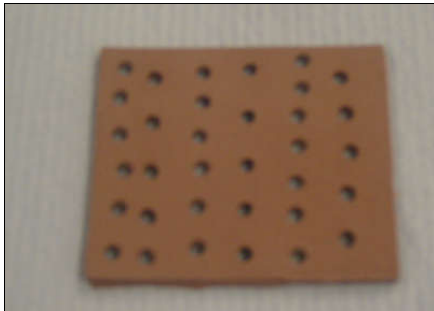


Figure-1: Polyethylene mould for specimen preparation

Fluoride Release Analysis

Ion Electrode method was employed to calculate the amount of fluoride release. A fluoride electrode model 96–09 Ionplus, Thermo Orion (Figure-2) was used to measure the amount of fluoride leached out from the specimen into the solution.

Following the manufacture’s protocol, the filling solution, Thermo Orion was used to fill the electrode’s chamber. The entire internal surface of the electrode was wetted with the filling solution including the probe. An initial calibration was done for the electrode using standard solutions of known concentrations (10 mMol, 0.1 mMol, 0.01 mMol, 0.001 mMol). For calibration, 1ml of distilled water was

pipetted out and transferred it to an electric stirrer. 1 ml of standard solution was added into it and stirred well on an electric stirrer. The tip of electrode was brought in contact with the stirrer and a stable reading was obtained. The procedure was repeated thrice for each standard solution and the mean of the readings were taken. The electrode was rinsed with distilled water after each reading.

Once the calibrations were done, fluoride release was measured in the stored sample using the same protocol. 1ml of sample solution was pipetted out and transferred to an electric stirrer. 1ml of distilled water was added and stirred well. The electrode was used to take the readings while making sure to rinse the electrode with distilled water after each reading. In this way, the stable reading was recorded. The entire experiment was executed in thrice for all three sample solutions and the mean of the readings was calculated. The entire experiment was repeated on day 3, 7, 14, 21 and 28. After day 28, the results were transferred on excel sheets and the graphs were plotted against readings.



Figure-2: Fluoride release measured by Ion Electrode

RESULTS

The results obtained for the amount of fluoride release from different materials at different time intervals are plotted in graphs as shown in figures 3–8.

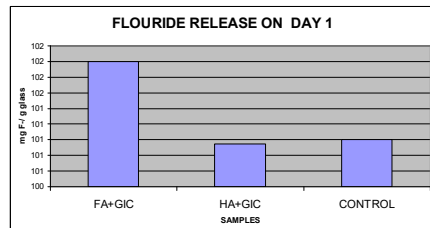


Figure-3: Fluoride released from FA+GIC, HA+GIC and control group on day 1.

On day 1, the combination of FA +GIC showed the highest amount of fluoride release followed by the control group (GIC) which released the second highest amount whereas the combination of HA+GIC released the least amount of fluoride (Figure-3).

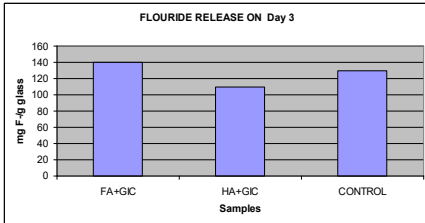


Figure-4: Fluoride released from FA+GIC, HA+GIC and control group on day 3.

The fluoride released for all three materials showed continuous increase on day 3 in comparison to day 1 (Figure-4). Fluoride released was maximum from FA+GIC followed by control group (GIC) and then HA +GIC.

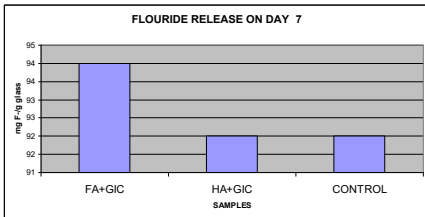


Figure-5: Fluoride released from FA+GIC, HA+GIC and control group on day 7.

On day 7, the amount of fluoride release started declining in all three groups as shown in figure-5. FA +GIC still gave the highest amount of activity as compared to HA +GIC and control group.

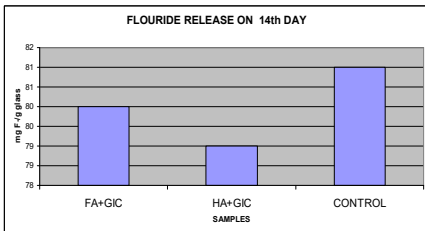


Figure-6: Fluoride released from FA+GIC, HA+GIC and control group on day 14.

The declining pattern of fluoride release was also evident on day 14. However; on day 14, the control group (GIC) was shown to release the greatest

amount of fluoride which was in contrast to the results obtained on day 1, 3, 7. The group FA+GIC released the second highest amount and HA +GIC still released the least amount of fluoride (Figure-6).

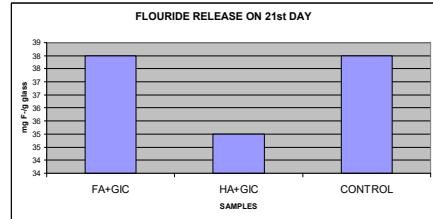


Figure-7: Fluoride released from FA+GIC, HA+GIC and control group on day 21.

The amount of fluoride release continued decreasing on day 21. The combination of FA +GIC and the control group are shown to release equal amount of fluoride whereas the combination of HA+GIC gave the least activity (Figure-7).

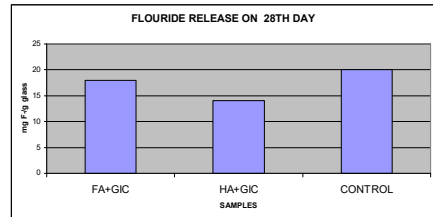


Figure-8: Fluoride released from FA+GIC, HA+GIC and control group on day 28.

The amount of fluoride release fall to a minimum level in all three groups by day 28 (Figure-8).

DISCUSSION

The materials used in this experiment study were Glass ionomer cement containing Fluoroapatite and Hydroxyapatite as an experimental group whereas GIC (Fuji IX, GC) alone was used as a control group.

GIC has recently attracted considerable interest in the field of dentistry due to its many favorable characteristics like biocompatibility, chemical bonding to tooth structure, similar coefficient of thermal expansion to that of tooth structure and anticariogenic property due to fluoride release. Sustain release of fluoride from glass ionomer cements prevents recurrent and secondary caries.¹² Fluoride ions release occurs via fluoride ion diffusion, in which hydroxyl group of hydroxyapatite can be replaced by fluoride ions to form fluoroapatite.¹³ Fluoroapatite is more caries resistant and more durable against acidic attack due to its

lower solubility and lower crystal energy in comparison to hydroxyapatite.¹⁴ The rate of fluoride release is depends on time and concentration gradient. It reduces as the time passes according to concentration gradient.¹⁵ Release of fluoride occurs in two stages, firstly, there is a quick surface elution followed by steady continuous bulk diffusion of fluoride ions.¹⁶ However, the use of GIC is limited due to its poor mechanical properties like brittleness and inferior mechanical strength. Therefore, various fillers are incorporated into GIC to enhance its mechanical properties. These include carbon fiber inserts, aluminosilicate glass fiber inserts, hydroxyapatite and fluorapatite particles. Various researches have shown an improvement in the mechanical properties of GIC with the addition of Hydroxyapatite and Fluorapatite.^{17,19} However, the data on effect of fluoride release with the addition of fluorapatite and hydroxyapatite is limited.

The result of the present study showed that the amount of fluoride release increased significantly in FA+GIC group as compared to control group except on 14th and 21st day in which amount of fluoride from control group is higher than FA+GIC group. Amount of fluoride release is higher in FA+GIC group because fluoride is already present in the structure of fluoroapatite and after immersion in water very small amount of fluoroapatite dissolve in it thereby, increasing the chances to release. On the other hand, fluoride release in HA+GIC group is lesser than the control group in most of the days which means HA may hinder the fluoride release from GIC.

These results are in agreement with earlier studies,^{17,18} which indicated an increase in fluoride release in modified GIC as compared to the unmodified GIC. Lin *et al*¹⁹ also reported a higher amount of fluoride release from resin modified glass ionomer when nonfluorapatite and nanofluorohydroxyapatite were further incorporated into it. However, our results differ from the findings of Moshaverinia A *et al*.⁹ A higher fluoride release and recharge capacity of Hydroxyapatite modified GIC was also reported by Arita *et al*²⁰ which are in contrast with our results, that showed no additive effect on fluoride release with the addition of hydroxyapatite. This difference in findings is probably due to small sample size and the presence of multiple variables. Current study was limited by the fact that only one weight ratio was tested for nanoparticles of fluoroapatite, hydroxyapatite and the study period was of relatively short, evaluation of long term fluoride release and uptake is desirable.

Evaluation of fluoride release was analysed by Ion Electrode Model 96–09 ion plus. This type of

electrode is user friendly, detect fluoride concentration quickly with good reproducibility. However, calibration in each session is needed as the electrode is unable to measure concentration directly. Moreover, the calibration curve is not linear in case of low concentration of fluoride.

In future investigations, dimensional stability and water absorption from this type of modified glass ionomer cement should be studied, preferably in prospective clinical trials.

CONCLUSION

It is concluded that addition of fluoroapatite in to GIC has significant effect on the amount of fluoride release as compared to GIC alone; however, addition of hydroxyapatite into GIC has no additive effect on the amount of fluoride release.

AUTHORS' CONTRIBUTION

SM: Topic selection, conceptualization of study design, performed procedure. MAA: Drafting of manuscript and abstract. ZC: Analysis and interpretation of data. NM: Critical revision. MA: Referencing. MAL: Discussion write up.

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