Aim of the study:

The aim of the present study was to:

Evaluate and compare the effect of application of:

- Topical fluorides (1.23% Acidulated phosphate fluoride gel),
- Casein phosphopeptide-amorphous calcium phosphate and,
- Casein phosphopeptide-amorphous calcium phosphate with fluoride;

On fluoride release and diametral tensile strength of a highly viscous glass ionomer cement.

Conclusions

Based on the findings of the present study, it can be concluded that:

- Fluoride release of highly viscous GIC is characterized by an initial burst in the first 24 hrs after mixing, followed by a rapid decline reaching a constant low level after 1 week.
- Highly viscous glass ionomer can be recharged using APF gel which increased fluoride release at 24 hrs 48 hrs and 4 days.
- Casein phosphopeptide-amorphous calcium phosphate paste does not have any recharging effect on glass ionomer fluoride release.
- Casein phosphopeptide-amorphous calcium phosphate with fluoride paste causes a decrease in fluoride release which was statistically significant at 24hrs and 4 days.
- Different remineralizing agents used in the study do not cause a statistically significant effect on DTS of glass ionomer.
- Diametral tensile strength of Fuji IX Highly viscous GIC is stable over a period of six weeks.

Discussion:

Fluoride is a major constituent of GICs but it has no role in the cement forming process as reported by Crisp and Wilson. [88] The other components such as calcium and aluminum are involved in cement-matrix formation and play a basic role in hardening and strengthening the ionomer salt hydrogel. [89] Swift and Dogan [90] reported that fluoride is dispersed throughout the matrix region of the set cement homogeneously and is available for elution for long period after setting.

The content of fluoride in the restorative materials should be as high as possible without adverse effects on their physico-mechanical properties and the release should be also as great as possible without undue degradation of the material. [46]

Fuji IX GIC was chosen for the present work as it is specifically designed for use as a permanent restorative material in primary teeth. Highly viscous glass ionomer overcomes the drawbacks of conventional glass ionomer by decreasing the mean particle size. The viscosity is increased with the use of smaller grain size particles and the use of low solubility glass that achieves fast maturing property. Fuji IX also contains fewer monovalent ions which results in greater cross linking of the polymer chains. [24, 39]

[39]All the specimens were prepared with a dimension of 6 mm diameter × 3 mm thickness to allow maximum surface area to be exposed; as required in in-vitro studies and since fluoride release is dependent on exposed surface area and not on sample weight. Moreover, these dimensions are required for

measuring the DTS using the universal testing machine. [10, 44.48, 91]

All specimens were stored in deionized water for 1 week before exposure to remineralizing agents in order to discharge a large amount of the fluoride out of the specimens and to compare fluoride release before and after application of these agents. [92] In this study standardization of intrinsic and extrinsic factors governing fluoride release was achieved; intrinsic factors are related to preparation of the material, its powder: liquid ratio, mixing time and temperature and specimen geometry. The extrinsic factors are related to the storage medium (its pH, temperature and impurities), experimental design (volume of storage solution, frequency of solution change and stirring) and analytic method.

APF gel 1.23% (pH 3.5) was used since it is the most widely used topical fluoride agent and is one of the American Dental Association recommended agents for professionally applied topical fluorides**.** [93] Moreover it has several advantages over NaF and SnF_2; it requires single semiannual application, does not produce staining of enamel and has an expected caries reduction of about 30% to 40%. It was applied for four minutes as recommended for professionally applied topical fluoride solutions, gels, or foams**.** [94, 95]

Casein phosphopeptide-amorphous calcium phosphate (CPP-ACP) is a remineralizing agent used as an adjunct to fluoride treatment in the non-invasive management of early caries lesions. It has been demonstrated to inhibit enamel and dentin demineralization and promote remineralization in in-vitro and in-vivo studies. [63, 68, 72, 74]

In addition to the benefits of CPP-ACP nanocomplexes on tooth tissues, *Mazzaoui et al.* demonstrated a strengthening effect on GIC specimens when 1.56% (w/w) CPP was incorporated into self-cured GIC while mixing. [80]

Abdou J et al supported the outcomes of *Mazzaoui et al*., and suggested that even if CPP-ACP is not originally incorporated in the GIC may still have a strengthening effect if applied to the external surface. As modern GIC material is capsulated, it is more applicable to apply CPP-ACP to the surface than to incorporate it into the GIC paste. [82]

In the present study, our results showed increased fluoride release in the first 24hrs (mean=3.58 ppm) which was followed by a sharp decline after 48hrs and 4 days reaching its lowest level after 1 week (0.38 ppm), (table 1, fig. 16). This pattern of fluoride release was in agreement with theories suggesting an initial burst of fluoride from the surface of the cement associated with its setting followed by slower but prolonged release. [82, 81, 95, 96, 97]

The high fluoride release observed in the initial periods after mixing may be explained by the fact that the glass-ionomer cement setting reaction is processed in a gradual manner within a time of approximately 12 to 24 hours. Thus, there is great ionic movement, facilitating the release of ionically active elements, fluoride being among them. [46, 98]

After application of different remineralizing agents, results showed statistically significant increase in fluoride release after 24 hrs in APF gel group (G1), which increased from 0.38 ppm to 0.81 ppm. Where, in (G2, G3 and G4) there was a significant decrease in fluoride release from 0.38 ppm to 0.34, 0.29 and 0.28 ppm respectively (table 2, fig. 17). This shows that only APF gel caused increase in fluoride release while there was no obvious recharging effect in the other groups However, this data did not reveal whether this property would contribute to the reduction of caries activity.

Measurements of fluoride release in each group showed that treatment of glass ionomer with APF gel (in G1) resulted in a significant increase in the first 24hrs followed by a rapid decline at 48 hrs, until a nearly constant low level was reached after 4 days returning to its initial level at about 0.3 ppm for the ongoing period (table 3, fig. 18). This finding was in agreement with previous studies where application of different topical fluorides resulted in increased fluoride release during the first 24hrs, followed by a sharp decrease over the first week. [2, 44, 45, 46]

The present results showed that although fluoride release after recharging had a similar pattern to that of the initial fluoride release, it occurred at a lower level with a mean fluoride release 0.81 ppm in the first 24 hrs compared to 3.51 ppm in the initial phase and for a shorter duration (4 days) compared to 1 week in the initial phase.

This increase in fluoride release after recharging could be due to erosion of the material by the low pH of APF gel, the washing-out of the remnants of the gel from the porosities of the material, or by subsequent diffusion of fluoride ions taken up by the matrix of the restorative materials. [34, 46, 99]

After application of CPP-ACP paste (in G2) there was a gradual decrease in fluoride release which was not significant among follow up periods.

However, after CPP-ACP+F application (in G3) there was a non significant decrease in fluoride release after 24 and 48 hrs followed by a significant decrease after 4 days, and then an increase in Fl release after 1, 3, and 5 weeks to values which were not significantly different from those noticed after 24 hrs and 48 hrs (graph 5. fig. 20).

It was reported that CPP-ACP and fluoride have a synergistic effect in reducing caries experience which may be attributable to the formation of CPP-stabilized amorphous calcium fluoride phosphate and the ability of the CPP to localize and stabilize the ions at the tooth surface. [100] It is likely that a combination of CPP–ACP and fluoride resulted in co-localization of calcium and phosphate ions with fluoride ions at the surface of GIC, presumably as CPP–ACFP nanocomplexes**,** resulting in decreased fluoride release.

Results of the control group (table 6, fig. 21) revealed that constant low level of fluoride release was reached after 1 week and continued for the ongoing follow-up periods which come in accordance to similar pattern described by *Mazzaoui SA et al, Al Zraikat H et al and Prabhakar AR et al*. [80, 81, 95]

However, our results disagree with *Mithra H. N. et al* who reported that sudden drop in fluoride release reaching a plateau occurred after 1 month. [39] This could be due to the different methodology used in their study including the temperature of the storage medium which was 4 ˚C that could have had a delaying effect on fluoride release from the specimens.

Comparing the 4 groups (table 7) revealed that fluoride release in APF gel group (G1) was significantly increased in comparison to the other groups at 24 hrs and 48 hrs, while G2, G3, and G4 showed no significant difference in fluoride release at 24 hrs and 48 hrs.

At 4 days, CPP-ACP+F group showed significant decrease in fluoride release compared with APF group, and showed lower levels, although not significant, compared to G2 and G4.

These results may come in accordance with the study made by Al *Zraikat H. et al* as they reported that the incorporation of CPP-ACP into GIC significantly decreased fluoride release in comparison to the control group. **[81]**

However, these results were found to be in contrast with *Mazzaoui SA et al* where incorporation of CPP-ACP into GIC resulted in a significantly higher fluoride release. **[80]**

Comparing APF gel and CPP-ACP+F groups showed higher fluoride release of APF gel group compared to CPP-ACP+F group at 24 hrs, 48 hrs and 4 days, and the difference was statistically significant. This could be due to the difference in fluoride concentration between the two agents (1.23% or 12,300 ppm in APF gel and 0.2% or 900 ppm in the fluoride containing CPP-ACP paste) which may affect the rechargeability of GICs. **[101,102]** The high acidity of APF gel which has an erosive effect on the surface of glass ionomer may lead to increased fluoride uptake by the cement. [103] The gel form and high viscosity of APF may also be more readily absorbed into glass ionomer than the paste form of CPP-ACP or may have been trapped in the pores and cracks of the specimens; eventually releasing fluoride ions as the gel dissolves in the storage medium. [34]

After 1 week and 5 weeks all groups showed a constant low level of fluoride release and values showed no statistical significant difference between groups.

However, APF gel group showed higher levels of fluoride release compared to the other groups at all time intervals, which was found to be statistically significant only after 24hrs and 48hrs.

Regarding testing of diametral tensile strength, there was no significant effect of remineralizing agents used on DTS of glass ionomer (table 8). However, numerical difference in each group showed the following:

In G1 there was a non significant increase in DTS after application of APF gel from 9.1 to 10.7 MPa. Study results of *Hassan L.*, however, showed a significant increase in DTS of highly viscous glass ionomer after treatment with APF gel. The author reported that fluoride release could result in creating porosities within the material, these porosities may allow deeper diffusion of the topical fluoride agent into the sample, occluding the defected porosities of the material through the formation of calcium fluoride (CaF_2), thus enhancing mechanical porosities. [59, 84] The difference between the two results could be related to the difference in storage periods between the two studies.

On the other hand, a number of studies [106-109] have shown that phosphoric acid, one of the components of APF gel, significantly alters the surface morphology of various restorative materials leading to changes in the physical properties, such as hardness, roughness, and erosion resistance.

In G2 there was a non significant increase in DTS after appli cation of CPP-ACP paste from 9.1 to 9.4 MPa.

This result comes in contrast with *Mazzaoui et al* who expected that calcium and phosphate ions from CPP-ACP may preserve the cross-linking of polyalkenoate chains as the original hydrogel ions dissolve out of the set GIC [80] resulting in a strengthening effect on GIC. This could be due to the different methodologies used in the two studies. *Mazzaoui et al* had incorporated CPP-ACP into GIC, while in our study CPP-ACP was applied topically. Our results also disagree with *Abdou J. et al* who reported that CPP-ACP had a significant strengthening effect when applied to the external surface of glass ionomer [82], this again may be caused by the different methodologies used in the two studies, where *Abdou J. et al* had used a solution containing CPP-ACP which could be more readily absorbed into GIC than the paste used in our study.

In G3 there was a non significant increase in DTS after application of CPP-ACP+F paste from 9.1 to 9.9 MPa, which is numerically higher than values of both G1 and G3, in which no fluoride was used and lower than G2 in which fluoride gel was used.

Results of the control group (G4) showed no significant effect of time over DTS since there was no significant difference between mean value of DTS after 24 hrs and 5 weeks in this group. This result comes in agreement with *Yap A.U.J.* et al who reported no change in DTS of highly viscous glass ionomer when measured after 24hrs, 1 week and 1 month, **[51]** and also in accordance to *Bresciani E et al* who reported no significant difference in DTS of Fuji IX GIC when measured after 1 hour and 1 week. **[50]** This could be due to the faster setting reaction and maturation of the material as a result of increased powder/liquid ratio and smaller powder particle size, which can improve the mechanical properties mainly in the first hours. **[104, 105]**

Results of this study had shown that fluoride release of GIC was characterized by an initial burst in the first 24 hrs after mixing, followed by a rapid decline reaching a constant low level after 1 week. It had been shown that the remineralizing agents tested had different effects on fluoride release after application. However, only APF gel had a significant recharging effect on glass ionomer. Our results had also shown that the three agents had no significant effect on DTS of glass ionomer.

EFFECT OF DIFFERENT REMINERALIZING AGENTS ON FLUORIDE RELEASE AND TENSILE STRENGTH OF GLASS IONOMER CEMENT

Thesis

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