Short- and long-term fluoride release from glass ionomers and other fluoride-containing filling materials in vitro

LENNART FORSTEN

Institute of Dentistry, University of Turku, Turku, Finland


Abstract — Test specimens of seven different glass ionomer filling materials and one fissure sealant were exposed to running water for 2 yr. One amalgam and one composite, both containing fluoride, were included for comparison. The fluoride release from the specimens was measured periodically after storing the specimens for 24 h or 1 wk in a small amount of water. The fluoride release from the glass ionomers decreased with time and a constant level was reached for most products during the 2-yr period. The release was increased by lowering the pH of the storage solution. The release from the glass ionomers was clearly greater than from the amalgam and the composite.

Key words: dental materials; fluoride release; glass ionomers.

Institute of Dentistry, University of Turku, Lemminkäisenkatu 2, 20520 Turku, Finland.

Accepted for publication 3 May 1989.

The caries preventive effect of silicate cement due to fluoride release is well established from vast clinical experience for over 50 yr and by numerous studies (1). Laboratory experiments have shown fluoride release also from glass ionomers (2—6). The experiments have, however, usually been of short duration and with conflicting results. Fluoride release has even been shown in connection with fluoride-containing composites (7—11) and an amalgam (12, 13). These studies have been of short duration, too. Radiopacity of glass ionomer fillings can be achieved by incorporating silver particles during the processing of the glass powder (cermet) (14) or by adding amalgam alloy to the powder before mixing (Miracle Mix) (15). There are only a few studies on the fluoride release from cermets (6) and, as far as I know, none on the release from alloy-glass ionomer cements.

The purpose of the present study was to determine the fluoride release over 2 yr from different conventional glass ionomer filling cements, from a fissure sealant, a cermet, two alloy-glass ionomer materials, and from an amalgam and a composite, both also containing fluoride.
Material and methods

The materials are listed in Table 1. All are filling materials except Fuji III, which is recommended for fissure sealing. ASPA, Fuji II and III are conventional glass ionomers whereas ChemFil II and Ketac-Fil are of the anhydrous type (powder containing vacuum dried polyacrylic acid). Ketac-Silver is called a cermet and contains silver particles fused to the glass.

Miracle Mix is a mixture of Fuji II and Lumi-lloy (G.C.). In addition, a mixture of Fuji II and Sybralloy (Kerr, batch No. 1024752289) was made. In both mixtures the proportion of glass ionomer powder to alloy was 15/17 by weight.

Heliomolar radiopaque is a posterior composite containing 5% ytterbium trifluoride according to the manufacturer. Fluor Alloy contains 1% stannous fluoride.

The materials were handled according to the manufacturer's instructions and 2.8-mm-thick round plates with a diameter of 10.7 mm were made in a plastic mold. The surface of the specimen was pressed even with the mold top using a glass plate. After hardening the glass ionomer specimens were protected with petroleum jelly and stored in water for 24 h. The surface of the specimens was then cleaned by grinding lightly against a 600 grit paper. Two samples were made of each material.

After the 24 h initial storing period the specimens were continuously exposed to running non-fluoridated (F<0.1 ppm) tap water (0.5 liter/min, pH 8.0), except when the fluoride release was determined.

For measuring the fluoride release each specimen was periodically transferred into 5 ml of double distilled deionized (Milli-Q treated, Millipore) water (pH 6.1) at room temperature (22±2°C) for 24 h during the first 2 months. At the end of the 2-month period the release was also measured by storing the specimens for 1 wk in the deionized water. During the later period of the experiment only the 1-wk storage was used.

The specimens were once (after 8 months) stored for 1 wk at pH 5.0 in a buffered 20 mM Na-acetate solution after storing them for 1 wk at pH 6.1.

After storing the specimens in deionized water for 24 h or 1 wk the fluoride content of the water was measured with an expandable ion analyzer (EA 94, Orion Research Inc., USA) using a fluoride electrode (94-09) and a reference electrode (900100, 4 M KCl + AgCl).

The temperature of the running water changed between 10 and 22°C depending on the time of the year.

Results

Fig. 1 shows the release of fluoride into 5 ml of water during 24 h when the specimens had been in running water for 24 h, 1 wk, 3 and 8 wk. The columns represent the mean fluoride release from two specimens. The

<table>
<thead>
<tr>
<th>Material</th>
<th>Batch No. powder/liquid</th>
<th>Symbol</th>
<th>Manufacturer</th>
</tr>
</thead>
<tbody>
<tr>
<td>ASPA capsule</td>
<td></td>
<td>AA</td>
<td>Amalgamated Dental</td>
</tr>
<tr>
<td>Fuji II</td>
<td>230421/280971</td>
<td>FII</td>
<td>G.C.</td>
</tr>
<tr>
<td>Fuji III</td>
<td>300761/090761</td>
<td>FIII</td>
<td>G.C.</td>
</tr>
<tr>
<td>ChemFil II</td>
<td>DE11284-06</td>
<td>C</td>
<td>DeTrey/Dentsply</td>
</tr>
<tr>
<td>Ketac-Fil</td>
<td>N328</td>
<td>KF</td>
<td>Espe</td>
</tr>
<tr>
<td>Ketac-Silver</td>
<td>N352</td>
<td>KS</td>
<td>Espe</td>
</tr>
<tr>
<td>Miracle Mix</td>
<td>230671/280971</td>
<td>MM</td>
<td>G.C.</td>
</tr>
<tr>
<td>Fuji II + Sybralloy</td>
<td>230421/280971</td>
<td>FII + a</td>
<td>G.C. and Kerr</td>
</tr>
<tr>
<td>Fluor Alloy</td>
<td>3980</td>
<td>A</td>
<td>Dentoria</td>
</tr>
<tr>
<td>Heliomolar Radiopaque</td>
<td>329402</td>
<td>H</td>
<td>Vivadent</td>
</tr>
</tbody>
</table>
FLUORIDE RELEASE FROM FILLING MATERIALS

Fig. 1. Fluoride release (ppm) during 24 h after exposing filling material specimens to running water for 1 day, 1 and 3 wk, and 2 months. Half of the range is indicated at the top of the columns. In some cases the range was so small that it could not be shown.

Fluoride release from MM was not measured after 24 h and 1 wk.

A strong initial booster effect is obvious for all materials except for the composite. There were clear differences both initially and later in the fluoride release from different products. FII with and without alloy showed the greatest release whereas the release from the cermet (KS) and the amalgam was small and the release from the composite almost negligible. After 2 months in running water the 24 h release of fluoride from some products was too small to allow measurements. Fig. 2 shows the total amount of released fluoride during the 4 days when the release was measured.

Fig. 3 reveals the fluoride release during 1 wk after the specimens had been in running water for 2, 8, 14, and 22 months (the release after 5 and 12 months was also measured but is not shown in the diagram).

For one glass ionomer (FII) the release decreases almost evenly during the observation period whereas another product (C) reached a level of constant release already after the 2nd month. The other of the two alloy-containing glass ionomers (FII+a)
Fig. 3. Fluoride release (ppm) during 1 wk after exposing the specimens to running water for 2, 8, 14, and 22 months. Half of the range is indicated at the top of the columns.

seems to have reached the constant level before the second month.

The other materials seemed to have reached a level of constant release during the second year.

After 1 yr all glass ionomers except the cermet were able to raise the fluoride content of the water above 0.5 ppm whereas the release from the amalgam and the composite was near the detection limit.

Fig. 4 shows the total amount of fluoride released during the 6 wk (2nd, 5th, 8th, 12th, 14th, and 22nd) when the release was measured. The release differs clearly between the two different types of glass ionomer filling materials. The anhydrous brands (C, KF, and KS) released less than the conventional brands (AA, FII).

When the pH of the storage media was lowered to 5.0 (Fig. 5), the fluoride release after 8 months was clearly greater from all glass ionomers, except from FII+a, than at pH 6.1. KF released three times as much and FII twice as much fluoride than at higher pH.

Discussion

The experimental setup does not simulate the clinical situation correctly. In the mouth fluoride is probably not washed away as completely as in this experiment. Films and even layers (e.g. plaque) on the fillings may reduce the release and part of the released fluoride.
Fluoride will accumulate in the layers covering the filling. In addition there is the possibility of an uptake of part of the released and accumulated fluoride back into the glass ionomer fillings (16). On the other hand the release may be increased when the pH drops in the plaque. Furthermore, fluoride release in water has been reported to differ from that in artificial saliva in that different products behaved in different ways (17). Consequently, the fluoride can not be expected to be released at the same rate from glass ionomer fillings in the mouth as in this experiment.

The glass ionomers released initially many times the amount of fluoride that was released later. The clinical significance of this early release has not yet been clarified. Tooth-saving caries removal methods, like “tunnel” preparations, are, however, increasing. Especially in these cases it is desirable to have a strong initial fluoride effect to inhibit bacterial activity and arrest the process in unintentionally left carious dentin. Furthermore, it may be expected that the fluoride will activate the remineralization of uninfected inner dentin and demineralized enamel.

The main advantage of a long-term fluoride release is protection against secondary caries. The results of some earlier studies (4, 6) indicate that a constant level of release is reached already after some weeks. Although the rate of release was slowing down for most of the glass ionomers after the 2nd month in the present study, a level of constant release was clearly shown only by two materials (G and FII + a) during the first year. The conflicting results may be due to the difference in methods. In the present study the specimens were exposed to a more effective fluoride “extracting” process than in the earlier studies.

The initial release differed a lot between the various products, but high initial release seemed to be related to a fast decrease in the release rate. Thus the differences between the materials diminished with time.

Judging from over 10 yr clinical experience with the first glass ionomer, ASPA (AA), the level of long-term fluoride release from this material should be enough to main-
tain an anticariogenic effect. Shorter clinical experience with KF and KS indicates that even a lower level of release may be enough to inhibit secondary caries.

An increase in fluoride release was shown at lower pH in the present study. This was not surprising and may be caused by faster dissolution of the filling material, which is not desirable. On the other hand, higher fluoride release in connection with a pH drop results in better protection of tooth substance against demineralization. This should be noted especially with KF, which was more influenced by low pH than the other materials. It has been shown that the dissolution of Ketac-Fil is more susceptible to pH changes than that of Fuji II (18). The release from Ketac-Fil after 14 months had decreased to approximately the same degree as from most other materials indicating that the 1 wk in pH 5.1 had not deteriorated the samples of this product. In an earlier 1-yr study (4) Ketac-Fil was shown to release as much fluoride as Fuji II and the silicate control and less than ASPA. This is in contrast to the present results and may be due to the less eroding method of the earlier study. At this stage it is difficult to decide which one of the methods better simulates the clinical situation.

In this study FIII released less fluoride than FII, which is contrary to the findings of an earlier study (19) where the release from FIII was twice that of FII. No explanation for these conflicting results can be offered at this stage.

The release from the amalgam was, during the first 2 months, about the same as from the cermet but after a year it was almost too small to measure. Since the main need for an anticariogenic effect of an amalgam filling would be after some years, the advantage of adding fluoride to amalgam alloy is doubtful.

It has been claimed that the fluoride release from the composite Heliomolar will inhibit caries around the composite filling (9). Although the composite released a little more fluoride than the amalgam after 1 yr, the release was minimal compared with the release from the glass ionomers and compared with earlier observations on the release from silicate cements and an early fluoride-containing composite (1, 2, 7).

Before the clinical significance of such a small release is stated, the possible anticariogenic effect should be confirmed by clinical studies.

When the results of this study are compared with those of earlier studies it is evident that the in vitro method of exposing the specimens to dissolution has a great influence on the results. It would therefore be desirable to find a method that simulates the clinical situation as correctly as possible.

The results of this study suggest that fluoride is released for at least 2 yr from glass ionomer filling materials. This release seems to be maintained at a level that, according to earlier observations, will have an anticariogenic effect. The release is also much greater than the release from fluoride containing amalgams and composites. Furthermore, the release is increased by a drop in pH, which thus increases the caries protective properties of the glass ionomer filling.

Acknowledgments – This study was supported by Linda Gadd's grant from Finska Läkarealliansen. The materials were provided by their manufacturers.

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