Release of fluoride and metal ions from root surfaces after topical application of TiF_4, SnF_2, and NaF in vitro

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Aqueous solutions of TiF_4 cause a rapid uptake and a long-lasting retention of fluoride when applied to dentin. The aim of this study was therefore to investigate the pattern of fluoride release after TiF_4 application in vitro, compared with SnF_2 and NaF application. TiF_4, SnF_2, and NaF were applied for 4 min and 1 min to standardized areas of six groups of root surface specimens immersed in distilled water. Untreated specimens were used as controls. The water was changed daily for 30 days, and F concentrations measured by an ion-selective electrode. All test groups showed a rapid decline in F concentration. In the 4-min group F concentration more than double the detection limit of the F electrode could be registered the first 28 days for TiF_4, 11 days for SnF_2, and 7 days for NaF. In the 1-min group periods of F registration were shorter. Analysis of Sn by atomic absorption spectrophotometry showed decreasing concentrations in the first 12-day samples in the 1-min and 4-min groups. Traces of Ti were found in the first few samples, but no pattern of release could be observed. □ *Cariostatic agents; dentin; F electrode; in vitro study; preventive dentistry*

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Root surface caries is common among elderly people (1–3). Because the etiology of root caries is assumed to be similar to that of enamel caries, the same preventive strategies have been suggested (4, 5).

Aqueous solutions of TiF_4 seem suitable for clinical use, since the uptake of fluoride is comparable to that from fluoride varnishes when applied to root surfaces in vitro and in vivo (6, 7). Fluoride uptake is rapid (8), and both fluoride and titanium remain on dentin surfaces for an extended period of time (9, 10).

The purpose of the present study was to examine the magnitude and duration of fluoride release from root surfaces treated with a single topical application of a TiF_4 solution, compared with equimolar solutions of the more commonly used NaF and SnF_2 agents. Since it is possible that the presence of metal ions may influence the release pattern, the release of titanium and tin was also recorded.

Materials and methods

Roots from young human premolars and third molars were used in the experiment. The teeth were stored in glass vials containing wet cotton and crystals of thymol until used. The roots were scaled with curettes and lightly polished with pumice in water to remove all soft-tissue remnants. The crowns were removed and the pulp chamber sealed with wax before the teeth were assigned to two groups for each of the three fluoride agents plus a control group. To obtain experimental areas of standardized size, adhesive paper discs with a diameter of 5 mm were attached all over the roots before they were painted with an acid-resistant nail varnish. A total unvarnished area of 981 mm^2 was used in each experimental group. In each group several teeth in one vessel were used to obtain sufficient solution volume and reliable concentration readings.

After a pilot study the roots were immersed in the following solutions for the two assigned time periods: a) 3.4% TiF_4, (1.1 M F), pH 1.0, for 1 min and 4 min; b) 8.6% SnF_2, (1.1 M F), pH 2.3, for 1 min and 4 min; c) 4.6% NaF, (1.1 M F), pH 7.0, for 1 min and 4 min; and d) distilled water, control.

After immersion the specimens were immediately rinsed in 500 ml of distilled water for 30 sec under continuous stirring and transferred to 10 ml of distilled water.

The water was changed every 24 h for 30 days, and an ion buffer was added (TISAB III, Orion Research Inc., Cambridge, Mass., USA). Fluoride concentrations were determined every day with an ion-selective electrode (cat. no. 96-09-00, Microprocessor ionanalyzer/901, Orion Research Inc.) until a concentration less than twice the detection limit given by the manufacturer (0.02 μg/ml) was reached.

Analyses of Ti and Sn were performed with flameless atomic absorption spectrophotometry. The instrument was a Perkin-Elmer HGA-76B graphite furnace connected to a Perkin-Elmer 372 atomic absorption spectrophotometer and equipped with a deuterium background corrector. The instrument settings were as suggested by the manufacturer (11).

The samples used for metal analyses were made acidic by addition of hydrochloric acid and then centrifuged at 1000 *g* to sediment solid matter that might interfere with the analyses. The solutions analyzed appeared clear as judged by the naked eye.

Results

The registered fluoride concentration in distilled water for each group of roots is presented in Figs. 1, 2, and 3. The fluoride concentration after release from the TiF_4-treated specimens was 21.2 μg/ml on the 1st day, whereas SnF_2 and NaF treatment resulted in fluoride concentrations of 9.65 μg/ml and 9.50 μg/ml. The fluoride release decreased rapidly during the first days, then decreasing much slower in all groups (Figs. 1, 2, and 3). After application for 4 min the concentration of fluoride was more than twice the detection limit given by the manufacturer (0.02 μg/ml) for 7 days in the NaF group, for 12 days in the SnF_4 group, and for 28 days in the TiF_4 group. Fluoride released after 1 min of treatment could be registered for approximately half the number of days compared with the 4-min results (Figs. 1, 2, and 3). A slightly decreased pH was observed in the water used for initial rinsing of the TiF_4- and SnF_2-treated specimens. Measurements of Sn release after 1 day showed 0.67 μg/ml Sn (1-min exposure time) and 0.32 μg/ml Sn (4-min exposure time). The values decreased to below the detection

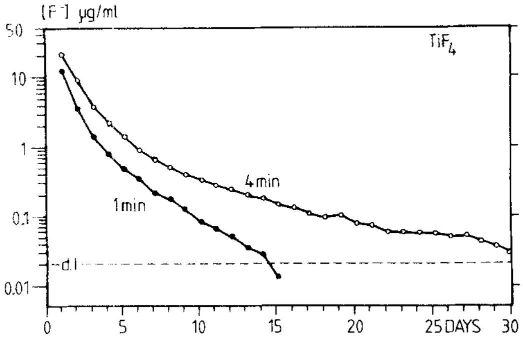

Fig. 1. Fluoride concentration (μg/ml) registered every 24 h in daily changed distilled water containing root surface specimens treated with 3.4% TiF_4 (1.1 M F) for 1 min and 4 min. d.l. = Detection limit of the electrode.

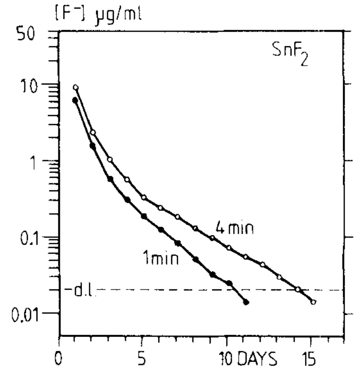

Fig. 2. Fluoride concentration (μg/ml) registered every 24 h in daily changed distilled water containing root surface specimens treated with 8.6% SnF_2 (1.1 M F) for 1 min and 4 min. d.l. = Detection limit of the electrode.

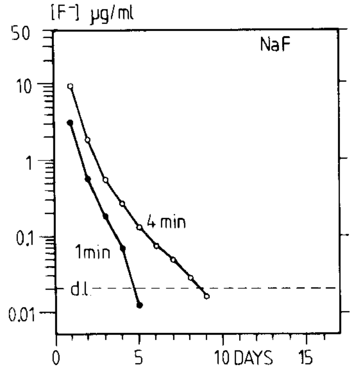

Fig. 3. Fluoride concentration (µg/ml) registered every 24 h in daily changed distilled water containing root surface specimens treated with 4.6% NaF (1.1 M F) for 1 min and 4 min. d.l. = Detection limit of the electrode.

limit (approximately 0.05 µg/ml) after 10 days. Titanium showed highly varying values even when the same sample was analyzed several times. Titanium was found in 1- to 5-day samples. However, no clear pattern of titanium release with time could be established.

Discussion

Since fluoride incorporated in dental hard tissues is considered to be important as a reservoir that can be released at the site of caries challenge, the pattern of release and the possibility for further uptake from the oral environment is important. The results in the present in vitro study showed that fluoride was released from root surfaces for a longer period of time after TiF_4 than after NaF treatment. This was not entirely unexpected, since previous studies have shown a much greater fluoride uptake with the TiF_4 agent (6–8). The higher release from the SnF_2-treated specimens than from the NaF-treated ones is in accordance with the high uptake of fluoride that has been reported from this agent as well (9, 12). On the other hand, the release of fluoride seems to be limited, since large amounts of fluoride are retained 22 weeks after treatment with both TiF_4 and SnF_2 (9).

One reason for the high uptake of fluoride from the Ti and Sn fluorides is the low pH, lower for Ti than for equimolar Sn fluoride solutions (13). The metal ions probably also play a part in the fluoride enrichment process because they are complex formers, binding concomitantly to fluoride and tooth structures. Ti is a more potent complexer than Sn (14). The complex-binding ability may explain the long-lasting fluoride retention registered after TiF_4 treatment in previous studies (9, 10). Owing to the great release of fluoride found in the present study, the complex-binding ability cannot be the only mechanism involved in the fluoride reactions. Possibly, several compounds are formed.

The high release of F from TiF_4-treated specimens indicates that at least part of the fluoride absorbed is not tightly bound. It is, however, likely that considerable amounts of fluoride remain in the hard tissue when release to distilled water can no longer be registered (9, 10). The remaining fluoride possibly has a caries-protective effect, since acid solubility is reduced after TiF_4 application to enamel (15–19) and to root surfaces (20). It is not known which part is played by the incorporated fluoride and which by the acid-resistant titanium-containing coating formed by this application (16, 17, 21).

The results indicate that Sn is released more readily than Ti from root surfaces. This finding may be explained by the stronger binding of the Ti ion to the tooth tissue (14). The variable results obtained with Ti suggest that the metal is given off as minute particles, possibly as a result of mechanical removal of the surface layer. Sn, on the other hand, showed a continuous pattern of release resembling the rate of fluoride release.

Fluoride concentrations necessary to inhibit artificial root caries has been reported to be a few micrograms per milliliter (5), whereas lower concentrations inhibit demineralization of enamel (22, 23). TiF_4-treated root surface specimens released greater amounts of fluoride for a longer period of time than did both SnF_2-and NaF-treated surfaces. NaF-treated specimens appeared to be rapidly depleted of fluoride. Thus, TiF_4 in this respect seems to be more advantageous than the more commonly used

SnF_2 and NaF solutions, because TiF_4 has a long-lasting fluoride delivery and, in addition, leaves considerable amounts of Ti in the surface layer (9, 10). The polyvalent Ti ion may attract fluoride when available and thereby maintain an enhanced concentration of fluoride in the surface layer (14). This process could not be studied in the present experiment, as the water was changed daily. Such redeposition has, however, been reported in a previous in vitro study in which the metal fluorides SnF_2, AgF, and CuF_2 were applied before immersion in distilled water (24).

Although the subsequent fluoride release decreased after 1 min of application compared with 4 min, the reduction was not in proportion to the shorter duration. Previous studies have indicated that TiF_4 has the most rapid reaction and consequently appears to be convenient for achievement of a fluoride depot in the clinical situation (8, 10). A greater caries-inhibiting effect than NaF, as shown in root caries models in vitro, supports this assumption (25).

Solubility properties of the topical agents used in vivo can be expected to be different from those in distilled water, because deposition of pellicle and plaque may modify release in the oral cavity (26, 27). The in vitro results nevertheless suggest that TiF_4 possesses desirable properties as a topical agent for root caries prophylaxis.

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