Effects of TiF_4 solutions on root surfaces in vitro after different application periods

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Skartveit L, Tveit AB, Tøtdal B, Selvig KA. Effects of TiF_4 solutions on root surfaces in vitro after different application periods. Acta Odontol Scand 1989;47:25–30. Oslo. ISSN 0001–6357.

Topical application of aqueous solutions of TiF_4 to root surfaces has been shown to result in a rapid uptake of fluoride. The purpose of this study was to assess whether the application period and/or the ionic strength of the TiF_4 solution can be reduced without a corresponding reduction in the fluoride uptake. Root halves from human teeth were exposed to (a) 1% TiF_4 for 10 sec and 1, 2, and 4 min, and (b) 3.4% TiF_4 for the same application periods. Root surface areas protected by nail polish served as controls. Transverse ground sections through the treated root surface areas were then prepared and analyzed for F by electron microprobe analysis. Most treated specimens showed F concentrations in the 0.60–1.94% range, whereas control surfaces contained less than 0.30%. Similar F concentrations were found in the surfaces treated with the two solutions. Reducing the application period resulted in a shallower penetration of F into the hard tissue but only a slight reduction of the F concentration in the surface layer. High concentrations of Ti were found on the surface of all treated specimens. This shows that both the application period and the ionic strength of TiF_4 solutions can be considerably reduced and still enable a high uptake of fluoride. The results indicate that TiF_4 may be an efficacious agent for F application to root surfaces under clinical conditions. □ *Electron microprobe analysis; fluoride uptake*

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In the search for fluoride agents suitable for the prevention of root caries, TiF_4 appears to have interesting properties. It has a strong complex-binding ability (1) and hence binds to F and the tooth structure at the same time. It forms a glaze on the surface (2–4) which is not removed by KOH washing (5). Less artificial lesion formation has also been reported in TiF_4-treated as compared with APF-treated enamel (4, 6).

An in vivo study in dogs has shown that topical application of a 3.4% TiF_4 solution for 4 min results in a high uptake and retention of fluoride (7). After 3 weeks the fluoride concentration in the dentin surfaces was at least as high as after treatment with fluoride varnishes and much higher than after application of a conventional 2% NaF solution. A subsequent study (8) showed that even a 1-min application of the TiF_4 solution resulted in high concentrations of F and Ti, which were retained for at least 21 weeks.

Previous observations have indicated that application of a TiF_4 solution, which has a very low native pH, may cause an undesirable demineralization of the tissue (8). Since TiF_4 is assumed to react very rapidly with dental hard tissues, the question arises as to whether the application period can be further reduced, to arrive at a clinically acceptable procedure. The purpose of this study was to assess whether the application period and the ionic strength of the TiF_4 solution can be reduced without a corresponding reduction in the amount of Ti and F taken up in the dentin.

Materials and methods

Twenty-four root halves from premolars extracted for orthodontic reasons were used. The teeth were stored in 100% humidity in glass vials containing wet cotton and crystals

of thymol until used. They were then rinsed in tap water and the root surfaces lightly scaled with curettes to remove soft-tissue remnants and polished with pumice. The crown of each tooth was cut off and the roots cut in two parts longitudinally. In each root half a longitudinal furrow, prepared with a carborundum disk, separated the experimental part of the surface from the control surface. The control surface was covered with two layers of nail polish. The specimens were divided into groups of three, which were then exposed to one of the following solutions of TiF_4 in distilled water: (a) 1% TiF_4 (0.32 M F) applied for 10 sec, 1 min, 2 min, or 4 min; and (b) 3.4% TiF_4 (1.1 M F) applied for 10 sec, 1 min, 2 min, or 4 min.

The fluoride agents were applied by cotton pellets to the three root halves in each group. The specimens were immediately after the application washed briefly in tap water and then prepared for electron microprobe analysis, to register and compare the uptake of fluoride in the root surfaces. The depth of the F increase and the deposition of titanium were also registered.

For this analysis two 200-μm-thick sections were prepared from each root half in a direction perpendicular to the long axis of the root. The ground sections were mounted on methacrylate specimen holders with two-sided tape, dried at room temperature, coated with a thin layer of carbon, and examined in an Applied Research Laboratories electron microprobe, which operated at 10 kV with a sample current on brass of approximately 60 nA.

Line scans of the electron beam were made at right angles to the root surface. Simultaneous analyses were carried out for Ca, F, and Ti. Four scans were made across the experimental part of each root and two analyses across the control surface. Analysis of Ca was included to determine the exact position of the edge of the root surface.

Analyses for F used the K_α emission of this element, and the concentration was estimated by assuming a linear relation between the concentration and the intensities in the specimens as compared with those in a standard of fluorapatite (38.94% Ca, 17.77% P, 3.85% F). Measurements of the concentration of this element with fluorapatite as reference standard is regarded as quite reliable (9, 10). Under the given operating conditions, the minimum detection limit for fluoride was about 0.15% by weight (11). For the analyses of titanium, biotite (1.83% Ti) was used as a standard. The techniques used have been fully described by Halse & Hals (10) and by Tveit & Tøtdal (12).

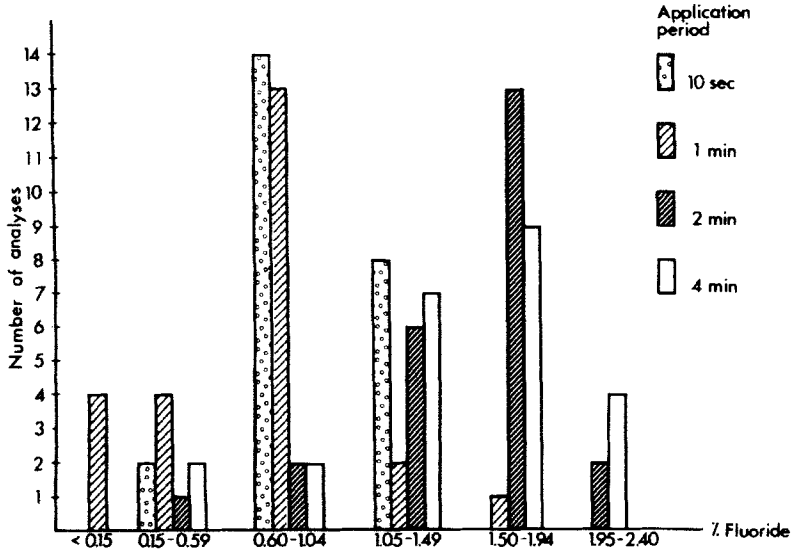

Fig. 1. Fluoride concentrations in root surfaces after application of 1% TiF_4 for different application periods. The bars indicate the number of scans showing fluoride values within each concentration range.

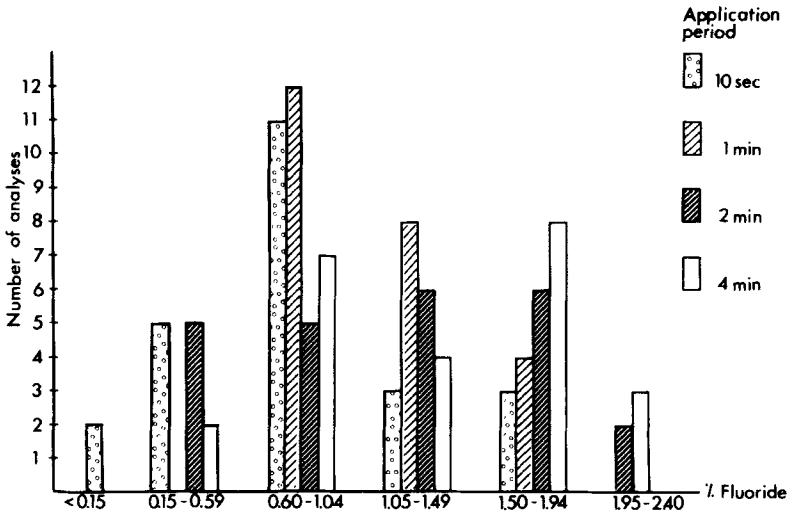

Fig. 2. Fluoride concentration in root surfaces after application of 3.4% TiF_4 for different application periods. The bars indicate the number of scans showing fluoride values within each concentration range.

Results

The results are summarized in Figs. 1 and 2 and Tables 1–3. Typical fluoride and titanium distribution curves are illustrated in Fig. 3.

In the control surfaces, 68 of 96 analyses did not show F concentrations exceeding the detection limit (0.15%). The remaining 38 analyses showed F concentrations from 0.15% to 0.30%, which are within the normal range of root surfaces (13).

In the experimental surfaces, four scans in one specimen in the 1% group and two scans in one specimen in the 3.4% group showed F concentrations below the detection limit. These data were regarded as artifacts due to misplaced varnish or handling error and were excluded from further analysis. All other analyses showed F concentrations well above the detection limit. After a 10-sec application of the TiF_4 solutions the F content in most of the root surfaces was higher than 0.60% (Figs. 1 and 2). Increasing the application period from 10 sec to 1 min did not result in an increase in the F concentration in the surfaces, whereas prolonging the application period up to 2 min gave an increase with both of the TiF_4 solutions. A 4-min application gave approximately the same result as the 2-min application, and the resulting F concentration in the outermost surface after application of the two solutions was of the same order (Figs. 1 and 2).

Table 1. Zone of increased F concentration in root surfaces after application of TiF_4 in accordance with solution strength and application period

Solution strength	Application period	Depth of F-rich zone, μm		
		Mean	SD	*n*
1%	10 sec	139	89	24
	1 min	185	85	20*
	2 min	171	70	24
	4 min	201	161	24
3.4%	10 sec	90	50	22*
	1 min	280	244	24
	2 min	234	31	24
	4 min	349	166	24

* A total of six analyses showing F concentration below the detection limit were excluded.

In the control surfaces the fluoride content registered was always localized in the outer 20–30 μm of the root tissue. In the experimental root surfaces, however, the fluoride concentrations showed maximum values at the surface and decreased to the normal level of dentin at depths ranging from 40 μm to 500 μm, with great variation within the groups (Table 1). The zone showing increased fluoride content was more uniform in depth within each specimen than between specimens. Fluoride penetration was deeper

Table 2. Titanium content on root surfaces after applications of 1% TiF_4 in accordance with application period. Number of analyses showing Ti values within each concentration range

Application period	Total no. of analyses	% Ti					
		<0.2	0.2–1.99	2–3.99	4–5.99	6–9.99	10–13.99
10 sec	24		8	8	8		
1 min	24	5	3	4	4	8	
2 min	24	4		2	5	10	3
4 min	24	2	4	7	5	4	2

Table 3. Titanium content on root surfaces after application of 3.4% TiF_4 in accordance with application period. Number of analyses showing Ti values within each concentration range

Application period	Total no. of analyses	% Ti					
		<0.2	0.2–1.99	2–3.99	4–5.99	6–9.99	10–13.99
10 sec	24	2	7	1	5	8	1
1 min	24		5	8	5		6
2 min	24	5	5	7	2	5	
4 min	24		6	3	5	6	4

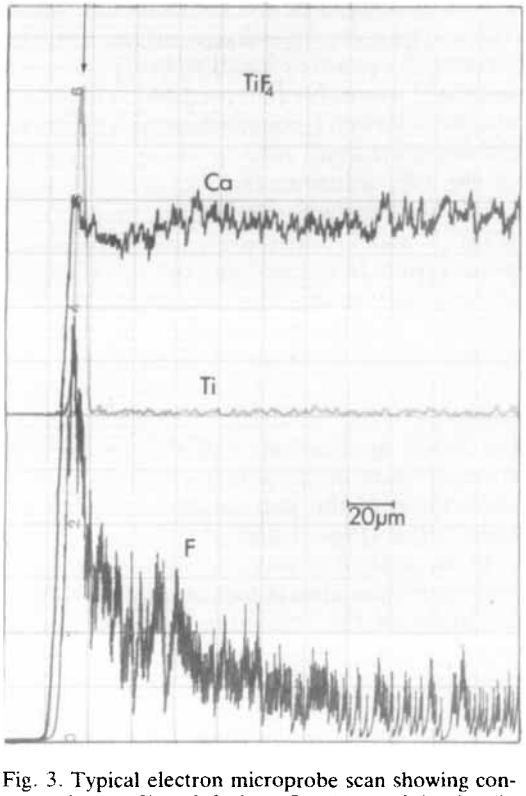

Fig. 3. Typical electron microprobe scan showing concentration profiles of clacium, fluoride, and titanium in a root surface after application of 1% TiF_4 for 4 min. The root surface is indicated by an arrow. Note the increased fluoride content in a 100-µm-wide surface zone and the Ti-rich coating on the surface.

when longer application periods and a stronger test solution were used.

An application period of 10 sec gave a similar fluoride penetration in specimens treated with 1% and with 3.4% TiF_4 solution. Prolonging the application period, however, resulted in a broader zone of increased fluoride concentration for the strongest test solution.

The Ti analyses did not show concentrations above the detection limit within the hard tissue. On the experimental surfaces, however, values up to 14% Ti were registered (Tables 2 and 3). There were great variations both within and between specimens, and a relationship between TiF_4 concentration in the solution, the application period, and the resulting Ti deposit was not established.

Discussion

Although the relationship between caries resistance and F content in tooth substances has not been uniquivocally shown, improving the availability of F at the surface is considered to be important, for instance by increasing the F content in the surface itself. This can be attained in various ways. Incor-

porating F into gels and varnishes to prolong the period of F exposure results in increased F content in the tooth surface (14, 15). Increasing the concentration and/or lowering the pH of the topical fluoride agent, and combining the fluoride with cations, as in TiF_4, are other methods to increase the F uptake (16–18).

Application of 3.4% TiF_4 for 4 min has been shown to result in a high fluoride uptake in dentin (7, 19, 20). Reduction of the application period to 1 min still gives a marked increase in fluoride content (8). Compared with a 4-min application of an acidulated phosphate fluoride (APF) solution, 1% TiF_4 applied for 1 min gave better caries protection in a clinical trial (21). Ti has a good complex-binding ability, and TiF_4 forms a glaze on the tooth surface (2–5, 20). These properties may in part explain the better effect of TiF_4 than of the APF solution (21). Moreover, it seems that TiF_4 reacts very rapidly with dental hard tissues and that a clinically significant effect may be obtained by using a shorter application period than with other fluorides.

The present results show that F is taken up within 10 sec after application of a 1% TiF_4 solution to a root surface. Increasing the concentration of the TiF_4 solution threefold did not give a corresponding increase in F uptake. The resulting F concentration in the surface layer of specimens treated with 1% and 3.4% solutions were of the same order, although total F uptake may have been greater with the stronger solution, as indicated by an, on the average, deeper zone of penetration. Similarly, prolonging the application period beyond 1 min did not result in a proportional increase in F uptake.

Furthermore, the results show that a 10-sec application period is sufficient for the formation of a TiF_4 glaze.

The great variations in fluoride concentration and penetration depth within each specimen group are presumably related to local differences in cementum thickness and permeability, peripheral obliteration of dentinal tubules, and the type of root surface left by the scaling procedure. Although such factors impart limitations on the statistical evaluation of the data, they reflect a source of variation which will also exist under clinical conditions.

Even though the pH is very low, the acidity of the TiF_4 solution (pH 1.0 for the 3.4% solution) does not result in the same surface demineralization as SnF_2 (8). The possibility of a certain amount of demineralization cannot be excluded, because the electron microprobe technique is not suitable for detecting a small mineral loss at the very top of the surface. This question therefore needs further study by transmission electron microscopy.

In conclusion, the results show that after topical application of TiF_4 to root surfaces, a high uptake of F and deposition of a Ti-rich glaze occur with much shorter application periods and more dilute solutions than previously examined. These findings indicate that TiF_4 would be a convenient topical agent for clinical use.

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Received for publication 6 October 1987