Influence of tooth surface conditions on enamel fluoride uptake after topical application of TiF_4 in vitro

Zhiling Gu, Jianguo Li and Rune Söremark

Center for Oral Biology and Department of Clinical Research, Faculty of Odontology, Karolinska Institute, Huddinge, Sweden

Gu Z, Li J, Söremark R. Influence of tooth surface conditions on enamel fluoride uptake after topical application of TiF_4 in vitro. Acta Odontol Scand 1996;54:279–281.

Three groups of extracted human teeth were sectioned longitudinally into experimental and control halves. The enamel surfaces of the three groups of teeth were pretreated with 37% H_3PO_4. for 1 min, 0.5 M $NaClO_3$ for 2 h, or water rinsing for 10 min. The experimental halves of the three groups received an application of 1% TiF_4 for 1 min and were washed with deionized water. The tooth specimens were then immersed in synthetic saliva for 24 h at 37 °C before sampling for measurement of fluoride uptake. An acid etching technique was used to determine the uptake of fluoride by measuring both fluoride and calcium concentrations in the solution collected from each etched sample. A significantly higher uptake of fluoride was found in teeth rinsed in water and etched with 37% H_3PO_4 than in teeth pretreated in 0.5 M $NaClO_3$. It seems that the organic components in enamel play an important role in the fluoride uptake after the topical application of TiF_4. □ *Dental caries; enamel; surface modification; titanium tetrafluoride*

Jianguo Li, COB, Box 4064, S-141 04 Huddinge, Sweden

Fluoride is the most effective and widely used caries-inhibitory agent. The topical application of fluoride in dentistry has two major advantages: inhibition of demineralization and increase in remineralization of the hard tissues of the tooth (1). Caries reductions of up to 40% have been reported in clinical trials after applications of topical fluoride agents (2). The role of fluoride is explained by the formation of calcium fluoride (3). However, the postulated correlation between enamel solubility in vitro and caries resistance has been challenged by many studies.

Titanium tetrafluoride (TiF_4) is a useful fluoride agent owing to its fast reaction with tooth substance. In vivo tests have confirmed the caries-inhibitory effect of TiF_4 (4, 5). Recently, we reported desensitizing effects of TiF_4 in a clinical study (6). Investigations have shown that TiF_4 reduces enamel solubility more effectively than other prophylactic agents after a short application time (7). Longer application times are needed for other fluoride prophylactic agents to achieve fluoride levels similar to those seen with TiF_4. A short application time provides an important clinical advantage.

The interaction between tooth surfaces and TiF_4 appears to differ from that of other fluoride preparations. Both titanium and fluorine seem to be involved in the interaction with the tooth. Several papers have been published exploring the interactions and mechanisms of high fluoride uptake during short application times. Mundorff et al. (8) assumed that an organo-metallic bridge was formed between TiF_4 and tooth tissue. Wei et al. (9) suggested that TiO_2 was formed on the surface of the enamel. However, more studies are needed to confirm these suggestions.

The purpose of this study was to explore the mechanisms of fluoride uptake by tooth enamel after brief topical applications of TiF_4 on the pretreated enamel surface of human teeth.

Materials and methods

Tooth preparation

Thirty extracted human premolars were cleaned with a hand instrument followed by ultrasonification for half an hour. Each tooth was then longitudinally sectioned into halves, one half serving as a control and the other as an experimental sample. All the pairs of tooth halves were stored in saline solution before treatment, and they were equally divided into three groups. The enamel surfaces of all the tooth halves were pretreated as follows: group I: rinsed in deionized water for 10 min; group II: etched for 1 min with 37% H_3PO_4 and rinsed in deionized water for 10 min; group III: kept in 12% $NaOCl_3$ (to remove organic components in the enamel) in accordance with Holma et al. (10) for 2 h and rinsed in deionized water for 10 min.

Fluoride application

One per cent TiF_4 was brushed onto the enamel surface of the experimental halves in the three groups for 1 min. The teeth were rinsed for 2 min with deionized water and immersed in synthetic saliva for 24 h at 37 °C before the measurements were performed.

Fluoride and calcium determinations

The consecutive acid etching technique, with 10 µl of 0.5 M $HClO_4$, 10 sec for each layer, was used to sample

Table 1. Mean values and standard deviations of fluoride uptake (ppm) in consecutive enamel layers after experimental treatments (the values are the net fluoride increases in the experimental halves of teeth)

Group	Layer 1, depth/15 μm	Layer 2, depth/30 μm	Layer 3, depth/45 μm
I	2488 ± 504	1284 ± 345	1284 ± 345
II	6721 ± 685	3346 ± 280	3380 ± 351
III	88 ± 257	20 ± 402	56 ± 231

the surface layers of enamel from the controls and the TiF_4-treated experimental specimens (11). Three layers from a 7 mm^2 biopsy window were taken. A cotton pellet was held against the window to collect the sample solution. The window was then washed twice with 5 μl of 20% TISAB and collected with cotton pellets. All the pellets were placed in 1 ml of 10% TISAB. The fluoride content was determined by using a fluoride electrode (Orion Research Inc., Boston, Mass., USA). The calcium content was analyzed by using an atomic absorption spectrophotometer (Pye Unicam SP9, Philips, Eindhoven, The Netherlands). The etched depth obtained with 0.5 M $HClO_4$ was calculated from the measured calcium content by assuming that the density of enamel is 2.95 g/cm^3 (12). The difference in fluoride uptake between experimental and control samples was calculated for each specimen pair, and the mean value of this net increase of 10 pairs was tabulated. The fluoride concentration was corrected to the standard sample depths of 15, 30, and 45 μm, to enable a comparison of the fluoride content in different etched layers.

Statistical analysis

The data were statistically evaluated by using Student's *t* test. The 95% confidence limit was used as a significant level.

Results

The results of the fluoride uptake in different groups after TiF_4 topical applications are summarized in Table 1. The uptake of fluoride in group II was significantly higher than that in groups I and III, and the fluoride uptake in group I was significantly higher than that in group III for all three depths ($P < 0.05$). The net increase in fluoride in group III after TiF_4 treatment was low. The etching depths of $HClO_4$ on enamel in the three groups are shown in Table 2. Group II showed significantly less etching depth than the other two groups. For depth 1 the etching depth was significantly less in group I than group III. The differences were not significant between group I and group III with the other two depths.

Table 2. The etching depths (means and standard deviations; μm) in enamel under different tested conditions (calculated depth after 10 μl 0.5 M $HClO_4$ etched for 10 sec)

Group	Depth 1	Depth 2	Depth 3	Total depth
I	25.1 ± 1.5	26.9 ± 1.5	29.0 ± 2.0	81
II	15.2 ± 1.5	18.1 ± 1.0	17.7 ± 1.0	51
III	34.1 ± 3.0	32.0 ± 2.0	36.9 ± 2.0	103

Discussion

The experimental methods used in the present study are in accordance with methods often used in this kind of study. It should be emphasized, however, that some variables are difficult to control. Thus, when using human teeth one has to accept that there are differences between teeth with regard to structure and composition of the enamel surface and differences along the tooth surface of the same tooth. Such differences may affect the test results. The pretreatment by means of acid etching removes a layer of the surface. This will influence the uptake of fluoride. The extent to which these differences affect the statistical treatment is difficult to say. Nevertheless, it is possible to discuss the results and draw conclusions, as follows.

A high fluoride uptake by the enamel was obtained after a short-term topical application of 1% TF_4. The results indicate that the fluoride uptake mechanisms of tooth enamel with TiF_4 differ from those with other fluoride agents, such as NaF, for which the uptake of fluoride is a comparatively slow process with a lower rate of uptake (13). Mundorff et al. (8) suggested that TiF_4 interacts rapidly with tooth tissues, both chemically and physically. The formation of fluoridated apatite reduces the solubility of the enamel, and the formation of a titanium dioxide glaze on the surface protects the enamel from acid attack.

The combination of 37% H_3PO_4 etching and 1% TiF_4 application (group II) resulted in a much higher fluoride uptake than did 1% TiF_4 application alone (group I). We assumed that the effect was related to the increased surface area and to relatively more organic components being exposed on the enamel surface after etching; there was practically no uptake of fluoride after the organic components in the tooth enamel were removed by $NaOCl_3$ (group III). It therefore seems that the organic components play an important role in the fluoride uptake of enamel from TiF_4.

The etching depth with 0.5 M $HClO_4$ correlates to the amount of fluoride taken up from TiF_4. The greater the fluoride uptake, the thinner is the acid-etched layer, because of the higher acid resistance. We believe that this effect is also due to the action of titanium dioxide, which may act as a barrier to acid penetration and fluoride loss. It is well known that titanium dioxide is a stable oxide, even in an acid environment (14).

It is concluded that the organic components in tooth enamel contribute to a high fluoride uptake after a brief topical application of 1% TiF_4, and the precipitated titanium dioxide on the enamel surface may be one of the reasons for the increased acid resistance of the enamel.

References

1. Featherstone J, ten Cate JM. Physiochemical aspects of fluoride-enamel interactions. In: Ekstrand J, Fejerskov O, Silverstone L, editors. Fluoride in dentistry. Copenhagen: Munksgaard, 1988: 127–30.
2. Marthaler T. Clinical cariostatic effects of various fluoride methods and programs. In: Ekstrand J, Fejerskov O, Silverstone L, editors. Fluoride in dentistry. Copenhagen: Munksgaard, 1988: 252–5.
3. Saxegaard E, Rölla G. Fluoride acquisition on and in human enamel during topical application in vitro. Scand J Dent Res 1988;96:523–35.
4. Shrestha B. Effect of systemic titanium tetrafluoride (TiF_4) on fluoride uptake by developing rat enamel. Caries Res 1983; 17:264–6.
5. Skartveit L, Spak C, Tveit A, Selvig K. Caries-inhibitory effect of titanium tetrafluoride in rats. Acta Odontol Scand 1991;49:85–8.
6. Charvat J, Söremark R, Li J, Vacek J. Titanium tetrafluoride for treatment of hypersensitive dentin. Swed Dent J 1995;19:41–6.
7. Shrestha B, Mundorff S, Bibby B. Enamel dissolution. I. Effects of various agents and titanium tetrafluoride. J Dent Res 1972; 51:1561–6.
8. Mundorff S, Little M, Bibby B. Enamel dissolution. II. Action of titanium tetrafluoride. J Dent Res 1972;51:1567–71.
9. Wei S, Soboroff D, Wefel J. Effects of titanium tetrafluoride on human enamel. J Dent Res 1976;55:426–31.
10. Holma B, Granath L, Gustafson G. A model for the study of tooth by scanning electron microscopy. Odontol Rev 1970;1: 1–11.
11. Retief D, Harris B, Bradley E. In vitro enamel fluoride uptake from topical fluoride agents. Dent Mater 1985;1:93–7.
12. Manly RS, Hodge, Ange LE. Density and refractive index studies of dental hard tissues. J Dent Res 1939;18:203–6.
13. Acuna V, von Beetzen M, Caracatsanis M, Sundström F. In vitro fluoride uptake by enamel and dentin. A comparative study of two varnishes. Acta Odontol Scand 1990;48:89–92.
14. Forberg S. Ceramic containers for spent nuclear fuel: on the corrosion resistance of rutile. Adv Ceram 1986;20:321–7.

Received for publication 7 November 1995
Accepted 19 December 1995