Titanium penetration in human enamel after TiF_4 application

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The TiF_4 application produces a titanium coating on enamel surface, reducing solubility in presence of cariogenic challenge. However, it is not established if this titanium also penetrates inside the enamel. The aim of this study was to evaluate in vitro *the presence of this superficial coat and titanium penetration into human sound and decayed enamel after TiF_4 application. Twenty-four unerupted third molars were mesiodistally cut and divided into two groups (GA - sound and GB – artificial decayed). After a 4% TiF_4 application, each sample was fractured longitudinally (occlusal-cervical). Through microprobe analysis with energy dispersive spectrometer (EDS), titanium penetration could be observed inside the enamel. The McNemar test (p=0.267) showed that there was no difference between the groups analyzed regarding to titanium penetration, although in group A the titanium penetrated more deeply (Wilcoxon test, p=0.047). It could be concluded that there was no difference between the groups regarding the titanium penetration, but titanium penetrated more deeply into sound enamel compared to artificially decayed enamel.*
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INTRODUCTION

Titanium tetrafluoride (TiF_4) presents an interaction with dental surface, through high penetration and retention of fluoride and low enamel acid solubility after application.[9]

The reaction is completed in seconds and both titanium and fluoride ion seem to be involved in this process.[12] When applied on dental structure, titanium breaks up its connections with fluoride ion and quickly links to oxygen on enamel surface, forming a superficial coat of titanium dioxide (TiO_2), chemically atoxic in biological systems.[11,12]

Studies show TiF_4 application as a preventive agent on sound enamel[6] and as a therapeutic one on artificial lesions.[22] Before that, some questions appear, such as the possibility of titanium, besides forming this superficial covering, also penetrates inside the enamel and if this penetration would be similar in sound or demineralized enamel.

The aim of this study was to evaluate *in vitro* the presence of a superficial coat and the titanium penetration into the human sound and decayed enamel after a 4% TiF_4 application.

MATERIALS AND METHODS

Sample

Twenty-four unerupted third molars were extracted for clinical reasons and stored in a 0.1% solution of distilled water with thymol crystals in pH 7.0.[10] These teeth did not present enamel defects (fractures, hypoplasias or hypocalcifications) as be observed to optic microscope.

After root portion removal, each tooth was mesiodistally cut using a diamond saw (Figure 1) and randomly divided into two groups: GA - 24 sound halves (12 buccal surfaces and 12 lingual surfaces) and GB - 24 artificial decayed halves (12 lingual surfaces and 12 buccal surfaces), so that each half in group A possessed its tally in group B. All segments were submitted to prophylaxis with rubber cup and pumice, followed by 10 seconds distilled water washing with spray and drying for the same time.

Demineralization Process

Group B samples were isolated with nail varnish[8] leaving a window of exposed enamel (2 x 2mm) on middle third to produce artificial demineralization according

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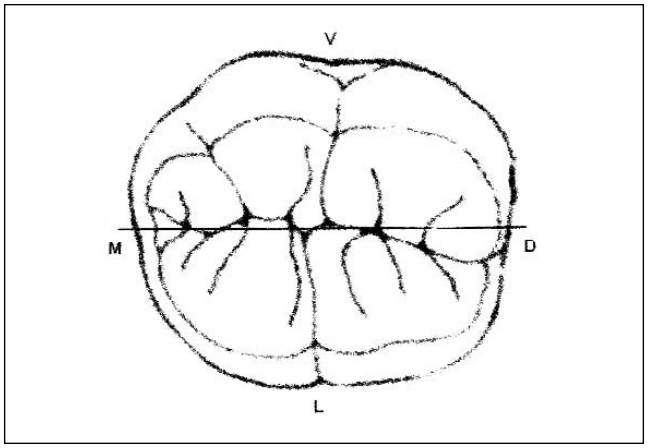

Figure 1. Third molar occlusal view.

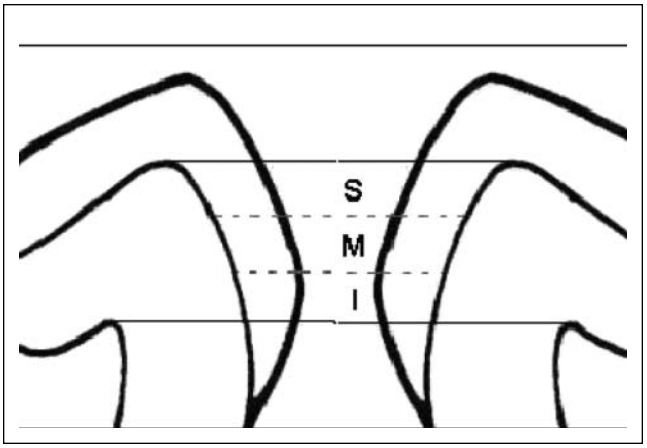

Figure 2. Internal view of left and right sides of segments. (S=superior area; M=middle area;I=inferior area.

to Boyle *et al.* [3] modified for this study: 2% hydroxyethylcellulose, 1M lactic acid, pH 4.5, for 24 hours at a temperature of 37º C. The halves were placed in individual containers with 6 ml, so that they were totally immersed in gel. At the end of this stage, each sample was washed in distilled water and dried for 10 seconds.

Application of the TiF_4

All segments received a 4% TiF_4 application (pH 1.0) for 60 seconds with a brush, followed by distilled water washing by the same time and drying by 10 seconds.

With diamond saw, fracture guides were accomplished in occlusal, posterior and cervical surfaces of each sample, except middle thirds. Through chisel and hammer all segments were longitudinally fractured.

SEM and EDS analysis

Only middle third area was observed at scanning electronic microscope (SEM). For this, the halves were glued in stubs, with enamel internal portions turned upward and with its buccal/lingual faces returned one for the other, so that each one corresponded to the left and right sides of sample. All segments were covered with a carbon layer. Of each half were made three electronmicrographs and respective titanium mapping through the microprobe analysis with energy dispersive spectrometer (EDS) in superior, middle and inferior areas of enamel middle third (Figure 2).

STATISTICAL ANALYSIS

The obtained results were tabulated in Excel 8.0 program, imported to statistical program SPSS 11.0 and analyzed through McNemar Chi-squared test, to determine if there was a significant difference in titanium penetration inside the enamel between groups A and B; and through Wilcoxon test, to determine if there was a significant difference in titanium penetration depth inside the enamel between groups A and B. For this, a significance level of 5% was considered.

RESULTS

The superficial titanium layer (TiO_2) was present in 100% of analyzed teeth, independently of titanium penetration. In group A, frequency of penetration was 66.7% (n=16) and 45.8% (n=11) in group B, without statistical difference through McNemar Chi-squared test (p=0.267).

After mapping the samples it could be verified titanium penetration inside the sound and artificially decayed enamel (Figures 3 A and B), although this titanium layer was irregular along the enamel.

Analyzing groups A and B regarding titanium penetration depth, there was a statistical difference (Wilcoxon test, p= 0,047), with a bigger titanium penetration depth at group A (Figure 4).

DISCUSSION

Several authors describe the presence of a titanium superficial layer over dental structure after application of titanium tetrafluoride.[6,9,12,17] In this work, the presence of this layer could be found in all samples, reinforcing the idea that, due to titanium likeness for the oxygen, titanium dioxide is formed before that free titanium penetrates inside the enamel.[11]

The titanium layer formation observed in the samples, was also verified on deciduous teeth occlusal surfaces, staying there 1 year after its application, as well as maintained these surfaces without caries lesions.[7] The use of TiF_4 as fissures sealants presents advantages when compared to other sealants types, such as easiness and reduction on application time, lower cost[7] and absence of biofilm retention areas, related to fractures and partial loss of sealants,[21] what would favor its employment in patients who carriers special needs, with disturbances of behavior, babies, institutionalized children or in patients that possess risk or high caries activity.

Still seeking prevention, Langer and Galon[13] accomplished a study of caries incidence in a population living at a non-fluoride water area. After 2 years and a

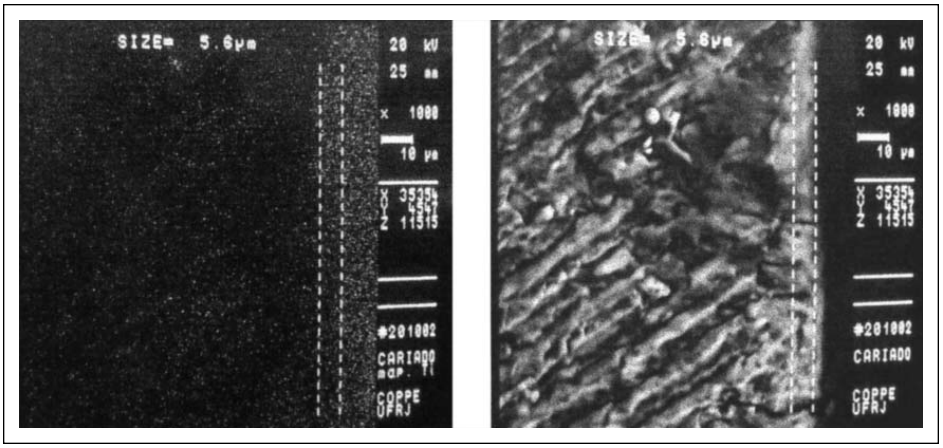

Figure 3. Titanium penetration inside enamel

half, the incidence was four times lower in study group that used TiF_4 as mouthrinse than in control group that received a placebo. These authors attributed study success to the layer formed on teeth after its TiF_4 contact. However, fluoride rinsing should be limited to children in preschool age, that possess spit reflex.[23]

Decalcified lesions around brackets are commonly observed during and after orthodontic treatment.[1] Due to that, Buyukyilmaz *et al*.[5] investigated the TiF_4 caries-preventive potential applied around brackets and observed after 4 weeks a reduction on depth and on mineral loss of these lesions. However, this titanium coat can be harmful in cases of brackets reposition, because it can reduce shear bond strength.[8] According to Santos *et al*.[16] this happens because TiF_4 provides a significant resistance to etched enamel. This fact should also be considered before making composite restorations.

However, when prophylaxis is accomplished with Robinson brush, this removes about 10.7µm of enamel, and with rubber cup, about 5µm.[15] In analyzed samples, the largest titanium penetration mean was 3.25µm (Figure 4), showing that prophylaxis should be indicated before enamel etching, in order to make shear bond strength between enamel and composite.

In samples of both groups, areas of titanium penetration were not evenly arranged along the enamel. This fact can be explained by the method used in samples preparation, because through fracture, surface observed at microscope becomes irregular and rough,[14] what may have hide the presence of titanium in some analyzed areas, due to perpendicular electrons incidence.[20]

Even if the difference on titanium penetration seem to be very small between the groups, it was observed that on group A the titanium penetration depth was statistically significant compared to group B (p=0.047). This fact was not expected, because due to the bigger porosity attributed to demineralized enamel,[2] it was speculated that titanium penetration would be larger in artificial decay group. However, demineralized enamel possesses smaller amount of water and carbonate (sources of oxygen) than sound enamel after drying,[4] that would harm titanium connection with oxygen on dental structure.

Table 1. Descriptive data of titanium penetration between groups A and B.

	Group A	Group B
Mean	3.25 µm	1.76 µm
Standard Deviation	2.84 µm	2.09 µm
Median	0.00 µm	0.00 µm
1st Quarter	0.00 µm	0.00 µm
Medium	3.50 µm	0.00 µm
3rd Quarter	4.50 µm	3.58 µm
Maximum	9.10 µm	6.70 µm

Regarding the titanium penetration, it was shown to be smaller in teeth with caries like lesions, according to Wefel[22] these lesions are significantly reduced after TiF_4 application when compared to acidulated phosphate fluoride. Later, Soyman *et al*.[19] concluded that titanium tetrafluoride application on enamel surface increases fluoride levels propitiating remineralization through fluorapatite formation.

In conclusion, this study demonstrated that that there was not difference between the groups regarding the titanium penetration, but titanium penetrated more deeply into sound enamel compared to artificially decayed enamel.

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