

TiF₄ varnish protects the retention of brackets to enamel after *in vitro* mild erosive challenge

Abstract

The effect of fluoride agents on the retention of orthodontic brackets to enamel under erosive challenge is little investigated. Objective: The aim of this study was to evaluate the effect of titanium tetrafluoride (TiF₄) and sodium fluoride (NaF) agents on the shear bond strength of brackets to enamel and on the enamel microhardness around brackets under erosive challenge. Methods: Brackets were bonded to bovine incisors. Five groups were formed according to fluoride application (n=10): TiF₄ varnish, TiF₄ solution, NaF varnish, NaF solution and control (without application). The specimens were submitted to erosive challenge (90 s cola drink/2h artificial saliva, 4x *per* day for 7 days). Solutions were applied before each erosive cycle and varnishes were applied once. Vickers Microhardness (VHN) was obtained before and after all cycles of erosion and the percentage of microhardness loss was calculated. Shear bond strength, adhesive remnant index and polarized light microscopy were conducted after erosion. The data were analyzed by ANOVA, Tukey, Kruskal-Wallis and Mann-Whitney U tests ($\alpha=0.05$). Results: The %VHN had no statistically significant differences among the experimental groups. However, considering the comparisons of all groups with the control group, TiF₄ varnish showed the highest protection from enamel demineralization (effect size of 2.94, while the effect size for the other groups was >2.4). The TiF₄ varnish group had significantly higher shear bond strength compared to other groups. There was no difference among groups for adhesive remnant index. Polarized light microscopy showed higher demineralization depth for the control group. Conclusions: Application of NaF and TiF₄ agents during mild erosive challenge minimized the enamel mineral loss around brackets, however only the experimental TiF₄ varnish was able to prevent the reduction of shear bond strength of brackets to enamel.

Keywords: Brackets. Fluorides. Shear bond strength. Tooth erosion.

Maria Isabel Dantas de MEDEIROS¹
Hugo Lemes CARLO²
Rogério Lacerda dos SANTOS²
Frederico Barbosa SOUSA¹
Ricardo Dias de CASTRO¹
Renata Cristina Sobreira FRANÇA¹
Fabiola Galbiatti de CARVALHO²

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Corresponding address:
Fabiola Galbiatti de Carvalho
Universidade Federal de Juiz de Fora
Instituto Ciências da Vida -
Departamento de Odontologia
Rua Israel Pinheiro, 2.000 - Bloco D9 -
Bairro Universitário - 35020-200 -
Governador Valadares - Minas Gerais - Brazil.
Phone: +(55)-33-3301100 - Fax: +(55)-33-3301101
e-mail: fabigalbi@yahoo.com.br

¹Universidade Federal da Paraíba, Programa de Pós-Graduação em Odontologia, João Pessoa, Paraíba, Brasil

²Universidade Federal de Juiz de Fora, Departamento de Odontologia, Governador Valadares, Minas Gerais, Brasil.



Introduction

Dental erosion is the teeth mineral loss due to a chemical process, by exogenous or endogenous acids, without bacterial involvement¹⁸. The prevalence of dental erosion among 12 to 21 years-old-students in the world population varies approximately between 15 to 75%, with mild erosion being the most prevalent condition^{1,7,14}. Currently, the most important acid sources come from dietary habits due to the increased consumption of soft drinks by the population¹⁸.

Consumption of acidic beverages decreases the pH in the oral environment, and factors other than pH, such as type of acid, pKa, titratable acidity, buffering capacity and temperature of acidic beverages can also influence on its erosive potential, causing enamel demineralization around brackets and interfering in their retention to enamel^{12,22}. Thus, the topical application of fluoride is also recommended to minimize the enamel demineralization and to improve the shear bond strength of brackets to enamel¹².

Sodium fluoride (NaF) is a monovalent fluoride compound and the most commonly found fluoride salt in toothpastes, mouthwashes and varnishes²⁴. The NaF has shown positive results in the reduction of enamel erosion progression^{6,13} and its protective effect is associated with the precipitation of calcium fluoride material on eroded dental surfaces, especially when used in high concentration and acidic formulation^{13,18}. As the anti-erosive effect of conventional monovalent fluorides requires a very intensive fluoridation regime¹⁷, current studies have focused on polyvalent metal ions of fluoride compounds that might have higher efficacy, as in the case of titanium tetrafluoride (TiF₄)^{13,17}. Studies have demonstrated that TiF₄ increases the uptake of fluoride because of its acidic pH and can form a glaze-like surface layer that acts as an acid-resistance diffusion barrier^{4,19,23}.

Although some studies have shown that the retention of orthodontic brackets to enamel is decreased when subjected to erosive challenge^{12,22}, there have been no data published concerning whether NaF and TiF₄ agents (varnish and solution) can protect the retention of metal orthodontic brackets to enamel during erosive challenge. Thus, the aim of this study was to evaluate the effect of TiF₄ and NaF agents on the shear bond strength of brackets to enamel and on the enamel microhardness around brackets under erosive challenge. The hypotheses tested were

that the TiF₄ and NaF agents (varnish and solution) applied to enamel during an erosive challenge can minimize: 1) the demineralization of enamel around orthodontic brackets, as measured by a microhardness test and visualized by polarized light microscopy; 2) the decrease of the shear bond strength of metal orthodontic brackets on enamel, as measured by the shear bond strength test and the adhesive remnant index (ARI).

Materials and methods

Specimen preparation

Fifty freshly extracted lower bovine incisors were used in this study. The teeth were cleaned and the buccal surfaces were ground flat with SiC paper discs (400, 600 and 1200 grids) to expose the enamel bonding. The specimens were allocated into five groups (n=10) according to the fluoride application:

- 1) experimental TiF₄ varnish group (TiF₄ V) – FGM, Joinville, SC, Brazil;
- 2) experimental TiF₄ solution group (TiF₄ sol) – 4 g power TiF₄ (Sigma-Aldrich, St. Louis, MO, USA) dissolved in 100 mL deionized water;
- 3) NaF varnish group (NaF V) – Duraphat - Colgate Palmolive Ltd., São Bernardo, SP, Brazil;
- 4) NaF solution group (NaF sol) – FlúorSol Clear – Dentsply, Petrópolis, RJ, Brazil;
- 5) Control group – without fluoride application.

The pH of solutions was measured by electrodes and the pH of varnishes were informed by the manufacturer. The composition of materials is described in Figure 1.

Application of brackets

The roots were vertically embedded in acrylic resin (Vipi Flash, Pirassununga, SP, Brazil) up to the clinical crown level, using a set-square supported on the buccal surface to maintain the enamel surface perpendicular to the base of the acrylic resin. After prophylaxis with pumice and water, the specimens were etched with 35% phosphoric acid gel for 30 s, washed and dried by air-blowing. The primer and the resin of Transbond™ XT Light cured system (3M Unitek, Monrovia, CA, USA) were used to bond the maxillary central incisor bracket (Edgewise system, Morelli, Sorocaba, SP, Brazil) in the central area of the middle third of the buccal surface. The resin excess

was removed. Light curing was performed for 60 s by LED (1200 mW/cm² – Rádi Cal. SDI, Bayswater, Victoria, Australia).

Fluoride treatment

The enamel was covered with acid-resistant nail varnish (Colorama, CEIL Ltda., SP, Brazil) around the bracket at a distance of 2 mm. This area was submitted to application of fluoride agents and erosive challenge. The specimens were immersed in artificial saliva for 24 h¹⁶, the saliva was produced according to study held by McKnight-Hanes and Whitford²⁰ (1992). Subsequently, the fluoride agent was applied on the enamel surface around the bracket using a 0.3 mL insulin syringe (BD Ultra-fine, Franklin Lakes, NJ, USA) to standardize the amount applied.

For groups 1 and 3, 20 µL of each fluoride varnish was applied on enamel around the bracket and spread with a microbrush. Samples were immersed in artificial saliva for 6 h to simulate clinically the contact time with the tooth surface¹⁹. Afterwards, the varnishes were carefully removed using a scalpel blade. For groups 2 and 4, 20 µL of each solution was applied on enamel around the bracket for 1 min. In the control group, no product was applied. The varnishes were applied only once before the erosive challenge, and solutions were applied before each erosive cycle¹⁹.

Mild erosive challenge

Specimens were immersed in Coca-Cola (Coca-Cola, SP, Brazil – pH 2.3), using separate containers (30 mL/specimen) at room temperature, 4 times *per* day for 90 s each time¹⁶. After thorough rinsing with deionized water, specimens were immersed in artificial saliva, pH 7.0 (30 mL/specimen), at room temperature for 2 h, between erosive challenges and overnight. This erosive challenge was repeated for 7 days and the specimens were stored in 100% humidity for shear bond strength and microhardness tests.

Shear bond strength test (SBS)

The direction of the debonding force was parallel to the enamel surface in an occlusogingival direction. A stainless steel rod with a chisel configuration was used for bracket debonding in a universal testing machine (Instron model 4411; Canton, MA, USA). Crosshead speed was 0.5 mm/min. The SBS was described in MPa.

Microhardness test

A microdurometer (HMV II; Shimadzu Corporation, Kyoto, Japan) was used with a Vickers indentator, under a 1.961 N load for 15 s. The area selected for indentation was the enamel located in the direction of the bracket slot, at a distance of 50 µm from the area of bracket debonding. The enamel had five indentations made in the mesial and distal regions of the bracket in the area described. Each indentation was at a distance of 100 µm from the other. The mean of the vickers microhardness values (VHN) was obtained. Measurements were conducted before and after erosive challenge in the same location. In addition, the percentage of vickers microhardness loss (%VHN) was calculated using the following formula⁵.

$$\%VHN = 100(VHN_F - VHN_I / VHN_I),$$

where VHN_I is the average of the initial (baseline) microhardness measurements, and VHN_F is the average of the final (after erosive challenge) microhardness values.

Evaluation of adhesive remnant index (ARI)

After removal of the brackets, the ARI was observed using a stereomicroscope with 10x magnification by a single examiner (intra-examiner kappa=0.92) according to Artun and Bergland² (1984): score 0) no composite left on the tooth; score 1) less than half of the composite left on the tooth; score 2) more than half of the composite left on the tooth; score 3) all composite left on the tooth, with distinct impression of the bracket mesh.

Material	Composition (batch number)
Experimental TiF ₄ varnish (FGM, Joinville, SC, Brazil)	2.45% titanium tetrafluoride, ethanol, synthetic resin and natural resin (pH 3.4)
Experimental TiF ₄ solution	2.45% titanium tetrafluoride, deionized water (pH 1.4)
Duraphat (Colgate-Palmolive Ind. Com. Ltda, São Bernardo, SP, Brazil)	2.26% sodium fluoride, alcohol, natural resins, wax, saccharine, flavor (pH 4.5) (05 10 01)
Flúor Sol Clear (Dentsply Ind. Com., Petrópolis, RJ, Brazil)	0.05% sodium fluoride, alcohol, preservative, sweetener, surfactant, flavor and water (pH 6.0) (791842F)

Figure 1- Compositions of fluoride agents tested in the study

Polarized light microscopy (PLM)

Teeth sections of 500 µm, containing an area of bracket adhesion and enamel, were obtained from each specimen. The sections were manually polished with SiC paper discs (600 and 1200 grits) under water refrigeration to a thickness of 100-120 µm. Polished tooth sections were placed in water and visualized under PLM (Axioskop 40, Carl Zeiss, Germany), and standard 35 mm photomicrographs were taken with 10x magnification.

Sample size calculation

Based on a pilot test, a difference was predicted in the shear bond strength between the two groups with the highest difference of 4.0 MPa ± 3.0 MPa, corresponding to a Cohen's *d* effect size of 1.33. For the microhardness testing, a difference was predicted between groups with the highest difference of 33.5 VHN ± 25.0 VHN, corresponding to a Cohen's *d* effect size of 1.32. The Cohen's effect size *d* between the two groups with the highest difference can be used to calculate the sample size per group for a study with various groups using ANOVA⁸. Considering a two-tailed type I error of 5% (*z* score of 1.96), and statistical power of 80% (*z* score of 0.842), the calculated sample size using equation 12.2.1 of Cohen⁸ was 10 teeth *per* group for both shear bond strength and microhardness experiments.

Statistical analysis

Data analysis was performed with the GraphPad Instat version 2.0 (GraphPad software, CA, USA) and Excel Microsoft software at a significance level of $\alpha=0.05$. Two hypotheses of difference were tested: one related to %VHN, and other related to SBS. Because the variables tested satisfied the assumptions of equality and normal distribution (Bartlett and Kolmogorov–Smirnov tests, respectively), one-way ANOVA and Tukey's tests with corresponding Cohens' effect size *d* and its 95% confidence intervals (CI) were carried out for statistical comparisons of SBS and %VHN. The correlation between the group ranks and the size of the effect of %VHN (determined in relation to the effect size of %VHN between each group and the control group) was tested using the Spearman's rank correlation coefficient. Evaluation of ARI scores was carried out using the Kruskal-Wallis and Mann-Whitney *U* tests.

Results

Following the recently published guidelines of the American Statistical Association²⁵, our statistical analysis was not restricted to *p*-values. According to ASA guidelines²⁵: (i), scientific conclusions should not be based whether a *p*-value passes as specific threshold; (ii) researchers should disclose the number of hypotheses explored during the study; and (iii) a *p*-value does not measure the size of an effect. Thus, the hypotheses tested in the statistical analysis of this study were reported using *p*-values, effect size (and its 95% CI), and power. The effect size measures the intensity of the difference (or correlation) between groups⁸.

The mean values (± standard deviation) of SBS and %VHN are shown in Figure 2. The treatments affected significantly both SBS ($p<0.0001$, ANOVA) and %VHN ($p=0.0002$, ANOVA). For both SBS and %VHN, the effect size of the erosive challenge in the control group was the highest and in the TiF₄ varnish group was the lowest. The pairwise comparisons are shown in Table 1. For all treatment groups, the %VHN was significantly lower (with a large effect size) compared with the control group. For other comparisons, no statistically significant differences were found, and the 95% CI either crossed the null hypothesis value or were very close to it (Table 1).

Considering the different sizes of the positive effect of each treatment relative to the control group, the hypothesis that there was correlation between effect size (scalar data) and treatment type (ranked data) was tested. For that, the group ranks were: TiF₄ varnish, rank=5; TiF₄ solution, rank=4; NaF solution, rank=3; NaF varnish, rank=2, and control group, rank=1. The Spearman's correlation of 0.99 indicates that the higher the group rank, the higher the size of the protection against enamel demineralization. The group ranks are positively correlated with the size of the protection against enamel demineralization. Thus, based on the Cohen's effect size *d* values, pairwise comparisons were ranked in the following order (from highest to lowest effect size): TiF₄ varnish x control (effect size=2.94); TiF₄ solution x control (effect size=2.37); NaF solution x control (effect size=2.26); NaF varnish x control (effect size=2.11), and control x control (effect size=0.0). The corresponding correlation coefficient was 0.99 (95% CI=0.999/0.903; $p=0.0012$; power=96.6%).

Regarding SBS, TiF₄ varnish group had significantly higher SBS values (with large effect sizes) in all pairwise comparisons, while the other comparison showed no statistically significant differences (Table 2). The largest difference was between TiF₄ varnish group and the control group.

Figure 3 shows the percentage of ARI scores for each group. No significant difference was found among

groups. All groups had large amounts of resin left on the tooth, with a distinct impression of the bracket mesh (score 3) (*p*=0.58).

Figures 4 and 5 show PLM images of patterns of demineralization around brackets after fluoride application and erosive challenge. All groups showed enamel demineralization compared to sound enamel (Figure 4A). Control group (Figure 4B) showed a higher

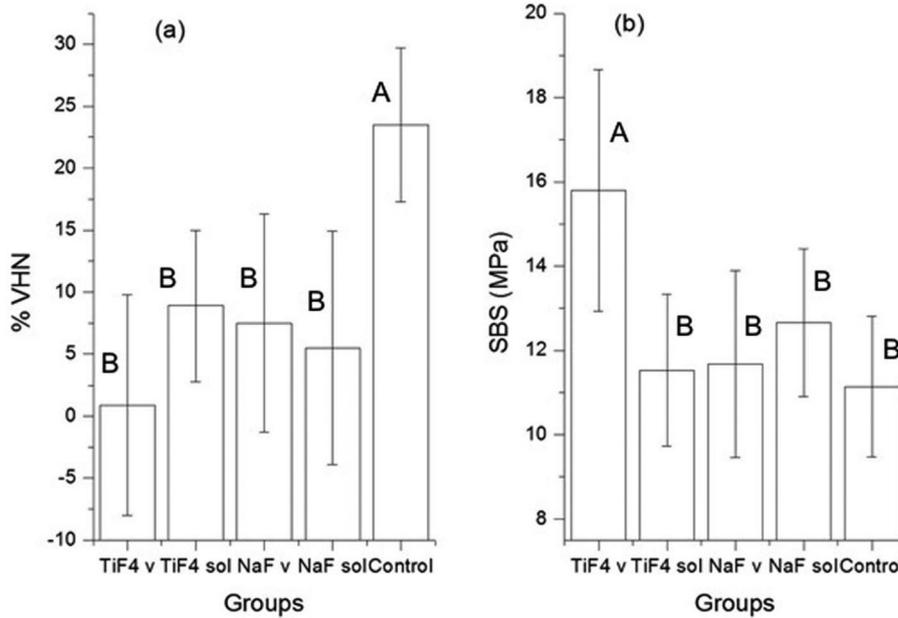


Figure 2- (a) Value plots (mean±SD) of microhardness percent loss (%VHN) for all groups. (b) Value plots of Shear Bond Strength (SBS) (mean±SD) in MPa. Different capital letters indicate statistical difference among groups (one-way ANOVA and Tukey's tests, *p*<0.05)

Table 1- Results of pairwise comparisons of microhardness percent loss (%VHN): Cohen's d effect size (ES) and its 95% confidence interval (upper limit of confidence interval/lower limit of confidence interval), and *p*-value (Tukey)

	TiF ₄ varnish	TiF ₄ solution	NaF varnish	NaF solution
TiF ₄ varnish	---	---	---	---
TiF ₄ solution	ES=1.05(1.98/0.11) p=0.0553	---	---	---
NaF varnish	ES=0.75(1.65/-0.16) p=0.6653	ES=-0.19(0.69/-1.06) p=0.597	---	---
NaF solution	ES=0.50(1.39/-0.39) p=0.232	ES=0.43(1.32/-0.45) p=0.9588	ES=0.22(1.10/-0.66) p=0.9381	---
Control	ES=2.94 (4.21/1.68) p<0.0000	ES=2.37(3.52/1.23) p<0.0004	ES=2.11(3.20/1.01) p<0.0000	ES=2.26(3.38/1.14) p<0.0000

Table 2- Results of pairwise comparisons of shear bond strength test: Cohen's d effect size (ES) and its 95% confidence interval (upper limit of confidence interval/lower limit of confidence interval), and *p*-value (Tukey)

	TiF ₄ varnish	TiF ₄ solution	NaF varnish	NaF solution
TiF ₄ varnish	---	---	---	---
TiF ₄ solution	ES=1.79(2.83/0.75) p=0.0004	---	---	---
NaF varnish	ES=1.61(2.62/0.60) p=0.0007	ES=0.07(0.95/-0.80) p=0.9998	---	---
NaF solution	ES=1.33(2.30/0.34) p=0.0406	ES=0.64(1.53/-0.26) p=0.5042	ES=0.49(1.38/-0.40) p=0.6086	---
Control	ES=1.99(3.07/0.92) p<0.003	ES=0.23(1.11/-0.65) p=0.9662	ES=0.28(1.16/-0.60) p=0.9887	ES=0.89(1.81/-0.03) p=0.8737

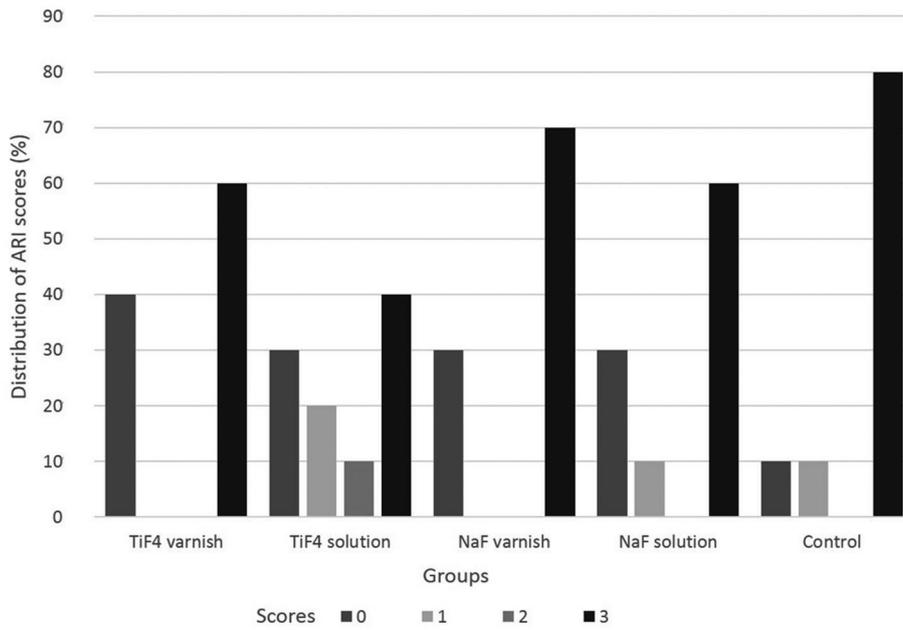


Figure 3- Distribution of Adhesive Remnant Index (ARI) scores (%) for each group

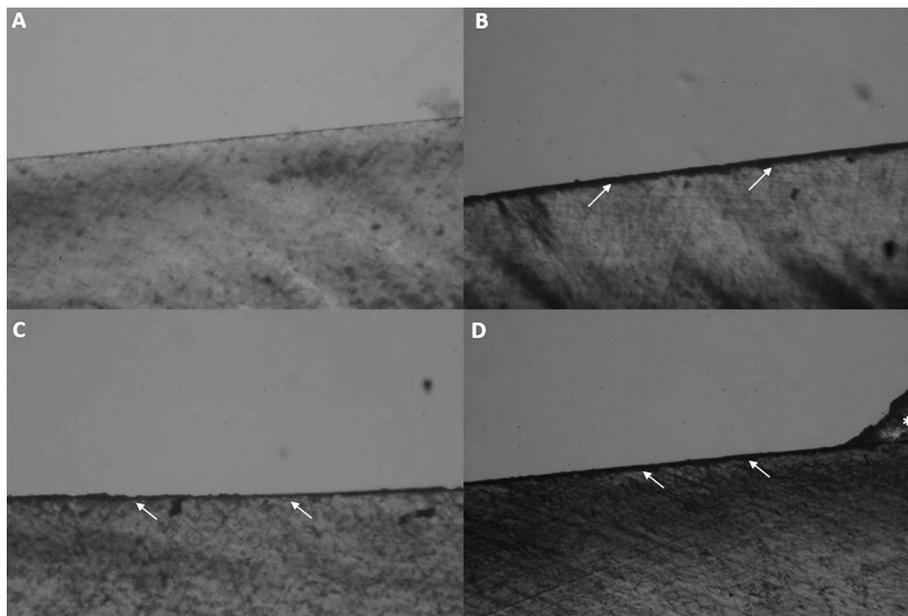


Figure 4- Polarized Light Microscopy images (10x). (A) Sound enamel. (B) Enamel demineralization in control; (C) NaF varnish; (D) and NaF solution groups. (→) Enamel demineralization. (*) Remaining resin left on enamel after shear bonding test

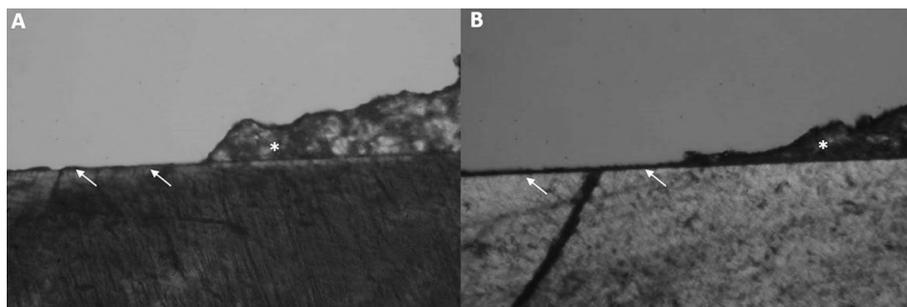


Figure 5- Polarized Light Microscopy images (10x). (A) Enamel demineralization in TiF₄ varnish; (B) and TiF₄ solution groups. (→) Enamel demineralization. (*) Remaining resin left on enamel after shear bond test

demineralization depth compared to other groups. NaF and TiF₄ varnishes (Figures 4C and 5A) and the solutions (Figures 4D and 5B) groups showed similar demineralization patterns on the enamel.

Discussion

The orthodontic bracket acts as an additional retention site for acidic substances, which contribute to tooth demineralization^{10,15} and decrease the bracket retention to the enamel surface^{12,22}. This study was the first to evaluate solutions and varnishes containing monovalent or polyvalent metal ions fluoride compounds applied on enamel during erosive challenge and their influence on the retention of brackets to enamel and on the enamel microhardness around brackets.

In the oral cavity, the contact of the enamel with an acidic beverage is usually limited to a few seconds before clearance by saliva³. Thus, the dynamic erosive model with immersion in saliva simulated the typical consumption of soft drinks by individuals considered to be at risk for dental erosion^{9,16}. Coca-Cola was chosen because it is widely consumed by the world population and has high erosive potential due to its low pH^{12,21,22}. The immersion times of specimens in acidic beverages vary widely among studies. Oncag, Tuncer and Tosun²² (2005) and Navarro, et al.²¹ (2011) used longer times of immersion in soft drinks. However, the immersion time used in these studies^{21,22} does not represent the clinical situation, and they did not test the effect of fluoride agents. In general, studies that evaluated the anti-erosive effect of fluoride agents on enamel performed the fluoride application during 4-5 days of dynamic erosive challenge with immersion in acid beverage from 90 s to 5 min^{9,16,19}.

This study found that after fluoride application and mild erosive challenge, the treatments had significant effect on the reduction of VHN values. While all treatment groups had significantly lower reduction in VHN values compared to the control group, they did not differ from each other (Table 1). However, the high correlation between effect size and treatment type (Spearman's correlation results) suggests that the protection against erosive challenge is expected to be the highest with TiF₄ varnish group. This is consistent with other studies, which also showed better results with the TiF₄ varnish compared to the NaF varnish, NaF

solution and TiF₄ solution to protect the enamel against erosion^{16,19}. The fluoride concentration (2.45%) and pH (3.4) of TiF₄ varnish (Figure 1) may have influenced the VHN results, because high concentration and low pH could increase the fluoride uptake by enamel¹³. However, the reduction in enamel erosion by TiF₄ agents is primarily attributed to the precipitation of a metal-rich layer on a tooth surface^{16,19}.

Titanium ions may substitute calcium in the apatite lattice and show a strong tendency to complex with phosphate groups, forming a new compound (hydrated hydrogen titanium phosphate) or organometallic complexes^{4,23}. This reaction forms a glaze-like surface layer that can act as an acid-resistance diffusion barrier^{4,23}. The different surface effects between TiF₄ formulations can be related to the better ability of the varnish to adhere on enamel compared to solution; therefore, the varnish was applied only once whereas the solution was frequent¹⁹. This adherence characteristic of varnish prolongs the reaction between TiF₄ and enamel¹⁹.

A previous study showed that the experimental TiF₄ varnish had better protective effect against erosion of enamel than the experimental NaF varnish with the same fluoride concentration (2.45%)¹⁶. Thus, our study chose to compare the protective effect of TiF₄ varnish with a commercial varnish (Duraphat – NaF varnish 2.26%). However, the higher fluoride concentration of TiF₄ varnish can be influenced in the results. The NaF varnish group was ranked lower than the TiF₄ varnish group. Concerning the Cohen's effect size values of the protection in enamel against erosion, the difference between the TiF₄ varnish and the NaF varnish group was the largest among treatments, being consistent with the aforementioned report. The difference can also be associated with the different protective layers formed on enamel. The calcium fluoride protective layer has been speculated to be less resistant to erosive challenge than the glaze-like layer^{9,16,19,23}.

The lower %VHN of all groups compared to the control group (Figure 2a) showed that after erosion, the application of NaF or TiF₄ agents (varnish and solution) minimized the enamel mineral loss around brackets, but they were not able to arrest mineral loss completely. These results were also demonstrated by PLM images. The control group seemed to have a demineralization depth higher than other groups (Figure 4B), and there was enamel demineralization

after fluoride application, but with no apparent difference among NaF and TiF₄ groups (Figures 4 C, D and 5 A, B).

The values of shear bond strength after erosion (Figure 2b) were higher than the values showed in other studies^{21,22}, most likely due to the use of fluoride agents and the inferior immersion time of specimens in acidic beverage. The ARI results showed that the score of 3 was the most observed for all groups (Figure 3), indicating that the erosive challenge did not impair the bonding of resin to enamel and that the difference found among groups for shear bond strength can be associated to the fluoride agent used in each group. Thus, the TiF₄ varnish group was the only one that showed statistically higher shear bonding strength of brackets to enamel after the erosive challenge compared to all groups (Figure 2b and Table 2). The glaze-like surface layer formed probably protected the demineralization of enamel, as explained before, and was able to prevent the decrease of shear bond strength caused by erosion. Fidalgo, et al.¹¹ (2012) found that NaF fluoride treatments improved the shear bond strength of brackets to enamel after cariogenic challenge, because NaF forms fluoride hydroxyapatite, which is more resistant than hydroxyapatite¹¹. However, our study showed that most likely the reaction of titanium ions with enamel apatite caused more protection from shear forces than the NaF reaction.

Formulations with low concentration of fluoride, as in toothpastes, had minimal or no anti-erosive effect^{13,18}. Although fluoride varnish requires a professional application and the cost-effectiveness can be higher than home care products, the topical fluoride varnish treatments have a surface and a sub-surface effect, which may be relevant in the prevention of dental erosion²⁴. Furthermore, fluoride varnishes are easy to apply, safe and well-tolerated by infants and children¹⁶. The hypotheses tested were partially accepted because the application of NaF and TiF₄ agents (varnish and solution) during erosive challenge minimized the enamel mineral loss around brackets, but only TiF₄ varnish was able to protect the shear bond strength of brackets to enamel. Although this study has been conducted *in vitro*, the experimental TiF₄ varnish seemed to be a promising agent to reduce enamel loss and to improve the retention of brackets to enamel under mild erosive conditions. However, *in vivo* studies should be conducted to verify the efficacy of

TiF₄ varnish in preventing enamel demineralization and retention of brackets to enamel during comprehensive orthodontic treatment of patients with dental erosion diagnosis.

Conclusions

Application of NaF and TiF₄ agents (varnish and solution) during mild erosive challenge minimized the enamel mineral loss around brackets, however only experimental TiF₄ varnish was able to prevent the reduction of shear bond strength of brackets to enamel.

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Conflict of Interest

The authors declare no conflict of interest.

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