



***In vitro* effect of experimental nanocomposites solutions on the prevention of dental caries around orthodontic brackets**

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This study aimed to evaluate the *in vitro* effect of a single application of experimental nanocomposite solutions on the prevention of dental caries around orthodontic brackets. The specimens were exposed to mesoporous silica (MS) nanocomposites containing fluoride by association with titanium tetrafluoride (TiF₄) or sodium fluoride (NaF). Nanocomposites also could contain calcium and groups were described as MSCaTiF₄, MSTiF₄, MSCaNaF, MSNaF, and controls (TiF₄, and NaF). Specimens were subjected to the formation of a multispecies biofilm to generate a cariogenic challenge. After 24h, both pH and total soluble fluoride concentration of the culture medium were assessed. Mineral loss was evaluated by percentage of surface mineral loss (%SML), mineral volume variation (ΔZ) of inner enamel and polarized light microscopy (PL). Linear (Ra) and volumetric (Sa) surface roughness and scanning electronic microscopy (SEM) were used to assess enamel topography. Statistical analyses were conducted considering $p < 0.05$. MSNaF had the highest value of culture medium pH after cariogenic challenge, similarly to MSTiF₄. All nanocomposite solutions released less fluoride than their controls NaF and TiF₄ ($p < 0.05$). All nanocomposite solutions presented lower %SML compared to their respective control groups ($p < 0.05$). Lower Ra, Sa and ΔZ were observed for experimental groups compared to TiF₄ ($p < 0.05$). The results were confirmed by PL and SEM analysis. The experimental nanocomposite solutions contributed for lower enamel demineralization around orthodontic brackets.

Introduction

Patients who wear fixed orthodontic appliances may have problems with dental hygiene because of biofilm accumulation (1). Poor hygiene puts patients at risk for the development of carious lesions, and products aimed at reducing demineralization are therefore recommended (2). Decalcification of the enamel surface adjacent to orthodontic brackets, observed in the form of white spot lesions, is frequent well-known side-effect of orthodontic treatment. Fixed orthodontic appliances and the bonding materials increase the biofilm retention and enhance the formation of white spot lesions (1,2). Orthodontic patients at high risk of dental caries should be instructed for appropriate and frequent oral hygiene regime with the use of fluoride-based agents, including the complementary use of mouthwashes to enhance remineralization (3). Therefore, fluoride- and calcium-based products, either in association or alone, have been used to minimize enamel demineralization (4).

In the available literature, nanoparticulated products have caught the attention of researchers, since they can carry a larger number of elements compared to the surface area, modulate availability, have specific action, and improve the stability of the chemical structure (5). Among these composites, mesoporous silica (MS) deserves special attention as its specific surface and porosity properties allow controlling drug delivery and improving drug release kinetics (6).

In general medicine, the association of MS with calcium improved the loading capacity of alendronate particles, efficiently inhibiting the growth of HeLa carcinogenic cells when compared to free alendronate (7). In dentistry, studies have used this nanoparticle in a new method to encapsulate and release CHX from dental composite using MS (8,9), also using CHX-enriched nanoparticles

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against oral biofilms (10). Bioactive glass-coated MS was also used to investigate their effects on dentinal tubule occlusion (11,12), remineralization (13) and against dental erosion (14,15).

However, to the authors' knowledge, no studies have evaluated the incorporation of fluoride on MS-based nanocomposites, with or without calcium, for the prevention of enamel demineralization. Bearing in mind the properties of MS-based nanocomposites (6-15) described above, the association with fluoride and possible incorporation of calcium would contribute towards the prevention/reduction of dental caries lesions. Therefore, this study evaluated *in vitro* the preventive effect of a single application of experimental MS-based nanocomposite solutions containing fluoride, with and without calcium, on the demineralization of enamel surface around orthodontic brackets.

Materials and Methods

Study Design

Given the paucity of previous studies on the topic, this randomized, controlled, single-blind, *in vitro* study took into account a sample size (n=13 per group) based on the mineral loss of compounds containing calcium and fluoride (16,17). Therefore, a 0.8 statistical power was used to detect a significant difference of 50% in average mineral loss in each treatment group when compared to the control group (1.36% of NaF), running a one-tailed test at a 5% significance level (BioEstat 5.3[®], Instituto de Desenvolvimento Sustentável Mamirauá, Tefé, Brazil).

Preparation of the Experimental Nanocomposites

MS-based nanocomposites were obtained by nanoprecipitation, altering their molar ratio for lyophilization and physicochemical characterization (6). Thereafter, fluoridated titanium tetrafluoride (1% TiF₄, 6135 ppm of F⁻) and sodium fluoride (1.36% NaF, 6135 ppm of F⁻) solutions (Aldrich Chemical Co[®], Saint Louis, USA) were included, to which calcium (Ca) was added or not. The fluoride concentration within groups was primarily defined according to the TiF₄ group, since this substance is more difficult to synthesize. Therefore, all other solutions were manipulated in order to present similar fluoride concentration to that observed in 1% TiF₄ usually used in clinical practice. The following experimental nanocomposites were produced: MSCaTiF₄, MSTiF₄, MSCaNaF and MSNaF.

For characterization analyses, the MS-only and MSCa controls were considered. The characterization of these nanocomposites indicated that the MS-only had a mean diameter of 183.18 ± 1.5 nm and a polydispersion index (PdI) of 0.240 ± 0.021, using the scattering technique (DLS) and zeta potential of - 29.66 ± 0.18 mV (Table 1). In addition, the MS nanoparticle formed were spheroid, according to transmission electron microscopy (TEM) (Figure 1). Infrared spectrum of absorption or emission of nanoparticles was obtained through Fourier-Transform Infrared Spectroscopy (FTIR) and X-ray diffraction pattern of mesoporous silica nanoparticles was evaluated (Figure 2 and 3).

Table 1: Characterization of mesoporous silica particles.

Groups	Fluoride theorists* (%)	Calcium concentration (%)	Yield (%)	Z-average (d. nm)	Polydispersity Index (PdI)	pH	Zeta potential (mV)
MSCaTiF ₄	13,09%	11,10 ± 0,08	55,03 ± 0,05	185,7 ± 1,5	0,235 ± 0,004	6,45 ± 0,62	- 32,28 ± 0,41
MSTiF ₄	6,71%	-	47,22 ± 0,03	176,6 ± 1,3	0,261 ± 0,007	6,38 ± 0,03	- 30,21 ± 0,33
MSCaNaF	16,37%	11,32 ± 0,12	58,21 ± 0,05	188,3 ± 1,7	0,242 ± 0,005	6,51 ± 0,37	- 29,71 ± 0,11
MSNaF	6,57%	-	48,63 ± 0,05	178,2 ± 1,4	0,223 ± 0,005	6,82 ± 0,51	- 28,16 ± 0,28
MSCa	-	5,60 ± 0,08	43,03 ± 0,04	187,1 ± 1,8	0,241 ± 0,008	6,36 ± 0,34	- 27,96 ± 0,31
MS-Only	-	-	-	187,8 ± 4,3	0,250 ± 0,007	6,11 ± 0,13	- 26,47 ± 0,22

* means predicted theoretical values of fluoride theorists for nanocomposites considering the atoms present individually. Value calculated based on the mass of each of the reagents used in the preparation of MS.

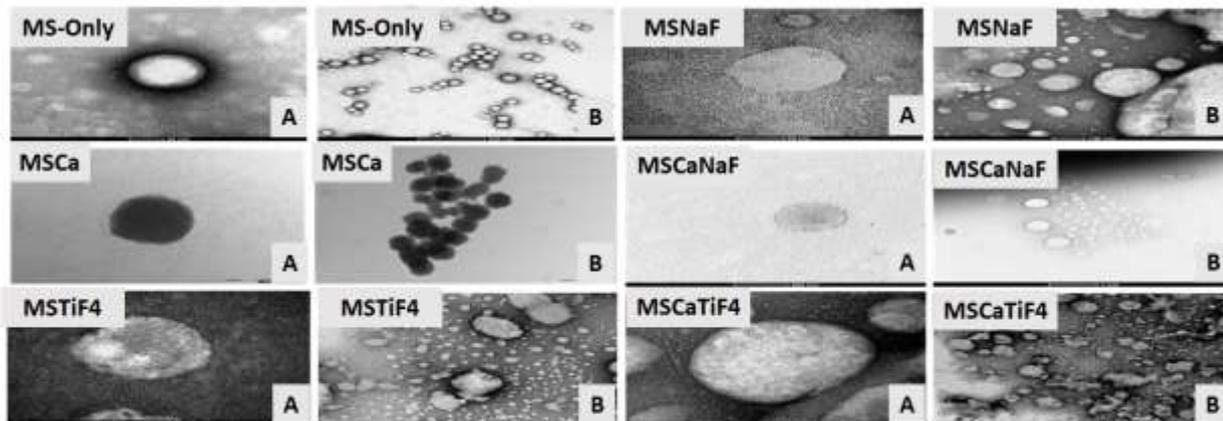


Figure 1: Photomicrographs obtained by TEM analysis of mesoporous silica nanoparticles.

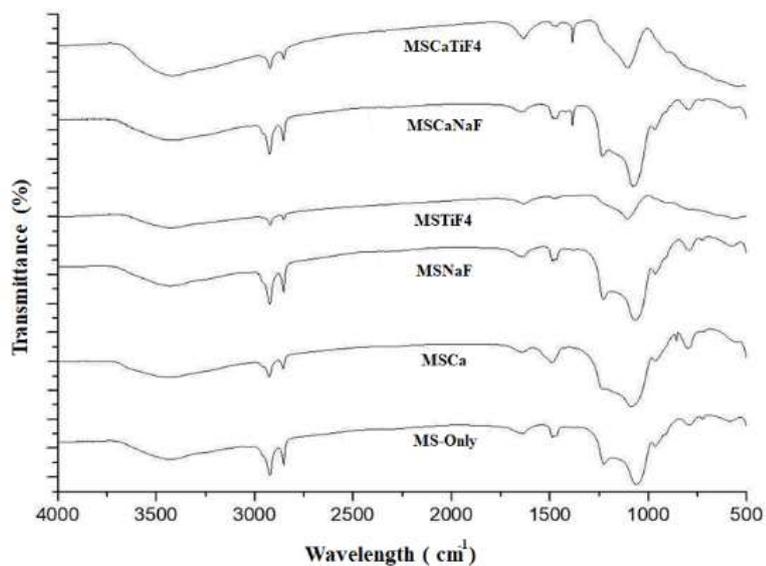


Figure 2: FTIR spectrum of mesoporous silica nanoparticles.

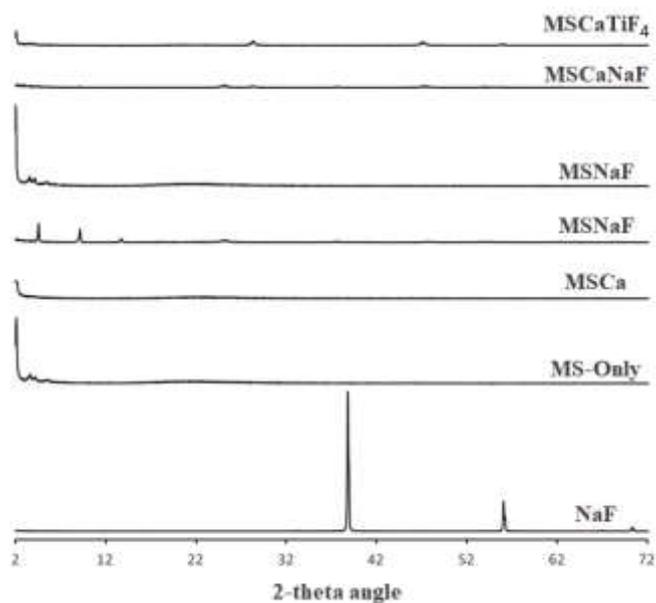


Figure 3: X-ray diffraction pattern of mesoporous silica nanoparticles.

Specimen Preparation

Bovine enamel blocks (8 x 8 x 2.5 mm) were obtained from sound bovine incisors and specimens were planned according to previously reported in the literature (18). After that, a window measuring 19.63 mm², whose surrounding area was protected with an acid-resistant varnish layer (Risque[®], São Paulo, Brazil), was exposed. Surface microhardness of the enamel was assessed by a Knoop diamond indenter using a load of 50 g for 5 s, with three equidistant indentations (100 μm) inside the delimited area, close to the margin. Enamel blocks in the ± 10% range were selected according to the total mean of the baseline microhardness.

A single trained operator used the Transbond Plus Self Etching Primer (3M Unitek[®], Monrovia, USA) and Transbond XT (3M ESPE[®], Saint Paul, USA) for bonding the metal orthodontic bracket (3.20 x 2.70 mm) (Edgewise Standard .022", Morelli, Sorocaba, Brazil) to the central area of the window. An orthodontic elastic (intermaxillary elastics 5/16", Morelli, Sorocaba, Brazil) band was placed around the bracket to mimic the clinical setting.

After random distribution of the specimens (Microsoft Excel[®]) into each of the groups (n=13), the specimens were transferred to a 12-well polystyrene plate (model K12-024, Kasvi[®], São José do Pinhais, Brazil), and then sterilized under ultraviolet light for 1h with 40 Watts power (19). The groups were as follows: MSCaTiF₄, MSTiF₄, TiF₄ (fluoridated control), MSCaNaF, MSNaF, NaF (fluoridated control), growth control (GC) and sterility control (SC).

A single blinded researcher actively applied the test products (100 μL) for 1 minute in the intervention area around the orthodontic brackets (11 mm²) using a microbrush (KG Sorensen[®], Cotia, Brazil). After application, specimens were dried at room temperature.

Cariogenic Challenge

After reactivation of *Streptococcus mutans* (ATCC 25175), *S. salivarius* (ATCC 7073), *S. sanguinis* (ATCC 20556), and *Lactobacillus casei* (ATCC 393) strains, a bacterial suspension was standardized according to CLSI standards (20). The inoculum consisted of the multispecies biofilm was standardized at a concentration of 1 × 10⁷ CFU/mL (0.1 absorbance under 625 nm wavelength).

Prior to biofilm formation, the specimens were submitted to the formation of artificial salivary pellicle (21), with 1% carboxymethyl, 0.0084% sodium chloride, 0.12% potassium chloride, 0.0342% phosphate monobasic potassium, 0.0146% calcium chloride, and 0.0052% magnesium chloride. Specimens were pre-conditioned with artificial saliva for 2 h, at 37°C, in 24-well polystyrene culture plates (model K12-024, KasviTM, São José do Pinhais, BRA). After removal of the saliva, 5 mL of the mixed inoculum (5 × 10⁵ CFU/mL of the final concentration) with BHI broth containing 2% sucrose (pH=7.10) was added and the specimens were incubated at 37 °C for 24 h.

GC (growth control) specimens contained bacterial suspension (multispecies biofilm of *Streptococcus* spp. and *Lactobacillus casei*) prepared in BHI broth containing 2% sucrose. SC (sterility control) specimens were incubated in BHI broth with 2% sucrose, without any microorganism. GC and SC groups did not receive treatment with any of the experimental solutions, nor respective controls (NaF and TiF₄).

Data Collection and Analysis

Analysis of pH and Total Soluble Fluoride in Culture Medium

After 24 h, the specimens were sonicated for 1 min using an ultrasonic cleaner to remove any remaining residues and then the brackets were debonded manually. Acidogenicity of the culture medium was assessed by measuring the pH of the culture medium using an electrode connected to a pH meter (PHOX[®], Colombo, Brazil). The procedure was carried out in duplicate by a blinded trained examiner.

The total soluble fluoride (TSF) concentration in the culture medium was determined using a fluoride ion selective electrode (Orion 9609) coupled to a potentiometer (Orion Star Series, Termo Fisher Scientific[®], Waltham, USA). Aliquots (2 mL) from culture medium were read at a 1:1 (v/v) ratio using a TISAB II solution after obtaining low-concentration (0.06 to 1 μg F⁻ / mL [r₂ = 0.994; slope = -58.9]) and high-concentration (1 to 32 μg F⁻ / mL [r₂ = 0.999; slope = -59.6]) standard curves.

Mineral Loss Analysis

All enamel blocks of each group were reassessed after cariogenic challenge by the same blinded and trained examiner to determine the final surface microhardness, in order to obtain the percentage of surface mineral loss (%SML) after the experiment (23). The %SML was calculated using the specimens' surface microhardness (SM, expressed in kg/mm²), measured before product application and after experiments. The %SML was obtained by the following formula:

$$\%SML = 100 \times \frac{(SM_{baseline} - SM_{final})}{SM_{baseline}}$$

For a volumetric analysis of mineral content in the lesion, randomly selected enamel blocks (n= 6, per group) were scanned on a high-energy micro-CT scanner (Skyscan 1173, Bruker, Kontich, Belgium) using the following acquisition parameters: 70 kVp, 114 mA, pixel size of 7.12 µm, and 1-mm Al filter. The images were later reconstructed into cross-sections using a proprietary software (NRecon, Bruker) and specific reconstruction parameters. Mineral loss was then determined by the integrated mineral loss analysis (ΔZ) in volumes of interest (VOI) selected from each specimen. After averaging gray values in each VOI, a linear profile was drawn across the enamel surface and the integrated area under the curve corresponding to the carious lesion was calculated. This represents the net mineral loss of the lesion (24).

A qualitative analysis of demineralization was performed using Polarized Light Microscopy. Randomly selected enamel blocks (n=2, per group) were sectioned longitudinally with a diamond disc (Isomet, model 11-1280-170, Lake Bluff®, Illinois, USA). Slices of approximately 300 µm were cut and then laterally worn to approximately 100 µm, using aluminum oxide sandpaper at granulation #600. After that, samples were stored in microtubes with 0.02% sodium azide solution for 72 h at 4 °C in order to keep them hydrated and to avoid the growth of fungi and bacteria for analysis of subsurface mineral loss under a polarized light microscope (Axioskop 40, Carl Zeiss®, Oberkochen, Germany) equipped with a 10x objective lens and 550-nm phase contrast filter (red filter I). The images were photographed with a digital camera (44 D7000, Nikon®, Tokyo, Japan) under the same lighting conditions. The distilled and deionized water was the environment used to analyze the change in enamel refraction, as indicative of the demineralization occurring on the surface of the block. Dental demineralization was observed as a dark area/band on the cross-sectional surface of specimen.

Surface Topography Analysis

Surface topography of the enamel in the regions around the bracket debonding area of all specimens (n=13 per group) was assessed by 3D non-contact profilometry (Nanovea PS50 Optical, NANOVEA®, Irvine, USA). A standardized 1-mm² assessment area was established on the enamel specimen. The mean value from three linear roughness (Ra) (500 µm) measurements and three volumetric roughness (Sa) (250 µm²) measurements were obtained for each specimen.

A qualitative analysis of surface topography was performed using Scanning Electron Microscopy (SEM). Specimens (n=2 per group) were gold-sputtered prior to SEM visualization. SEM (Fei Quanta 250®, Termo Fisher Scientific®, Waltham, USA) was operated at 20 kV. The area between the resin remnant after bracket debonding and the sound enamel surface was examined. Photomicrographs were obtained at 5.000× magnification. Both profilometry and SEM evaluations were carried out by a single blinded examiner.

Statistical Analysis

The data were statistically analyzed by SPSS version 20.0 (IBM®, Chicago, USA) using a 5% significance level. The data distribution was evaluated by the Shapiro-Wilk test. The Kruskal-Wallis and Mann-Whitney tests were used for the analyses of pH, TSF, %SML, profilometry parameters, and ΔZ values.

Results

Treatment with MSNaF resulted in the highest value of culture medium pH after the cariogenic challenge and was similar to MSTiF₄. As for the TSF of the culture medium the experimental nanocomposites released less fluoride than their controls NaF and TiF₄ (p<0,05) (Table 2).

Table 2: Ph, total soluble fluoride, microhardness, mineral loss analysis (ΔZ) and profilometry analysis (Ra and Sa).

Groups	pH	TSF ($\mu\text{g F-}/\text{mL}$)	%SML (kgf/mm^2)	ΔZ (8-bit gray values)	Ra (μm)	Sa (μm)
MSCaTiF ₄	5.14 \pm 0.60 ^A	3.52 \pm 1.68 ^A	15.02 \pm 5.93 ^A	42.72 \pm 18.15 ^A	0.43 \pm 0.11 ^A	0.57 \pm 0.17 ^A
MSTiF ₄	6.72 \pm 0.37 ^B	1.39 \pm 0.60 ^B	12.47 \pm 3.79 ^A	40.35 \pm 8.62 ^A	0.36 \pm 0.12 ^A	0.46 \pm 0.21 ^A
TiF ₄	4.61 \pm 0.10 ^C	19.76 \pm 9.88 ^C	31.41 \pm 7.77 ^B	58.15 \pm 36.97 ^B	0.58 \pm 0.11 ^B	0.86 \pm 0.71 ^{AB}
MSCaNaF	5.79 \pm 0.71 ^{AB}	3.25 \pm 0.48 ^A	11.85 \pm 7.00 ^A	40.35 \pm 11.63 ^A	0.39 \pm 0.19 ^A	0.55 \pm 0.12 ^A
MSNaF	6.86 \pm 0.69 ^B	1.16 \pm 0.31 ^B	14.61 \pm 8.12 ^A	41.53 \pm 14.53 ^A	0.40 \pm 0.11 ^A	0.58 \pm 0.17 ^A
NaF	4.57 \pm 0.09 ^C	10.18 \pm 4.34 ^D	28.01 \pm 8.00 ^B	45.09 \pm 11.63 ^A	0.45 \pm 0.16 ^{AB}	0.62 \pm 0.26 ^A
GC	4.11 \pm 0.04 ^D	0.15 \pm 0.02 ^E	64.49 \pm 9.09 ^C	199.36 \pm 75.22 ^C	1.02 \pm 0.16 ^C	1.76 \pm 0.62 ^B
SC	7.09 \pm 0.09 ^E	0.16 \pm 0.03 ^E	3.61 \pm 4.81 ^D	26.11 \pm 5.81 ^A	0.29 \pm 0.11 ^A	0.34 \pm 0.14 ^A

Ra: linear roughness; Sa: volumetric roughness; TSF: total soluble fluoride; Different uppercase letters in the same column indicate statistical difference (p<0.05).

In addition, all experimental nanocomposites were better than NaF and TiF₄ in preventing surface %SML, (p<0.05) (Table 2). Micro-CT analyses revealed that with exception of MSCa, all experimental nanocomposites were better than TiF₄ (p<0.05) and similar to NaF (p>0.05) in preventing mineral loss within the lesion (ΔZ) (Table 2 and Figure 4). These data were confirmed by polarized light microscopy (Figure 5), in which different degrees of enamel demineralization were detected in all treatment groups. Photomicrographs observed in Figure 5 show that groups treated with mesoporous-silica nanocomposite in association with fluoride had lower demineralization, similarly as observed in groups treated with NaF, TiF₄ and sterility control. Greater demineralization was observed in growth control, similarly as observed in the group treated with mesoporous-silica without fluoride.

With respect to Ra and Sa, the nanocomposites exhibited favorable and similar behavior between them, being similar to the NaF group (p>0.05) for Ra and Sa (p>0.05), and to the TiF₄ group only for Sa (p>0.05) (Table 2). Surface topography findings can be corroborated by SEM images (Figure 6).

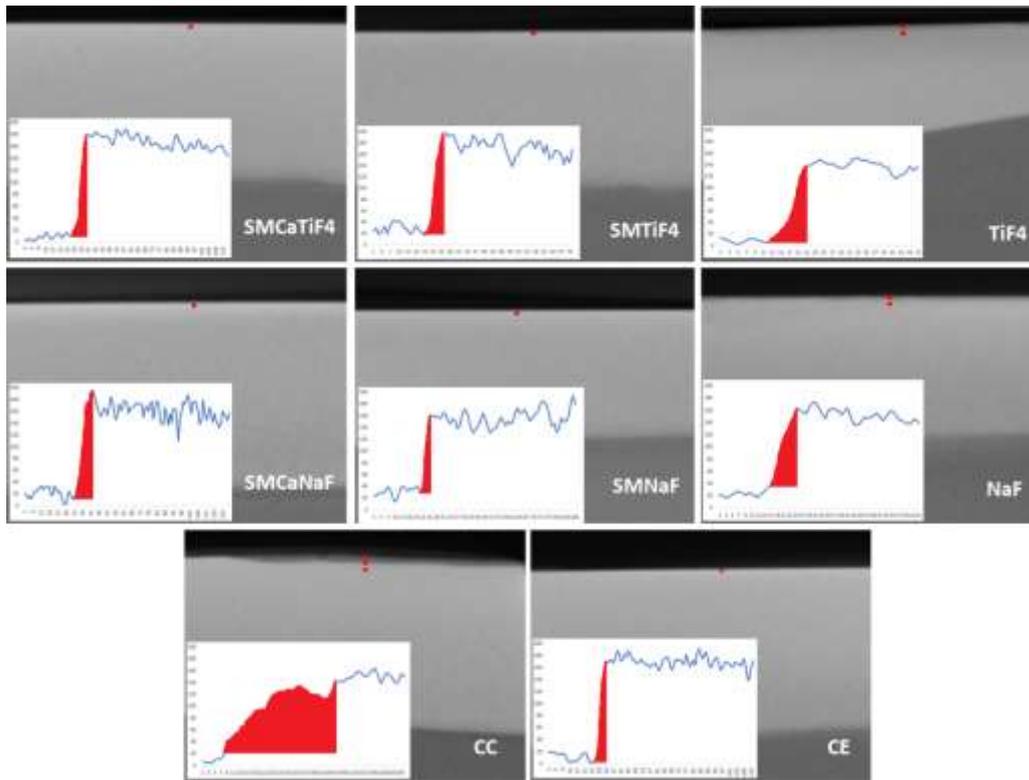


Figure 4: Photomicrograph of enamel surface assessed by micro-CT. The region in red indicates the area and depth of the carious lesion, from where ΔZ values were obtained. The unit of measurement on x axis corresponds to 8-bit gray value, whilst the unit of measurement on y axis corresponds to enamel depth (pixel).

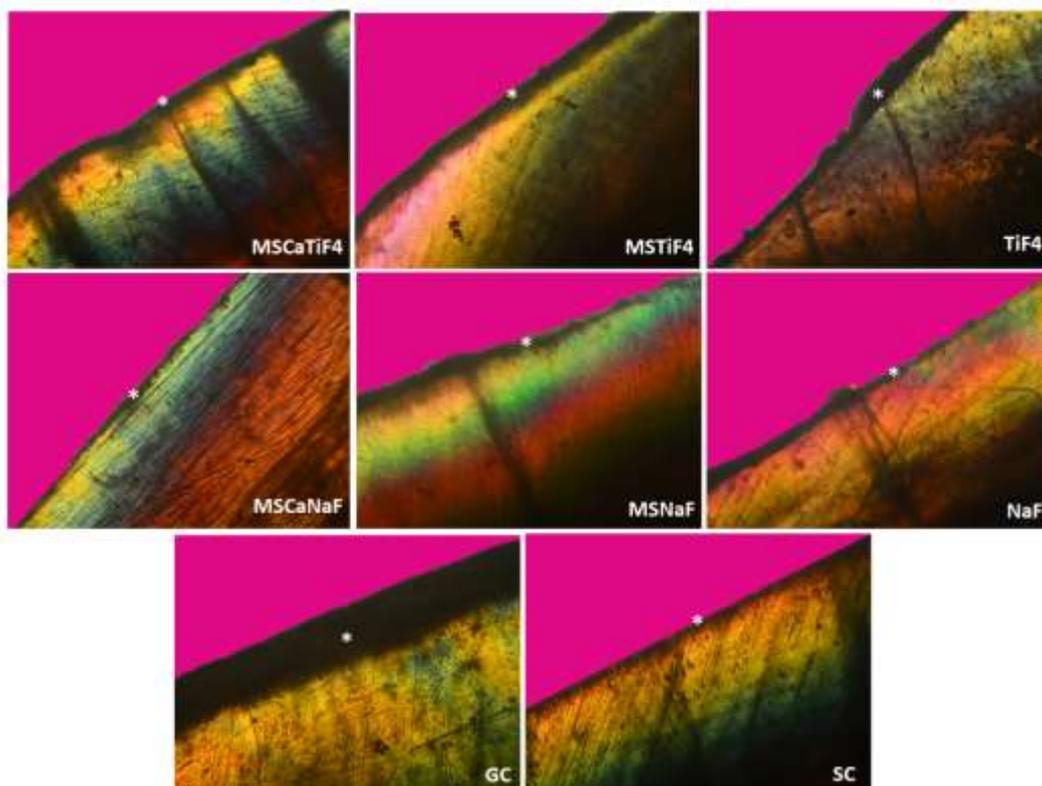


Figure 5: Photomicrograph of enamel surface assessed by polarized light. Demineralization is denoted by the dark area marked with an asterisk (*).

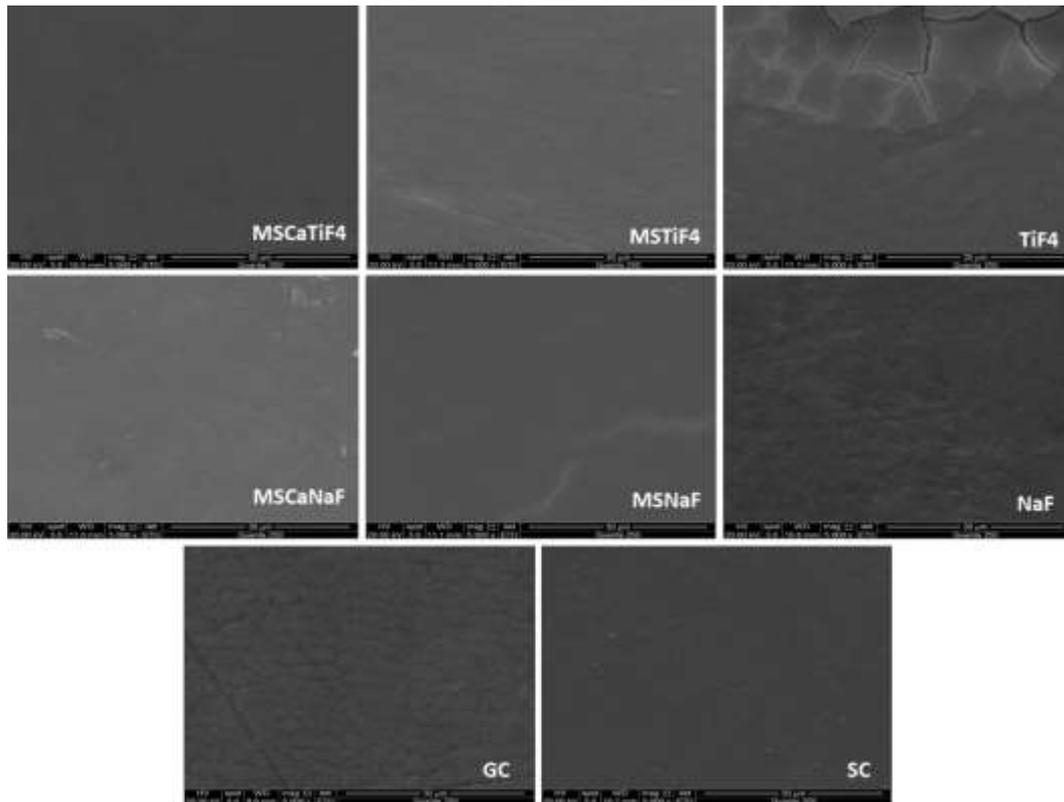


Figure 6: Surface of enamel blocks after treatment assessed by scanning electron microscopy at 5.000x

Discussion

The present study investigated the effects of new fluoridated products on inhibition of dental enamel demineralization around orthodontic brackets, analyzing the loading capacity of different sources/types of calcium fluorides using MS nanoparticles. There has been no report in the literature on the association of this nanoparticle with fluoridated products as those proposed here, even though the benefits of calcium-doped MS for the production of new materials with different applications have been confirmed, as this particle has mechanical resistance, permeability, thermal stability, and chemical stability (6,7).

The short period of biofilm formation, the use of bovine dental enamel slabs, and constant exposition to sucrose during biofilm development should be acknowledged as relevant aspects that differ from real clinical situation. In order to overcome some limitations of this *in vitro* assay, the retention of multispecies biofilm was simulated with the use of artificial saliva and orthodontic bracket.

Nevertheless, this study demonstrated that the *in vitro* multispecies biofilm simulation model was sufficient to reduce pH to levels below the hydroxyapatite critical level within 24 h. As evidenced by %SML, micro-CT profile and polarized light images, this cariogenic challenge model resulted in a surface mineral loss. It was noticed that only the preventive application of fluoridated nanocomposites was able to maintain the pH above the critical level of enamel demineralization, allowing for ionic balance and favoring remineralization. Calcium-doped MS has a high rate of drug release directly proportional to the acidity of the medium (7). According to Ribeiro et al. (6) MS particles are more stable at pH levels greater than 5, explaining the findings obtained in this study. Moreover, the remineralization conditions could not be developed only in the presence of calcium, because of the effect of the cariogenic challenge associated with the previous application of the test product. This fact justifies the need of the presence of fluoride to optimize the ionic changes in the interface tooth biofilm leading the prevention of surface and subsurface mineral loss.

In this experimental model, as the test products were applied before the cariogenic challenge, the nanocomposites containing calcium and fluoride probably lost their chemical stability, which could have influenced the ionic behavior of fluoride. The presence of an acidic microenvironment, products

with this association tend to produce better outcomes, thus being indicated for therapeutic use rather than for preventive use as proposed in this study.

Regarding the results of total soluble fluoride, all nanocomposites had better outcomes than conventional TiF_4 and/or NaF solutions, where these solutions that released more fluoride into the medium showed poorer protection of the enamel. The nanocomposites may be explained by the high concentration of fluoride adhered to the enamel, as it may reduce demineralization, stopping the release of calcium and phosphate from the enamel into the medium (25).

The development of white spot lesions as consequence of successive dental demineralization exposure during orthodontic treatment may compromise treatment outcomes (1). To minimize that, the complementary use of TiF_4 or NaF solutions may help to maintain oral health, but it is necessary that they are used regularly (2), because of the action of saliva and swallowing will only allow fluoride for only a short period of time. The solutions used in the present study were able to reduce demineralization in a single preventive application, despite differences between the inclusion of nanoparticles with TiF_4 and with NaF associated or not with calcium. On the other hand, when compared to conventional products, nanocomposites reduced demineralization of surface and subsurface lesions adjacent to orthodontic brackets after exposure to the biofilm. TiF_4 and NaF nanocomposites showed the best outcomes, and since these mesoporous silica products can release this composite gradually, the presence of residual fluoride can act as a protective factor against dental demineralization.

The enamel surface of areas around orthodontic brackets is often rough, because biofilm removal in these areas is difficult (3). To minimize such damage, the experimental nanocomposites evaluated herein could be recommended, as they resulted in less rough enamel surfaces (lower Ra and Sa), however further *in situ* and *in vivo* studies must be conducted to confirm these results.

Dental enamel demineralization analysis by %SHL and Micro-CT showed that all experimental groups minimized the demineralization process, mainly those treated with MS associated with fluoride. The polarized light microscopy revealed that groups treated with fluoride-enriched nanocomposites exhibited lower demineralized area on the enamel surface, similarly to that observed in NaF, TiF_4 and sterility control groups. Further analyses should consider differences regarding demineralization depth of groups treated with different nanocomposite solutions and their respective controls.

Moreover, as pointed out by SEM, changes on the enamel surface were more evident in the groups MSCa and growth control. On the other hand, enamel integrity was preserved by all of the assessed nanocomposites, and these findings support the therapeutic use of these solutions as complement to caries treatment, (2,3) especially around orthodontic brackets.

Considering both %SML and micro-CT analyses, nanocomposites reduced the demineralizing capacity when compared to conventional TiF_4 and NaF solutions, and these positive outcomes indicate that these test products seem to be efficient in inhibiting the demineralizing potential of multispecies biofilms, since the inclusion of these composites can produce a synergistic effect, arresting caries progression. Therefore, these test products may be recommended, providing patients on orthodontic treatment with a lower risk of dental caries.

The nanocomposites used in this study could be deemed appropriate for the prevention of dental caries, but, ideally, it would be necessary to verify the mineral content of the enamel after exposure to this test product to assess the presence of calcium and fluoride deposits by future studies. Nevertheless, given the innovative nature of this study, it is also necessary to assess the dose-response effect of these test products in order to elucidate their therapeutic effect, as the association of calcium-doped MS with fluorides had a preventive effect in a single application, which might have extended the release of these composites. Even though cytotoxicity assays were not conducted in the present study, there are no indicative signs in the literature that calcium-doped MS would cause changes in cell integrity (7), which turns this gradual release system into an efficient mechanism for the delivery of drugs or composites.

Conclusion

A single application of the experimental nanocomposites was able to improve protection against dental enamel demineralization around orthodontic brackets. In general, the association of calcium-doped MS with fluorides had a preventive effect for parameters analyzed there are no differences between these solutions.

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RESUMO

Este estudo teve como objetivo avaliar o efeito in vitro de uma única aplicação de soluções experimentais de nanocompósitos na prevenção de cárie dentária em braquetes ortodônticos. Os espécimes foram expostos a nanocompósitos de sílica mesoporosa (MS) contendo fluoreto por associação com tetrafluoreto de titânio (TiF₄) ou fluoreto de sódio (NaF). Os nanocompósitos também podem conter cálcio e os grupos foram descritos como MSCaTiF₄, MSTiF₄, MSCaNaF, MSNaF e controles (TiF₄ e NaF). Os espécimes foram submetidos à formação de um biofilme multiespécie para gerar um desafio cariogênico. Após 24h, o pH e a concentração de flúor solúvel total do meio de cultura foram avaliados. A perda mineral foi avaliada pela porcentagem de perda mineral superficial (% SML), variação do volume mineral (ΔZ) do esmalte interno e microscopia de luz polarizada (PL). A rugosidade superficial linear (Ra) e volumétrica (Sa) e a microscopia eletrônica de varredura (MEV) foram utilizadas para avaliar a topografia do esmalte. As análises estatísticas foram realizadas considerando $p < 0,05$. MSNaF apresentou o maior valor de pH do meio de cultura após o desafio cariogênico, semelhante ao MSTiF₄. Todas as soluções de nanocompósitos liberaram menos flúor do que seus controles NaF e TiF₄ ($p < 0,05$). Todas as soluções de nanocompósitos apresentaram % SML menor em comparação com seus respectivos grupos de controle ($p < 0,05$). Ra, Sa e ΔZ menores foram observados para os grupos experimentais em comparação ao TiF₄ ($p < 0,05$). Os resultados foram confirmados por análises PL e SEM. As soluções experimentais de nanocompósitos contribuíram para a menor desmineralização do esmalte ao redor dos braquetes ortodônticos.

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