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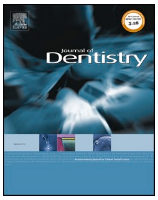

Experimental toothpastes containing β-TCP nanoparticles functionalized with fluoride and tin to prevent Erosive Tooth Wear

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ABSTRACT

Objectives: The present study aimed to synthesize toothpastes containing Beta- TriCalcium Phosphate (β-TCP) nanoparticles, functionalized with fluoride and tin, and test their ability to reduce erosive tooth wear (ETW).

Methods: Toothpastes were synthesized with the following active ingredients: 1100 ppm of fluoride (as sodium fluoride, F^-), 3500 ppm of tin (as stannous chloride, Sn^{2+}), and 800 ppm of β-TCP (Sizes a – 20 nm; and b – 100 nm). Enamel specimens were randomly assigned into the following groups ($n = 10$): 1. Commercial toothpaste; 2. Placebo; 3 F^-; 4. $F^- +$ β-TCP_a; 5. $F^- +$ β-TCP_b; 6. $F^- + Sn^{2+}$; 7. $F^- + Sn^{2+} +$ β-TCP_a and 8. $F^- + Sn^{2+} +$ β-TCP_b. Specimens were subjected to erosion-abrasion cycling. Surface loss (in μm) was measured by optical profilometry. Toothpastes pH and available F^- were also assessed.

Results: Brushing with placebo toothpaste resulted in higher surface loss than brushing with F^- ($p = 0.005$) and $F^- +$ β-TCP_b ($p = 0.007$); however, there was no difference between F^- and $F^- +$ β-TCP_b ($p = 1.00$). Commercial toothpaste showed no difference from Placebo ($p = 0.279$). The groups F^-, $F^- +$ β-TCP_a, $F^- +$ β-TCP_b, $F^- + Sn^{2+}$, $F^- + Sn^{2+} +$ β-TCP_a and $F^- + Sn^{2+} +$ β-TCP_b were not different from the commercial toothpaste ($p > 0.05$). Overall, the addition of β-TCP reduced the amount of available fluoride in the experimental toothpastes. The pH of toothpastes ranged from 4.97 to 6.49.

Conclusions: Although toothpaste containing β-TCP nanoparticles protected enamel against dental erosion-abrasion, this effect was not superior to the standard fluoride toothpaste (commercial). In addition, the functionalization of β-TCP nanoparticles with fluoride and tin did not enhance their protective effect.

Clinical Significance: Although β-TCP nanoparticles have some potential to control Erosive Tooth Wear, their incorporation into an experimental toothpaste appears to have a protective effect that is similar to a commercial fluoride toothpaste.

1. Introduction

Dental hard tissues can be damaged as a consequence of tooth decay, trauma, and erosive tooth wear (ETW). ETW has become a common condition worldwide, with high prevalence among children and young adults [1]. This could be attributed to changes in lifestyle, with an increase in the consumption of acidic foodstuff. ETW is characterized by dissolution of the tooth surfaces promoted by erosive acids [2]. These acids are classified as having either an extrinsic (from the diet: consumption of citric acid-rich fruit drinks) [3] or an intrinsic origin (disorders, such as gastroesophageal reflux) [4]. The interaction of these acids with the tooth surfaces results in a softening of enamel, its most superficial layer. Eroded enamel becomes more susceptible to wear by mechanical forces, such as the ones resulting from tooth brushing [2].

The use of monovalent fluorides, such as sodium fluoride salts (NaF) reduces ETW, due to the formation of a calcium-fluoride-like (CaF_2-like)

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layer on the tooth surfaces, which acts as a mechanical barrier or a first layer of dissolution until the acid reaches the mineral underneath [5]. However, the magnitude of protection from this approach is limited, as these deposits are removed under highly erosive environments [6]. A higher degree of protection is observed with the use of polyvalent metal fluoride compounds, such as stannous fluoride, due to their ability to incorporate into the tooth structure reducing dissolution or by forming more acid-resistant metal-rich precipitates [7]. Nonetheless, this effect is also temporary [8], and currently, several studies have shown that the regular use of tin can promote allergic reactions in the oral mucosa [9,10] and tooth staining [11], highlighting that alternative strategies to prevent ETW are still needed.

With the advent of nanomedicine, dental science has been improved with the use of nanoparticles for several purposes, such as the prevention and treatment of oral diseases, restorative and repair procedures (increasing adhesive strength), and diagnosis [12,13]. For clinical conditions that require mineral deposition on tooth surfaces, the use of mineral systems in nanometric dimensions appears to be advantageous, as a higher and easier integration with the tooth structure can occur [14]. Additionally, these nanometric mineral systems can be functionalized with other elements, such as fluoride and tin, thus enhancing their ability to promote surface protection [15].

Recently, Beta TriCalcium Phosphate (β-TCP) compounds were synthesized in nanometric dimensions and tested under erosive-abrasive conditions, showing promising results when offered in solutions [15] or restorative materials [16]. β-TCP is a system that simultaneously provides calcium and phosphate ions, and has biocompatibility, biodegradability, and osteoinductivity properties [17]. Additionally, it has solubility close to that of hydroxyapatite [18]. Hydroxyapatite is the principal constituent of the teeth inorganic matrix, forming crystallites with nanorod-like shape [19]. For all these reasons, the use of β-TCP in dental applications has increased.

Among the different vehicles available to offer β-TCP nanoparticles to the oral cavity, toothpastes seem more relevant as tooth brushing is the most common oral hygiene habit worldwide. In addition, the vast majority of toothpastes do not require professional prescription. Therefore, new toothpastes are constantly synthesized and/or reformulated, aiming to offer multipurpose active ingredients that can contribute to maintaining oral health. Currently, the use of nanoparticles in toothpastes for tooth protection and remineralization is encouraged [20].

Hence, the present study aimed to synthesize toothpastes with β-TCP nanoparticles, functionalized with fluoride and tin, to measure the amount of fluoride available in these experimental formulations, and to test their ability to reduce enamel wear under erosive and abrasive challenges. The null hypotheses tested were that the experimental toothpastes with β-TCP nanoparticles would not differ in (1) reducing enamel surface loss from the placebo toothpaste and (2) in the amount of potentially available fluoride from the commercial toothpaste.

2. Materials and methods

2.1. Study design

The present study had two experimental phases. The first phase aimed to synthesize the experimental toothpastes and measure the potential available fluoride in these formulations. The second phase aimed to test the ability of the toothpastes to reduce enamel surface loss under erosive-abrasive challenges. The study had one experimental factor: toothpastes, at eight levels (see Table 1). β-TCP nanoparticles were tested in two different sizes: a) smaller (5–20 nm) and b) larger (agglomerates of nanoparticles formed during the synthesis - around 100 nm), as shown in Fig. 1. The response variables were the amount of potentially available fluoride (in μg/ml) and surface loss (in μm).

PHASE 1 – SYNTHESIS AND CHARACTERIZATION

Table 1
Experimental groups.

Code	Group
Commercial Toothpaste	Commercial toothpaste "Gengiva Detox" stannous fluoride (1100 ppm of fluoride); Oral-B® Procter & Gamble - LOT: L2268GR
Negative Control	Placebo toothpaste (without active ingredients)
F^-	1100 ppm F^- as NaF
F^- + β-TCP$_a$	1100 ppm F^- as NaF + 800 ppm β-TCP$_a$
F^- + β-TCP$_b$	1100 ppm F^- as NaF + 800 ppm β-TCP$_b$
F^- + Sn^{2+}	1100 ppm F^- as NaF + 3500 ppm Sn^{2+} as $SnCl_2$
F^- + Sn^{2+} + β-TCP$_a$	1100 ppm F^- as NaF + 3500 ppm Sn^{2+} as $SnCl_2$ + 800 ppm β-TCP$_a$
F^- + Sn^{2+} + β-TCP$_b$	1100 ppm F^- as NaF + 3500 ppm Sn^{2+} as $SnCl_2$ + 800 ppm β-TCP$_b$

2.2. Synthesis of the toothpastes

Toothpastes were formulated by mixing 1.5% sodium lauryl sulfate (Sigma-Aldrich Co, CAS 151–21–3), 0.8% xanthan gum (Labsynth, CAS 1138–66–2), 20% glycery (Labsynth, CAS 56–81–5), 10% sorbitol (InLab®, CAS 50–70–4), 0.15% soluble saccharin (InLab®, CAS 82,385–42–01), 1% titanium dioxide (Sigma-Aldrich Co, CAS: 1317–70–0), 0.1% methylparaben (Nipagin, C84803), 6% silica (Sigma-Aldrich Co, CAS 112,945–52–5) and 100% distilled water [21]. Toothpastes were stored for 48 h protected from light and heat to check their stability. β-TCP nanoparticles (800 ppm) were incorporated into the basic formulation, functionalized with fluoride (as sodium fluoride, NaF, 1100 ppm) and/or stannous (as stannous chloride, $SnCl_2$, 3500 ppm). The synthesis and the characterization of the β-TCP nanoparticles used were previously described in the literature [15].

2.3. Potential of hydrogen – pH

The slurry was prepared by diluting the toothpaste in distilled water in a ratio of 1:3. The equipment HI 221 Calibration Check was calibrated using the following standard solutions: pH 4, 7, and 10. The pH of the slurries was measured in triplicate by the same operator and the average was calculated [22].

2.4. Amount of potentially available fluoride

The slurry was prepared as aforementioned. Then, 1 mL of the slurry was mixed with 1 mL of TISAB II buffer. The amount of potentially available fluoride was determined in triplicate and by using an ion-selective electrode of fluoride (Orion EA940). The equipment was previously calibrated using standard fluoride solutions (in ppm F^-), as follows: 0.01; 0.1; 1; 10; 100; and 500. The values obtained (in μg/ml) were multiplied (4×) due to the dilution carried out to prepare the slurry [22].

PHASE 2 – TOOTHPASTE ABILITY TO REDUCE ETW

2.5. Sample size

A pilot study was conducted to define the number of specimens required for the enamel surface loss test. Enamel bovine blocks ($n = 3$) were used for the Negative Control (Placebo Toothpaste) and Experimental Toothpaste with Fluoride (F^-) groups. The ANOVA Sample Size Test of SigmaPlot 13.0 software was used (Systat Sotware Inc., Chicago, IL, USA), considering a power of 80%, a significance level of 5%, and eight experimental groups. Data on enamel surface loss was used for sample size calculation. A difference in means of 5.25 and a standard deviation of 1.89 among the tested groups resulted in five specimens per group. For standardization purposes and based on a previous study with the same methodology [22], ten specimens per group were used.

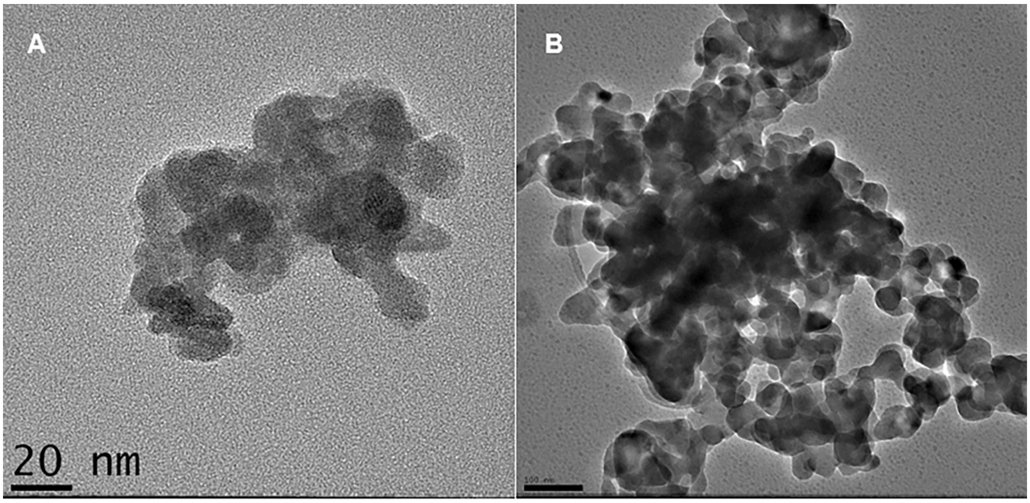

Fig. 1. Transmission electron microscopies of β-TCP nanoparticles tested in two different sizes: A) around 5–20 nm and B) around 50–100 nm. Scale bars 20 and 100 nm, respectively.

2.6. *Specimens' preparation*

Bovine lower incisor teeth were obtained from a slaughterhouse. Teeth were cleaned using periodontal scalers, rotary brushes, and pumice paste. Each tooth was then visually examined for the presence of cracks, fractures, and stains. Only sound teeth were selected for the study. The roots were separated from the crowns. Fragments of enamel measuring 4 mm × 4 mm were obtained from the middle of the crown, using an automatic cutting machine (Isomet, Buehler, Lake Bluff, IL, USA). The back of the enamel blocks was flattened, and the enamel surface was polished, using aluminum oxide abrasive discs (Al_2O_3) with increasing granulation sequence (800, 1200, and 4000), under water cooling (Struers ApS, Pederstrupvej 84 DK – 2750 Ballerup, Denmark). Between every abrasive disc change, the specimens were subjected to an ultrasonic bath with distilled water to remove debris.

2.7. *Baseline measurement*

Eighty enamel blocks were analyzed using an optical profilometer (Proscan 2100 – Sensor Model S11/03) connected to the computer software Proscan Application software *v. 2.0.17* to evaluate the surface curvature. The center of the specimen surfaces was scanned in an area of 2 mm long (X) × 1 mm wide (Y). On the x-axis, the step size was set to 0.01 mm and the number of steps was 200. On the y-axis, these values were 0.1 mm and 10, respectively. Only curvatures lower than 0.3 μm were accepted [22]. According to the surface curvature values, specimens ($n = 10$) were randomly assigned into eight experimental groups (Table 1).

Unplasticized polyvinyl chloride (UPVC) tapes were used to create a window of 4 mm × 1 mm on the enamel surface. This window was created to leave a central area subjected to the treatments (erosion-abrasion cycling with experimental toothpaste) and two reference areas (not treated-protected). Fig. 2 represents the specimens prepared for cycling.

2.8. *Erosive-abrasive challenges*

Specimens were daily immersed in 1% citric acid (natural pH, ~2.4) for two minutes, followed by one hour immersed in artificial saliva. This procedure was repeated four times per day. Thirty minutes after the first and last immersion in artificial saliva, specimens were subjected to abrasion–toothbrushing simulation, using an automatic brushing machine (BIOPDI©, São Carlos, SP, Brazil) as follows: 45 brushing strokes (15 s), under 1.5 N load, using the slurry of experimental toothpastes (toothpaste + artificial saliva, using ratio 1:3) and soft toothbrushes (Oral-B® Indicator 30S). Specimens were exposed to the slurry for two minutes. After brushing, specimens were washed and again immersed for 30 min in artificial saliva. The daily erosion-abrasion cycling was

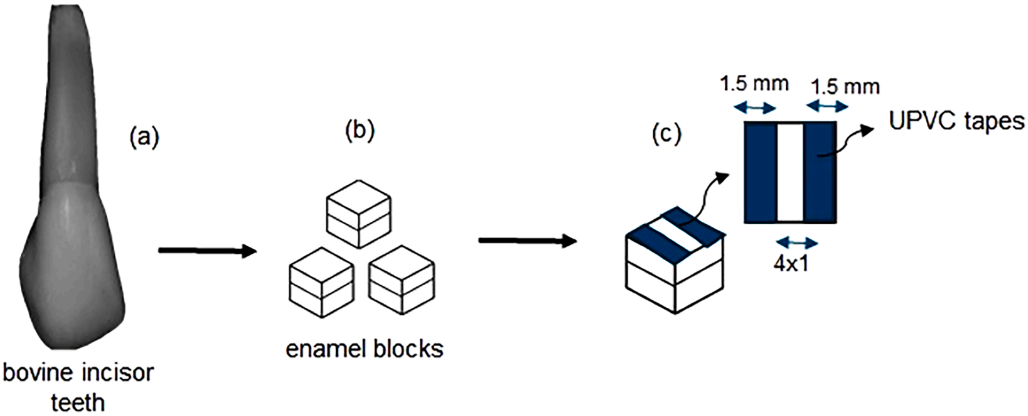

Fig. 2. Specimens preparation.
(a) Bovine incisor teeth; (b) enamel blocks (4 mm × 4 mm) obtained from the crown; (c) surface protected with UPVC tapes, leaving a central area (window) of 4 mm x 1 mm exposed to the subsequent tests.

repeated for 5 days. This protocol was adapted from a previous study [22] and aimed to simulate a patient with a high risk for ETW.

The artificial saliva used was prepared by mixing: 0.213 g/L $CaCl_{2\times}2H_2O$; 0.738 g/L KH_2PO_4; 1.114 g/L KCl; 0.381 g/L NaCl and 12 g/L of Tris buffer [23]. The pH was adjusted to 7.0 using hydrochloric acid.

2.9. Surface loss assessment

After cycling, the UPVC tapes were removed and profilometric analyses were performed again, using the same protocol described in the "Baseline measurement" section. The depth of the treated area was calculated based on the subtraction of the average height of the test area from the average height of the two reference surfaces, using the tool 3-point height.

2.10. Statistical analysis

Data of enamel surface loss and available fluoride followed normal distributions (Shapiro-Wilk test, $p = 0.084$ and $p = 0.122$, respectively) and homoscedasticity (Brown-Forsythe test, $p = 0.278$ and $p = 0.354$, respectively). Data was then analyzed by One Way Analysis of Variance followed by Tukey tests. A significance level of 5% was considered and tests were performed with the statistical program SigmaPlot (version 13, Systat Software Inc., San Jose, CA, USA).

3. Results

3.1. pH and potentially available fluoride

The values of pH ranged from 4.97 to 6.49 (Table 2). Regarding fluoride, negative control (placebo) and distilled water presented the lowest amount of fluoride, with no difference between them ($p = 1.00$). Commercial toothpaste (used as a reference product) presented a higher content of available fluoride than all groups, except than groups $F^- + Sn^{2+}$ ($p = 0.088$), $F^- +$ β-TCP$_b$ ($p = 0.513$) and F^- ($p = 1.00$). The group containing only fluoride (F^-) had no difference from $F^- + Sn^{2+}$ ($p = 0.116$) and $F^- +$ β-TCP$_b$ ($p = 0.600$). $F^- +$ β-TCP$_b$ presented a higher content of available fluoride than all groups, except $F^- + Sn^{2+} +$ β-TCP$_b$. ($p = 0.059$) and $F^- + Sn^{2+}$ ($p = 0.963$). The group $F^- + Sn^{2+}$had no difference from $F^- +$ β-TCP$_a$ ($p = 0.06$) and $F^- + Sn^{2+} +$ β-TCP$_b$ ($p = 0.392$). $F^- + Sn^{2+} +$ β-TCP$_a$; $F^- + Sn^{2+} +$ β-TCP$_b$ and $F^- +$ β-TCP$_a$ had a higher mean than water ($p < 0.001$) and placebo – negative control ($p < 0.001$). The means and standard deviation of fluoride (μg/ml) are shown in Table 2.

3.2. Enamel surface loss

The negative control (placebo toothpaste) presented a higher enamel surface loss than the groups $F^- +$ β-TCP$_b$ ($p = 0.007$) and F^-($p = 0.005$). There were no differences among the other groups tested ($p > 0.05$). The means (SD) of enamel surface loss are shown in Fig. 3.

Table 2
Means of pH and fluoride measurement (μg/ml).

Groups	pH	Fluoride
Commercial toothpaste	6.49	694.21 (10.88) A
F^-	5.45	690.26 (9.19) A
$F^- +$ β-TCP$_b$	5.21	637.89 (15.99) AB
$F^- + Sn^{2+}$	4.98	607.49 (1.75) ABC
$F^- + Sn^{2+} +$ β-TCP$_b$	5.56	545.34 (3.74) BCD
$F^- +$ β-TCP$_a$	5.39	515.25 (10.46) CD
$F^- + Sn^{2+} +$ β-TCP$_a$	5.52	494.93 (5.69) D
Negative control	4.97	1.09 (0.03) E
Distilled water	–	0.00 (0.00) E

4. Discussion

The present study aimed to synthesize a new formulation to manage ETW since the available treatment modalities may not present a long-term effect [24,25]. If not managed in earlier stages, ETW has several consequences for oral health, such as dentin hypersensitivity [26] and darkening (yellowish aspect) of the tooth [27], due to dentin exposure. Calcium and phosphate minerals are the main constituents of dental hard tissues. For several years, calcium and phosphate ions have been provided in oral care products to improve the acid resistance of the tooth. However, premature reactions with fluoride limit their remineralizing potential [28]. Hence, a range of compounds was developed to offer these ions simultaneously, especially because higher calcium content seems to prevent and reduce tooth wear, as proven in a recent systematic review [29]. According to the calcium and phosphate (CaP) ratio, different bioceramic systems can be obtained with a range of solubility, such as amorphous calcium phosphate (ACP), octacalcium phosphate (OCP), hydroxyapatite (HA) and tricalcium phosphate (TCP), which will modulate the ions release. TCP can be obtained through different processing methods, resulting in different chemical phases (α, β, γ) and allotropic forms, which seem to affect its stability [30]. Although β-TCP was already tested for bone repair and tooth remineralization [31,32], the use of β-TCP nanoparticles remains innovative [33].

According to our results, brushing with placebo toothpaste (no active ingredients) resulted in a higher enamel surface loss than brushing with experimental toothpastes containing fluoride (F^-) or β-TCP nanoparticles functionalized with fluoride ($F^- +$ β-TCP$_b$). Firstly, this result validates the model adopted, as the absence of treatment (negative control) did not protect enamel against erosive wear. Later, this result leads us to reject the first null hypothesis tested. The use of β-TCP nanoparticles neither promotes a higher degree of protection than the standard fluoride nor increases the fluoride effect in the toothpastes formulated. Although there are no studies testing nanoparticles in toothpastes on enamel erosive wear, our data is in accordance with a previous study that evaluated the effect of hydroxyapatite nanoparticles and fluoride on enamel caries demineralization [34] and found no relevant protection. The same was observed in a study that tested β-TCP nanoparticles using a solution as a vehicle and found no enamel erosion protection in comparison with solutions containing fluoride and stannous [15]. There are some factors that can explain our findings, which are related to the type of particles, the carrier tested, and the cycling model.

It is known that the shape and size of the particles impact the formation of agglomerates, due to the higher reactive surface area and stiffening efficiency of nanoparticles [35]. Hence, two sizes of nanoparticles were tested in the present study. It is possible that the nanoparticles formed agglomerates (during the synthesis of toothpaste and during the dilution of the slurry), and consequently, the nanoparticles lost their ability to be retained in the deposition sites (micro spaces created by acids) present on the tooth surface. It is also possible to hypothesize that nanoparticles have been thrown off the tooth surface during the abrasive movement of the toothbrushes, due to the size of the agglomerates, reducing their mineralizing potential [36]. Our TEM images confirm this hypothesis of agglomerates formation. For nanoparticles of 20 nm, agglomerates of 150 nm were observed; while for nanoparticles of 100 nm, agglomerates of 400 nm or larger were observed.

The absence of differences observed among the groups can be attributed to toothpaste's characteristics. Toothpaste has a complex formulation (as rheology modifiers, abrasives, surfactants, enzymes) [37] and it is a semisolid gel [38]; hence, its viscosity may have not favored the delivery and viability of β-TCP nanoparticles. Additionally, specimens were in contact with slurry no more than four minutes per day. Therefore, active ingredients did not have a long time in contact with tooth structure to promote mineral deposition on the surface. This

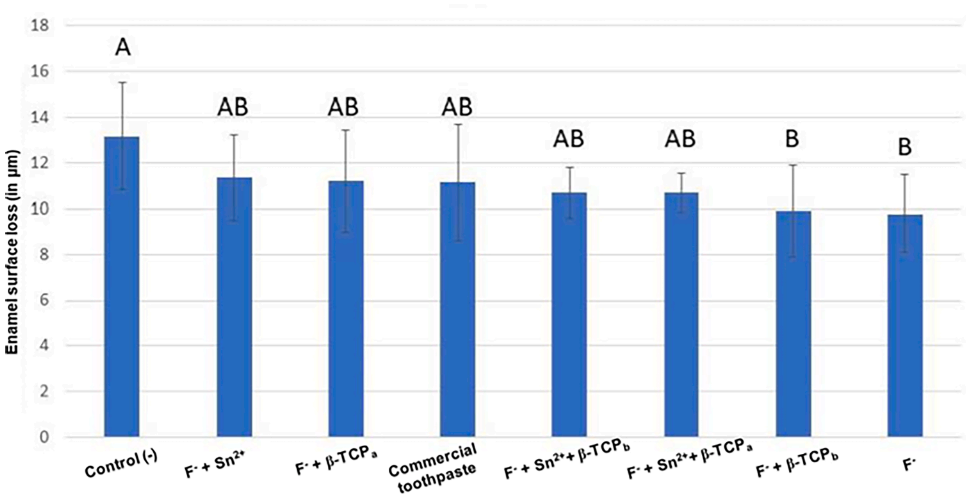

Fig. 3. Means and Standard Deviations of Enamel Surface Loss.

possibility is corroborated by a study that tested the effect of β-TCP nanoparticles in the context of dentin hypersensitivity under erosive-abrasive challenges [16]. The authors used a resin-based material that was light-cured on a dentin surface (long-time contact). As a consequence, some degree of protection was observed, despite the presence of fluoride. It is important to bear in mind that oral health products used without professional supervision have a lower weight (.wt %) of active ingredients compared with restorative materials (used by dental professionals). The lower concentration of fluoride, stannous, and nanoparticles incorporated into the toothpaste formulation may have also limited their protective action. Additionally, the lack of difference between the placebo toothpaste and most of the toothpastes tested (experimental and commercial) can be justified by the interaction of tin with silica (an abrasive with negative zeta potential). For this reason, only the groups without tin in the composition (F- + β-TCPa and F-) reduced enamel surface loss compared with the placebo toothpaste [39].

In the present study, the amount of β-TCP nanoparticles used was defined in a pilot study (data not shown), testing a range of concentrations, while the concentrations of fluoride and stannous were defined based on commercial toothpastes. By contrast, a previous study found greater anti-erosion protection with the use of TCP when compared to fluoride alone [40]. However, the authors used TCP combined with silica (Si) and urea (Ur): TCP-Si-Ur, which might have enhanced its affinity with demineralized enamel. In addition, a pH cycling was used, without the abrasion protocol. Based on these results, it could be suggested that although the erosion-abrasion cycling adopted in the present study reproduces in vitro the clinical conditions, it seems to reduce the performance of the materials tested. For this reason, future studies are needed to synthesize different vehicles with a professional approach (long time of contact with tooth surface) to deliver β-TCP nanoparticles and test their ability to reduce ETW.

In our study, the use of tin did not increase enamel resistance to erosive wear. This result was not expected, since tin has a significant affinity with hard mineralized tissues, such as enamel, which allows the formation of a less soluble layer, resulting from a recrystallization of the surface layer under erosive conditions [41]. A recent study showed that the effect of tin-containing fluoride toothpastes on erosive wear protection is dependent on some characteristics, such as%weight of solid particles [42]. Hence, we encourage future studies to assess the chemical and physical characterization of toothpastes containing β-TCP nanoparticles.

The present study measured the pH of experimental formulations since some chemical reactions are dependent on the pH. For example, the tin mechanism of action in the context of erosion occurs under lower pH [43,44]. However, the pH did not seem to impact erosion protection, which is in accordance with a previous study showing no correlation between the pH of toothpaste and its protection against ETW [45]. Concerning fluoride measurement, overall, the commercial toothpaste showed a higher fluoride content than all groups containing β-TCP nanoparticles, leading us to reject our second null hypothesis. It can be assumed that the commercial formulation has a higher chemical stability than experimental formulations and that the groups containing β-TCP$_a$, which has presented a lower availability of fluoride content, possibly indicate the retention of the fluoride ions adsorbed in the nanoparticles surface area, since it is very unlikely β-TCP nanoparticles have been dissolved during the assay. The smallest are the particles, and the highest are their surface area and their reactivity.

However, based on the results of surface loss, the fluoride content, as the pH, did not affect the surface loss. For both tests (pH and fluoride), the slurry was prepared with distilled water and not with artificial saliva (as in the cycling). This methodology was used to avoid the mineral content of artificial saliva modifying the results of pH and fluoride.

The doses of calcium phosphate nanoparticles applied in biomedicine, oral care products, and cosmetics present a low potential health risk [46] and for this reason, the authors did not perform a cytotoxicity test. One of the limitations of the present study is that the β-TCP nanoparticles were not tested alone without association with fluoride and/or stannous. Consequently, it was not possible to verify their individual effect in reducing enamel surface loss. However, the authors decided not to synthesize a fluoride-free toothpaste as it is usually not recommended for oral hygiene and dental caries prevention [47]. In addition, it is known that the abrasives and other ingredients present in the toothpaste formulation modulate their protective effect against ETW [45], as they can change surface roughness, contributing for surface loss. However, the present study did not evaluate the particles characteristics (such as hardness, shape, and size), the relative enamel abrasivity (REA), and the relative dentin abrasivity (RDA). RDA is the most usual parameter of the toothpastes' abrasiveness and can be used as a safety measurement [48]. Hence, the authors encouraged future investigations to test the RDA of the toothpastes tested in the present study and also their effect on dentin erosive wear.

5. Conclusions

Although toothpaste containing β-TCP nanoparticles protected enamel against ETW, this effect was not superior than the standard fluoride toothpaste, in the formulation tested in this study. In addition, the functionalization of β-TCP nanoparticles with fluoride and stannous did not enhance their ability to control ETW.

CRediT authorship contribution statement

Guilherme Stangler Weiss: Writing – review & editing, Writing – original draft, Visualization, Validation, Software, Resources, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Flávia Rodrigues Oliveira Silva:** Writing – review & editing, Writing – original draft, Validation, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Raíssa Manoel Garcia:** Writing – review & editing, Writing – original draft, Methodology, Investigation, Conceptualization. **Letícia Oba Sakae:** Writing – review & editing, Writing – original draft, Methodology, Investigation, Conceptualization. **Ítallo Emídio Lira Viana:** Writing – review & editing, Writing – original draft, Methodology, Investigation, Conceptualization. **Anderson T. Hara:** Writing – review & editing, Writing – original draft, Formal analysis, Data curation, Conceptualization. **Leonardo Custódio Lima:** Conceptualization, Data curation, Formal analysis, Funding acquisition, Investigation, Methodology, Project administration, Resources, Software, Supervision, Validation, Visualization, Writing – original draft, Writing – review & editing. **Taís Scaramucci:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Software, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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