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Corrosion effects related to bending fracture resistance of orthodontic mini-implants

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Aim: this study is to evaluate the effect of corrosion on flexural fracture resistance in orthodontic mini-implants composed of two materials and submerged in salivary substitutes with or without fluoride. **Methods:** twenty mini-implants were used, 10 from SIN Company (Ti6AL4V alloys) and 10 from Morelli (steel alloys), (G1: Ti6AL4V in fluoride-free saliva solution; G2: Ti6AL4V in saliva solution with 1500 ppm of fluoride; G3: Steel in saliva without fluoride; and G4: Steel in saliva with 1500 ppm of fluoride). The samples were taken to a potentiostat to evaluate the corrosion, and then were evaluated under scanning electron microscopy (SEM). Then, the mini-implants underwent flexural fracture resistance tests. Kruskal-Wallis test with the Student-Newman-Keuls comparison evaluated the corrosion and pitting potentials of each group. ANOVA and Tukey's comparison test at a 1% significance level. **Results:** All groups suffered corrosion potential and pitting potential, but those that were in solutions with the presence of fluoride showed less resistance to the formation of corrosion pits (G1 and G3). In the SEM analysis after flexural resistance, small cavities suggestive of pitting corrosion were noted. The G4 was the only one that formed the passivation potential. In the fracture resistance test, mini-implants manufactured by Ti6AL4V fractured with less force applied (G1 and G2). Most steel mini-implants (G3 and G4) only deformed with a higher force application. **Conclusion:** Fluoride acts to corrode mini-implants, regardless of their manufacturing material. Regarding flexural resistence, the corrosion rate of the mini implants did`nt influence the fracture resistance values.

Keywords: Corrosion. Dental implants. Orthodontics.

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Introduction

Mini-implants have been used for over decades since their introduction to Orthodontics as a skeletal anchorage system. They are determining aspects of anchorage control and orthodontic treatment success[1]. The most used metallic mini-implants are made of titanium alloys (Ti6AL4V) and stainless steel[2]. Both mini-implants made from the Ti-6Al-4V alloy and stainless steel have superior mechanical resistance to the commercially pure titanium used in the manufacture of osseointegrated dental implants[3].

The mini-implant head is clinically exposed to the oral cavity and the coupling area for orthodontic devices such as elastic bands, springs, or ligature wires[4]. This area, in turn, is more susceptible to the action of saliva and fluoride from the oral environment and can undergo metal corrosion with higher or lower intensity. It is a slow and gradual process that may clinically interfere with the success of orthodontic therapy[5].

The corrosion of orthodontic mini-implants or Orthodontic anchoring devices has been suggested for contributing to the inflammation of oral tissues, favoring clinical failure because the release of titanium ions is considered part of the mechanism underlying the process. Further reinforcement of this effect occurs because titanium corrosion can be increased under inflammatory conditions[5-7]. The oral cavity can be a potentially corrosive environment, and the corrosion resistance of orthodontic alloys depends on the oral environment in which they are found. It is influenced by several variables, such as quantity and quality of saliva, pH of foods and drinks, among others[8].

Fracture is another risk factor and complication of using mini-implants. It typically occurs when inserting or removing mini-implants in the bone, but it can also happen when applying forces in the orthodontic treatment[4]. As using mini-implants became more popular in orthodontics, there was more attention to the clinical and mechanical factors contributing to their success. The literature reports failure rates from 6% to 30%[9,10].

There are still no scientific studies reporting when corrosion compromises the structure of a mini-implant and its behavior under tension, forces, and stresses in the oral cavity and orthodontic movement. This laboratory study aims to evaluate the effect of corrosion on the bending fracture strength of orthodontic mini-implants with Ti6AL4V alloys and stainless steel in two different solutions: artificial saliva and artificial saliva with 1500 ppm fluoride. The study tested the hypothesis that after the corrosion process, orthodontic mini-implants made of Ti6AL4V and stainless steel and subjected to artificial saliva with fluoride present lower bending fracture strength.

Materials and Methods

Study design

An *in vitro* laboratory study on corrosion and fracture testing was performed. Hence, 20 orthodontic anchorage new mini-implants from two manufacturers were selected: 10 mini-implants from SIN (Implant System – São Paulo, SP, Brazil) and 10 mini-implants from Morelli (Morelli – Sorocaba, SP, Brazil). No ethical committee approval was required for this study since no intervention was made.

Group division

Ten samples were kept in artificial saliva without fluoride, and 10 samples were placed in artificial saliva with 1500 ppm fluoride. An n=05 was obtained for each group. The sample size determination was conducted considering previously published references, notably the study by Souza et al.[11] (2015). The groups were divided as follows: G1 - Ti6AL4V in artificial saliva, G2 - Ti6AL4V in artificial saliva with fluoride, G3 - Steel in artificial saliva, and G4 - Steel in artificial saliva with fluoride.

Characterization of artificial saliva

Fusayama's artificial saliva formulation was used as the electrolyte solution in this *in vitro* study, its composition is (Nacl 0.4 g\l; kcl 0.4 g\l, cacl2·2h2o 0.795 g\l, na2s·9h2o 0.005 g\l, nah2po4·2h2o 0.69 g\l; ureia 1g\l) for Artificial Saliva with Fluoride, the same composition was used, adding (1500 ppm of Fluoride) and the electrochemical behavior of metallic materials in this solution was similar to human saliva with temperature of the buccal cavity 37° [12].

Corrosion test

For the corrosion test, a copper device was made to thread the active part of mini-implants, exposing their transmucosal region and head in fusayama artificial saliva, an araldite epoxy glue isolated the copper device from the saliva so it would not interfere with the corrosion test. Fusayama artificial saliva was added to a beaker, along with electrical wiring composed of a black plug to be connected to the working electrode (mini-implant threaded in the copper device), a second blue plug connected to the reference electrode (containing silver chloride), and a third red plug representing the counter electrode (platinum).

The electrochemical tests were performed with an AUTOLAB potentiostat, model 128N FRA32M, coupled to the Voltamaster 4 software used for electrochemical control and data analysis. This equipment can generate an open circuit potential (OCP) defined as the potential of an electron-conducting material immersed in an ion-conducting electrolyte and measured against a reference electrode[13].

Similar to the samples immersed in the electrolyte (Saliva), the OCP evolves, and a waiting time of 1 (one) hour was included until it stabilized. For corrosion occurrence, the equilibrium potential of the anodic reaction of metal dissolution must be lower than the equilibrium potential of the hydrogen reduction reaction (cathodic). A cathodic polarization was performed in the test by increasing this voltage until obtaining corrosion pits. Pitting corrosion is characterized by a highly localized metal attack, occurring only in a given medium at electrode potentials equal to or greater than a determined potential, known as pitting potential[13].

Numbers such as current density and applied potential were generated on the computer, the area of the mini-implant head was measured with the (SOLIDWORKS) software in (cm^2), and there was corrosion in this area. The current/area calculation measured the current density, and descriptive analysis graphs were generated in Excel with these data.

SEM analysis after corrosion

After the corrosion tests, the mini-implants were cleaned in an ultrasonic vat, using acetone to remove the Araldite epoxy glue, and then they were taken to the scanning electron microscope (SEM- TESCAN, LM3 Vega, Curitiba, PR, Brazil) was the equipment used. The surface of the head of the samples was analyzed and photographed at 130x and then at 4000x where there were cracks or corrosion of these materials[14].

Flexural fracture tests

After the electrochemical tests, the mini-implants were attached to an acrylic resin for a period of 1 week until the fracture tests are perfomed. This resin was manipulated, and when reaching the plastic stage, the mini-implant was inserted in its center, leaving only the mini-implant head out of the resin, with no standardized length in each sample. Next, bending fracture tests were performed.

The mechanical tests were performed in the Linha DL testing machine (EMIC, DL2000, São José dos Pinhais, PR, Brazil). The test speed of the machine was 0.5 mm/min, at which the piston drops into the sample. For the tests, compressive loads were applied to the free end of the mini-implant (mini-implant head) at 90 degrees. During the test, the machine recorded the force required, in Newtons (N), for mini-implant deformation or fracture.

SEM analysis after flexural fracture testing

After fracture or deformation of these mini-implants, one from each group was selected again for SEM analysis. The mini-implants were photographed at 130x and 4000x to verify signs of corrosion in the fractured or deformed region.

Statistical analysis

The electrochemical tests were statistically analyzed with the Shapiro-Wilk normality test, followed by the Kruskal-Wallis test with the Student-Newman-Keuls comparison, which evaluated the corrosion and pitting potentials of each group. As for the fracture tests, the Shapiro-Wilk normality test was used, followed by ANOVA two factors and Tukey's comparison test at a 1% significance level to determine the statistically different means among the groups.

Results

The corrosion test results showed current density (mA/cm^2) and two factors potential (mV) values. It is worth mentioning that this study only analyzes the potential (mV). The five samples from each group were tested and provided the following data for each group: Corrosion potential (early corrosion potential), pitting potential (corrosion pits), and passivation potential (formation of a protective layer for the corrosion material). Table 1 and Figure 1 display these data.

Table 1. Mean, standard deviations, and p-values of the corrosion potential, pitting potential, and passivation potential.

		Group 1	Group 2	Group 3	Group 4
Corrosion potential	Mean (SD)	-301.10 (255.76)	-616.55 (43.57)	-327.99 (78.64)	-545.87 (272.78)
	p	0.2899 a	0.2851 b	0.0624 a	0.4182 ab
Pitting potential	Mean (SD)	1266.32 (1005.93)	-480.37 (73.93)	572.54 (114.34)	806.97 (51.32)
	p	0.2899 A	0.2851 B	0.0624 A	0.4182 A
Passivation potential	Mean (SD)	_	_	_	800.12 (53,47)

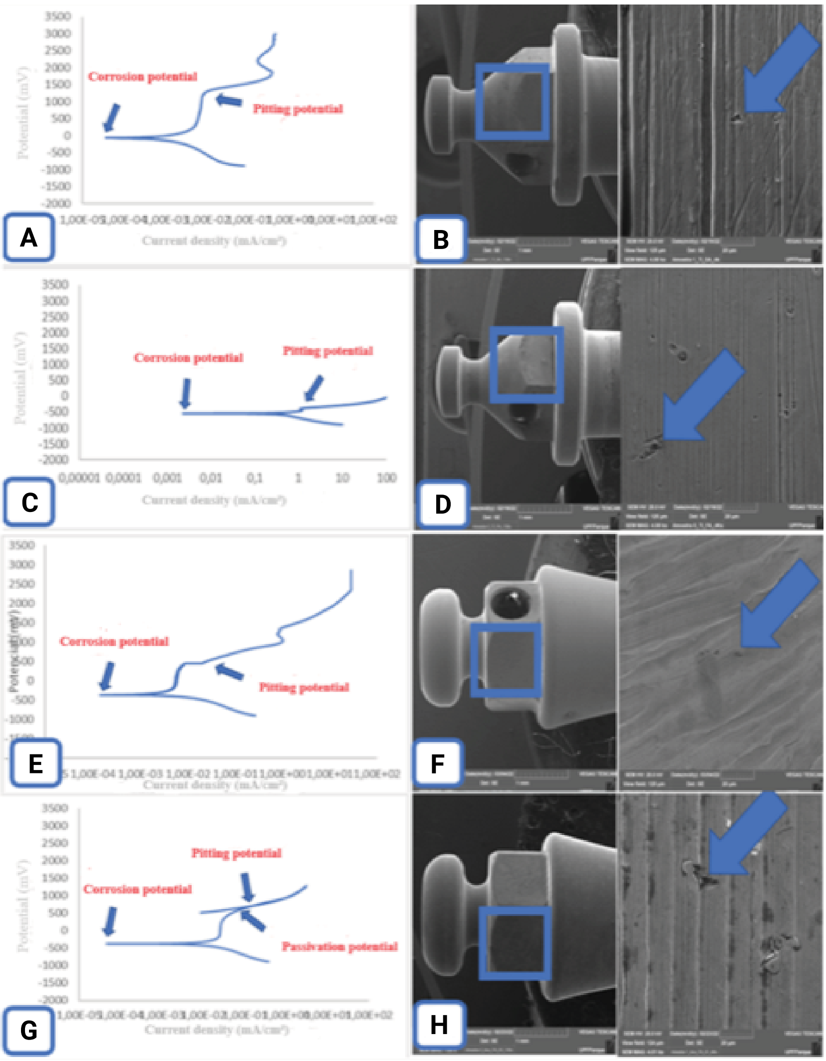

Figure 1. Graphs and SEM images of each group. (A) graph and (B) SEM image of sample 01 of group 1 (Ti6AL4V in artificial saliva); (C) graph and (D) SEM image of sample 05 of group 2 (Ti6AL4V in artificial saliva with fluoride); (E) graph and (F) SEM image of sample 04 of group 3 (Steel in artificial saliva); (G) graph and (H) SEM image of sample 03 of group 4 (Steel in artificial saliva with fluoride).

It was noted a significant difference between group 1 (Ti6AL4V in artificial saliva), which took the longest to form the corrosion potential, and group 2 (Ti6AL4V in artificial saliva with fluoride), which took the least amount of time to form the corrosion potential. There was also a difference between group 2 (Ti6AL4V in artificial saliva with fluoride) and group 3 (Steel in artificial saliva). In turn, the statistical analysis to assess the pitting potential and compare the groups showed a difference between group 2 (Ti6AL4V in artificial saliva with fluoride) and the other groups because group 2 needed a lower potential to form corrosion pits. Also worth noting, group 4 (Steel in artificial saliva with fluoride) was the only one that formed the passivation potential.

The bending fracture strength test results showed that all Ti6AL4V mini-implants (groups 1 and 2) fractured with a lower force application. Most steel mini-implants (groups 3 and 4) only deformed with a higher force application (Table 2). The statistical analysis showed a high difference between groups 1 and 2 compared to groups 3 and 4. When analyzing a sample of each group in SEM (Figure 2), fracture and deformation originated at the load application site.

Table 2. Mean, standard deviations, and p-values after two-way ANOVA tests and comparison between groups with TuKey test.

Group1	247.98 (18.92) Aa	Group 3	490.29 (53.27) Bb	P<0.001**
Group 2	254.52 (29.95) Aa	Group 4	508.60 (45.60) Bb	P<0.001**
	P>0,001*		P>0,001*	

The data were subjected to the Shapiro-Wilk normality test, showing homogeneity.
Group 1: Ti in artificial saliva without fluoride-A
Group 2: Ti in artificial saliva with fluoride-a
Group 3: Steel in artificial saliva without fluoride-B
Group 4: Steel in artificial saliva with fluoride- b
*Upper letters indicate comparison between groups with the same material. Upper letters that differ from each other have a significant difference
** Lowercase letters indicate comparison between groups with different solutions. Lowercase letter that differs from each other have a significant difference

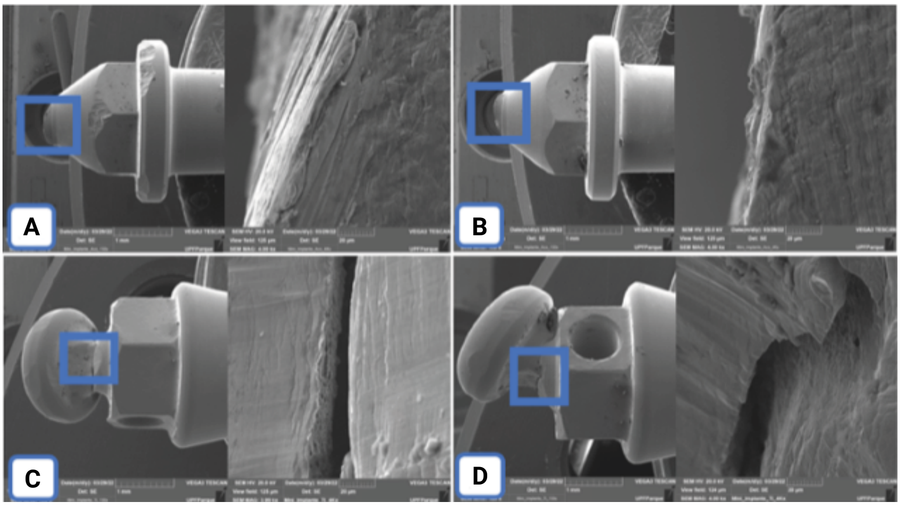

Figure 2. MEV images of each group. (A) Ti6AL4V in artificial saliva; (B) Ti6AL4V in artificial saliva with fluoride; (C) Steel in artificial saliva; (D) Steel in artificial saliva with fluoride.

Discussion

The corrosion tests in this study showed that mini-implants in group 2 (Ti6AL4V in artificial saliva with fluoride) and group 4 (mini –implants Steel in artificial saliva with fluoride) had a lower potential for pitting corrosion resistance. This occurred because fluoride in contact with titanium alloys affects the properties, destroying the titanium oxide layer and its alloys, which favors a lower resistance of this material[15]. Regarding clinical recommendations, authors have advised against using fluoride-containing gels for titanium implants. However, due to the temporary nature of orthodontic mini-implants and the satisfactory success rate, the advantages of fluoride treatment may outweigh the disadvantages.

Although it is widely acknowledged that Al, Ti, and V alloys are generally highly resistant to corrosion due to the stability of the metal oxide layer, it is important to note that, due to the chemical and mechanical characteristics of the oral cavity, along with the presence of microbial flora, enzymatic reactions, and thermal variations, there is a potential for the release of metal ions from titanium mini-implants[16]. Titanium alloys, such as Ti-6Al-4V, are often used instead of commercially pure titanium due to their higher strength, even though commercially pure titanium is more biocompatible[17].

The mini-implants in group 4 (Steel in artificial saliva with fluoride) showed the formation of a passive layer because steel alloys depend on high electrolytic potential, and chemical aggression (presence of fluoride) creates a protective layer, inhibiting the corrosive process. According to Gittens et al. 2011 corrosion events form small cavities on the surface of the metallic device, potentially compromising its mechanical stability, shortening implant life, and causing sudden failure[18].

According to Knutson and Berzins[5] (2013), when a thin, coherent and continuous layer of oxide forms on the surface of certain metals, it is observed in the electrochemical potentiometric measurement curves as a relatively vertical line at potentials higher than the corrosion potential. In other words, the current remains relatively constant, and this is attributed to the protective nature of the passive layer (passivity). In our study, 316L Steel samples exhibited a very stable passive layer, while it was less stable in Ti6Al4V samples.

The artificial saliva solution was selected because it is common in studies on the corrosion of dental and orthodontic implants and simulate the oral environment. The tested solutions are formulated to simulate physiological solutions in the human body. One of them is artificial saliva, with potential variations in chemical composition and pH, which are relevant for the corrosion of metal alloys. The metallic materials used to manufacture the mini-implants are susceptible to corrosion due to the diversity of the oral cavity[5]. Another factor regards the presence of corrosive therapeutic solutions such as fluoride[19].

Fluoride, found in most oral care products, can reduce the stability of the passive layer, normally formed on titanium-based surfaces, thus reducing its resistance to corrosion[20]. In fact, the corrosion resistance of titanium alloys decreases as the fluoride concentration increases from 227 ppm. Consequently, localized corrosion in the form of pitting was detected for the Ti6Al4V alloy being degraded in the form of integranular corrosion, while for 316L steel it occurred at a similar time to the beginning of the formation of corrosion pitting, the passivation of the same pitting, these results are in agreement with the studies by Alves et al.[21] (2016).

Both saliva and fluoride caused corrosion in the alloys studied, but there were no major differences between the solutions. Thus, further *in vivo* studies are required because there are still other factors in the oral cavity that may influence corrosion, such as diet habits, hygiene, and peri-implant toxicity.

In regions where the mini-implant have contact with saliva and fluoride solutions, all suffered corrosive potential[14]. Mini-implants of Ti6Al4V suffered fracture when the flexion fracture test was performed, some steel mini implants suffered deformation when the flexion fracture test was performed and few fractured

The fracture strength of mini-implants varies according to the manufacturer and type of mini-implant. Therefore, professionals must be aware of the characteristics of each material[19]. Differences in mechanical properties can be attributed to mini-implant design. Burmann et al observed cracks and grooves on the surface of these materials, concluding that these irregularities might facilitate mini-implant fractures[22]. The need to evaluate mini-implant deformation when applying perpendicular forces is because this is the most used axis for applying orthodontic forces. Fortunately, they were sufficient to participate in anchorage systems, as they did not fracture when subjected to the orthodontic forces of 50 N[23].

The Ti6AL4V mini-implants fracture due to torsional stress caused by their small diameter and composition[24]. Usually, this alloy has good mechanical strength and excellent osseointegration. Considering the reduction of the risk of fracture, stainless steel has been used because it has higher fracture strength than titanium alloy mini-implants, but lower osseointegration[25]. This corroborates our findings because the properties of mini-implants with stainless steel alloys guarantee higher fracture strength than Ti6Al4V alloys. However, it is worth noting that both mini-implants resist the forces applied to the orthodontic anchorage and can be used without clinical problems.

The SEM analysis was conducted to provide a detailed assessment of the morphology of the mini-implants after the corrosion process and, consequently, their fracture. Additionally, the study by Ranjan et al. 2023 observed, through SEM, signs of corrosion such as pits or fissures, especially attributed to milling defects[26]. It is worth noting that corrosion not only alters the surface nature but also affects the strength and other properties of the material.

The present study can contribute clinically to orthodontists because the variables in the mouth are saliva and the presence of fluoride. However, our hypothesis was rejected because, despite the lower corrosion resistance in the fluoride groups, it does not affect clinical practice. The limitations of this study are the small sample, *in vitro* testing, the lack of other variables in the oral cavity, and the lack of a control group for both tests.

In conclusion, all the groups studied in the corrosion analysis suffered from corrosion and pitting potentials. Fluoride acts to corrode mini-implants, regardless of their manufacturing material. Although the Ti6 Al4V mini-implants fractured and the steel ones deformed, for the most part, the corrosion rate did not significantly influence the flexural resistance values, influence the flexural resistance values, inferring that both mini-implant composition materials are capable of being applied clinically.

Conflict of Interest

The authors have no conflict of interest to disclose.

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Data availability

Datasets related to this article will be available to the corresponding author upon request.

Author contribution

All authors contributed significantly from manuscript findings, revision and final approval of the manuscript and all authors agree with the final version of the manuscript.

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