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Corrosion and Micromorphological Analysis of Temporary Stainless Steel and Titanium Alloy Anchorage Devices

Carolina Assis do Nascimento[1] · **Jurandir Antonio Barbosa**[1] · **Victor Angelo Martins Montalli**[1] · **Fábio Micheletti**[1] · **Rodrigo Milani**[2] · **Valter Pereira**[3] · **Lecino Caldeira**[3] · **Roberta Tarkany Basting**[4]

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Abstract

The aim of this study was to compare the surface micromorphology and corrosion resistance of different temporary anchorage devices (TADs) composed of titanium alloy (SIN®) and stainless steel (DAT Steel® and Bio Ray®). Ten samples of each TAD were submitted to qualitative analyses using energy-dispersive and scanning electronic microscopy before and after immersion in artificial saliva (1500 ppm of fluoride) for 30 days. The chemical analysis was done by X-ray fluorescence, and the corrosion tests were performed by electrochemical means (open circuit potential—OCP, potentiostat, and electrochemical impedance spectroscopy—EIS, using anodic potentiodynamic polarization curves). Passive film resistance (PFR) and corrosion current were established. The corrosion rate was determined by the mass loss test. Greater smoothness and fewer machining defects were observed for the stainless steel TAD before artificial saliva immersion. Comparatively, higher corrosion resistance was found for titanium alloy TAD after immersion in saliva. There was no release of ions into the TAD when immersed in artificial saliva. ANOVA and Tukey tests showed that OCP (V) was significantly lower for the titanium alloy TAD ($p = 0.030$) than the stainless steel brands. E_{pite} (V) and E_{pite}−OCP (V) were significantly higher for the titanium alloy TAD ($p = 0.0009$ and $p = 0.0005$, respectively). Stainless steel TADs presented lower roughness surface than titanium alloy TAD, although the latter presented higher corrosion resistance than the former.

Keywords Mini-implants · Temporary anchorage devices · Surface micromorphology · Corrosion · Stainless steel · Titanium

1 Introduction

Temporary anchorage devices (TAD), or mini-implants, are often used as anchorage aids during orthodontic treatment [1, 2]. They can be manufactured using several materials, including commercially pure titanium (Ti cp), titanium alloy (Ti-6Al-4V) and stainless steel [3, 4]. In terms of features, these materials should be non-toxic, biocompatible, present favorable mechanical properties, be able to withstand stress, deformation and corrosion [5, 6], and not induce adverse local or biological responses [7, 8]. In this respect, these devices should not exhibit surface degradation over time, so

✉ Roberta Tarkany Basting
rbasting@yahoo.com

Carolina Assis do Nascimento
ortonascimento@yahoo.com.br

Jurandir Antonio Barbosa
jurandir@jurandir.com.br

Victor Angelo Martins Montalli
victormontalli@gmail.com

Fábio Micheletti
fabiomichelletti@gmail.com

Rodrigo Milani
rodrigomilani13@gmail.com

Valter Pereira
valter.pereira@ifsudestemg.edu.br

Lecino Caldeira
lecino.caldeira@ifsudestemg.edu.br

1 Faculdade São Leopoldo Mandic, Campinas, SP, Brazil

2 Universidade Fernando Pessoa, Porto, Portugal

3 Instituto Federal de Educação, Ciência e Tecnologia do Sudeste de Minas Gerais, Campus Juiz de Fora, Juiz de Fora, MG, Brazil

4 Faculdade São Leopoldo Mandic, Rua José Rocha Junqueira 13, Bairro Swift, Campinas, SP 13045-755, Brazil

as to ensure very low or no-ion release and good corrosion resistance [6, 9].

The presence of saliva and biofilm, as well as changes in temperature and pH of a patient's diet, provides a favorable environment for metal corrosion [10]. The lower pH of some solutions makes the surface of metallic alloys more prone to pitting corrosion, and shows nobler corrosion potentials [11]. The surface properties of materials, such as their composition and roughness, may also contribute to greater corrosion by causing greater deposition of dental biofilm [12]. In addition, the metallic ions released by TAD components or fixed orthodontic appliances could lead to adverse physiological effects, including cytotoxicity, genotoxicity, carcinogenicity and allergenic effects [13–15]. In this regard, biomaterials should not produce any adverse local or systemic biological response [14], or cause material surface degradation over prolonged use, which leads to corrosion.

Some recent studies have shown that titanium alloys (TADs) may be considered corrosion-resistant [16–18], in compliance with the biosafety standards of alloys for human use [19], based on the stability of the passive titanium oxide layer on their surface [20]. However, Pavlic et al. [21] showed that chlorhexidine increased the roughness of stainless steel mini-implants, whose mechanism is associated with pit corrosion and may cause the release of some ions from the material to the solution. Nevertheless, there are no reports of a comparative corrosion analysis of titanium alloy and stainless steel TADs used for orthodontic purposes.

The composition and microstructure of alloys used for TAD fabrication may present strong corrosion resistance [21]. Furthermore, biomaterials should not present degradation of the surface over time; this would result in low or no-ion release [8]. Since the amount of corrosion in the different types of materials used in TAD composition is not known, the objective of this study was to analyze TAD surface micromorphology, and compare the electrochemical corrosion of titanium alloy and stainless steel TADs submitted to immersion in an artificial saliva solution.

2 Material and Methods

Ten TADs of each tested trademark (Table 1) were evaluated for surface micromorphology in a scanning electron microscope (SEM) (Tescan, Model VEGA 3 SBU, Brno, Czech Republic), equipped with an energy-dispersive spectroscopy microanalyzer (Bruker, Berlin, Germany). Chemical composition analyses by X-ray fluorescence spectroscopy (Shimadzu, model EDX 8000, Tokyo, Japan) were also performed with non-monochrome copper (Cu) Kα radiation ($I = 1.54$ Å), in a chamber vacuum at 1 °C × 10^{-7} Pa. Surface micromorphology analysis of the TADs was performed in the region corresponding to the head and transmucosal profile, with magnifications from ×16 to ×5000.

The TADs were then immersed separately into 3 ml of Fusayama–Meyer artificial saliva with 1500 ppm fluoride (F) (potassium chloride (0.4 g/l); sodium chloride (0.4 g/l); calcium chloride (0.6 g/l); sodium phosphate (0.690 g/l) and urea (1 g/l); pH = 5.8), and stored for 30 days at 37 °C ± 1 °C [22]. The TADs were removed, dried and reassessed for surface micromorphology and chemical composition. The saliva used for TAD immersion was also analyzed chemically by X-ray fluorescence spectroscopy.

Another three TADs of each trademark were subsequently prepared for open circuit potential (OCP) and anodic potentiodynamic polarization (APP) testing, and yet another three for electrochemical impedance spectroscopy (EIS). Test specimens were prepared for corrosion testing, in which the threaded portion of the TAD was welded to the end of a Cu wire (≈ 20 cm long) to make the electrical contact for corrosion simulation. The welded Cu region and the screw tip were prepared with epoxy resin for electrical isolation, leaving only the screw head area for testing.

According to the ASTM G-5 standard, the OCP and APP tests were performed 1 h after immersion in artificial saliva [8, 9]. A BiPotentiostat/Galvanostat Module (AUTOLAB, model PGSTAT 128N, Herisau, Switzerland) was used to assess behavior of the film as applied.

EIS was performed after the immersion process, using the same BiPotentiostat/Galvanostat Module. The behavior of the test materials in forming the film was investigated using a curve analysis to study the spectrum data and evaluate capacitance and polarization resistance. A 100 kHz 10 MHz scanning range, 10 mV perturbation amplitude, and 10-point data acquisition rate per decade were used to perform the OCP test at 24 ± 2 °C Each experiment was repeated three times to ensure its reproducibility, and the potentials were stated in relation to the silver/silver chloride electrode (Ag/AgCl).

Table 1 Materials used in this study and specifications

Brand	Composition	Origin	Dimensions (mm)	Lot
SIN	Titanium alloy (Ti-6Al- 4 V)	Jardim Anália Franco, SP, Brazil	1.6 × 1.8	Q060200494
DAT Steel	Stainless steel (F138)	São Bernardo do Campo, SP, Brazil	2 × 12	20,170,823-SL10939
Bio-Ray	Stainless steel (AISI 316L VM)	Taipei, Taiwan	2 × 12	1,604,132-1

The corrosion rate was determined by quantifying the difference between the initial and the final mass of the specimen. An analytical balance (Sartorius, Model ED224S, Gottingen, Germany) was used to weigh the TADs. The corrosion rate was expressed in mm/year, and calculated from mass loss data according to ASTM G 31-72.

The data were submitted to one-way analysis of variance (ANOVA) and Tukey's test. The R Core Team program [23] was used, and the significance level was set at 5%.

3 Results

Stainless steel TADs showed higher surface smoothness and fewer machining defects before immersion in artificial saliva (Figs. 1 and 2). However, after immersion, the steel TADs presented surface alterations, and fewer irregularities on the Bio Ray® surface. All the TADs showed minimal surface deterioration points after immersion, but deterioration was less significant for the titanium alloy.

X-ray fluorescence spectroscopy did not detect the presence of chemical elements in the composition of any TAD immersed in the test solutions (Table 2). The prevalent chemical element in the composition of steel TADs was iron (Fe), whereas titanium (Ti) prevailed for the Ti alloy TAD.

In the electrochemical tests, the OCP values, with small variations, indicated that the alloy surfaces behaved differently in the presence of the ions released in the immersion solutions. The OCP variation curves (Fig. 3) for steel TADs showed that OCP decreases as time increases; this means that the passive film has a hard time passivating in the presence of ions in the artificial saliva. The same behavior was observed for the Ti alloy TAD; however, this alloy presented considerably lower OCP levels than those of the steel alloy. Scanning potential started at OCP in steel alloys, at a rate of 0.333 mV/s (1.2 V/h) to E_{pit}. The region in which the potential values had current density $> 10^{-4}$ μÅ/cm^2 was considered the passive range. Current density values greater than this indicated onset of passive film dissolution, as the di/dt derivative grew more rapidly.

The APP curves (Fig. 4) showed a characteristic active–passive transition shape regarding the ions present in the artificial saliva. The anodic polarization curves were spontaneously passivated in the presence of saliva. The Ti alloy showed some instability, detected by an increase in the current density of the imperfections in the passive film. Analysis of the steel alloy behavior revealed instability with potential applications, until the current reached the density value of 1×10^{-5} A/cm^2. From this point on, overpotential applications caused the steel alloys to have the characteristic performance of materials that have passive films exhibiting corrosion resistant.

The E_{pit} for the Ti alloy was considered the potential value at which the inversion in behavior occurred, i.e. the point when current density began to decrease, and the test was stopped (Table 3). There was no significant difference

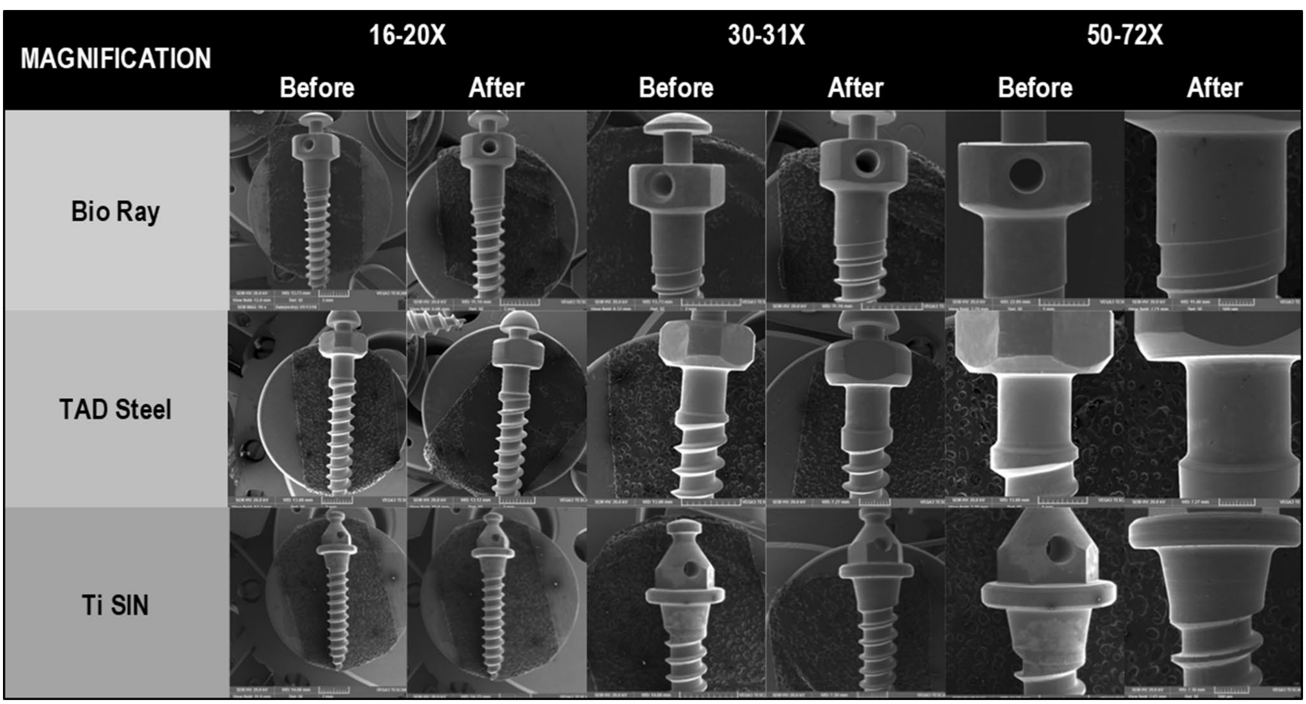

Fig. 1 Representative photomicrographs of the transmucosal TAD profile region before and after immersion in artificial saliva at lower magnifications

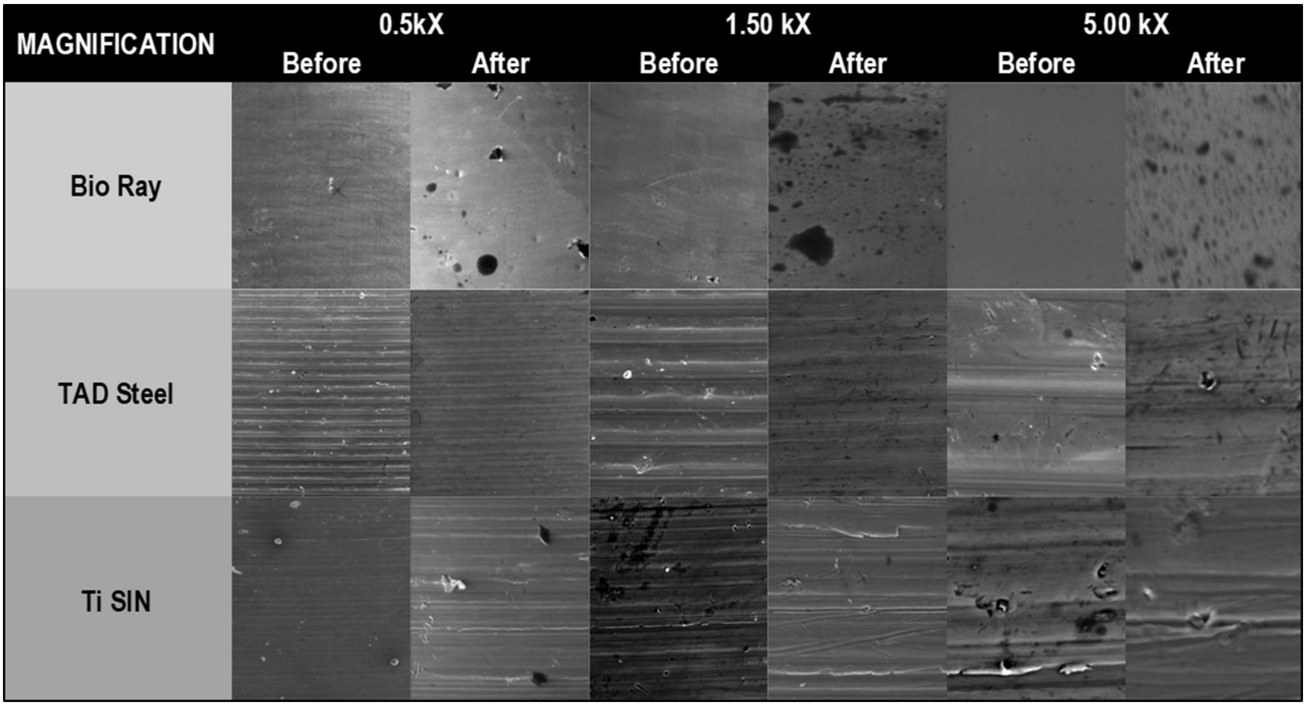

Fig. 2 Representative photomicrographs of the transmucosal TAD profile region before and after immersion in artificial saliva at higher magnifications

Table 2 Quantitative analysis of the main elements detected, and of the artificial saliva after 30 days of immersion

Element	DAT Steel	Saliva	Bio-Ray	Saliva	SIN	Saliva
Fe	67.336 ± 1.20	ND	64.758 ± 0.043	ND	–	–
Cr	18.871 ± 1.30	ND	17.996 ± 1.77	ND	–	–
Ni	11.944 ± 1.50	ND	14.578 ± 0.192	ND	–	–
Mo	1.850 ± 1.20	ND	2.667 ± 0.01	ND	–	–
Ti	–	–	–	–	87.731 ± 1.607	ND
Al	–	–	–	–	7.655 ± 1.587	ND
V	–	–	–	–	4.595 ± 0.020	ND

ND not detected

among the TADs regarding solution pH ($p = 0.1250$). OCP (V) was significantly lower for the Ti alloy TAD ($p = 0.030$) compared to the stainless steel brands. E_{pit} (V) and E_{pit}-OCP (V) values were significantly higher for SIN® TAD ($p = 0.0009$ and $p = 0.0005$, respectively), demonstrating a great decrease in these values in the presence of the fluoride ions in saliva. Stable passive film formation and corrosion resistance began when the potentials were applied, but were unable to reach the total loss of corrosion resistance potential (E_{pit}). This explains why the Ti alloy presented a high value of passivation domain.

Figure 5a shows that similar behavior occurred with the low zreal and zimag values at high frequencies, for both alloys. In the intermediate to low frequency region, the samples showed an increase in the capacitive arc, suggesting the formation of a more capacitive passive film, owing to the high values obtained for the zreal and zimag components. Zreal and zimag were found to have very close values at all frequencies, for both alloys. This indicates that the materials have good corrosion resistance at medium to low frequencies.

The Bode module diagram (Fig. 5b) shows the characteristic performance of materials that have passive films resulting from increased corrosion resistance at certain frequencies during the test period. The impedance module for frequencies higher than 1 kHz shows low values for all the materials, thus characterizing the system behavior as one that presents only electrolyte resistance. As the frequency decreased, the resistance values of passive films increased, but slightly more for Bio Ray® TAD, followed

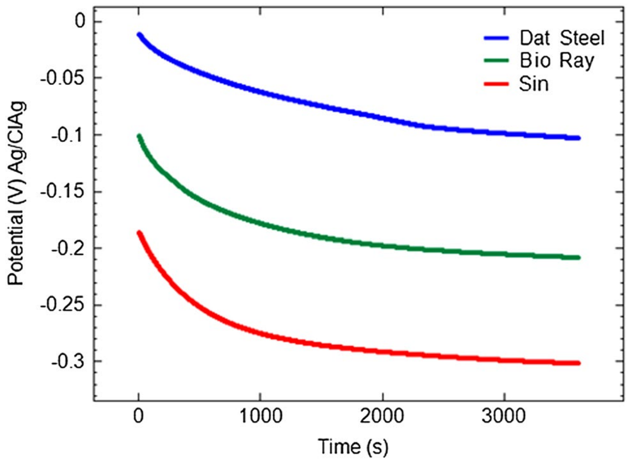

Fig. 3 TAD OCP after immersion for 1 h in artificial saliva

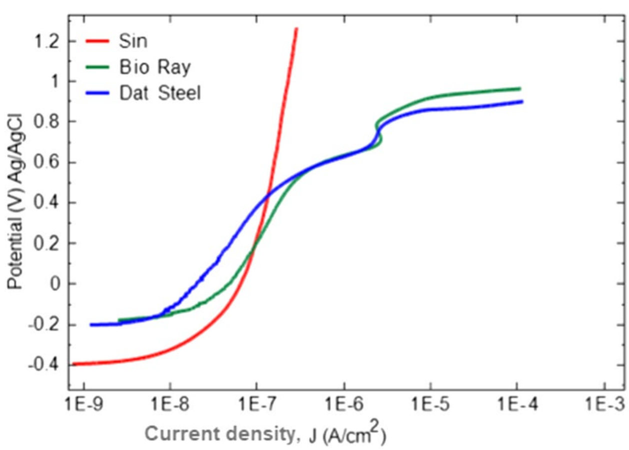

Fig. 4 TAD APP assessed after 1 h of immersion in artificial saliva

by SIN® TAD and DAT Steel®. In comparison, at a frequency of 10^{-2} Hz, the materials presented an impedance modulus greater than $1 \times 10^5\ \Omega\ cm^2$, suggesting that the steel films in this condition have good passivity, that is, good electrochemical resistance to the components present in the saliva solution. Figure 5c depicts the Bode phase plot, showing that the phase angle has relatively low values at higher frequencies (1 kHz). At this value, the alloy films did not show any corrosion resistance. The curves were observed as having the characteristic shape of materials with resistance to the load passage, thus indicating that the alloys were passivated and had good corrosion resistance. There was no mass loss or release of ions in fluoridated artificial saliva after immersion for 30 days (Table 4).

Table 3 Values obtained from the SIN, DAT Steel and Bio-Ray alloy anodic polarization tests in an artificial saliva solution containing 1500 ppm F

Variable	TAD	Means*	Standard deviation
pH	SIN	5.79	0.01
	Bio Ray	5.78	0.01
	DAT Steel	5.78	0.01
	$p = 0.1250$		
OCP (V) (Ag/AgCl)	SIN	−0.34	0.05
	Bio Ray	−0.18	0.02
	DAT Steel	−0.15	0.05
	$p = 0.0030$		
E_{pite} (V) (Ag/AgCl)	SIN	1.28	0.02
	Bio Ray	0.91	0.10
	DAT Steel	0.86	0.08
	$p = 0.0009$		
E_{pite}−OCP (V)	SIN	1.63	
	Bio Ray	1.10	
	DAT Steel	1.03	
	$p = 0.0005$		

*$p \leq 0.05$

4 Discussion

Knowledge of the surface micromorphology and the corrosion resistance of different TADs in biological fluids can enable metal dissolution to be controlled at permissible and harmless levels [5, 6, 8, 24]. In this study, the TAD analysis showed smooth and regular surfaces when observed at lower magnifications. However, when observed at higher magnifications, there were irregularities from the manufacturing process, derived from machining of the material. Material roughness may range depending on the process and the type of thread [25]. Therefore, roughness is a parameter inherent to the screw manufacturing process [26]. Priority should be given to ensure lower surface roughness, considering that the greater the irregularities from the surface finishing process, the higher the chance of impurities accumulating on the TAD surface. In this study, immersion of the TADs in acidic pH artificial saliva showed that they all had minimal surface deterioration points, but deterioration was more significant for the stainless steel TAD. These surface changes may lead to contamination and points of corrosion [13], which may enhance the pit corrosion [14]. Changes in surface roughness were also observed when stainless steel TADs were immersed in a chlorhexidine solution [21]. Thus, surface treatments in biomaterial applications should be performed on stainless steel TADs, specially to improve resistance to degradation [27].

However, the immersion test provided no differences regarding the initial and final mass, or the presence of

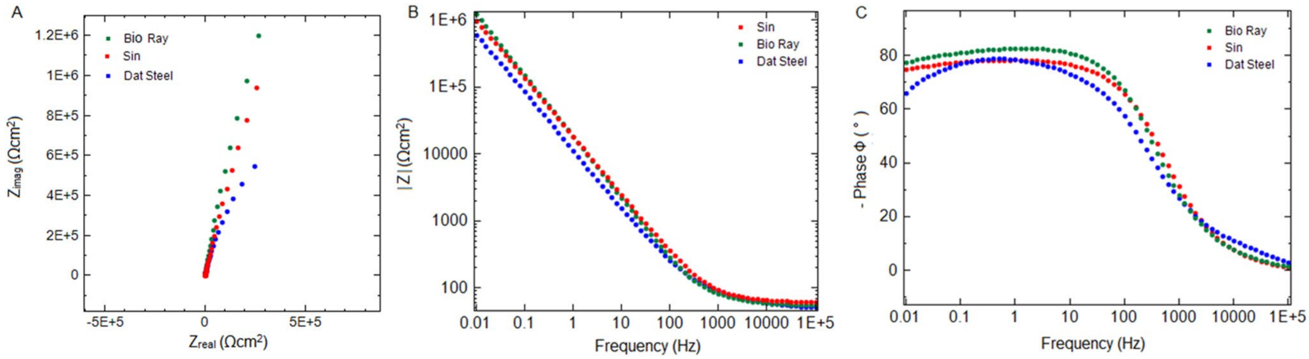

Fig. 5 Impedance diagrams measured at TAD OCP (**a** Nyquist; **b** Bode module; **c** Bode phase) after 1 h of immersion in an artificial saliva solution

Table 4 Initial and final TAD mass means before and after immersion in an artificial saliva solution containing 1500 ppm F, and mass difference between the assessment periods

Samples	DAT Steel	Bio Ray	SIN
Initial mass (g)	0.3017 ± 0.0073	0.2955 ± 0.0008	0.085 ± 0.006
Final mass (g)	0.3017 ± 0.0073	0.2955 ± 0.0008	0.084 ± 0.006
ΔM (g)	−4E−05	−4E−05	−4E−05

g gram, *ΔM* mass difference

Ti, aluminum, vanadium, iron, chromium, molybdenum or nickel metal ions in the artificial saliva solutions after immersion. The absence of mass and the ion loss in the solution after storage characterizes resistance to alloy degradation during the immersion period. This may be associated with how easily the chemical elements making up the Ti and stainless steel alloys formed oxide films on the surfaces of the metals [20], when in contact with the solution under the test conditions [18]. Passive film formation can occur by various means. One is by surface treatment with highly oxidizing solutions (passivating solution), such as nitric acid, which ensures a good passive film. Another means is through the spontaneous formation of this film, as occurs in 316 stainless steel and Ti alloys [20, 28]. The performance of the alloys during immersion in an acidic fluoride-containing solution confirmed their good resistance to pitting corrosion on their surface, in the form of a spontaneous passive film, since no ion release was observed after 30 days of immersion in artificial saliva. Similar results were found by Huang et al. [29] when evaluating a titanium alloy in artificial saliva containing fluoride, and by Suzuki et al. [19] after 120 days of immersion.

This declining potential value over time is characteristic of the lower corrosion resistance of alloys [8]. Considering the literature that associates this response to the environment and the exposure time, the OCP values found suggest that the decrease in OCP presented by the alloys reveals an increase in the electrochemical reactions on their surfaces, and consequent predisposition to corrosion [30]. This indicates that the test time may not have been sufficient to cause the formation of passive surface films.

Orthodontic brackets and wires are made of stainless steel, and remain in the oral cavity for an average of 24 to 36 months [14]. It is worth noting that TADs are used for a short time, an average of 6 months, and that even when fluoridated artificial saliva was used, no metal ions were released. This is because of the influence of other variables present in the oral cavity, such as changes in temperature, pH, and amount of fluoride and other chemical elements. Although an acidic fluoride-containing solution was used, the short immersion time of only 30 days could be a limitation of this study, since it did not cause any ion release, but did cause minimal degradation of the surface, as seen in the micromorphological analyses at higher magnification.

The anodic polarization results showed that SIN® caused slight variations in passive film behavior under the experimental conditions, and in the OCP intervals up to the final test potential. The superior performance of passive films in relation to steel alloys may be associated with non-degradation of titanium oxide (TiO_2) on the surface of the SIN® alloy, caused by the fluoride concentration in the solution. According to these results, it can be inferred that degradation of the passive titanium film and the titanium alloys, caused by the fluoride in the solution, depends not only on the pH value [31], but also on other variables, such as oscillations in time and temperature immersion, and the presence of mechanical wear. Steel alloys showed oscillations in the current density values during the test, but also showed good corrosion resistance. Since the chemical elements of steel are similar in its annealed structure, the differentiating factor in providing higher corrosion resistance is better finish [32].

The small variations that led to the better performance of the Bio Ray® TAD, in relation to SIN® and DAT Steel® alloys, at low-frequency mid-range intervals in the OCP, were sufficient to determine its superiority in terms of pitting corrosion resistance. This behavior can be associated with better surface finish, and better response to passive film formation in the OCP period. Thus, considering the results of this study, it can be inferred that the Ti alloy and the austenitic stainless steel alloys owe their corrosion resistance to the formation of a protective surface film of TiO_2 and chromium. When this film adheres to the surface of the metal, it acts as a barrier that inhibits corrosion and maintains ion release at very low or zero levels. The formation of this passive film on the alloys was little influenced by the electrolyte. This indicates that the passive layer of the Ti and steel alloys increased the alloy resistivity in the presence of the solution, and prevented the formation of pitting. In general, the TADs studied presented good corrosion resistance, as well as low or no ion release.

5 Conclusions

Titanium alloy and stainless steel TADs exhibited good corrosion resistance, but the titanium alloy showed superior performance. Stainless steel alloy TADs had better surface finish and fewer machining defects. No ion release was observed for the TADs immersed in a fluoridated artificial saliva solution. The OCP evaluation showed lower corrosion resistance of the TADs after immersion in saliva, and the titanium alloy TAD showed superior performance in the anodic potentiodynamic polarization tests. These results may serve to guide future industrial practices for promoting better product manufacturing processes.

Author Contributions CAN: investigation, data curation, writing—original draft, JAB: investigation, writing—original draft, VAMM: formal analysis, writing—review & editing, FM: conceptualization, resources, RM: conceptualization, resources, VP: methodology, validation, investigation, LC: methodology, validation, investigation, RTB: conceptualization, writing—review & editing, supervision.

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Data Availability The data will be available on request.

Compliance with Ethical Standards

Conflict of interest The authors declare that they have no conflict of interest.

Ethical Approval Approved by the Ethical Committee of Faculdade São Leopoldo, Process Number 2017/0912.

Informed Consent All the named authors involved in the paper had read it before its submission to publication and confirmed the approval of the complete content.

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