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Corrosion of Dental Alloys Used for Mini Implants in Simulated Oral Environment

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The aim of this study was to explore the resistance to general and pitting corrosion of stainless steel and titanium-based orthodontic mini implants in an oral environment. Studies were performed in artificial saliva and in the presence of two oral hygiene products (one containing chlorhexidine digluconate with sodium fluoride and another containing probiotic bacteria) that are usually recommended to orthodontic patients. The results showed that mini implants made from stainless steel have lower resistance to corrosion than titanium implants. General corrosion was increased in both steel and titanium implants when antiseptic agents containing fluoride ions were used, while the tendency towards pitting corrosion was not noticeably altered. Probiotics, on the other hand, had a beneficial effect on general corrosion resistance but were associated with an increased tendency towards pitting corrosion for both implant materials.

Keywords: mini implants, corrosion, antiseptic, probiotic, artificial saliva

1. INTRODUCTION

Dental materials in the oral cavity are continuously exposed to biochemical, mechanical, and microbiological influences [1]. Corrosion products, which are a consequence of the interaction between

these corrosive factors and dental materials, can cause inflammation or allergic reactions [2-4]. Thus, it is important for dental materials to be resistant to the abovementioned influences [5]. Furthermore, changes in their surface structure alter their mechanical properties, which have undesirable effects on dental and orthodontic treatments. Therefore, testing of these materials should be done in an environment that would simulate the conditions in the oral cavity, such as temperature, pH and influence of the bacteria and their by-products. Simulating oral conditions in vitro is difficult because saliva is a very unstable electrolyte. As salivary pH decreases, its corrosive effect increases [6]. Bacterial biofilms that coat dental materials accelerate the corrosion process due to lower local pH values [7].

Dental implants are components integrated in the bone of the jaw in favour of supporting a dental prosthesis (crown, bridge, or denture) when a tooth is missing or acting as an anchor during orthodontic treatment. Implants should have excellent biocompatibility, which refers to their ability to perform in an appropriate manner to the host [8]. Titanium presents the gold standard for dental implants and orthodontic mini implants, although stainless steel implants can also be used. Newly presented zirconia implants may prove to be a better option; however, further studies must be completed to confirm this hypothesis [9].

The corrosion stability of dental implants and orthodontic mini implants depends on the type of the materials they are made of, as well as the properties of the medium where they are placed. The stability and resistance to corrosion of the implants is determined by the durability of the protective passive layer of the alloy [10]. This passive layer spontaneously forms on the metallic surface, protects the implant from external influences, such as corrosion processes, and reduces the release of metal ions [11]. In orthodontic mini implants, the head of the implant is exposed to the influence of saliva and other substances in the oral cavity.

To maintain better oral hygiene, orthodontic patients often use oral antiseptics. Their purpose is to reduce the negative effect of microorganisms, which accumulate in dental plaques. Furthermore, these mouthwashes stimulate remineralization of the tooth structure. It is well known that chloride ions, which can be found in artificial saliva and other body fluids, have a negative effect on the stability of the passive layer on stainless steels by causing localized corrosion [12]. Recent studies indicate that the addition of fluorides to artificial saliva may additionally decrease the barrier properties of the stainless steel passive layer [13]. Titanium and its alloys exhibit a high corrosion stability and a high corrosion resistance in chloride media due to the formation of a titanium dioxide passive film [14, 15]. However, it has been shown that fluoride ions may decrease the corrosion resistance of titanium and its alloys, especially in solutions with low pH [16-21].

Recently, probiotic bacteria have been recommended to promote oral health, although research implies a greater effect on the reduction of bad breath in orthodontic patients with minimal effects on plaque accumulation and gingival inflammation [22]. However, there is no relevant research on how probiotic supplements affect the corrosion stability of dental materials.

In this work, the influence of two commercial products recommended for the improvement of oral health of dental patients on the corrosion stability of titanium and stainless steel mini implants was investigated. Studies have been conducted on commercial implants to gain more realistic results, as the alloy microstructure and surface pretreatment play significant roles in the corrosion behaviour of metallic materials.

2. EXPERIMENTAL

Stainless steel (SS) type 316 orthodontic mini implants (atomic weight percentage of 58.1% Fe, 18.5% Cr, 12.4% Ni, 6.7% C, 2.5% S and 1.3% Al) and mini implants made from titanium (Ti) alloy Ti-6Al-4V grade 5 (atomic weight percentage of 87.7-89% Ti, 5.5-6.8% Al, 3.5-4.5% V, <0.3% Fe, <0.2% O, <0.1% C, <0.1% N) were used in this study.

Electrochemical testing was performed in a three-electrode cell coupled to the PAR 263A potentiostat and PAR 1025 frequency analyser (Princeton Applied Research, Oak Ridge, USA). The working electrode was the head of the implant, while the other part of the implant was isolated by Teflon film and silicone. The surface of the head of the mini implant made from stainless steel was 32.5 mm², while the surface of the head of the mini implant made from titanium alloy was 35.8 mm². The counter electrode was a platinum wire, while the reference electrode was a saturated calomel electrode (SCE). The following parameters were assessed: corrosion current density (j_{corr}), corrosion potential (E_{corr}), anodic and cathodic Tafel line slopes (b_a) and (b_c), passivation current density (j_{pas}), breakdown potential of the passive layer (E_{bd}), repassivation potential (E_{rp}), electrolyte resistance (R_e), resistance of the electrolyte in the outer porous layer (R_p), resistance of the barrier inner layer (R_b), capacitive behaviour of the outer porous layer (Q_p) and barrier inner layer (Q_b), coefficient deviation from ideal capacitive behaviour in the outer porous layer (n_p), and barrier inner layer (n_b). The studies were conducted in three solutions: artificial saliva, artificial saliva with oral antiseptic a ratio of 1:1, and probiotic lozenges dissolved in artificial saliva (2 lozenges in 100 mL of artificial saliva). The composition of the oral antiseptic was chlorhexidine digluconate 0.05%, sodium fluoride (0.05%, 500 ppm), sodium benzoate, sodium metabisulfide, sodium citrate, purified water, xylitol, propylene glycol, Peg-40 hydrogenated castor oil, and L-ascorbic acid; pH=6.2. The probiotic lozenge contained the probiotic bacteria *Lactobacillus reuteri* Prodentis DSM 17938 and ATCC PTA 5289, which consists of a minimum of 200 million live *L. reuteri* in one lozenge. The chemical composition of artificial saliva was KCl (0.4 g/L), NaCl (0.4 g/L), CaCl₂ x 2H₂O (0.906 g/L), NaH₂PO₄ x 2H₂O (0.690 g/L), Na₂S x 9H₂O (0.005 g/L) and urea (1 g/L). The pH of such prepared saliva was 5.1. The total volume of the electrolyte was 200 mL. All measurements were conducted at a temperature of 37 °C ± 2 °C, which represents the temperature of the oral cavity. Measurements for each specimen were repeated three times in each of the electrolyte media.

Corrosion tests were carried out by cyclic polarization and electrochemical impedance spectroscopy (EIS). First, corrosion potential stabilization was monitored for 2 hours. EIS was then carried out at the open circuit potential with a 10 mV amplitude and a frequency range from 100 kHz to 10 mHz. Afterwards, cyclic polarization was carried out from an initial potential of -300 mV from the open circuit potential to 700 mV potential or a potential where the current density reached 100 μAcm⁻². Polarization was then continued in the cathodic direction to the open circuit potential or the passivation potential. The potential sweep rate was 1 mVs⁻¹. By Tafel's extrapolation method, corrosion current density was determined, as well as corrosion potential and anodic and cathodic Tafel line slopes. The breakdown potential of the passive film (a point where a sudden rise in anode current occurs) and repassivation potential (where the recurring polarization curve intersects the initial anodic part of the curve) were also determined. The surface morphology was studied using a JSM-7800F field emission

scanning electron microscope (FE-SEM) (JEOL, Tokyo, Japan), applying a secondary electron detector with an electron beam acceleration voltage of 10 kV and a working distance of 10 mm. The elemental composition of the samples was determined using an Xmax 80 energy dispersive X-ray (EDX) spectrometer (Oxford Instruments, Abingdon, UK).

3. RESULTS

The cyclic polarization curves for each sample in different media are presented in Figure 1, while the corrosion parameters, corrosion current density j_{corr} , corrosion potential E_{corr} , anodic and cathodic Tafel line slopes b_a and b_c , as well as the passivation current density (j_{pas}), breakdown potential of the passive layer (E_{bd}) and repassivation potential (E_{rp}), are presented in Table 1.

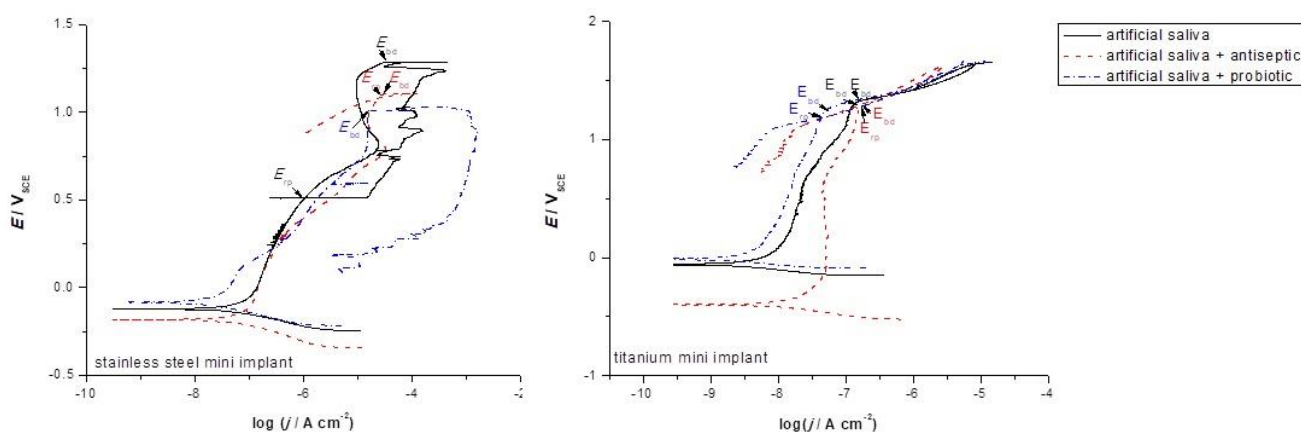


Figure 1. Cyclic polarization curves of stainless steel and titanium mini implants exposed to artificial saliva and oral antiseptic and probiotic supplements. The x-axis represents the current density (j), and the y-axis represents the difference between the working electrode and counter electrode potential (E).

Table 1. Average values and standard deviations of corrosion parameters determined by cyclic polarization for mini implants made from titanium alloy and stainless steel. The passivation current density (j_{pas}) was determined at a potential of 500 mV.

Sample	$j_{orr}/$ nA cm ⁻²	$E_{corr}/$ mV	$b_a/$ V dec ⁻¹	$b_c/$ V dec ⁻¹	$j_{pas}/$ nA cm ⁻²	$E_{bd}/$ V	$E_{rp}/$ V
Ti-saliva	2.7 ±0.4	-0.077 ±0.027	0.251 ±0.051	-0.067 ±0.006	27.7 ±0.6	1.338 ±0.032	1.338 ±0.032
Ti-antiseptic	7.4 ±6.4	-0.295 ±0.117	0.422 ±0.038	-0.092 ±0.029	33.9 ±14.1	1.254 ±0.013	1.254 ±0.013

Ti-probiotic			0.157	-0.043			
	1.7	-0.006			8.7	1.272	1.203
	±0.9	±0.065	±0.069	±0.003	±0.9	±0.017	±0.079
SS-saliva			0.536	-0.085			
	87.6	-0.139			1415.7	1.244	0.869
	±39.5	±0.022	±0.3950	±0.013	±384.6	±0.055	±0.515
SS-antiseptic			0.450	-0.106			
	116.9	-0.289			9078.6	1.071	1.049
	±29.4	±0.141	±0.318	±0.023	±10141.3	±0.066	±0.072
SS-probiotic			0.0357	-0.078			
	36.9	-0.120			619	1.135	270
	±15.5	±0.050	±0.091	±0.010	±73.4	±0.119	

The results show that both implant materials exhibit passive behaviour in the studied solutions with a low general corrosion rate ($j_{\text{corr}} \leq 0.1 \mu\text{Acm}^{-2}$). A lower corrosion current density and passivation current were observed for the titanium alloy than for the stainless steel mini implants. While titanium implants are easily repassivated, for steel implants, the observed difference between E_{bd} and E_{rp} points to the possibility of localized corrosion. The addition of antiseptic to artificial saliva resulted in increased corrosion current densities, especially for titanium implants. The passivation current density increased as well, but the increase was more pronounced for steel implants. The breakdown potential and corrosion potential values are shifted towards more negative values for both implant materials. However, the tendency towards localized corrosion of steel implants decreased as more positive E_{rp} values were observed.

The results of the studies conducted in saliva containing dissolved oral probiotics show that for both implant materials, corrosion and passivation current densities decrease compared to artificial saliva alone. For both alloys, the shift of corrosion potential towards more positive values and breakdown potential towards more negative values is observed. It should also be stressed that repassivation was observed only for one out of three stainless steel samples. Thus, it can be concluded that in the presence of probiotic agents, the general corrosion rate of both implant materials decreases, but the localized corrosion intensity increases for steel implants. For titanium implants, there also exists a slight possibility for localized corrosion, although the difference between E_{bd} and E_{rp} is not significant. To further analyse the properties of the studied samples, EIS measurements were conducted. Impedance parameters are shown in Table 2, and representative impedance spectra are shown in Figure 2.

Table 2. Impedance parameter spectra for stainless steel and titanium mini implants in the studied media (average ± standard deviation).

Sample	$R_p /$ $k\Omega\ cm^2$	$Q_p /$ $\mu S\ s^n\ cm^{-2}$	N	$R_b /$ $M\Omega\ cm^2$	$Q_b /$ $\mu S\ s^n\ cm^{-2}$	n
SS-saliva	0.042±0.021	21.73±4.89	0.99±0.01	1.906±0.692	37.92±5.82	0.84±0.01
SS-antiseptic	0.172±0.227	38.66±18.53	0.85±0.05	3.859±1.854	44.17±27.73	0.87±0.01
SS-probiotic	0.310±0.242	25.11±13.47	0.91±0.05	4.672±0.450	10.76±7.99	0.92±0.06
Ti-saliva	70.45±57.48	0.36±0.03	0.94±0.01	97.92±37.39	4.04±3.76	0.64±0.01
Ti-antiseptic	32.86±21.52	0.61±0.30	0.94±0.01	15.48±5.24	2.37±1.55	0.56±0.93
Ti-probiotic	82.35±75.00	0.66±0.55	0.91±0.04	303.33±189.98	1.82±1.77	0.63±0.03

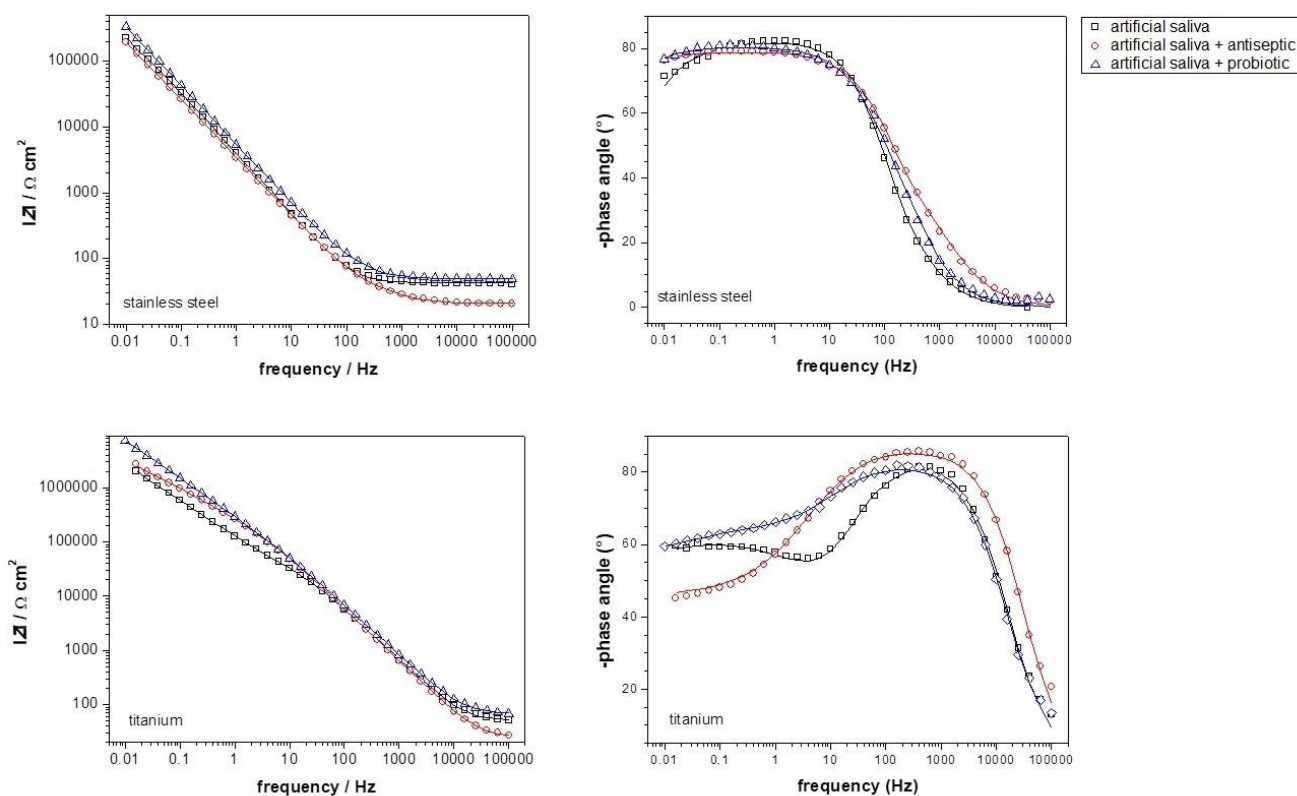


Figure 2. Electrochemical impedance spectra for stainless steel and titanium mini implants exposed to artificial saliva, oral antiseptic and probiotic supplement. Symbols represent measured and lines fitted data. The y-axis presents the impedance module (left figure) and phase angle (right figure), while the x-axis presents the excitation frequency.

The EIS spectra for stainless steel implants exhibit a broad phase angle maximum at medium and low frequencies, which is typical for metals exhibiting passive behaviour. However, a good match between the experimental and simulated data (χ^2 value below 10^{-3}) was obtained only by using the model with two time constants (Figure 3.). This type of equivalent electrical circuit can be regarded as an electrical representation of a two-layer oxide film consisting of a barrier compact inner layer and a

porous outer layer. It consists of the electrolyte resistance R_e in series with 2 RQ elements in parallel. The components R_p and R_b are the resistance of the electrolyte in the porous outer layer and resistance of the barrier inner layer, respectively. The nonideal capacitive behaviour is represented by a constant phase element CPE described by the function $Z(\text{CPE}) = (Q(j\omega)^n)^{-1}$, where Q is the constant, j is the imaginary number, ω is the angular frequency, and n is the CPE exponential factor. Q_p thus describes the capacitive behaviour of the outer porous layer, while Q_b describes that of the barrier inner layer.

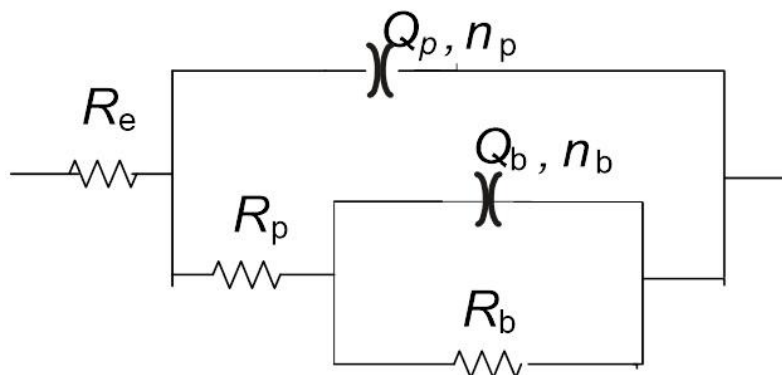


Figure 3. Electrical equivalent circuit used to fit the experimentally obtained impedance spectra (Figure 2) for stainless steel and titanium mini implants exposed to artificial saliva and oral antiseptic and probiotic supplements. R_e represents electrolyte resistance, R_p the resistance of the electrolyte in the outer porous layer, R_b resistance of the barrier inner layer, Q_p capacitive behaviour of the outer porous layer, Q_b capacitive behaviour of the barrier inner layer, n_p coefficient deviation from ideal capacitive behaviour in the outer porous layer, and n_b coefficient deviation from ideal capacitive behaviour of the barrier inner layer.

The impedance parameters obtained for stainless steel implants are presented in Table 2. The outer porous film contribution to the overall corrosion resistance is very low, as in all studied solutions, the R_p values were below $1 \text{ k}\Omega\text{cm}^2$. On the other hand, barrier film resistance values are very high, indicating excellent barrier properties. Contrary to the results obtained by polarization measurements, R_b values are higher for samples immersed in antiseptic than for those exposed to artificial saliva. However, it should also be noted that in antiseptic, larger standard deviations were observed for R_b and Q_b . The highest protective properties were observed for samples containing the probiotic. In this solution, both R_p and R_b increased, while both Q_p and Q_b decreased compared to artificial saliva alone.

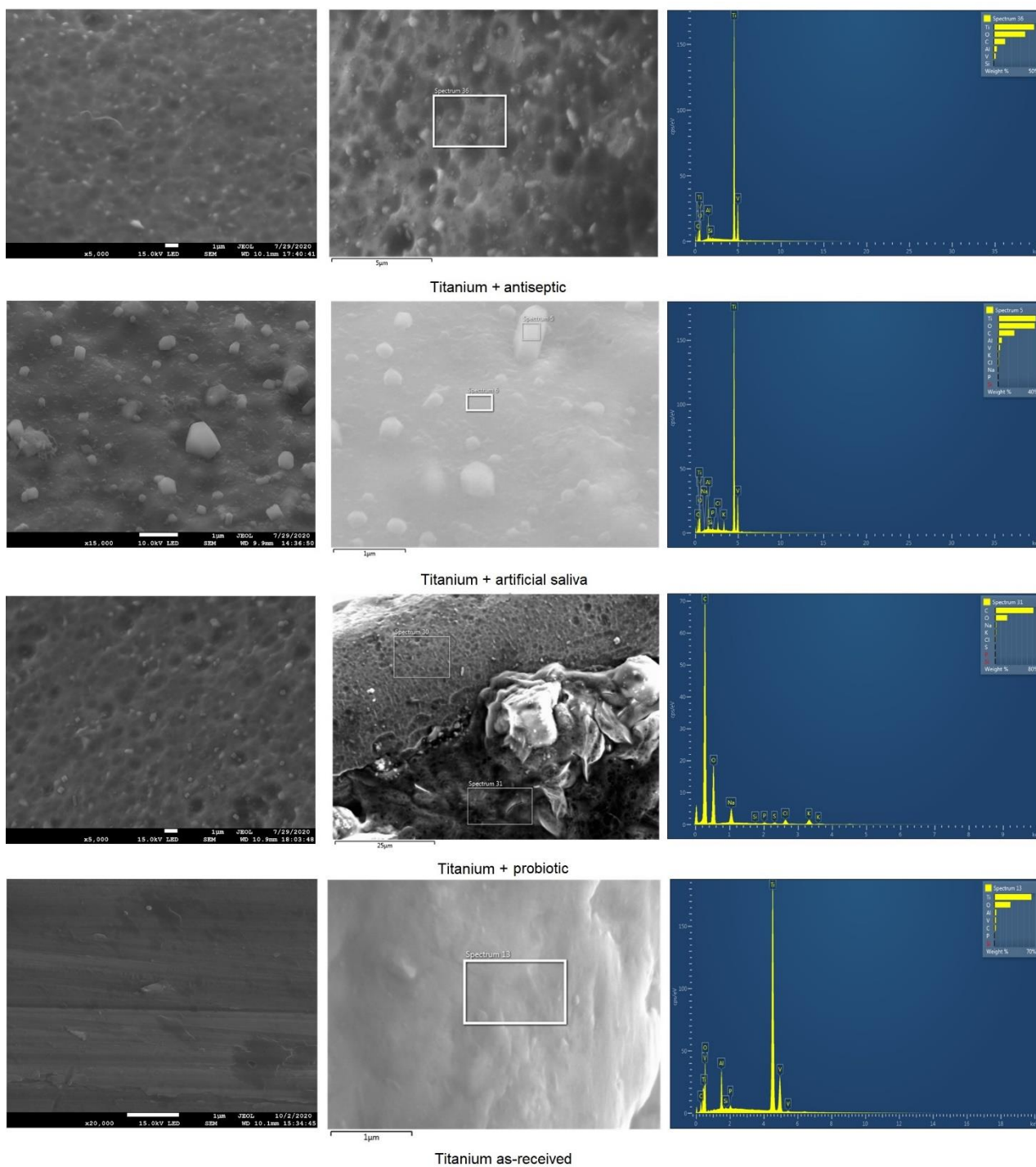


Figure 4. SEM and EDX of titanium mini implants exposed to artificial saliva, oral antiseptic, and probiotic supplement and unexposed as-received samples

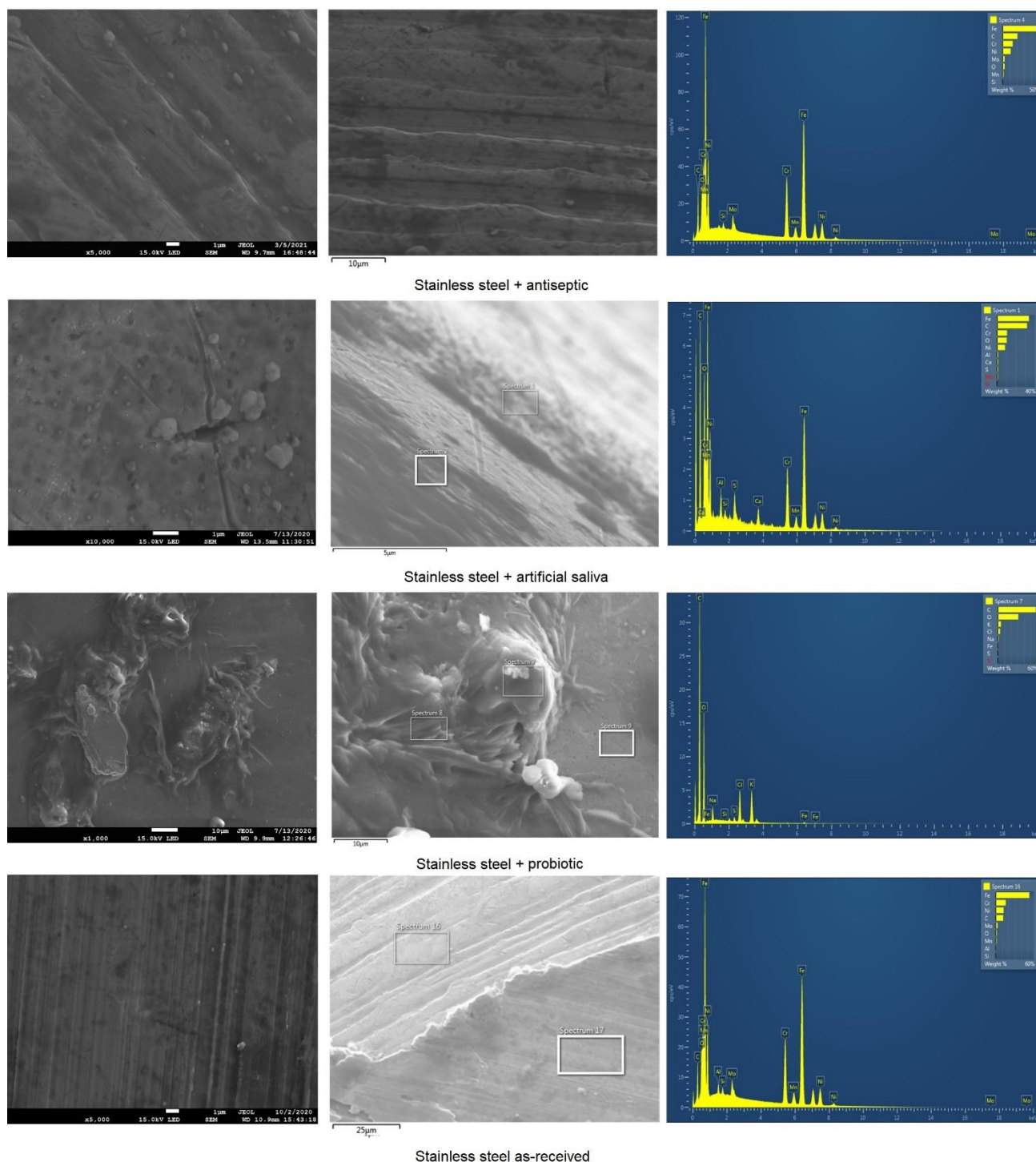


Figure 5. SEM and EDX of stainless steel mini implants exposed to artificial saliva, oral antiseptic, and probiotic supplement and unexposed as-received samples

EIS spectra for titanium mini implants (Figure 2) show higher impedance values than those of the stainless steel samples, which is in accordance with the results obtained by polarization measurements. These spectra also exhibit two phase maxima and can be modelled by the electrical equivalent circuit shown in Figure 3. For titanium implants, both the outer porous and inner barrier layers exhibited higher resistance and lower capacitance values than those observed for steel implants. Thus, it can be concluded that on the titanium implants, a thicker and more protective oxide layer was present.

Regarding the influence of the medium, the lowest R_b and R_p values were observed in antiseptic, while the highest were observed in the probiotic solution. Such results are in accordance with those obtained by polarization measurements.

The surface of the titanium sample exposed to antiseptic ~~Curasept~~ appears to be porous with some corrosion product deposits (Figure 4), while the surface of the sample exposed to artificial saliva appears to be much less porous, which explains the higher corrosion resistance of the artificial saliva sample. The sample exposed to solution with a probiotic surface appears to be porous as well, but it is partially covered with organic deposits, which is probably the reason why higher R_p and R_b are observed for this sample. Similar surface morphology was observed in unexposed stainless steel mini implants and those exposed to antiseptic (Figure 5). Mini implants exposed to saliva show the formation of corrosion products on the surface and a slightly higher number of defects, while those exposed to probiotics exhibit the formation of organic deposits.

4. DISCUSSION

The results conducted in this study show that titanium-made mini implants, when exposed to artificial saliva, exhibit low corrosion current density and passivation current density and vast passive areas with neglectable tendencies towards localized corrosion.

When an oral antiseptic was added to the solution of artificial saliva, there was a small increase in corrosion and passivation current density as well as the barrier properties of the surface oxide layer. This indicates that in the presence of antiseptic, the corrosion activity of titanium implants is altered, which could be ascribed to the fact that antiseptic contains fluoride ions in its formulation. It has been shown that the corrosion rate of titanium alloys depends on the concentration of fluoride ions as well as the pH value. A higher concentration of fluoride ions leads to increased porosity and thickness of this layer, thus decreasing its corrosion resistance [23]. It was reported that in the presence of fluoride ions, titanium becomes vulnerable to localized corrosion processes at pH=3.5 [24]. This could be due to the formation of HF, which can dissolve the passive layer. Some studies report the ability of titanium to repassivate the oxide layer even in a fluoride environment after a period of time. This could be due to the ageing of the oxide layer [25]. However, pitting corrosion was not detected in any of the samples examined in this work. It should be stressed that antiseptic slightly increases the pH of the artificial saliva to 6.1; thus, only low amounts of HF may be present. Nevertheless, it resulted in decreased barrier properties of the oxide layer and increased corrosion rate. That corrosion process on the metal surface largely depends on the environmental conditions was also confirmed in another study [26].

The results obtained for stainless steel mini implants show lower corrosion resistance to general and pitting corrosion in artificial saliva compared to titanium implants. In two out of four stainless steel implants, pitting corrosion was detected. Titanium-made oral implants have superior resistance to corrosion than those made from stainless steel, which is in accordance with previously published papers [27]. In artificial saliva with oral antiseptic, there is an increase in corrosion and passivation current density and a decrease in corrosion resistance, as is the case with titanium-made mini implants. On the other hand, oral antiseptic does not increase the tendency of implants towards pitting corrosion. Although

the breakdown potential is lower in media containing oral antiseptics than in artificial saliva, the difference between E_{bd} and E_{rp} is not significant. This is explained by fast repassivation of the damaged oxide layer. We can assume that although fluoride ions increase media aggressiveness towards the examined samples (higher pH value of the oral antiseptic of 6.1 compared to the pH of the pure artificial saliva 5.1), they have a positive effect on the repassivation of the layer of the stainless steel material, which is in accordance with the observed increased barrier properties of the oxide layer.

The addition of the probiotic has a beneficial effect on the barrier properties of the surface layer of both implant materials, resulting in a decrease in their corrosion rate. This could be attributed to the slight increase in the artificial saliva pH (to pH=6) and to the fact that this probiotic product contains various compounds (palm oil, peppermint oil) that could be adsorbed on the oxide surface acting as corrosion inhibitors. On the other hand, this was the only medium in which Ti implants showed a slight tendency towards localized corrosion, and for steel implants, repassivation was observed for only one out of three samples and occurred at much lower potentials than in the other two media. This could occur either because only part of the surface was covered by adsorbed organic compounds, leaving the rest of the surface more vulnerable to corrosion, or because the probiotic compound could not be fully dissolved and undissolved particles deposited on the metallic surface caused underdeposit corrosion.

The same model based on EIS spectra for both steel and titanium was also applied by other authors [28-31].

Previous research confirm that oral probiotic supplements do not influence the mechanical properties of stainless steel above the influence of saliva but also imply that probiotic bacteria alone have an even less corrosive influence on dental alloys than commercial probiotic supplements [32, 33]. Probiotic bacteria decrease the general corrosion rate of stainless steel and do not increase the possibility of pitting corrosion, which corresponds to fewer changes in mechanical properties [33, 34]. Chlorhexidine seems to induce surface changes in terms of increasing the roughness of steel mini-implants, while probiotics seem to increase the roughness of titanium mini-implants [35]. The corrosion-induced release of titanium ions from titanium alloys mainly supports findings of the general corrosion rate; however, the cytotoxic effect is not high or clinically important [36, 37].

5. CONCLUSIONS

This study evaluates the corrosion behaviour of stainless steel and titanium orthodontic mini implants. The results demonstrate that implants made from titanium alloy have significantly greater resistance towards both general and localized corrosion in all studied media.

In both types of mini implants, the tendency towards corrosion was greater when an oral antiseptic containing fluoride ions was used, while the tendency towards localized corrosion was not significantly altered. On the other hand, in the solution containing oral probiotics, a decrease in the general corrosion rate for both implant materials was observed but also an increase in the tendency towards localized corrosion. Studies performed in this work show that products used for improved oral hygiene or oral health influence the corrosion stability of dental implants and, in some cases, may lead to increased metal dissolution.

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