Corrosion Resistance of Titanium Alloy Orthodontic Mini-implants Immersed in Chlorhexidine, Fluoride, and Chitosan Mouthwashes: an in-vitro Study

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Abstract

Orthodontic mini-implants are widely used as an intraoral skeletal anchorages. Titanium alloy orthodontic mini-implants are known to have high corrosion resistance, but studies have found some corrosion behavior after contact with mouthwashes. The current in-vitro study aimed to examine surface topography and elemental composition as parameters of corrosion resistance for titanium alloy orthodontic mini-implants after being immersed in three different types of mouthwashes.

A total of 28 titanium alloy orthodontic mini-implants were divided equally into four groups and immersed for 28 days in chlorhexidine gluconate 0.2% mouthwash, sodium fluoride 0.2% mouthwash, chitosan 1.5% mouthwash, and distilled water. All the orthodontic mini-implants' heads and necks were then examined for surface topography using a scanning electron microscopy (SEM) and the elemental composition was assessed using energy-dispersive x-ray spectroscopy (EDS).

Surface topography of the orthodontic mini-implants immersed in chlorhexidine gluconate, sodium fluoride, chitosan, and distilled water exhibited some manufacturing defects and rough surfaces, but no signs of crevices or pitting corrosion on the heads and necks. The elemental composition of all groups was comparable, but there was a statistically significant difference between titanium and aluminum (at%) between the sodium fluoride group and the chitosan group.

Titanium alloy orthodontic mini-implants exhibited good corrosion resistance after immersion for 28 days in chlorhexidine gluconate 0.2%, sodium fluoride 0.2%, and chitosan 1.5%. Orthodontic mini-implants immersed in chitosan showed a smoother surface and higher titanium and aluminum (at%) than other groups.

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Introduction

Orthodontic anchorage, defined as resistance to unwanted tooth movement, is one of the key elements in successful orthodontic treatment.[1] Orthodontic mini-implants, a type of intraoral skeletal anchorage devices, give maximum or absolute anchorage. They have many advantages. They are reliable, simple to insert and remove, relatively affordable, and rely less on patients' cooperation.[2–4] One biomaterial widely used for orthodontic mini-implants is titanium alloy (Ti_6Al_4V).[5] Titanium (Ti) is known for its superior biocompatibility because of the high corrosion resistance provided by its stable protective titanium oxide layer.[5–7] Titanium alloy is commercially pure titanium combined with aluminum (Al) and vanadium (V) to improve strength, toughness, and modulus elasticity,[5,6] but several studies have found that aluminum and vanadium ions could be released from orthodontic mini-implants.[6,8–10]

An orthodontic mini-implant, especially the head, is exposed to various chemical agents that might affect the corrosion resistance of the titanium alloy.[11] Mouthwashes such as chlorhexidine gluconate and sodium fluoride are often used by orthodontic patients as an antibacterial agents to prevent inflammation of the soft tissue or white spot lesions on

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enamel.[12,13] In an in-vitro analysis using scanning electron microscopy (SEM), Abboodi et al. (2018) found that fluoridated mouthwashes could cause crevices and pitting corrosion on the surface of orthodontic mini-implants after 28 days of immersion.[14] Mandsaurwala et al. (2015) showed that chlorhexidine mouthwash could cause the release of metal ions after 45 days of immersion.[15] The release of titanium, aluminum, and vanadium ions from the surface of titanium alloy was reported to have harmful effects on the human body such as toxicity, allergic reactions, or mutagenicity.[6,16] Chitosan, a natural biomaterial obtained from crustaceans, is currently developed as a mouthwash because of its antibacterial effects and biocompatibility, but its effect on the corrosion resistance of titanium alloy orthodontic mini-implants is not known.[17,18]

Therefore, this study is aimed at examining the surface topography and elemental composition as parameters of corrosion resistance of titanium alloy orthodontic mini-implants after being immersed in chlorhexidine gluconate 0.2%, sodium fluoride 0.2%, and chitosan 1.5% mouthwashes.

Materials and methods

Samples of 28 titanium alloy orthodontic mini-implants (Dual Top, Jeil Medical, Korea) with head diameter and total length 1.6x8 mm were divided equally and randomly into four groups and immersed separately in chlorhexidine gluconate 0.2% (MINOSEP®, PT Minorock Mandiri, Indonesia), sodium fluoride 0.2% (Pepsodent Expert Protection Pro Complete, PT Unilever, Indonesia), chitosan 1.5% (KITOBE™, CV EcoShrimp, Indonesia), and distilled water (Aqua Pro Injection Sterile, PT Ikapharmaindo Putramas, Indonesia). The distilled water was used as a control. The study also included one as-received titanium alloy orthodontic mini-implant with the same specification and without any immersion.

Each group of seven orthodontic mini-implants was immersed in an individual centrifuge tube containing 0.3845 ml mouthwash or water. These were randomly labeled as red, blue, green, and yellow by the second author. All immersed orthodontic mini-implants were incubated at a constant temperature of 37 °C for 28 days. Then the 28 orthodontic mini-implants were washed with distilled water, sonicated three times, and dried before further analysis (Fig. 1)

Figure 1. (a.) Titanium alloy orthodontic mini-implant, (b.) Four groups of mouthwashes, (c.) Orthodontic mini-implant immersed in four groups of mouthwashes.

Scanning Electron Microscopy and Energy-Dispersive X-Ray Spectroscopy Analysis

The heads and necks of all orthodontic mini-implants were examined for surface topography using scanning electron microscopy (SEM) and the elemental composition using energy-dispersive x-ray spectroscopy (EDS). The SEM examination was performed to obtain a qualitative analysis of the surface topography of the one as-received orthodontic mini-implant and the 28 orthodontic mini-implants that had been immersed in mouthwashes or water. Surface topography was done using FEI type Inspect F50 at the Center for Materials Processing and Failure Analysis, Universitas Indonesia at 15kV with magnification of 100 x to 10.000 x. The elemental composition of Ti, Al, and V was analyzed using EDAX type Apollo X at the same place. EDS examination was performed at three spots that showed signs of corrosion or roughness found during SEM analysis.

Statistical Analysis

The Statistical Package for Social Science (SPSS) version 20 software (IBM Corp., Armonk, NY, USA) was used. Means and standard deviations were calculated and tested for normality with Saphiro–Wilk. Kruskal–Wallis and Mann–Whitney tests were performed to compare the mean atomic percentage for Ti, Al, and V in the four groups. Fisher's Exact test (expected count < 5) was performed to compare the surface topography of the orthodontic mini-implants in the four groups, using the categories smooth, rough, crevice, and pitting corrosion. A p-*value* < *.05* was considered to be statistically significant.

Results

Scanning Electron Microscopy

The SEM images of the as-received orthodontic mini-implant showed some manufacturing defects. Even without immersion, there were some surface imperfections in the form of scratches and spots on the head and neck part of that orthodontic mini-implant surface (Fig. 2).

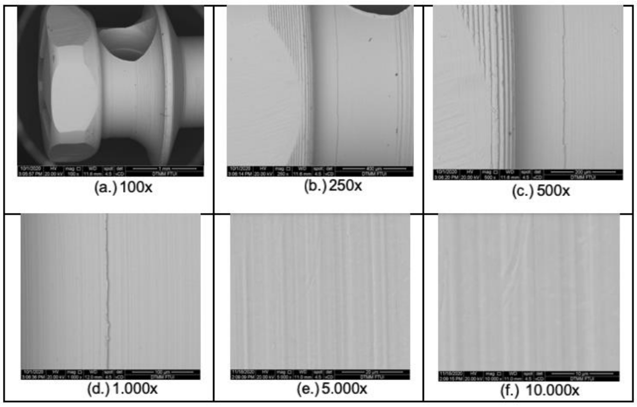

Figure 2. Surface topography of the as-received titanium alloy orthodontic mini-implant assessed by SEM with magnification (a.)100x, (b.)250x, (c.)500x, (d.)1.000x, (e.)5.000x, and (f.)10.000x.

The SEM images of the orthodontic mini-implants immersed in chlorhexidine gluconate 0.2%, sodium fluoride 0.2%, chitosan 1.5%, and distilled water also exhibited some manufacturing defects. The chlorhexidine gluconate and sodium fluoride groups showed rougher surfaces, but no sign of crevice or pitting (Fig. 3 dan Fig. 4). The chitosan and distilled water groups showed more samples with smooth surfaces and no sign of crevice or pitting corrosion (Fig. 5 and Fig. 6).

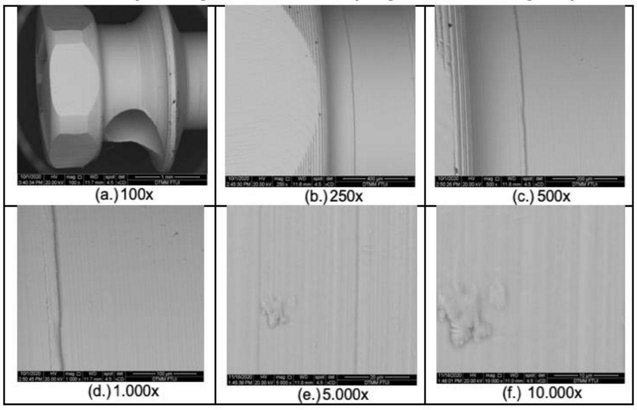

Figure 3. Surface topography representation of the titanium alloy orthodontic mini-implant immersed in chlorhexidine gluconate assessed by SEM with magnification (a.)100x, (b.)250x, (c.)500x, (d.)1.000x, (e.)5.000x, and (f.)10.000x.

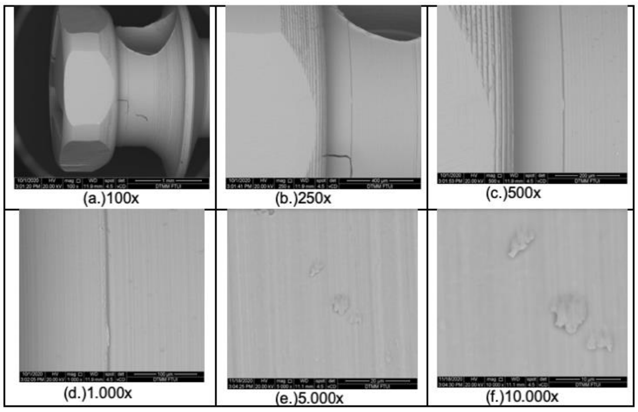

Figure 4. Surface topography representation of the titanium alloy orthodontic mini-implant immersed in sodium fluoride assessed by SEM with magnification (a.)100x, (b.)250x, (c.)500x, (d.)1.000x, (e.)5.000x, and (f.)10.000x.

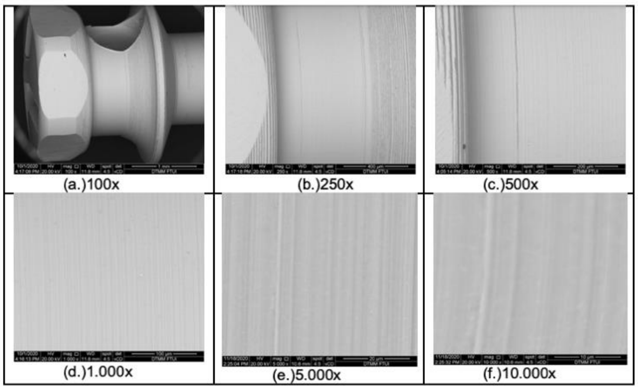

Figure 5. Surface topography representation of the titanium alloy orthodontic mini-implant immersed in chitosan assessed by SEM with magnification (a.)100x, (b.)250x, (c.)500x,

(d.)1.000x, (e.)5.000x, and (f.)10.000x.

The differences in surface topography between chlorhexidine gluconate, sodium fluoride, chitosan, and distilled water groups is shown in Table 1. There was no statistically significant difference in surface topography between the groups. The chitosan group contained more samples with smooth surfaces than the other groups.

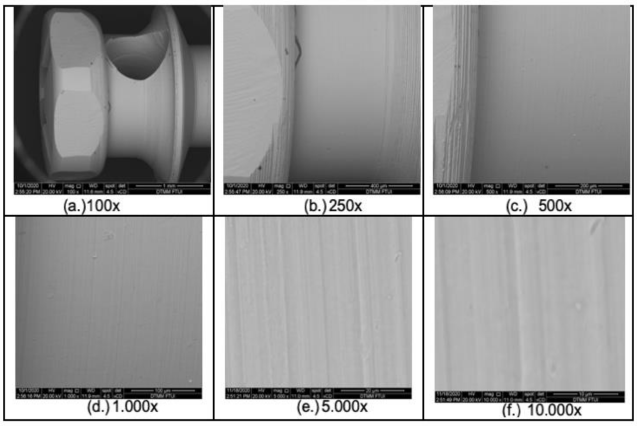

Figure 6. Surface topography representation of titanium alloy orthodontic mini-implant immersed in distilled water assessed by SEM with magnification (a.)100x, (b.)250x, (c.)500x, (d.)1.000x, (e.)5.000x, and (f.)10.000x.

Mouthwash Group	Smooth surface	Rough surface	Crevice and pitting corrosion	P-value
Chlorhexidine gluconate	3 (42.9%)	4 (57.1%)	0 (0%)	1
Distilled water	4 (57.1%)	3 (42.9%)	0 (0%)	
Sodium Fluoride	3 (42.9%)	4 (57.1%)	0 (0%)	1
Distilled water	4 (57.1%)	3 (42.9%)	0 (0%)	
Chitosan	6 (85.7%)	1 (14.3%)	0 (0%)	0.559
Distilled water	4 (57.1%)	3 (42.9%)	0 (0%)	
Chlorhexidine gluconate	3 (42.9%)	4 (57.1%)	0 (0%)	1
Sodium Fluoride	3 (42.9%)	4 (57.1%)	0 (0%)	
Chlorhexidine gluconate	3 (42.9%)	4 (57.1%)	0 (0%)	0.266
Chitosan	6 (85.7%)	1 (14.3%)	0 (0%)	
Sodium Fluoride	3 (42.9%)	4 (57.1%)	0 (0%)	0.266
Chitosan	6 (85.7%)	1 (14.3%)	0 (0%)	

Table 1. Comparison of surface topography of titanium alloy orthodontic mini-implant immersed in chlorhexidine gluconate, sodium fluoride, chitosan, and distilled water, assessed by SEM.

Energy-Dispersive X-Ray Spectroscopy

EDS analysis of the elemental composition of the orthodontic mini-implant in all groups showed that titanium (Ti) was the main element of titanium alloy orthodontic mini-implant, followed by aluminum (Al) and vanadium (V) (Fig. 7). The elemental composition of orthodontic mini-implants in chlorhexidine gluconate 0.2%, sodium fluoride 0.2%, chitosan 1.5%, and distilled water groups was comparable. A statistically significant difference was found only in titanium and aluminum (at%) composition between the sodium fluoride and chitosan groups, as shown in Table 2.

Ion	Mouthwash Groups	N	Elemental composition (at%) (Mean ± SD)	*P-value*
Titanium (Ti)	Chlorhexidine gluconate	21	13.87 (0.21125)	0.538
	Distilled water	21	13.6714 (0.28084)	
	Sodium Fluoride	21	13.0243 (0.35473)	0.365
	Distilled water	21	13.6714 (0.28084)	
	Chitosan	21	14.08 (0.11067)	0.174
	Distilled water	21	13.6714 (0.28084)	
	Chlorhexidine gluconate	21	13.87 (0.21125)	0.145
	Sodium Fluoride	21	13.0243 (0.35473)	
	Chlorhexidine gluconate	21	13.87 (0.21125)	0.554
	Chitosan	21	14.08 (0.11067)	
	Sodium Fluoride	21	13.0243 (0.35473)	0.024*
	Chitosan	21	14.08 (0.11067)	
Aluminum (Al)	Chlorhexidine gluconate	21	13.87 (0.21125)	0.88
	Distilled water	21	13.6714 (0.28084)	
	Sodium Fluoride	21	13.0243 (0.35473)	0.107
	Distilled water	21	13.6714 (0.28084)	
	Chitosan	21	14.08 (0.11067)	0.554
	Distilled water	21	13.6714 (0.28084)	
	Chlorhexidine gluconate	21	13.87 (0.21125)	0.078
	Sodium Fluoride	21	13.0243 (0.35473)	
	Chlorhexidine gluconate	21	13.87 (0.21125)	0.651
	Chitosan	21	14.08 (0.11067)	
	Sodium Fluoride	21	13.0243 (0.35473)	0.02*
	Chitosan	21	14.08 (0.11067)	
Vanadium (V)	Chlorhexidine gluconate	21	2.0286 (0.07634)	0.84
	Distilled water	21	2.0138 (0.08015)	
	Sodium Fluoride	21	2.1529 (0.07542)	0.365
	Distilled water	21	2.0138 (0.08015)	
	Chitosan	21	2.1405 (0.07384)	0.268
	Distilled water	21	2.0138 (0.08015)	
	Chlorhexidine gluconate	21	2.0286 (0.07634)	0.141
	Sodium Fluoride	21	2.1529 (0.07542)	
	Chlorhexidine gluconate	21	2.0286 (0.07634)	0.379
	Chitosan	21	2.1405 (0.07384)	
	Sodium Fluoride	21	2.1529 (0.07542)	0.97
	Chitosan	21	2.1405 (0.07384)	

Table 2. Comparison of elemental composition (at%) of titanium alloy orthodontic mini-implant immersed in chlorhexidine gluconate, sodium fluoride, chitosan, and distilled water, assessed by EDS.

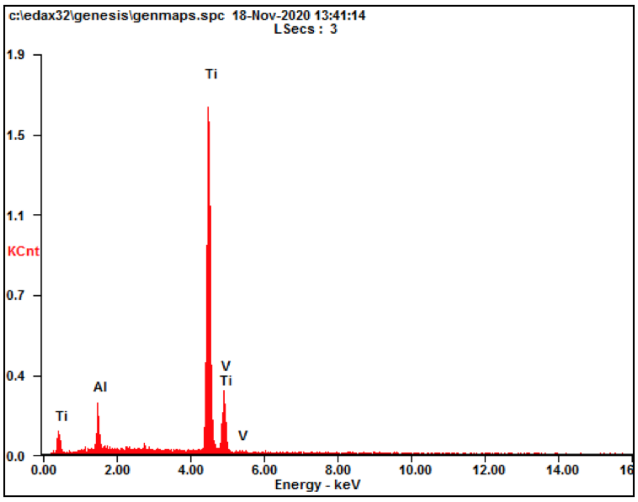

Figure 7 a. Chlorhexidine gluconate.

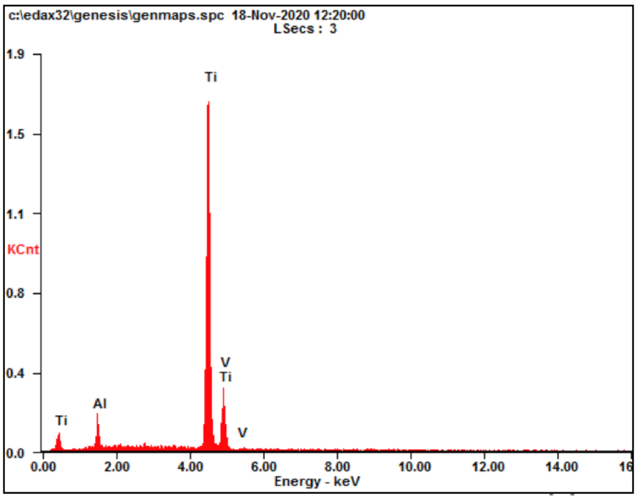

Figure 7 b. Sodium Fluoride.

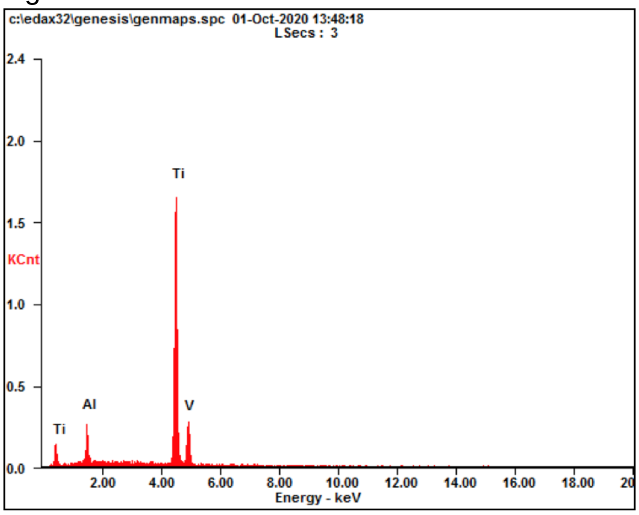

Figure 7 c. Chitosan.

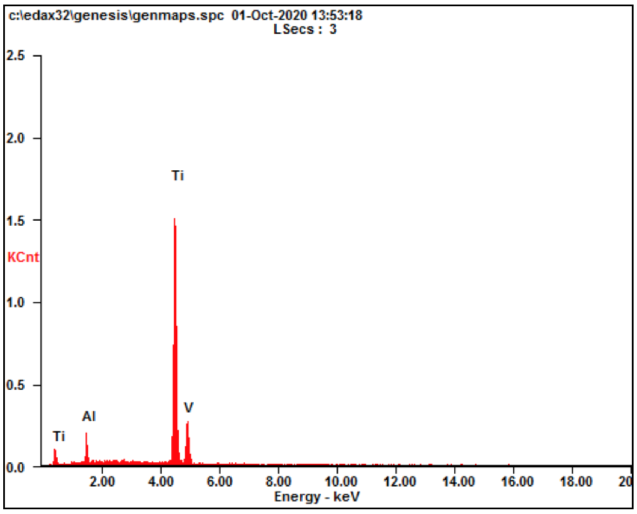

Figure 7 d. Distilled water.

Figure 7. Graphical representation of the elemental composition of titanium alloy orthodontic mini-implant immersed in (a.) chlorhexidine gluconate, (b.) sodium fluoride, (c.) chitosan, (d.) distilled water, assessed by EDS.

Discussion

The immersion period of 28 days was decided assuming that the patient had an orthodontic mini-implant for 12 months and used mouthwash twice a week. Each time the mouthwash was used, it stayed for 6 hours in a patient's mouth. Therefore, the mouthwash was present for approximately 40.000 minutes (or 28 days).[19] Abboodi et al. (2018) also demonstrated that orthodontic mini-implants showed signs of corrosion after 28 days of immersion in fluoridated mouthwash.[14] To compare the corrosion resistance of Ti alloy orthodontic mini-implants in different mouthwashes, we used distilled water as a control group, a neutral solution without any influencing ions as found in saliva. Saliva was shown to affect the corrosion resistance of orthodontic appliances.[10,20,21] Distilled water does not caused corrosion of titanium alloy.[22]

In the present study, the surface topography of the orthodontic mini-implant showed a smooth surface with some manufacturing defects in the form of scratches and spots on the head and neck. This result was similar to those of Ananthanarayanan et al. (2016), who found surface irregularities in the form of scratches in five different brands of orthodontic mini-implants.[9] Irregularities on the surface can result from the machining process, polishing defects, or deposits of crystal growth.[23] Manufacturing defects were also present in all groups immersed in mouthwash, with some loss of surface smoothness, but none showed any signs of crevice or pitting corrosion. This SEM analysis revealed results similar to those in an *in-vitro* study by Alves et al. (2006), where orthodontic mini-implants remained smooth and free from corrosion after 30 days of immersion in artificial saliva. Darkened spots of decreased corrosion resistance were shown only after 60 days of immersion.[10] This result confirms the high corrosion resistance of titanium alloy orthodontic mini-implants by the formation of a compact protective titanium oxide layer such as TiO_2, Ti_2O_3, dan TiO on the surface.[5,6]

According to SEM, there was no statistically significant difference in surface topography between the three groups of mouthwashes and distilled water. However, the chlorhexidine gluconate and sodium fluoride groups showed more samples of rough surface compared to the chitosan group. The study of elemental composition also showed that titanium, aluminum, and vanadium (at%) had statistically insignificant differences between the groups, except for titanium and aluminum (at%) between the sodium fluoride and chitosan groups. The chitosan group showed the highest mean atomic percentage of titanium and aluminum.

The greater number of samples of rough surfaces in the sodium fluoride and chlorhexidine gluconate groups in this study are thought to be related to the ability of fluoride and chloride ions to damage the protective titanium oxide layer of titanium alloy. An electrochemical study by Bhola et al. (2013) has shown that chlorhexidine gluconate mouthwash can increase the dissolution of the protective titanium oxide layer and increase the corrosivity of titanium alloy through a complex reaction between titanium and chloride ions, and also between titanium and gluconate. This, then hinders the process of re-passivation of the protective layer.[24] The SEM research by Abboodi et al. (2018) also showed that fluoridated mouthwashes could trigger corrosion of titanium alloy by damaging the protective titanium oxide layer on several part of titanium alloy orthodontic mini-implants, and seen principally at the sites of manufacturing defects.[14] The formation of a titanium-fluoride ion complex leads to a decrease of the resistance of the protective titanium oxide.[25,26]

Recent studies have shown that chitosan is an effective coating agent to improve corrosion resistance by giving titanium and titanium alloy a high inhibition efficiency.[27,28] This might explain the more samples of smooth surfaces and the highest mean atomic percentage of titanium and aluminum of the chitosan group in this study.

These findings showed that chlorhexidine gluconate, sodium fluoride, and chitosan mouthwash did not affect the corrosion resistance of titanium alloy orthodontic mini-implants, and chitosan can be considered not to cause corrosion on titanium alloy. Titanium offers high corrosion resistance because of the formation of a stable passivation layer of titanium oxide which forms spontaneously and instantaneously when contacted with oxygen and liquids.[5,6]

Conclusions

In conclusion, titanium alloy orthodontic mini-implants exhibited good corrosion resistance 28 days of immersion in chlorhexidine gluconate 0.2%, sodium fluoride 0.2%, chitosan 1.5%. Orthodontic mini-implants immersed in chitosan showed a smoother surface and higher titanium and aluminum (at%) than the orthodontic mini-implants immersed in other mouthwashes.

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Declaration of Interest

The authors report no conflict of interest.

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