International Journal of Mechanical Engineering Science and Technology

ISSN: 3163-5040 (Online)

Vol. 1, No. 2 (2026) | Page 62–73 | Received: 14 July 2026 Revised : 25 September 2026 Accepted: 26 September 2026

Corrosion Behavior of Ti-12Cr and Commercially Pure Titanium (CpTi) in AFNOR Artificial Saliva at 37°C

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A B S T R A C T

Metallic materials for orthodontic applications must exhibit good corrosion resistance, as they operate within the corrosive environment of the oral cavity. Titanium and its alloys are widely used as biomaterials owing to their favorable biocompatibility and corrosion resistance. This study aimed to evaluate the corrosion behavior of Ti-12Cr alloy under three heat-treatment conditions, namely solution treated (ST), aged for 30 ks (AT 30 ks), and aged for 60 ks (AT 60 ks), and to compare it with commercially pure titanium (CpTi) in AFNOR artificial saliva at 37°C. Corrosion testing was performed using the weight-loss method with immersion periods of 1, 2, and 3 weeks, using one specimen for each condition. Surface characterization was conducted using Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDX), while the mechanical properties were evaluated through Vickers hardness testing before and after immersion. The results showed that all materials experienced weight loss during immersion. Ti-12Cr AT 60 ks exhibited the lowest average corrosion rate at 3.88×10^{-6} mmpy, followed by Ti-12Cr AT 30 ks at 4.18×10^{-6} mmpy and Ti-12Cr ST at 5.00×10^{-6} mmpy, whereas CpTi showed the highest corrosion rate at 7.70×10^{-6} mmpy. Hardness testing revealed a decline in hardness values for all materials during immersion, with Ti-12Cr AT 30 ks maintaining the highest hardness throughout the observation period. The findings indicate that Ti-12Cr possesses better corrosion resistance than CpTi, and that increasing the aging time from 30 ks to 60 ks resulted in a further reduction in the average corrosion rate. The Ti-12Cr AT 60 ks condition yielded the highest corrosion resistance, whereas Ti-12Cr AT 30 ks retained the highest hardness after immersion. These results reveal a divergence between the corrosion resistance and hardness responses to heat treatment. Ti-12Cr demonstrates potential as a nickel-free orthodontic biomaterial with good corrosion resistance in artificial saliva solution.

K E Y W O R D S : Ti-12Cr; commercially pure titanium (CpTi); corrosion; AFNOR artificial saliva; orthodontic biomaterials.

1. INTRODUCTION

Orthodontic treatment is one of the dental procedures widely used to correct tooth alignment, improve masticatory function, and enhance oral aesthetics [1]. Orthodontic appliances consist of various components, such as brackets and archwires, which are generally manufactured from metals or their alloys [2]. During use, these components remain in continuous contact with the oral environment and saliva, causing their surfaces to be susceptible to degradation through corrosion [3]. Corrosion of orthodontic biomaterials warrants particular attention, as it can lead to the release of metal ions into the oral environment [4]. The release of such ions may affect the stability of the material and potentially trigger biological responses, including irritation, inflammation, and hypersensitivity in certain individuals [5]. The corrosion behavior of a material is influenced by several factors, including material composition, pH, electrolyte content, and environmental temperature [6]. The oral temperature,

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which is maintained at approximately 37°C, is also an important condition to consider when evaluating the corrosion resistance of orthodontic biomaterials [7].

Titanium and its alloys are widely used as biomaterials owing to their favorable biocompatibility and corrosion resistance. This resistance is primarily attributed to the ability of titanium to form a passive oxide layer on its surface [8]. Titanium-based materials are also considered an attractive alternative for orthodontic applications, as they do not contain nickel as a primary constituent, making them appealing for development as materials for patients with nickel sensitivity [9]. Ongoing development of titanium alloys continues to be pursued in order to obtain a combination of corrosion resistance and mechanical properties suited to the requirements of orthodontic applications [10]. One alloy of particular interest is Ti-12Cr. The addition of chromium can influence the phase stability and passive-layer characteristics of titanium alloys, potentially resulting in a corrosion response that differs from that of pure titanium [11]. As a comparative material, commercially pure titanium (CpTi) has been widely used as a biomaterial owing to its favorable biocompatibility and corrosion resistance.

In addition to alloy composition, heat treatment can alter the microstructural condition and properties of a material. In the Ti-12Cr alloy, solution treatment and aging can produce different microstructural conditions through changes in the distribution and formation of the α phase within the alloy matrix. Variations in aging time can lead to changes in microstructural characteristics, which in turn influence the mechanical properties and surface response of the material. Therefore, the evaluation of Ti-12Cr cannot be based solely on its alloy composition but must also take into account the heat-treatment condition applied. A comparison between the solution-treated condition and aging at different durations can provide insight into the influence of heat treatment on material behavior in a corrosive environment.

Corrosion testing of biomaterials intended for oral applications can be performed using artificial saliva solutions to simulate the physiological environment [12]. AFNOR artificial saliva contains various electrolytes used to represent the saliva environment under laboratory testing conditions [13]. Testing at a temperature of 37°C is employed to approximate the oral temperature during material use. Evaluation under such conditions is important for determining the changes in mass, surface condition, elemental composition, and mechanical properties of a material following exposure to a corrosive solution.

Based on the foregoing, research on the corrosion behavior of Ti-12Cr under different heat-treatment conditions in an artificial saliva environment remains necessary to obtain a more comprehensive understanding of the material's response. This study aims to analyze the corrosion behavior of Ti-12Cr in the solution-treated (ST), aged for 30 ks (AT 30 ks), and aged for 60 ks (AT 60 ks) conditions, with CpTi used as a comparative material, in AFNOR artificial saliva at 37°C. Corrosion testing was conducted using the weight-loss method with immersion periods of 1, 2, and 3 weeks. Surface and elemental composition changes were analyzed using Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDX), while changes in mechanical properties were evaluated through Vickers hardness testing before and after immersion. The findings of this study are expected to provide insight into the influence of heat treatment on the corrosion resistance and hardness changes of Ti-12Cr in an environment simulating oral cavity conditions.

2. METHODOLOGY

This experimental study was conducted to evaluate the corrosion behavior and hardness changes of Ti-12Cr under three heat-treatment conditions, namely solution treated (ST), aged for 30 ks (AT 30 ks), and aged for 60 ks (AT 60 ks), with CpTi used as a comparative material. The testing was carried out in several stages, comprising specimen preparation and initial characterization, immersion in AFNOR artificial saliva at pH 5 and a temperature of 37°C for 1, 2, and 3 weeks, measurement of mass loss and calculation of the corrosion rate, and surface and hardness characterization after immersion. The overall research procedure is illustrated in Figure 1.

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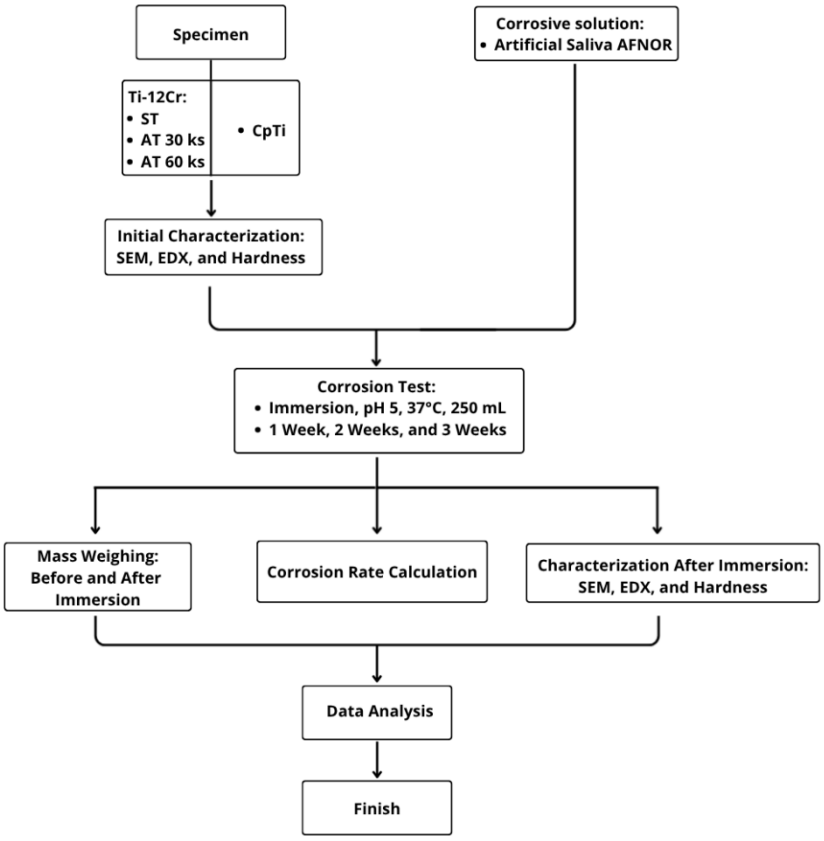


Figure 1. Schematic diagram of the research procedure.

2.1 Material and Specimen Preparation

The materials used in this study consisted of the Ti-12Cr alloy and commercially pure titanium (CpTi). The Ti-12Cr alloy originated from a single batch of material and was prepared in three heat-treatment conditions, namely solution treated (ST), solution treated followed by aging for 30 ks (Ti-12Cr AT 30 ks), and solution treated followed by aging for 60 ks (Ti-12Cr AT 60 ks). The ST condition was obtained by heating the material to a temperature of 885°C, followed by rapid cooling through water quenching. Subsequently, the specimens for the AT 30 ks and AT 60 ks conditions underwent an aging treatment at a temperature of 540°C, with holding times of 30 ks (8.3 hours) and 60 ks (16.7 hours), respectively. This variation in aging time was employed to produce different microstructural conditions, particularly with respect to the development and formation of α-phase precipitates.

All materials were subsequently cut into disk-shaped specimens for testing. The specimen surfaces were prepared through sequential grinding using silicon carbide (SiC) abrasive papers with grit sizes of 400, 800, 1200, 1500, and 2000. Grinding was performed successively until a smooth, uniform surface, free of visible scratches, was obtained and ready for corrosion testing. An example of a prepared specimen is shown in Figure 2.

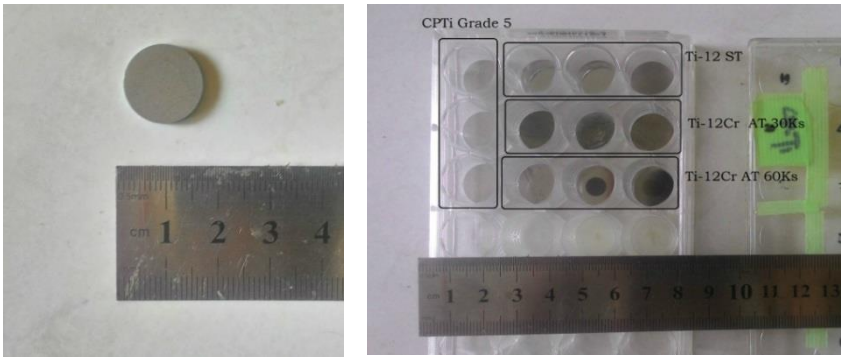


Figure 2. Ground specimen sample

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2.2 AFNOR Artificial Saliva Test Solution

The corrosive medium used was AFNOR-type artificial saliva, with the composition per liter presented in Table 1.

Table 1. Composition of AFNOR artificial saliva solution

Elemen	NaCl	KCl	$KH2PO_4$	$NaHCO_3$	NaH2PO4	KSCN	Urea
g/l	0,7	1,2	0,2	1,5	0,26	0,33	0,13

The solution pH was adjusted to 5, lower than the neutral condition, to represent the acidic tendency of the oral cavity resulting from daily dietary patterns. During testing, the solution temperature was maintained constant at 37°C using a circulating heating system, so that the testing environment closely approximated the physiological conditions of the human oral cavity during the clinical use of orthodontic biomaterials [14].

2.3 corrosion testing using the immersion method

The testing procedure in this study comprised initial material characterization, corrosion testing, post-corrosion surface characterization, and hardness testing. Prior to immersion, all specimens were characterized using SEM to observe the surface morphology and EDX to determine the elemental composition. The results of this initial characterization served as a baseline for evaluating changes in the surface and composition of the material after corrosion testing.

Corrosion testing was performed using the immersion (weight-loss) method based on the general principles of ASTM G31. Each specimen was immersed in 250 mL of AFNOR artificial saliva contained in a glass vessel [15]. The vessel was placed in an acrylic bath connected to a circulating heating system to maintain the solution temperature at 37°C throughout the test. The immersion time was varied at 1, 2, and 3 weeks, with a different specimen used for each time variation. Upon reaching the designated immersion time, the specimen was removed, cleaned of corrosion products, dried, and weighed using an analytical balance with a readability of 0.0001 g. The mass loss was calculated from the difference between the mass before and after immersion, and was then used to determine the corrosion rate (CR) using Equation 1:

$$\text{Corrosin rate} = \frac{(K \times W)}{(A \times T \times D)} \quad \ldots\ldots\ldots\

3. RESULTS AND DISCUSSION

3.1 Initial Material Characterization

SEM observation of the four material conditions prior to corrosion testing revealed relatively uniform surfaces. Fine, unidirectional scratch patterns, resulting from the sequential grinding process, were observed on all specimens. No corrosion products, cracks, or distinct surface contamination were detected. This condition indicates that the specimens exhibited a relatively clean surface prior to immersion. Uniformity of the initial surface condition is important in corrosion testing, as surface characteristics can influence the interaction between the material and the corrosive solution. With a relatively uniform initial condition, morphological changes after immersion can be observed more clearly. The initial morphology of each material is shown in Figure 3.

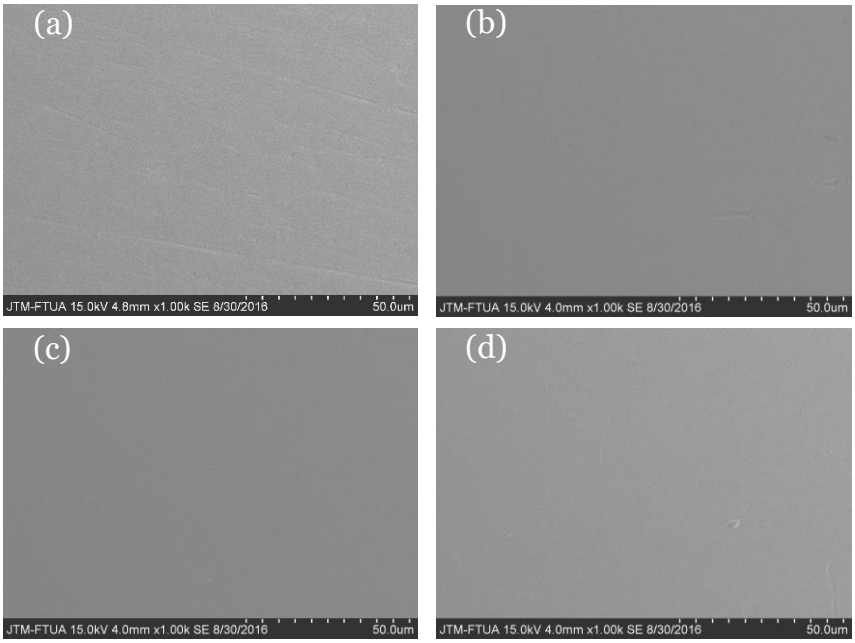
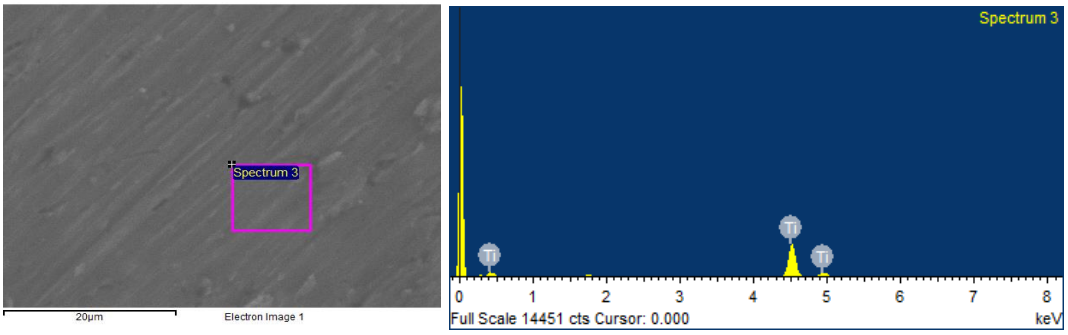


Figure 3. Microstructural observation results of (a) CpTi, (b) Ti-12Cr AT 30 ks, (c) Ti-12Cr AT 60 ks, and (d) Ti-12Cr ST.

EDX characterization was subsequently performed to confirm the elemental composition of each material. The quantification results are presented in Table 2, while the analysis point locations and EDX spectra are shown in Figure 4.

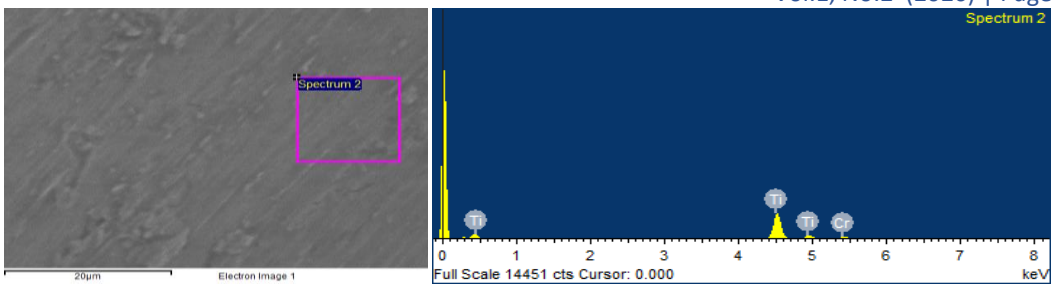
Table 2. Initial chemical composition prior to corrosion testing

Material	Ti (%)	Cr (%)
CpTi	100,00	–
Ti-12Cr AT 30 ks	89,09	10,91
Ti-12Cr AT 60 ks	88,63	11,37
Ti-12Cr ST	88,08	11,92

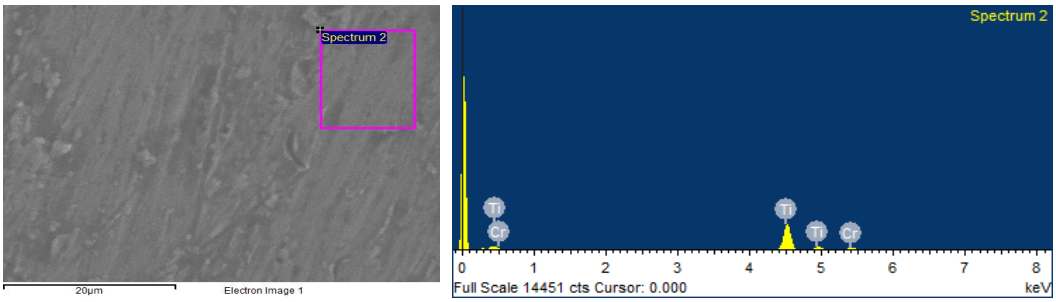


(a) CpTi

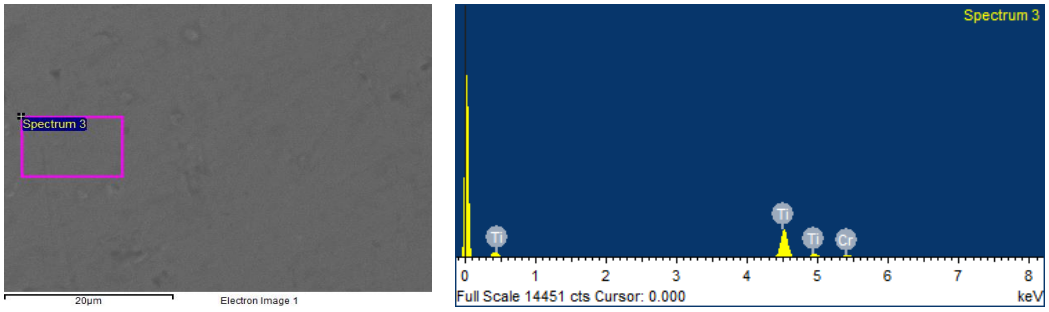
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(b) Ti-12Cr AT 30 ks



(c) Ti-12Cr AT 60 ks



(d) Ti-12Cr ST

Figure 4. Analysis point locations (left) and EDX spectra (right) of (a) CpTi, (b) Ti-12Cr AT 30 ks, (c) Ti-12Cr AT 60 ks, and (d) Ti-12Cr ST.

The spectrum of CpTi in Figure 4a shows only the characteristic peaks of titanium, namely Ti Lα at approximately 0.45 keV and Ti Kα in the range of 4.5–4.9 keV. No peaks indicating the presence of other alloying elements were observed. In contrast, the three Ti-12Cr specimens in Figure 4b–d exhibit an additional peak at approximately 5.4 keV, corresponding to the characteristic Cr Kα line. This difference in spectral pattern indicates that chromium was detected only in the Ti-12Cr specimens, consistent with the designed alloy composition. The EDX quantification results further show that the Cr content in the three Ti-12Cr conditions is close to the nominal composition of 12% Cr. The slight differences among the specimens indicate that the ST treatment and the variation in aging time did not produce a significant change in the primary chemical composition within the measurement scale of EDX. Accordingly, the three Ti-12Cr specimens possess a relatively consistent base composition, so that the differences in corrosion behavior observed in the subsequent stage can be attributed primarily to the influence of heat treatment and the resulting microstructural changes.

3.2 Corrosion Behavior of Ti-12Cr and CpTi

The immersion test showed that all materials experienced an increase in weight loss with increasing immersion time, as a result of the continuous interaction between the metal surface and the AFNOR artificial saliva solution. The corrosion rate calculated from the weight loss is presented in Table 3, while the average value for each material condition is shown in Figure 5.

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Table 3. Corrosion rate of Ti-12Cr and CpTi at various immersion times

Material	1 week	2 weeks	3 weeks	Average
Ti-12Cr AT 60 ks	$4{,}49\times10^{-6}$	$3{,}45\times10^{-6}$	$3{,}71\times10^{-6}$	$3{,}88\times10^{-6}$
Ti-12Cr AT 30 ks	$4{,}44\times10^{-6}$	$4{,}44\times10^{-6}$	$3{,}68\times10^{-6}$	$4{,}18\times10^{-6}$
Ti-12Cr ST	$6{,}37\times10^{-6}$	$5{,}66\times10^{-6}$	$2{,}97\times10^{-6}$	$5{,}00\times10^{-6}$
CpTi	$8{,}05\times10^{-6}$	$7{,}95\times10^{-6}$	$7{,}09\times10^{-6}$	$7{,}70\times10^{-6}$

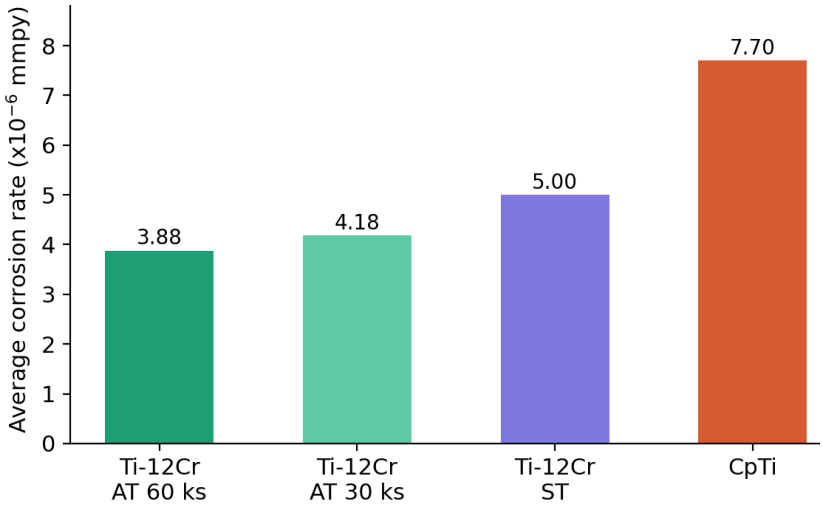


Figure 5. Comparison of the average corrosion rates of the four material conditions.

The immersion test results show that Ti-12Cr exhibited a lower corrosion rate than CpTi throughout the entire immersion period. The average corrosion rate of CpTi reached 7.70×10^{-6} mmpy, whereas Ti-12Cr ST, AT 30 ks, and AT 60 ks recorded 5.00×10^{-6}, 4.18×10^{-6}, and 3.88×10^{-6} mmpy, respectively. This difference indicates an improvement in corrosion resistance following the addition of Cr to titanium. This finding is consistent with previous research [17] on Ti-Cr systems in an artificial saliva environment. The addition of Cr enhances the stability of the passive layer on the alloy surface through the formation of Cr-containing oxides, thereby providing greater protection of the surface against dissolution reactions during contact with the electrolyte. A passive layer based on TiO_2 and Cr oxide constitutes an important factor in maintaining the corrosion resistance of Ti-Cr alloys.

The differences in corrosion rate among the three Ti-12Cr conditions also reflect the influence of the aging treatment. The ST condition exhibited an average corrosion rate of 5.00×10^{-6} mmpy, which decreased to 4.18×10^{-6} mmpy after aging for 30 ks and to 3.88×10^{-6} mmpy after aging for 60 ks. This pattern indicates that increasing the aging time from 30 ks to 60 ks resulted in a progressively lower corrosion response. This change is consistent with the microstructural characteristics of Ti-12Cr reported in [18] under the same heat-treatment conditions. That study showed that the aging process produced α precipitates within the β matrix, with finer precipitates observed at an aging time of 60 ks compared with 30 ks. The increase in aging time also resulted in increased hardness from the ST condition through to 60 ks. These findings indicate that the microstructural changes were accompanied not only by changes in mechanical properties but also by changes in the material's response to the corrosive environment.

Overall, the results indicate that Ti-12Cr exhibited a lower corrosion rate than CpTi, and that the aging process produced a further reduction in the corrosion rate of the alloy. The AT 60 ks condition yielded the lowest average corrosion rate. These results extend previous findings on Ti-12Cr, which had previously focused on microstructural and mechanical property changes resulting from aging, by showing that the same treatment also produces differences in corrosion behavior in an artificial saliva environment.

3.3 Surface Morphology and Chemical Composition

SEM observation was performed to evaluate the changes in surface morphology during immersion in AFNOR artificial saliva. The observation results of Ti-12Cr AT 60 ks from the initial condition up to 3 weeks of immersion are shown in Figure 6.

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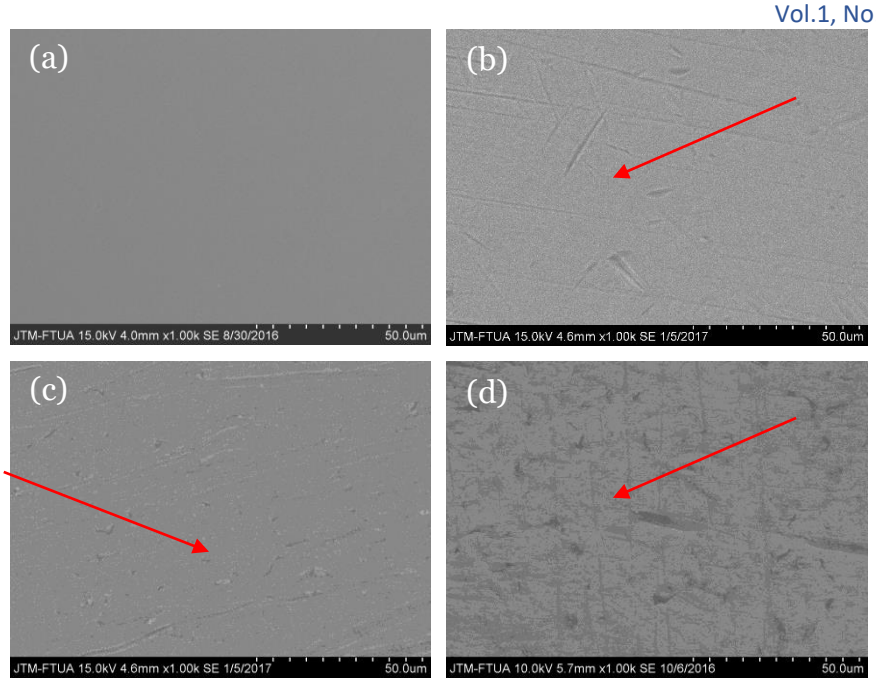


Figure 6. Surface morphology of Ti-12Cr AT 60 ks under the conditions of (a) before testing, (b) 1 week, (c) 2 weeks, and (d) 3 weeks of immersion.

In the initial condition, the surface of Ti-12Cr AT 60 ks was dominated by fine, unidirectional scratches originating from the grinding process. After 1 week of immersion, the surface pattern began to show changes in several areas, marked by the appearance of surface features with differing contrast. These changes became more pronounced at the second and third weeks, when the surface exhibited a more non-uniform texture compared with the initial condition. This morphological change indicates alterations in the surface layer as the material interacted with the artificial saliva solution.

The surface morphology of CpTi is shown in Figure 7. The initial condition likewise exhibited scratch patterns resulting from the grinding process. After immersion, the scratch pattern remained visible throughout the observation period, although the surface texture changed progressively. These changes were evident from the appearance of areas with different contrast and texture compared with the initial surface, particularly after longer immersion times.

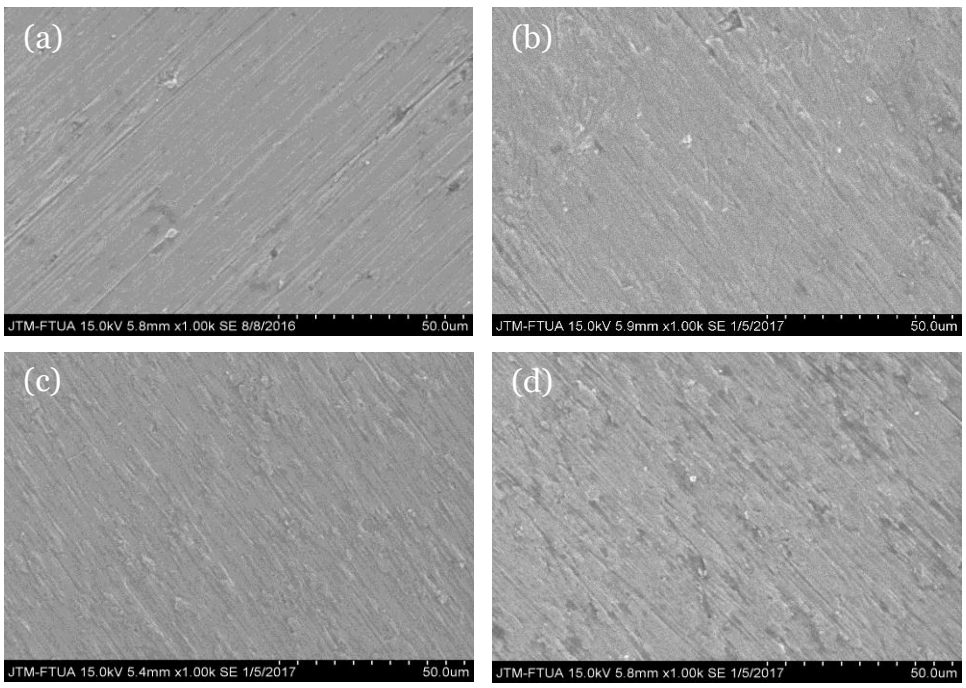


Figure 7. Surface morphology of CpTi under the conditions of (a) before testing, (b) 1 week, (c) 2 weeks, and (d) 3 weeks of immersion.

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A comparison of the two materials shows that surface changes occurred during immersion, but the characteristics of these changes differed. Ti-12Cr AT 60 ks retained a relatively uniform morphology during the early stage of immersion, whereas the change in surface texture of CpTi occurred progressively up to the third week. These SEM observations provide visual evidence that exposure to the solution caused changes in the surface layer, which warrants further examination in terms of changes in elemental composition.

To determine the chemical changes on the surface, EDX analysis was performed before and after immersion. The results of the elemental composition analysis are presented in Table 4.

Table 4. Surface elemental composition before and after immersion

Material	Element	Before	1 week	2 weeks	3 weeks
CpTi	Ti	100,00	78,00	59,97	52,81
	O	0,00	22,00	40,03	47,19
Ti-12Cr AT 30 ks	Ti	89,09	70,58	72,73	24,98
	Cr	10,91	16,86	7,06	30,54
	O	0,00	12,56	20,20	44,48
Ti-12Cr AT 60 ks	Ti	88,63	60,07	73,76	77,40
	Cr	11,37	11,36	8,49	13,90
	O	0,00	28,56	17,15	8,70
Ti-12Cr ST	Ti	88,08	64,93	53,19	83,79
	Cr	11,92	17,94	15,52	3,12
	O	0,00	17,14	31,29	13,09

The EDX data show that all specimens underwent changes in surface composition after immersion. For CpTi, the Ti content decreased from 100% to 52.81% at the third week, while O increased from 0.00% to 47.19%. In the Ti-12Cr alloy, the change in O content exhibited a different pattern for each heat-treatment condition. AT 30 ks showed an increase in O up to 44.48% at the third week; ST reached 31.29% at the second week before decreasing to 13.09%; whereas AT 60 ks showed a decrease in O from 28.56% at the first week to 8.70% at the third week. Cr remained detectable in all Ti-12Cr conditions after immersion. For AT 60 ks, the Cr content remained relatively stable throughout testing, reaching 13.90% at the third week, whereas the changes in Cr content for AT 30 ks and ST were greater, particularly at the third week. These changes in Ti, Cr, and O content indicate alterations in the surface layer during the immersion process.

The EDX results also show that the O content did not follow the same pattern across all materials. Therefore, the low corrosion rate of Ti-12Cr AT 60 ks cannot be explained solely on the basis of its surface O content. The microstructural condition formed during aging, as well as the stability of the surface layer during immersion, also contribute to determining the corrosion response of the material [19]. This is consistent with the weight-loss results, in which AT 60 ks exhibited the lowest average corrosion rate of 3.88×10^{-6} mmpy. Overall, the SEM and EDX results indicate changes in surface morphology and composition during immersion. These changes provide insight into the processes occurring on the material surface and support the corrosion rate results obtained for Ti-12Cr and CpTi.

3.4 Effect of Corrosion on Material Hardness

Hardness testing showed that all materials experienced a decrease in hardness value after immersion in AFNOR artificial saliva. The change in hardness value with immersion time is shown in Figure 8.

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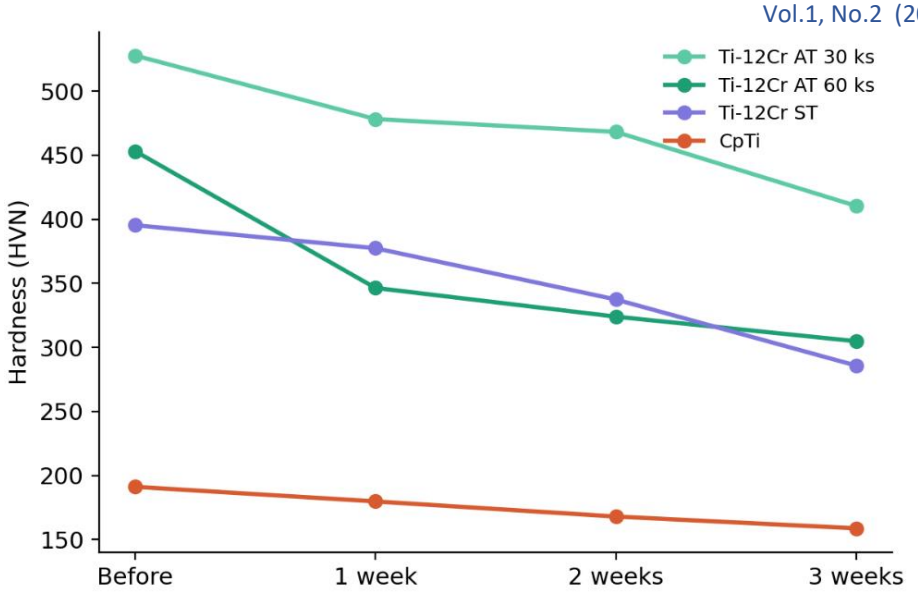


Figure 8. Hardness values of the four material conditions with respect to immersion time.

The Vickers test results show that all materials experienced a decrease in hardness after immersion in AFNOR artificial saliva. Ti-12Cr AT 30 ks had the highest initial hardness, at approximately 530 HVN, followed by AT 60 ks at approximately 450 HVN and ST at approximately 390 HVN, whereas CpTi had the lowest value, at approximately 190 HVN. After immersion, the hardness values of all materials decreased progressively up to the third week. At the end of testing, AT 30 ks retained the highest hardness, at approximately 410 HVN, whereas AT 60 ks and ST were at approximately 300 and 280 HVN, respectively. CpTi exhibited the smallest change, decreasing from approximately 190 to 160 HVN.

The decrease in hardness with increasing immersion time indicates changes in the surface condition of the material during contact with the solution. The corrosive environment can alter the surface layer, which subsequently affects the material's response to penetration by the Vickers indenter. Research [20] on titanium materials for oral environments has similarly shown that surface condition plays an important role in both corrosion behavior and mechanical characteristics. That study showed that surface modification of Ti-6Al-4V affects both its surface characteristics and its corrosion behavior in artificial saliva.

The difference in hardness among the heat-treatment conditions reflects the influence of the aging process on the structure and mechanical properties of Ti-12Cr. The higher initial hardness of AT 30 ks compared with ST indicates that the aging process produced strengthening of the alloy. Recent studies on Cr-containing titanium alloys have shown that aging promotes the precipitation of the α phase within the β matrix and increases hardness through precipitation strengthening. Increasing the aging time and temperature can alter the amount, size, and distribution of the α phase, thereby resulting in changes to the hardness and strength of the material.

Interestingly, the hardness pattern after immersion did not follow the same order as the corrosion rate. AT 30 ks retained the highest hardness up to the third week, whereas AT 60 ks exhibited the lowest average corrosion rate. This indicates that hardness and corrosion resistance exhibit different responses to the aging treatment. Studies on heat-treated titanium alloys in artificial saliva have also shown that microstructural changes can influence corrosion behavior through changes in the stability of the passive film on the surface. These results therefore indicate that the aging treatment affects two material characteristics differently: microstructural change results in differences in hardness, whereas surface condition and passive film stability determine the corrosion response during immersion.

CONCLUSION

Based on the study of the corrosion behavior of Ti-12Cr and CpTi alloys in AFNOR artificial saliva at a temperature of 37°C, the following conclusions were obtained:

1. The average corrosion rate indicates that the Ti-12Cr alloy exhibits better corrosion resistance than CpTi. Ti-12Cr AT 30 ks had an average corrosion rate of 4.18×10^{-6} mmpy, Ti-12Cr AT 60 ks of 3.88×10^{-6} mmpy, and Ti-12Cr ST of 5.00×10^{-6} mmpy, whereas CpTi had a rate of 7.70×10^{-6} mmpy. The lowest corrosion rate was obtained for Ti-12Cr AT 60 ks, while the highest was obtained for CpTi. These results indicate that the aging treatment applied to Ti-12Cr improved its corrosion resistance compared with the solution-treated condition.

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2. The hardness values of CpTi and the Ti-12Cr alloy decreased with increasing immersion time. After three weeks of immersion, Ti-12Cr AT 30 ks had the highest hardness value at 410.4 HVN, whereas CpTi had the lowest hardness value at 158.8 HVN. These results indicate that heat treatment influences the material's ability to retain its hardness during immersion in artificial saliva.

AUTHORS' CONTRIBUTIONS

AAA and **G** contributed to the conceptualization of the study, methodology development, supervision, formal analysis, data validation, as well as writing the original draft and reviewing the manuscript. **AAA** and **JA** were responsible for investigation, data curation, formal analysis, data visualization, and preparation of the original draft. Meanwhile, **G** and **JA** contributed to conceptualization, methodology development, project supervision, project administration, and manuscript review and editing.

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