Corrosion Characteristics Of Titanium Ti-12Cr And Cp-Ti In Artificial Saliva Afnor At Human Body Temperature

Sir Anderson, Aguswan, Jon Affi, Yuli Yetri, Gunawarman

Abstract: Corrosion behavior of ß-type titanium alloy, Ti-12Cr, has been investigated within artificial saliva solution to acquire its potential as orthodontic application. Solution treated alloy (Ti-12CrST), aging treated alloy (Ti-12CrAT 60 ks, Ti-12CrAT 30 ks), and CpTi, as a comparator for these two materials, have been immersed in solution at constant temperature 37°C and pH 5 to imitate human body condition. Corrosion behavior was carried out by applying weight loss method with different exposure times (1, 2 and 3 weeks). Surface morphology was analyzed by using scanning electron microscopy (SEM). Energy dispersive X-Ray (EDX) was used to determine thechemical compositions. The results reveal that the corrosion rate of Ti-12CrAT 60ks, Ti-12CrAT 30ks, Ti-12CrST, and Cp-Ti alloys are 3.88x10-6mmpy, 4.18x10-6mmpy, 4.50x10-6mmpy, and 7.70 x10-6mmpy, respectively. The corrosion rate of all Ti-12Cr types is lower than that of the Cp-Ti. The hardness of Ti-12Cr and Cp-Ti alloys decreased by the exposure time. Ti-12CrAT 30ks with 410.4 HVN has the highest hardness value while Cp-Ti with 158.8 HVN has the lowest one. Low oxygen content on the Ti-12CrAT 60ks surface indicates its corrosion resistance. It can be concluded that the best material for orthodontic application from the corrosion resistance view is Ti-12CrAT 60ks.

Index Terms: Afnor, Artificial Saliva, Corrosion Rate, CpTi, SEM-EDX, Temperature, Ti-12Cr.

1 INTRODUCTION

Orthodontic treatment, or mouth and dental treatment,has been used by quite many people. One kind of orthodontic treatment primarily applied is the use of dental braces. The function of dental braces is to improve the irregularity of dental formation, defend the support dental structure, produce occlusion contact that is good, and improve the aesthetic value of the mouth, especially for the tooth [1]. The main components of orthodontic orders arebracket, auxiliary, and archwire [2]. Some components are made of metal. However, if all of the components are made of metal, the metal component will react with saliva or saliva inside the mouth cavity. Metal has the potential to cause allergy related to corrosion. Corrosion is a process of releasing ions from a combination as the formation element goes back to its original shape [2,3]. The process of corrosion causes a reaction in the mouth. Some users of orthodontic devices talk about allergy reaction caused by metal's contact with skin. Among the reactions are mouth edema, tongue, mouth lining, and anaphylaxis. The reaction occurs as the orthodontic device made of metal reacts with saliva and causes corrosion. The corrosion is not dependent upon metal composition but the influence of temperature and pH of the workplace. One solution to solve the problem is using titanium as a main matter of bracket. Titanium has been used for some decades for implant matter and has been a great success for a light allergy sufferer. Thus, titanium can be used for orthodontic treatment at hypersensitive allergy1 as an alternative material for bracket matter. As an alternative allergen formetal-allergic surferers, non-metal material such as titanium □□that has 1 or more chemical elements4 has been developed for biomedical applications4-6. Titanium alloy □ has the best corrosion endurance and low modulus elasticity compared to titanium alloy α type or (α +β)type [4,5]. Titanium type □ that has more than one alloy element has a high cost. TNTZ (Ti-29Nb-13Ta-4,6Zr) [4,5] that has fatigue resistant and good pull power compared to Ti6Al4V Eli for biomedical application [6] is expensive. Thus, titanium has been improved to have little alloy such as Cr and Fe, one of Ti-12Cr [7], with a lower cost.

Research of Titanium Ti-12Cr that has been concerns with mechanic characteristic tests such as bending and determining itsmodulus elasticity [8]. However, the resistance of this material has not yet clinically tested. The resistance included corrosion resistance to artificial saliva. So, if it is used for a long time, the mechanic characteristic or the dimension of the orthodontic material does not decrease. To prove this, some corrosion tests were conducted to know the corrosion rate of the matter. By doing this, the age of the use of the matter as an alternative orthodontic matter can be predicted.

There has been some research about corrosion behavior of titanium Ti-12Cr in the body simulation liquid. The behavior of the corrosion Ti-12Cr in artificial saliva was carried out at room temperature. The result showed that the corrosion rate of Ti-12Cr is 1.57x10-7 mmpy, lower than CpTi as big as 15.3x10-7 mmpy [9]. However, the result of the test examining the value of corrosion rate and the influence of the corrosion toward the hardness of the matter used as orthodontic alternative matter conducted at 370C (agree with human body temperature condition) has not yet known. Considering the problems described above, a research entitled "The behavior of titanium corrosion Ti-12Cr and CpTi inside artificial saliva at a 370C controlled temperature"has been conducted. The objective of the research was to examine the value of corrosion rate and the influence of the corrosion toward the value of the hardness of Ti-12Cr and CPTi alloy inside artificial saliva liquid.

2 EXPERIMENTAL PROCEDURE

2.1 Preparation of the sample

The samples tested are Ti-12Cr AT 60ks, Ti-12Cr AT 30 ks, Ti-12Cr ST, and CpTi with the diameter 1.50 cm, and variation of the thickness are 0.205 cm, 0.300 cm, and 0.320 cm. The surface of the sample was sanded,with the rudeness of mesh are 400, 800, 1200, 1500, and 2000, until the surface is smooth and free from scratches (glow). A small hole, with a diameter 1.50 cm, was made in the middle of the sample as a place to hang the sample in yarn in the scuttle of liquid. The corrosion liquid used is afnor artificial saliva, whose condition resembles human saliva liquid, with the composition as presented in Table 1, at controlled temperature 370C and pH of liquid 5.0. The sample is submerged inside the liquid for 1 week, 2 weeks and 3 weeks.

TABLE 1

Composition of Artificial Saliva Afnor Liquid

Element	NaCl	KCl	KH_2PO_4	$NaHCO_3$	NaH_2PO_4	KSCN	Urea
g/L	0.7	1.2	0.2	1.5	0.26	0.33	0.13

2.2 Analysis of Microstructure, Chemical Composition, and Hardness Test

The microstructure of the sample is analyzed using optic microscopic and SEM (Scanning Electron Microscope). Examining the chemistry Composition at the surface of the sample used EMAX x-act liquid nitrogen less X-ray Detector. Measuring the hardness of Ti-12Cr refers to ASTM 384; standard test methods for Knoop and Vickers Hardness Materials [10]. Indentor that used in this test is a rectangular pyramid with angle 1360 and the loaded value as big as 9.8 N with indentation time 10-15 seconds.

2.3 The Rate of Corrosion

Before the test, the weight of the samples was measured. Immersion test, with the steep sample inside the corrosion liquid (artificial saliva Afnor), for 1 week, 2 weeks, and 3 weeks was used.After steeping, the sample was measured again and the rate of corrosion was counted using formula (1) [11]:

$$CR = \frac{(W \times K)}{(D \times A \times T)} \quad .... (1)$$

where,

CR = Corrosion Rate (mmpy or millimeter/year)

W = Lost Weight (gram)

K = Factor of corrosion rate, Constanta (8.76 x 104)

D = Density of corrosion good tests (gram/cm3)

T = Time (hour)

3 RESULT AND DISCUSSION

3.1 The Characteristics before the Immersion

The result of microstructure observation using an optic microscope and SEM (Scanning Electron Microscope) shows that every type of matter has a smooth surface. However, at the sample, CpTi still has little scratches (Fig. 1), which is siilar with the result of another study (Fig.2) [12].

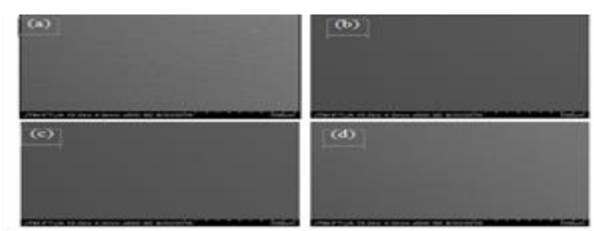

***Fig.1**. The result of microstructure alloy (a) CpTi (b)Ti-12Cr AT 30 ks. (c) Ti-12Cr AT 30ks. (d) Ti-12Cr ST.*

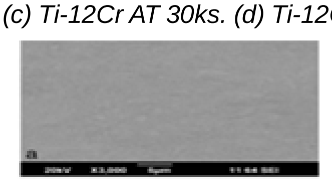

***Fig.2**. The result of microstructure alloy CpTi in other research [12].*

Fig.3 shows the result of the test to the initial chemical composition of every alloy using EDX. It can be seen that the chemical composition of CpTi alloy has 100% titanium. This result is in accordance with the type of a material namely Commercially Pure Titanium. So, it can be sure that the matter which will be tested for corrosion rate test is CpTi.

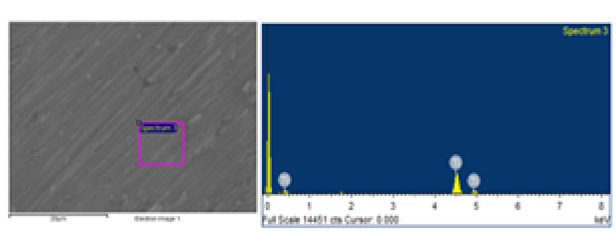

***Fig.3.** (a).CpTi Chemistry composition obsevation area (b) CpTi Chemistry composition spectrum.*

The result of EDX test to the composition alloy of Ti-12Cr AT 30ks revealed that this alloy has 89.09% titanium atom and 10.91% chrome atome. Theoretically, Ti-12Cr has 12 % atom so that the result of the value obtained shas 1% difference for chrome (Fig. 4).

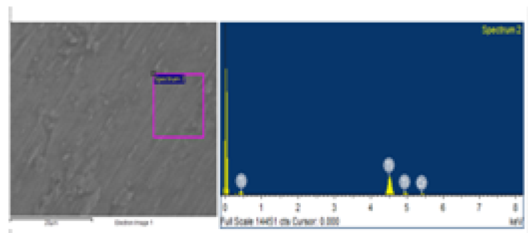

***Fig.4**. (a). Ti-12Cr AT 30 ks chemistry composition obvervation area (b) Ti-12Cr AT 30 ks test spectrum.*

The result of composition test for Ti-12Cr AT 60 ks obtained in surface areas (Fig. 5) indicates that it has 88.63% titanium atom and 11.37% chrome. This then proved that alloy tested is Ti-12Cr.

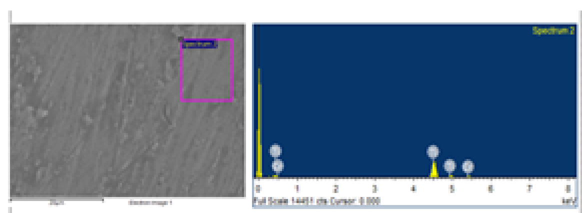

***Fig.5**. (a) Ti-12Cr AT 60 ks chemistry composition obsevation area (b) Ti-12Cr AT 60 ks.*

The same compositions were also found for Ti-12 Cr ST alloy; the percentage of atom degree is not different from the degree mentioned in the theory (Ti-12Cr has 12% chrome atom). The result of the composition test (Fig. 6) is 88.08% titanium atom and 11.92% chrome.

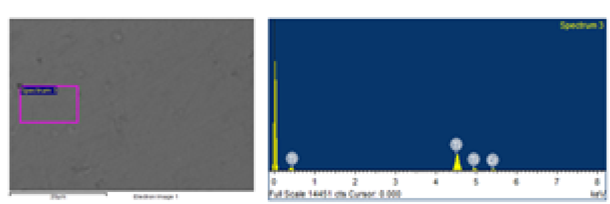

***Fig.6**. (a) Ti-12Cr ST chemistry observation test*

(b) Ti-12Cr ST test Spectrum.

3.2 Corrosion Behavior

3.2.1 Mass Reduction

After tested with a variation of immersion, 1 week, 2 weeks, and 3 week, all samples soaked in artificial saliva experienced mass reduction. The mass reduction occured due to the corrosion process on the material surface. As presented in Table 2, the lost mass varies for each sample. Ti-12Cr AT 60 ks alloy and Ti-12Cr AT 30 ks alloy soaked for 1 week are the materials with the lowest mass reduction, which is 0.0002 grams. Meanwhile, the CpTi alloy soaked for 3 weeks experienced the highest mass loss, which is 0.0008 grams.
Table 2 shows that the longer the immersion time the greater the mass reduction. This is true for every type of material, which is in line with the result obtained in the previous studies [9,13]. The biggest mass reduction occurred in 3-weeks immersion time while the lowest is in 1-week.

TABLE 2
Reduction of Specimen Mass After Immersion

Immersion Time	Mass Reduction (g)			
	Ti-12Cr AT 60 ks	Ti-12Cr AT 30 ks	Ti-12Cr ST	Cp-Ti
1 week	0.0002	0.0002	0.0003	0.00003
2 weeks	0.0003	0.0004	0.0005	0.0006
3 weeks	0.0005	0.0005	0.0004	0.0008

When compared with other studies, the result of the mass reduction of Ti-12Cr with 3% NaCl solution of 9 mgat room temperature13 was higher than that of the saliva solution (4 mg). This is because saliva solution contains a small amount of NaCl 0.7% even though the test was carried out at a higher temperature, which is 370C. The loss of mass in various types of materials also varies. Fig. 7 shows that in the 1-week immersion while the greatest mass loss occurred in the CpTi and Ti-12Cr ST material, which is 0.0003 grams, while the lowest occured in Ti-12Cr AT 60 ks and Ti-12Cr AT 30 alloys. ks, which is 0.0002 grams. Similar values occur due to the accuracy of the scale of only 0.0001 grams. The 1-week immersion variation treatment results in a low mass reduction value as well. In 2- weeks immersion variation, the lowest mass reduction is performed by Ti-12Cr AT 60 ks, which is 0.0003 grams, while the largest by CpTi, which is 0.0002 grams. The similar value of each sample can be obtained as the accuracy of weighting is 0,0001. For 1-week immersion, the mass loss is also small. For 2-weeks, Ti-12Cr AT 60 ks shows the lowest loss mass, which is 0.0003 grams while the greates loss mass is found in CpTi, which is 0.0006 grams. So, there is the value of the mass loss for each material is not the same. For 3-week immersion, the largest mass reduction occurred in CpTi material, which is 0.0008 grams while the lowest is in Ti-12Cr ST, which ia 0.0004 grams.

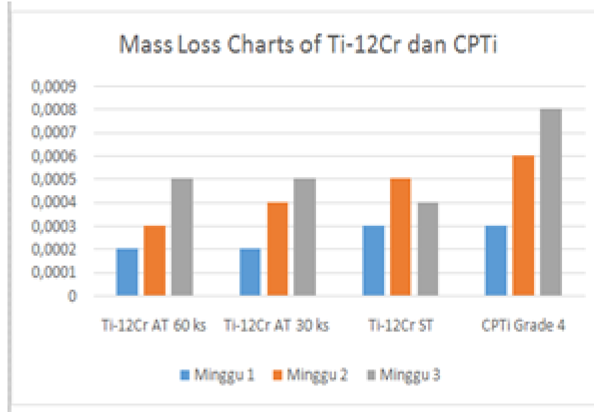

***Fig.7**. Mass loss chart of Ti-12Cr and CPTi*

3.2.2 Corrosion Rate

The results of calculation of the corrosion ratepresented in Table 3 show that the corrosion rate of Ti-12Cr AT 30 ks of 4.18x10-6 mmpy is lower than that of the Ti-12Cr ST of 4.99x10-6 mmpy. This value is consistent with the one obtained in other studies of corrosion rate, where Ti-12Cr AT 30 ks at 7.00977x10-6 mmpy is also lower than the Ti-12Cr ST of 26.6341967x10-6 mmpy [13]. The difference in the value of the corrosion rate is due to the NaCl of the corrosive solution used in this study contains more NaCl, which is 3% [13], than that of saliva solution, which isonly 0.7%.It can be seen in Table 3 that the CpTi alloy has the largest corrosion rate, which is 7.70x10-6 mmpy while Ti-12Cr AT 60 ks alloy has the lowest, which is 3.88x10-6 mmpy. This is in line with the values obtained in other studies where the greatest corrosion rate in CpTi alloy is 153x10-8 mmpy while the lowest is inTi-12Cr AT 60 ks, which is 15.67x10-8 mmpy [9]. The difference in the value of the corrosion rate in this study is because the corrosive solutions of artificial saliva are carried out at room temperature [9]. It can be said that the CpTi material is the most corroded material for the Ti-12Cr ST alloy, the Ti-12Cr AT 30 ks alloy, or the Ti-12Cr AT 60 ks alloy, which is in line with the previous studies [9].

TABLE 3
Corrosion rate values for Ti-12Cr and CpTi alloys soaked for 1 week, 2 weeks and 3 weeks

Material	Variation (week)	Weight Loss (g)	Corrosion Rate (mmpy)	Average Corrosion Rate (mmpy)
Ti-12Cr AT 60 ks	1 week	0.0002	4.49x10^{-6}	3.88x10^{-6}
	2 weeks	0.0003	3.45x10^{-6}	
	3 weeks	0.0005	3.71x10^{-6}	
Ti-12Cr AT 30 ks	1 week	0.0002	4.44x10^{-6}	4.18x10^{-6}
	2 weeks	0.0004	4.44x10^{-6}	
	3 weeks	0.0005	3.68x10^{-6}	
Ti-12Cr ST	1 week	0.0003	6.37x10^{-6}	4.99x10^{-6}
	2 weeks	0.0005	5.66x10^{-6}	
	3 weeks	0,0004	2.97x10^{-6}	
Cp-Ti	1 week	0.0003	8.05x10^{-6}	7.70x10^{-6}
	2 weeks	0.0006	7.95x10^{-6}	
	3 weeks	0.0008	7.09x10^{-6}	

3.2.3 Material Surface Morphology

The results of surface morphology analysis with SEM, as in Fig. 8, did not show significant morphological differences. This occured due to relatively short immersion variations and that the titanium material used is very resistant to corrosion so that it is only visible as black spots on the scrape marks on the material surface.

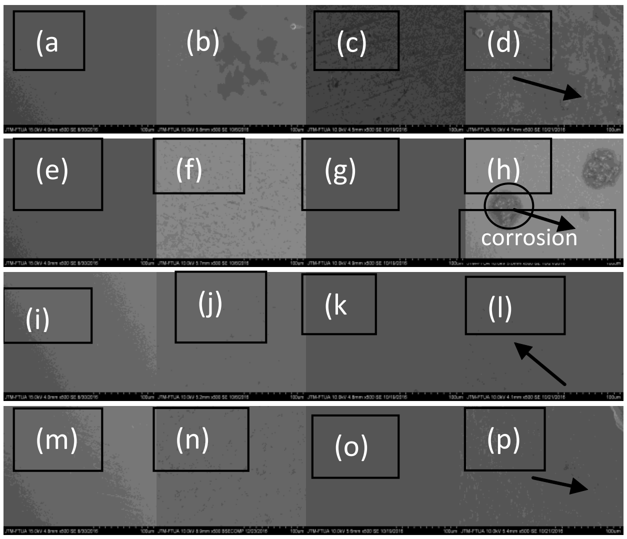

***Fig.8**. Microstructure of Ti-12Cr AT 30 Ks ((a), (b), (c), (d)), Ti-12Cr AT 60 Ks ((e), (f), (g), (h)), Ti-12Cr ST ((i), (j), (k), (l)), and CpTi ((m), (n), (o), (p)) with Optical Microscopes (100X magnification) before immersion ((a). (e), (i), (m)), 1 week immersion ((b), (f), (j), (n)), 2 weeks immersion ((c), (g), (k), (0)), 3 weeks immersion ((d), (h), (l), (p)) in saliva solution.*

Fig. 8 (a) shows that before soaking, the surface is smooth and slippery, but in the first week, the surface of the material begins to have small dots, indicating the presence of corrosion. At 2-wee immersion, the alloy surface has more corrosion than before. At 3-week immersion, the corrosion becomes wider and larger. Corrosion examples can be seen from the direction of the arrow in the image. Fig. 8 (f) shows indications of corrosion taking place in the cracks between scratches. This occured due to the surface of the material is not smooth enough. Fig. 8 (h) shows that the samecorrosion behavior of the alloy for 3-week immersion.Fig. 8 (m), (n), (o), (p) shows that the surface quality tends to be flat in a variation of 1-week immersion, with almost all corrosion on the entire surface and no holes or scratches. This shows the entire corrosion of the CpTi material.

3.2.4 Composition Analysis

The chemical composition tests using EDX is aimed to see whether corrosion process corrosion occur in all materials. As it is known, the corrosion process produces oxygen on the surface of the material. As shown in Table 4, there is no oxygen found in the initial composition before the immersion. Oxygen is found after 1-week, 2-weeks, and 3-weeks immersions. The weight of Ti elements reduced (eroded). For examples, Ti-12Cr AT 30 Ks weight reduced from 89.09% to 24.98%, resulting in 44.48% oxygen, Ti-12Cr AT 60 Ks Ti elements reduced from 88.63% to 77.40%, resulting in 8.7% oxygen, Ti-12Cr ST Ti elements reduced from 88.08% to 83.79%, resulting in13.09% oxygen, and CpTi Ti elements reduced from 100% to 52.81%, resulting in 47.19% oxygen. The production of oxygen during test indicates that corrosion has occurred on the metal surface.

TABLE 4
Chemical composition of Ti-12Cr and CpTi alloys on variations before 1-week, 2-weeks, and 3-weeks immersions

Material	Unsur Paduan	Atomic Content (%)			
		Before	1 week	2 weeks	3 weeks
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	28.56	17.15	8.70
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	12.56	20.20	44.48
Ti-12Cr ST	Ti	88.08	64.93	53.19	83.79
	Cr	11.92	17.94	15.52	3.12
	O	0	17.14	31.29	13.09
Cp-Ti	Ti	100	78	59.97	52.81
	O	0	22	40.03	47.19

3.3 Hardness

Before the immersion time, all the material is firstly tested to check their hardnessin order to determine the effect of immersion on the value of the material's hardness. The value of hardness tested using Vicker Hardness Tester is presented in Table 5. Before the immersion, the highest to lowest hardness values are alloy Ti-12Cr AT 30 ks, 527.8 HVN, alloy Ti-12Cr AT 60 ks, 453.2 HVN, alloy Ti-12Cr ST, 395.4 HVN, and CPTi, 191.2 HVN. It appears that titanium alloy has higher hardness value than pure titanium.

TABLE 5
Hardness values of Ti-12Cr and Cp-Ti alloys soaked for 1 week, 2 weeks and 3 weeks

Immersion Variations	Average Hardness Values (HVN)			
	Ti-12Cr AT 60 ks	Ti-12Cr AT 30 ks	Ti-12Cr ST	Cp-Ti
Before	453.2	527.8	395.4	191.2
1 week	346.4	478.2	377.4	179.8
2 weeks	324	468.2	337.4	168
3 weeks	304.8	410.4	285.8	158.8

The hardness value of Ti-12Cr during 3-weeks immersion can be seen in Table 5. The highest hardness value is shown by Ti-12Cr AT alloy 30 ks, which is 410.4 HVN, while the lowest is by Ti-12Cr ST, which is 285.8 HVN. The results during 6 weeks immersion are in line with those in the previous studies [13]. The highest hardness price on Ti-12Cr AT alloy 30 ks is 406 HVN while the lowest is Ti-12Cr ST, which is 326 HVN (Table 6). The difference in hardness value is caused by the difference in the immersion time.

TABLE 6
Value of the hardness of Ti-12Cr before and after soaking for 2, 4, and 6-week immersion in 3% NaCl solution [13].

Immersion Variations	Average Hardness Values (HVN)		
	Ti-12Cr	Ti-12 Cr (ST)	Ti-12Cr (AT 30 ks)
Before	286	309	390
2 week	340	385	395
4 weeks	370	386	501
6 weeks	366	326	406

Table 5 shows the comparison of the value of the hardness of Ti-12Cr and CpTi during 3-week immersion. Ti-12Cr AT 30 ks has the highest hardness value is, which is 410.4 HVN, while

CPTi has and the lowest, which is 158.8 HVN. In other studies (Table 7), during 1000 hour immersion (5.9 weeks) [9], the highest hardness value is Ti-12Cr AT 60 ks, 296 HVN, while the lowest is CpTi, 139 HVN. This test was carried out in saliva solution at a room temperature [9].The value of hardness in all types of material tends to decrease in longer immersion variations, which results in a greater corrosion process on the surface of the entire material (Table 5).This result is in line with the one obtained in another study (Table 7) [9] but is in contrast to another study (Table 6) of which the corrosion rate of Ti-12Cr tends to increase but showsno linearity with the increasing immersion time13. This is becuse this study uses 3% NaCl solution, a more corrosive solution. The increase in the hardness value is due to the increase in the oxide layer that occurs on the surface of Ti alloy after immersion (in 3% NaCl solution) such as TiO2 and Cr2O3. This oxide layer has a high hardness value [13].

TABLE 7
Comparison of hardness values (HVN) of Ti-12Cr and CPTi alloys soaked for 10 hours, 100 hours and 1000 hours [9]

Immersion Variations	Average Hardness Values (HVN)			
	Ti-12Cr AT 60ks	Ti-12Cr AT 30ks	Ti-12Cr ST	Cp-Ti
Before	321	309	305	162
1 hour	312	305	295	162
10 hours	308	301	291	153
100 hours	302	295	289	149
1000 hours	296	273	261	139

Corrosion produces 2 reactions, namely oxidation and reduction. In the oxidation process, the material will release electrons which are more anodic, while in the reduction process, material will use electron which are more cathodic. If titanium reacts with H2O, it will form titanium oxide and hydrogen [14]. Titanium corrosion consists of cathodic oxygen withdrawal and anodic metal release [15,16].

$$Ti\ (s) + 2H_2O\ (g) \rightarrow TiO_2\ (s) + 2H_2\ (g)$$

Excessive release of Ni and Cr ions will cause a negative influence on braces [17]. Ni and Cr are heavy metals which cause allergy (carcinogenic) in the human body. Cr can protect the oxide surface because of the epresence of oxygen on its surface [18]. When Cr ions come into contact with oxygen in the electrolyte medium, it will settle on its surface to become chromium oxide (Cr2O3), so that the Cr element in Ti-12Cr can increase the corrosion resistance of implants and braces. Salivary solutions that contain sulfur can also cause corrosion more susceptible to occur [19]. Other research on the corrosion behavior of titanium alloys for biomedicine associated with Ti-12Cr and salivary solutions. Zheng, et al., (2006) [20] stated that Ti-11.3Mo-6.6Zr-4.3Sn (TMZS) titanium alloy obtained low Icoor values (good corrosion resistance) in all 3 body simulation solutions, namely artificial saliva solution, hanks, and 0.9% NaCl. TMZS corrosion resistance is best obtained in artificial saliva solution compared to hanks and 0.9% NaCl.

4 CONCLUSIONS

The following conclusions are reached:

1. The corrosion rate depends on the length of immersion. The lowest corrosion rate is found in Ti-12Cr AT alloy 60 ks, which is 0.0000038821 mmpy, while the highest corrosion rate is obtained from CpTi material, which is 0.0000076959 mmpy.
2. The CpTi and Ti-12Cr hardness values decrease with the increase of immersion time. The highest hardness value for Ti-12Cr AT 30 ks is 191.2 HVN while the lowest is for CpTi, which is 158.8 HVN, during the 3-week immersion time.
3. Based on corrosion resistance values, Ti-12Cr AT 60 ks alloys are better than other alloy for biomedical applications.

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