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Corrosion of galvanic couplings of Ni-Cr and Co-Cr alloys with Ti-6Al-4V in artificial saliva using electrochemical methods

Corrosão de acoplamentos galvânicos de ligas Ni-Cr e Co-Cr com Ti 6Al 4V em saliva artificial por métodos eletroquímicos

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ABSTRACT

In this work, the open-circuit corrosion potentials of the alloys studied in aerated artificial saliva were determined; in the same solution, the potentiodynamic polarization curves of said alloys have been drawn. The corrosion potentials and current intensities of the galvanic coupling of the Ti-6Al-4V alloy with the Co-Cr and Ni-Cr alloys were determined using the Evans method, finding that the galvanic couples more resistant to corrosion in the electrolytic medium considered are those formed between the Ti alloy and the Co-Cr alloys; the least resistant are those formed between Ti alloy and Ni-Cr alloys. The corroded surfaces of the seven alloys considered in this work were also studied, and by EDAX the corrosion products due to the ions detached in the electrolytic medium were analyzed.

Keywords: Galvanic couplings, dental alloys, Co-Cr, Ni-Cr, corrosion.

RESUMO

Neste trabalho foram determinados os potenciais e densidades de corrosão das ligas Ti-6Al-4V, Co-Cr e Ni-Cr e, usando o método Evans, os potenciais e densidades de corrosão dos acoplamentos galvânicos formados pela liga de titânio com cada uma das ligas dentárias de Co-Cr e Ni Cr, mostrando que os pares galvânicos mais resistentes à corrosão na saliva artificial ai-reada são aqueles formados entre Ti-6Al-4V com as ligas de Co-Cr e o menos resistente entre a liga de titânio e as ligas de Ni-Cr. A superfície das sete ligas consideradas neste trabalho também foi estudada e pela EDAX os produtos de corrosão foram analisados.

Palavras-chave: Acoplamentos galvânicos, ligas dentárias, Co-Cr, Ni-Cr, corrosão, EDAX.

1. INTRODUCTION

Dental alloys have applications in the restoration and correction of lost, deteriorated or misaligned dental pieces. They are used for crowns, bridges, incrustations, implants, or in the form of wires in orthodontic appliances [1]. To fulfill these functions, a variety of materials must be chosen that meet biocompatibility requirements, have adequate physical properties, resistance to wear and corrosion deterioration, and acceptable and stable appearance [2].

The different types of restorations frequently require the use of different materials whose dissimilarity in the mouth increases the galvanic interaction, whose electrolytic knowledge in the oral cavity has been known since the time of Galvani [3,4].

In recent years, investigations of biocorrosion, galvanic corrosion, wear corrosion, fatigue corrosion and stress corrosion of titanium have been carried out with Co-Cr, Ni-Cr and stainless steels in different

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physiological solutions to determine their resistance to corrosion and use them in various biomedical applications, including orthodontic wires with memory, crowns and dental bridges, endo-osseous dental implants and plates for oral and maxillary surgery [5-14].

Currently there is a large variety of non-precious dental alloys in the national and international market whose properties have not been seriously studied by users, due to the lack of adequate laboratories in Latin American countries and norms that regulate their use, forcing us to use the materials according to the specifications of the manufacturers. Therefore, it is necessary to implement and disseminate appropriate techniques to classify said alloys, so that dentists and dental technologists use it with scientific criteria, thereby minimizing the electrochemical potentials of the galvanic pairs formed in the oral cavity, generating electric batteries that end corroding the restored pieces and affecting the health of the patients when ingesting the liberated ions.

For this reason, in this work we will study the corrosion of galvanic pairs of Ti 6Al 4V with dental alloys Co-Cr and Ni-Cr in aerated artificial saliva, determining the potentials and densities of corrosion current of the individual alloys and of the pairs or Galvanic couplings of titanium alloy with Co-Cr and Ni-Cr alloys using the Evans method, and by EDAX we will analyze the corrosion products in the simulated oral cavity.

2. MATERIALS AND METHODS

2.1 Materials

An alloy of Ti-6Al-4V and six dental alloys, three of Co-Cr and three of Ni-Cr whose chemical composition and tradename are presented in Table 1 were used.

Table 1: Chemical composition of Ti-6Al-4V and dental alloys Co-Cr and Ni-Cr in % mass [15,16].

MATERIAL	TRADENAME	CHEMICAL COMPOSITION (% m/m)
Alloy Ti	Ti - 6Al - 4V	Ti 89,60 - Al 6,12 - V 4,0 - Fe 0,3
Alloys Co-Cr	Vera PDN	Co 63,5 - Cr 27,0 - Mo 5,5 - Fe, Si, Mn
	Co-Cr in Bulk	Co 65,4 - Cr 26,0 - Si 4,5 - Fe 1,4, Ni 0,8
	Premium	Co 59,9 - Cr 24,0 - W 8,5 - Nb 2,2- V 2,2- Mo 0,95- Fe, Si, Mn
Alloys Ni-Cr	VeraBond II	Ni 76,5 - Cr 11,5 - Mo 3,5 – Nb, Al, Si, Ti
	VeraBond	Ni 77,9 - Cr 12,6 - Mo 5,0 - Al, Be, Co
	VeraBond V	Ni 74,8 - Cr 12,7 - Mo 9,0 - Al, Co, Be

2.2 Preparation of test pieces

- The Co-Cr and Ni-Cr test pieces were prepared by the method of the lost wax with an oxygen - butane - propane flame and subjected to a centrifugation process; these specimens were sheets of 1.0 cm^2 of surface by 0.2 cm thick.
- The Ti-6Al-4V test specimens were prepared on a *Casmatic* machine for fully automatic of Ti casting and pouring. This process was carried out in a closed room with two chambers, which were alternately filled and evacuated with argon. The material was melted in the upper chamber with electric arc in a copper crucible and protective gas (argon), then the casting was carried out in a cylindrical mold of zirconium of 1 cm high by 0.8 cm of internal diameter.

2.3 Electrochemical methods

In order to determine the corrosion current density of galvanic pairs in artificial saliva, potential measurements were made and polarization curves of the alloys under study were determined:

- **Potential measurements**

The electrode potential of each alloy was measured against a saturated calomel electrode in a three-electrode electrochemical cell containing aerated artificial saliva at 25 °C. A Princeton Applied Research Model 173 potentiostat was used. The measurements were made in quintuplicate with different test pieces of each alloy. The potencial was previously stabilized for one hour in the solution, airing it continuously with a fish tank aerator at 80 bubbles per minute.

Polarization curves in artificial saliva were plotted in triplicate using the same cell and the same potentiostat as the one used to measure the electrode potential. The reference electrode was saturated calomel and the counter electrode was platinum. The scanning speed was 12 mV / min controlled with a Universal PAR 175 Programmer and the recording of the curves was performed with a XT PAR model REO 151. Before the start of recording of the curves, the corrosion potential was measured in the same way than in the previous section. The global curves (anodic and cathodic) of each alloy were plotted with different specimens. Once the tests were finished, the specimens were analyzed with a scanning electron microscope to determine the type of corrosive attack. The corrosion products were analyzed by EDAX.

- **Potentials of corrosion of galvanic pairs-** Dental alloys corrode in the oral environment by the action of saliva and oral fluids, so that when they are electrically coupled, both are polarized and corrode at a new speed. When metals A and B are coupled, the mixed potential of the galvanic coupling, $V_{corr\ PG,}$ is at the intersection of the polarization curves where the total oxidation rate is equal to the total reduction velocity and the current density polarization is $I_{corr\ PG}$ [17]; this process that allows to determining the potential and the current density of the galvanic pair is called *Evans method*.

2.4 Microanalysis of corrosion products

The corrosion products were analyzed by the Energy Dispersive Spectroscopy X-Ray Method, consisting in analyzing the characteristic X-rays emitted by a sample reached by a high-energy electron beam from a scanning electron microscope, allowing the identification of the elements that makeup said sample through a multichannel analyzer. In this work, a Philips SEM 500 scanning electron microscope was used, to which is attached an X-ray energy dispersive detector, EDAX DX4, and whose detection limit was approximately one cubic micron.

2.5 Electrolyte

The electrolyte used in the potentiodynamic tests was an artificial saliva that reproduces the electrochemical behavior of natural saliva [18], whose composition in grams per liter (g / L) is: NaCl (0.600), KCl (0.720), $CaCl_2{\cdot}2H_2O$ (0.220)), KH_2PO_4 (0.680), $Na_2HPO_2.12H_2O$ (0.856), KSCN (0.060), $KHCO_3$ (1.500) and $H_8C_6O_7$ (0.030), prepared with deionized water.The pH of the solution was 6.5 measured with a pH-Meter Millivoltmetre type P61 Tacussel; to prevent this value from being modified, $KHCO_3$ was added at the end of the process, shortly before starting the tests.

3. RESULTS AND DISCUSSION

3.1 Corrosion potentials

Table 2 shows the open-circuit corrosion potentials of the alloys in study ordered from the noblest to the most active for the Co-Cr and Ni-Cr alloys. These form a kind of electrochemical series in aerated artificial saliva. The average standard deviation, $\sigma(V_{sce})$, calculated according to standard G: 16-95 ASTM [19] is also shown. The numerical code of each alloy will be used to accurately identify the galvanic pairs in the respective graphs.

Table 2: Potentials of open-circuit corrosion of Ti-6Al-4V, alloys Co-Cr, Ni-Cr and standard deviations in aerated artificial saliva.$V_{corr}(V_{sce})$ is the corrosion potencial of the galvanic pair referred to the saturated calomel electrode.

MATERIAL	TRADENAME	CODE	V_{Corr} (V_{sce})	$\sigma(V_{sce})$
Alloy Ti	Ti-6Al-4V	1	-0,304	0,011
	Vera PDN	2	-0,216	0,007

Alloys Co-Cr	Co-Cr in bulk	3	-0,245	0,007
	Premium	4	-0,259	0,013
Alloys Ni-Cr	VeraBond II	5	-0,185	0,006
	VeraBond	6	-0,192	0,005
	VeraBond V	7	-0,209	0,007

The most active dental alloy of Co-Cr is Premium, whose corrosion potential is -0.259 V_{sce}, probably due to the lower Cr content (24%). The noblest of these alloys is Vera PDN followed by Bulk Co-Cr whose corrosion potentials are -0.216 V_{sce} and -0.245 V_{sce} respectively in accordance with their chromium content of 27 and 26%. These alloys will always act as cathodes when they form galvanic pairs with Ti-6Al-4V because they have higher corrosion potentials than the titanium alloy.

The least active Ni-Cr dental alloy is VeraBond II, probably due to its content of chromium and aluminum as well as niobium and titanium, which also increase corrosion resistance [20]. The less corrosion resistant alloys are VeraBond and VeraBond V due to the beryllium content, an element that produces a eutectic structure with NiBe and α-NiCrMo phases (matrix material) susceptible to corrosion [21]. In addition, beryllium greatly reduces the thickness of the passive film by decreasing the corrosion resistance in the oral cavity [22,23].

3.2 Polarization curves

Polarization curves were determined under static conditions at room temperature (25 °C). Figure 1 shows the anodic and cathodic polarization curves of the Ti-6Al-4V alloy and the Co-Cr alloys considered in this work. The alloy of Ti-6Al-4V has a passive zone that extends from -0.180 V_{sce} up to 1.200 V_{sce} with an average current density of 0.30 μA / cm^2 in that region; from 1.250 V_{sce} the current density increases due to the dissolution of the passivating oxide film and the beginning of a process of pitting on the surface of the specimen, which is accompanied by the oxygen evolution, as determined by Speck and Franker [24]. In the same figure, it is also observed that the passive zone of the Co-Cr alloys extend approximately from -0.060 V_{sce} to their rupture potentials of 0.300 V_{sce} with average current densities of 0.25 μA / cm^2, 0.30 μA / cm^2 and 0.34 μA / cm^2 for Vera PDN, bulk Co-Cr and Premium respectively. A decrease in the nobility of the alloys is clearly observed due to the lower content of chromium. In general, the corrosion resistance of Co-Cr alloys is due to the presence of the Cr (III) oxide-hydroxides present in the passive layers of these alloys [25]

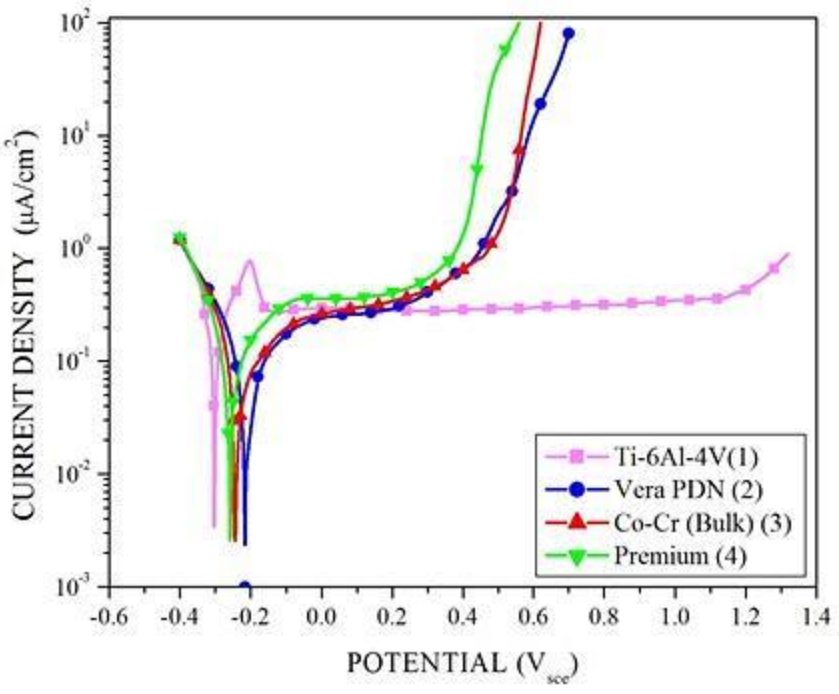

Figure 1: Polarization curves of Co-Cr and Ti-6Al-4V alloys in aerated artificial saliva.

Figure 2 shows the anodic and cathodic polarization curves of the Ti-6Al-4V alloy and the Ni-Cr alloys considered in this work. The passive zone of the Ni-Cr alloys extends approximately from -0.040 V_{sce} with average current densities ordered from lowest to highest of 0.42 μA / cm^2, 0.53 μA / cm^2 and 0.75 μA / cm^2 for VeraBond V, VeraBond and VeraBond II, up to their potentials of rupture that are 0.22 V_{sce}, 0.14 V_{sce} and 0.420 V_{sce} respectively. The current density of all Co-Cr and Ni-Cr alloys under study increases rapidly from their rupture potential, due to the dissolution of the interdendritic zone rich in Ni and poor in Cr and Mo [26,27].

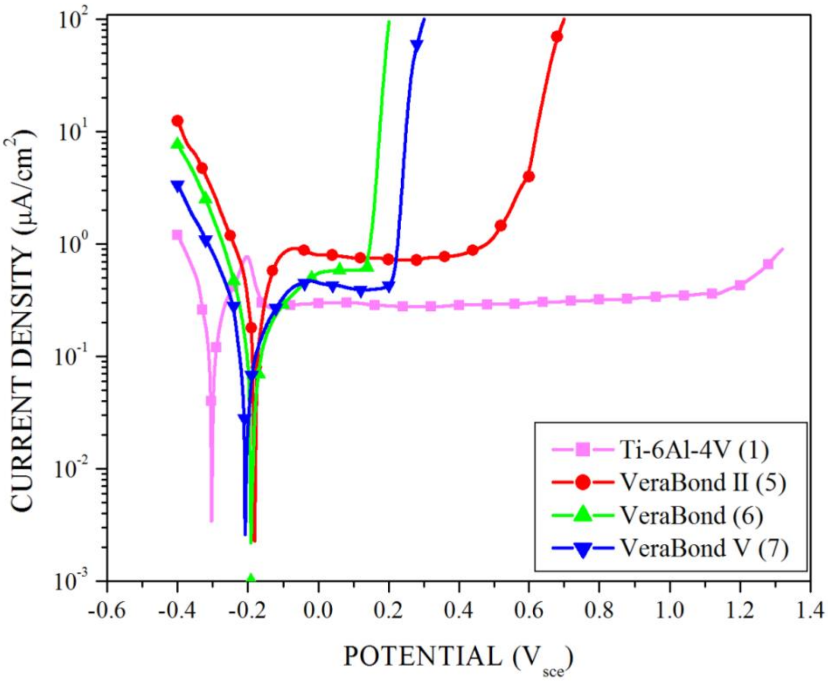

Figure 2: Polarization curves of Ni-Cr and Ti-6Al-4V alloys in aerated artificial saliva.

3.3 Potentials and densities of corrosion current of galvanic couplings of dental alloys

The potentials and instantaneous corrosion current densities of the galvanic couplings between different materials are obtained using the Evans method described above. In this case the anodic curve of the Ti-6Al-4V alloy is superimposed with the cathode curves of the Co-Cr and Ni-Cr alloys as shown in Figures 3 and 4 respectively. The intersection of the cathodic curve with the anodic curve allows determining the potentials and current densities of the galvanic pairs under study whose results are shown in Table 3.

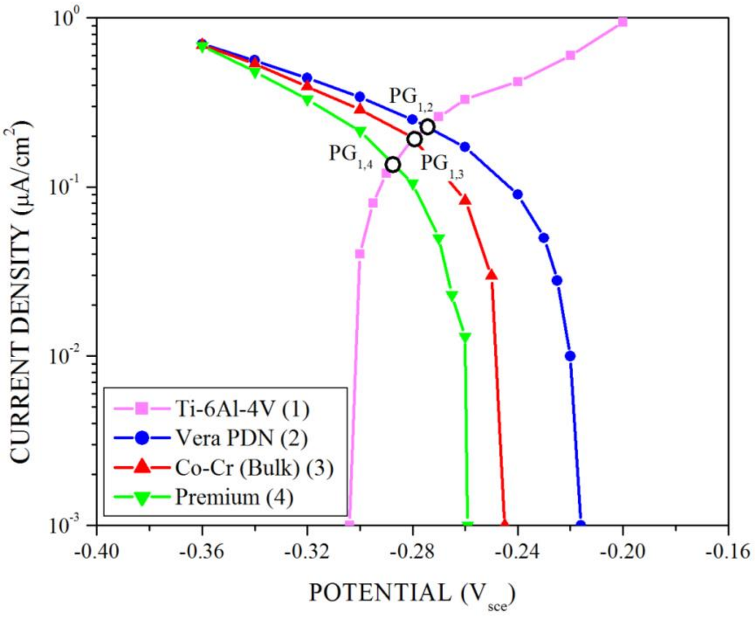

Figure 3: Evans diagrams of cathodic polarization curves of Co-Cr alloys and anodic polarization curve of Ti-6Al-4V showing the position of the respective galvanic pairs.

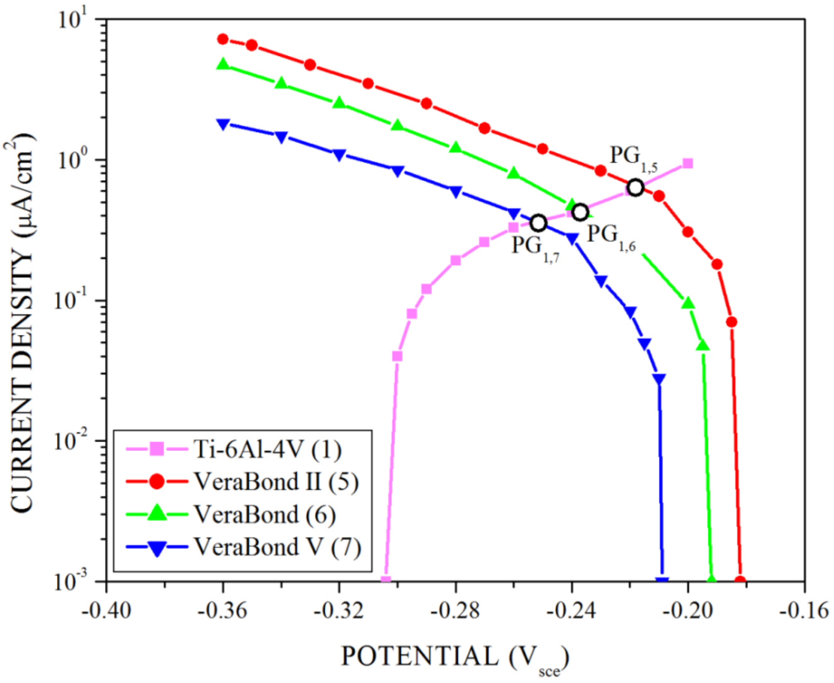

Figure 4: Evans diagrams of cathodic polarization curves of Ni-Cr alloys and anodic polarization curve of Ti-6Al-4V showing the position of the respective galvanic pairs.

Table 3 shows that the corrosion current densities of the galvanic couples of Ti-6Al-4V with the Co-Cr alloys under study are lower than the current densities of the galvanic couplings formed between the Ti alloy with Ni-Cr alloys, meaning that the most stable galvanic couples are those formed between Ti-6Al-4V with dental alloys Co-Cr.

Table 3: Potentials and corrosion current density of galvanic pairs of Ti-6Al-4V with dental alloys Co-Cr and Ni-Cr by the Evans method obtained from figures 3 and 4. The first sub-index of PG represents the titanium alloy and the second the Co-Cr or Ni-Cr alloys as appropriate and that are mentioned in table 2.

ALLOYS	**I_{Corr}(μA/cm^2)**	**GALVANIC PAIRS**		
		CODE	**$V_{Corr.PG}$(V_{sce})**	**$I_{Corr.PG}$(μA/cm^2)**
Ti-6Al-4V	0,138	1		
Vera PDN	0,110	$PG_{1,2}$	-0,275	0,227
Co-Cr in bulk	0,128	$PG_{1,3}$	-0,279	0,192
Premium	0,113	$PG_{1,4}$	-0,287	0,136

VeraBond II	0,403	$PG_{1,5}$	-0,218	0,639
VeraBond	0,235	$PG_{1,6}$	-0,237	0,432
VeraBond V	0,180	$PG_{1,7}$	-0,251	0,360

3.4 Dental alloy microstructure

Figures 5 shows SEM photographs of the microstructure of Ti-6Al-4V and of the dental alloys Co-Cr and Ni-Cr under study after removing the corrosion products from the surfaces of the specimens, observing:

1) The surface of the apparently intact Ti-6Al-4V alloy shown in Figure 5A is due to the passive layer of corrosion-resistant TiO_2 and Al_2O_3 oxides [28].

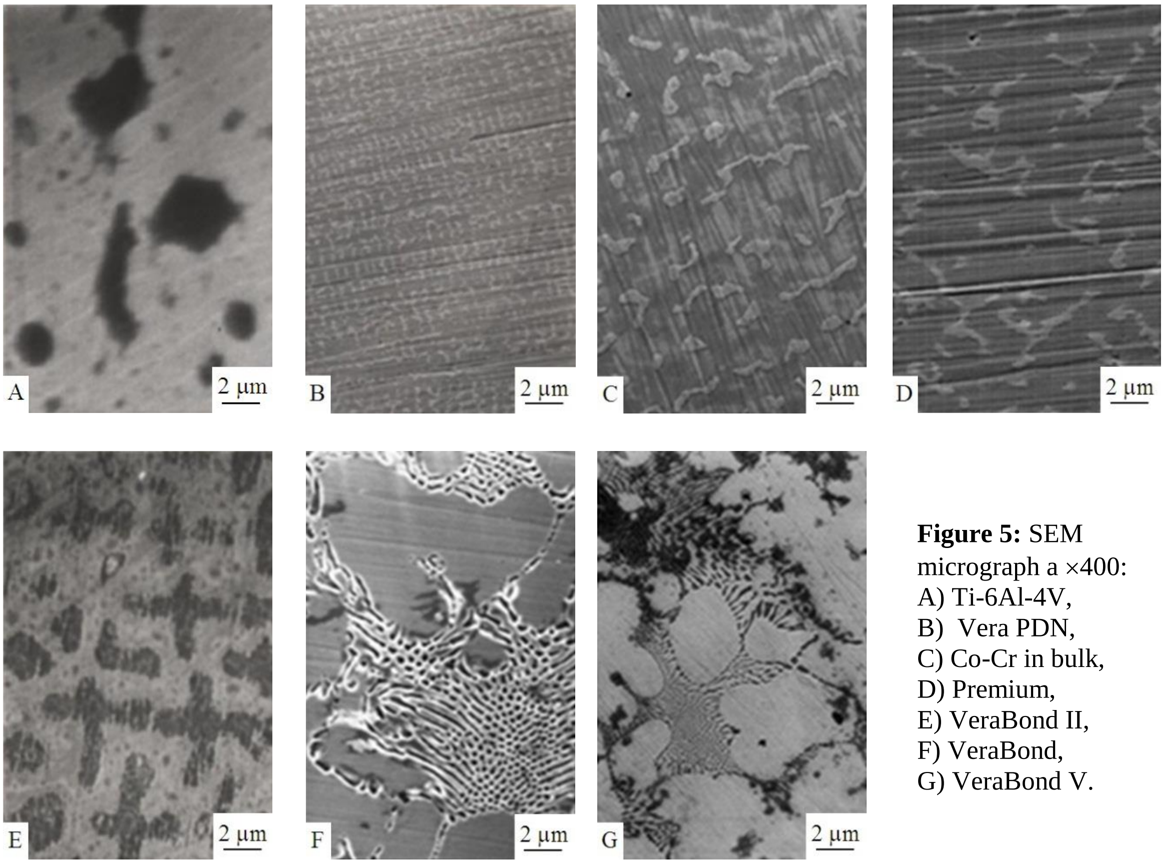

Figure 5: SEM micrograph a ×400:
A) Ti-6Al-4V,
B) Vera PDN,
C) Co-Cr in bulk,
D) Premium,
E) VeraBond II,
F) VeraBond,
G) VeraBond V.

2) In the Co-Cr and Ni-Cr alloys, the material of the interdendritic zone of the microstructure of the dental alloys is corroded due to the presence of precipitates in said zone [27, 29]. The dendritic regions are slightly rich in Cr and poor in Co. These regions, dendritic and interdendritic, are characterized by having a crystal structure hcp and fcc [30].

3) The most aggressive attack of the solution occurs in the interdendritic zone of the Ni-Cr alloys containing beryllium (VeraBond and VeraBond V) due to the eutectic microstructure that these alloys possess, as seen in the micrographs of figures 5F and 5G. The corrosion process implies the preferential dissolution of regions rich in Ni and poor in Mo [31].

3.5 Corrosion products

The EDAX spectrum of the corrosion products of the alloys under study shown in figure 6 correspond to a general sweep over these samples. The spectra show large peaks of Ti, Co, Cr, Ni, K and small peaks of Al and V corresponding to the dissolution of the alloys. On the other hand, the presence of the peaks of Cl, Ca and P are due to the electrolyte components that precipitated on the surface of the specimens. As oxygen and hydrogen are not determined by this method, it is to be assumed that oxides and hydroxides of these elements are formed in addition to chlorides and phosphates.

On the other hand, most mouth rinses contain fluoride, so the dissolution of Cr_2O_3 from the passive layer of alloys containing chromium, according to Porcoyo-Calderón et al [25], it must be:

$$Cr_2O_3 + 12F^- + 8H^+ \rightarrow 2CrHF_6^{2-} + 3H_2O$$

and the formation and repair of said passive film according to the reaction:

$$2Cr + 3H_2O \rightarrow Cr_2O_3 + 6H^+ + 6e^-$$

The passive oxide layer on the Ti alloy is also destroyed by the action of fluorine according to the reaction [25]

$$TiO_2 + 6F^- + 4H^+ \rightarrow TiF_6^{2-} + 2H_2O$$

and the formation and repair of the anodic titanium oxide film according to the reaction:

$$Ti + 2H_2O \rightarrow TiO_2 + 4H^+ + 4e^-$$

Nickel is released into the oral environment through the anodic reaction:

$$Ni \rightarrow Ni^{2+} + 2e^-$$

Metal ions from dental restorations released into the oral cavity as a result of corrosion enter the body by penetrating the enamel, dentin, pulp, gums and gastrointestinal system. These produce discoloration of adjacent soft tissues, allergic reactions and skin rashes in some people due to the oxides of nickel, chromium and molybdenum that are formed mainly [32-35]. No metal or alloy is completely inert in the living being. All remove ions from its surface to a greater or lesser extent. The Co-Cr and Ni-Cr alloys produce chromium oxides but the only ion captured by intracellular red blood cells is Cr^{6+} which then quickly converts to Cr^{3+} [36]. Titanium is a potential allergic, so care must be taken not only of the first effects such as the rejection or fracture of the prosthesis, but also its dissolution within the human body for long periods of time that the material will remain implanted [37-39].

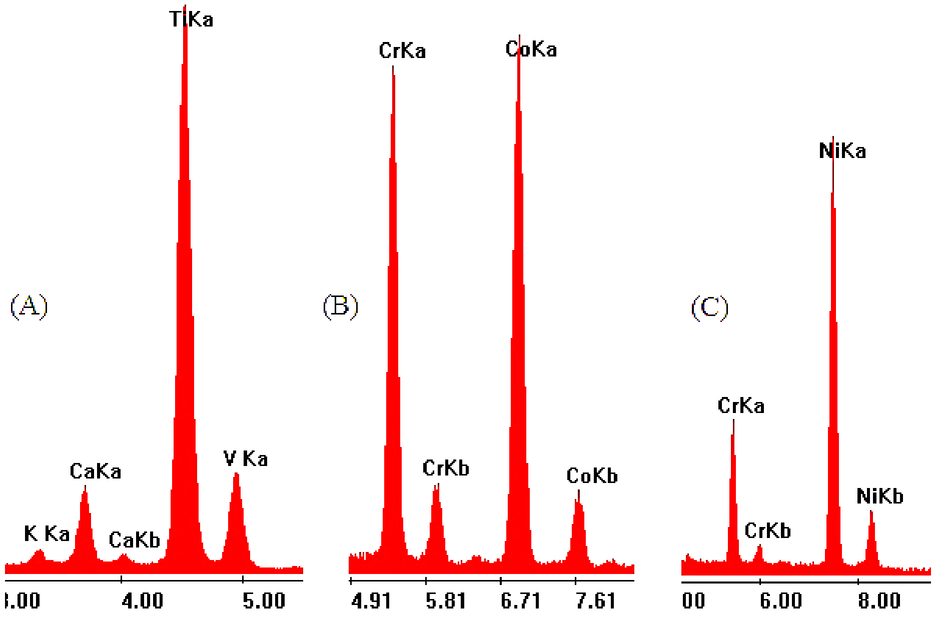

Figure 6: EDAX spectra of corrosive products in artificial saliva of: (A) Ti-6Al-4V, (B) Co-Cr and (C) Ni-Cr.

4. CONCLUSIONS

The corrosive behavior of the alloys of Ti-6Al-4V, Co-Cr and Ni-Cr have been studied using electrochemical methods in aerated artificial saliva to determine the potentials and densities of corrosion current of the isolated alloys using potentiodynamic polarization curves and the Evans method for galvanic couplings, obtaining:

1. Galvanic couplings of Ti-6Al-4V with Co-Cr alloys are more resistant to corrosion in artificial saliva than the galvanic pairs formed by Ti alloy and Ni-Cr alloys.

2. The corrosion of Ni-Cr alloys in artificial saliva is more aggressive in those alloys that contain beryllium due to its eutectic structure.

3. The corrosion products formed by the release of Co, Cr, Ti and Al ions form oxides, hydroxides, phosphates and chlorides of these metallic elements which can affect the health of patients.

4. The choice of galvanic pairs should be made based on the corrosion resistance and the biological data of the corrosion products of the alloys involved so that they do not produce allergies to the users.

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