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Corrosion of Ni-Cr-Mo alloys for fixed dental prostheses in an aqueous solution of 0.05% NaF and in commercial mouthwashes by means of EIS.

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Abstract: - Ni-Cr-Mo alloys have been used as dental prostheses due their properties such as good mechanical strength, high corrosion resistance and also because they are economically viable. The corrosion protection of these alloys in salt solutions which are typical of physiological media is due to the phenomenon called passivation with an oxide superficial layer formation, particularly chromium oxides. This protective film, subjected to mechanical stress in a corrosive environment, can be broken partially releasing ions that have deleterious effects in a human body. Fluoride ions, existent in hygiene products, change the oral environment and their presence may enable the beginning of a localized corrosion process. The objective of this work is to evaluate the corrosion resistance of three Ni-Cr-Mo alloys, of different compositions: SC (73% Ni, 14% Cr, 8.5% Mo, 1.8% Be, 1.8% Al) SB (61% Ni, 25% Cr, 10.5% Mo, 1.5% Si) and W (65% Ni, 22.5% Cr, 9.5% Mo, 1.0% Nb, 1.0% Si, 0.5% Fe) in media containing fluoride which simulate oral rinse solutions. This study has been performed in solution of 0.05% NaF, NaCl 0.05% and commercial mouthwashes on pH 6.0 at 37°C using metallography analyses, immersion techniques, and electrochemical tests, impedance tests and analysis. SC, with the highest nickel and the lowest chromium content, does not passivate in the studied means showing a continuous increase in current density due to increasing potential, while the other alloys exhibit passivation range of 600 mV and passive current density 10^{-6} A/cm^2. In general, considering the immersion and electrochemical corrosion tests, it is concluded that the worst performance has been attributed to the alloy SC, while W and SB have shown the best performance and very similar behaviors.

Keywords*: -* *Ni base alloys, Dental prostheses, Corrosion, Mouthwash.*

a. INTRODUCTION

Biomaterial can be defined as any synthetic material which replaces or restores the function of body tissues and keeps intermittent or continuous contact with organic fluids. Considering that there is contact with body fluids, it is essential that the material presents biocompatibility, produces no adverse immune response, induces no systemic effects, be not toxic, carcinogenic, mutagenic or antigenic. However, the biomaterials use may cause adverse effects in the human body, due to release of cytotoxic metals. Every implanted metal biomaterial has some interaction with surrounding tissues, by occurring release of ions by dissolution, wear or corrosion. The intra-oral corrosion is considered to be a complex process which depends on composition and thermomechanical alloy state, in combination with manufacturing, surface finishing, mechanical aspects, environment and host systemic state [1,2].

Evidence of metallic elements in the human body is often derived from biomaterials implanted in the body. Metal ions can be released from metallic biomaterials, such as orthopedic prostheses, plates, screws, implants, etc.. This metal biodegradation can cause several phenomena such as transport, metabolizing, accumulation of this material in the organs; moreover can cause allergy and carcinomas. A large amount of metal ions released can be detrimental to individual health. The metal ion release from biomaterials should be understood for a better discussion about safety and biocompatibility of these materials [3,4].

Resistance and biocompatibility of some alloys in oral means are of fundamental importance due to formation possibility of allergenic, toxic or even carcinogenic corrosion products [5,6].

Since the development of dental caries results from bacteria accumulation on the teeth and from frequent sugar intake, one of the primary measures for its control would be the periodic removal of dental plaque. Nonetheless, the greatest impact measurement has been attributed to the widespread use of fluoride in several use forms.

Saliva naturally protects both enamel and dentin, because it has calcium and phosphate, the main mineral components of crystal structure of teeth. On the other hand, this biological property of saliva is pH dependent. Thus, pH variations will determine the limit of protection capacity of saliva and by considering that nowadays people are exposed to fluoride, either by drinking water or by the use of fluoridated toothpaste, the constant presence of fluoride in saliva changes its physico-chemical properties with respect to critical pH of dissolution [7,8].

Sodium fluoride and other fluorides are often used as prophylactic agents in dental treatments to prevent dental plaque formation and caries development. Fluoride is present in all dental creams that are in general use on the market. Despite being beneficial, it should be used or consumed in the correct dosage, so that prevention does not result in adverse health effects. Creams, gels and mouthwashes are not the only available fluoride sources which population has access to.

II. MATERIALS AND METHODS

For this work, it was used three Ni-Cr-Mo alloys used in the dental market: SupremCast, SuperBond (American Dent All, USA) and Wiron 99 (BEGO, Germany). Table 1 shows the chemical compositions provided by the manufacturers.

Table 1 - Nominal chemical composition of the alloys (% m/m)

Alloy	Ni	Cr	Mo	Be	Si	Al	Nb	Fe	Ce	C
SC	73	14	8.5	1.8	-	1.8	-	-	-	-
SB	61	25	10.5	-	1.5	-	-	-	-	-
W	65	22.5	9.5	-	1.0	-	1.0	0.5	0.5	< 0.02

The samples of approximately 1 cm^2 of exposed area were sanded with sandpapers 80, 220, 600 and 1200, afterwards it was made a polishing with alumina 1µ and then dipped in acetone in an ultrasonic bath Thorton model Metasom -14 per a period of 15 minutes, washed with distilled water, then dried with hot air and then weighed on an analytical balance accurate to 0.1 mg Shimadzu, obtaining the initial mass ($m_{initial}$). Each sample was hung with nylon and placed in a flask with about 500 mL NaF 0.05% (w/w), pH 6.0 at 37 °C. The specimens were kept in static state for a period of 12 months.

After this period, the samples were removed from the solution, washed with distilled water, dried with hot air and weighed to obtain the final mass (m_{final}). Then the solutions were analyzed by atomic absorption spectrometry for the detection and quantification of ions nickel, chromium and molybdenum.

The electrochemical measurements were made using as electrolytes four commercial mouthwashes, a solution of sodium fluoride and another sodium chloride. A solution of sodium fluoride and 0.05% sodium chloride 0.05% were prepared in laboratory with analytical grade reagent using a Shimadzu Analytical Balance for weighing the solute. The pH of this solution was adjusted to a value of 6.0 by adding sodium hydroxide, with the aid of a magnetic stirrer, using a pH-meter model DIGIMED DM20 combined of glass and a magnetic stirrer Model TE-TECNAL 085 [9,10].

In order to evaluate corrosion resistance of films or covers in electrolyte medium, it was carried out the Electrochemical Impedance Spectroscopy (EIS).

In this work, electrochemical impedance measurements of the three alloys were performed in four different media, namely: NaF (0.05%), NaCl (0.05%), and two commercial mouthwashes, CP and C.

The choice of these two solutions is because the mouthwash CP has in its formulation fluoride ion and has not chloride ion, making the comparison with 0.05% NaF solution possible, and the mouthwash C has in its formulation the chloride ion present in cetylpyridinium chloride and has no chloride ion, making the comparison with 0.05% NaCl solution possible.

III. RESULTS AND DISCUSSION

3.1 Corrosion tests

3.1.1 Immersion tests

The immersion tests were carried out in NaF solution 0.05% (w/w), pH 6.0, aerated at controlled temperature (37 ± 1) °C using the alloys W, SB and SC and further a solution of NaF 0.05% as a reference standard. After the period of 1 year, the alloys were removed, washed with distilled water, dried in an air stream, and finally weighed. It was performed calculus of mass loss using the expression presented in equation (1) and the solutions were analyzed by atomic absorption spectrometry for detection and quantification of ions nickel, chromium and molybdenum.

$$\text{Mass loss } \%.\text{cm}^{-2} = \left(\frac{\left(m_{initial} - m_{final} \right) / A_{exposed}}{m_{initial}} \right) 100 \qquad (1)$$

where: $A_{exposed}$ = sample area

Results indicated that in these alloys the percentage mass loss after immersion is virtually zero, and SC was the one that lost more weight (0.08%), as shown in Table 2. This small mass loss is attributed to formation of surface film composed mainly of Cr oxides, which dissolve and impede the metal contact with the aggressive medium [10,11-13]. In this medium, the solution pH increased as a result of corrosion, the initial pH was 6.0 and the final around 7.92, as displayed in Table 2.

Quantitative analysis by flame atomic absorption was made in all solutions, with an average of 3 determinations. The alloy SC showed higher Ni ions concentration in relation to the alloys W and SB. The concentrations of Cr and Mo ions were less than 0.2 mg/L for all alloys, whereas for Ni the amount it was approximately 8.4 mg/L for the alloy SC, as shown in Table 2. This mass loss can also be observed due to a yellow powder formation in the bottle bottom, probably Ni corrosion products, further emphasizing the low corrosion resistance of this alloy.

Table 2 – Results of quantitative analysis by atomic absorption and weight loss calculations for all the three alloys.

Alloys	Ionic concentration (mg/L)					Mass loss ($\%cm^{-2}$)	pH Increase
	Ni	Cr	Mo	Total	Concentration /Unit ($mg.L^{-1}cm^{-2}$)		
Wiron	< 0.2	< 0.2	< 0.2	< 0.6	$7.6x10^{-2}$	$8.6x10^{-3}$	1.13
SB	< 0.2	< 0.2	< 0.2	< 0.6	$7.9x10^{-2}$	$5.4x10^{-2}$	1.92
SC	8.4 ± 0.1	< 0.2	< 0.2	< 8.9	$8.3x10^{-1}$	$8.0x10^{-2}$	1.92
Reference solution (NaF)				8.3 < % < 8.9	----------	--------	1.12

3.2 Measurements of open-circuit potential

A comparison of stability of the alloys W, SB and SC in medium 0.05% NaF pH 6.0 at 37 °C in aerated system can be seen in Figure 1. The potential values about 200 mV more positive to alloys W and SB were achieved at steady state. It is important to note that this significant increase in values of open-circuit potential is consistent with the increase in the alloy chromium content. The SC alloy which has a lower percentage of chromium presented the lowest potential in the whole time interval, indicating that the passive film is less protective, which makes this alloy less corrosion resistant [14,15].

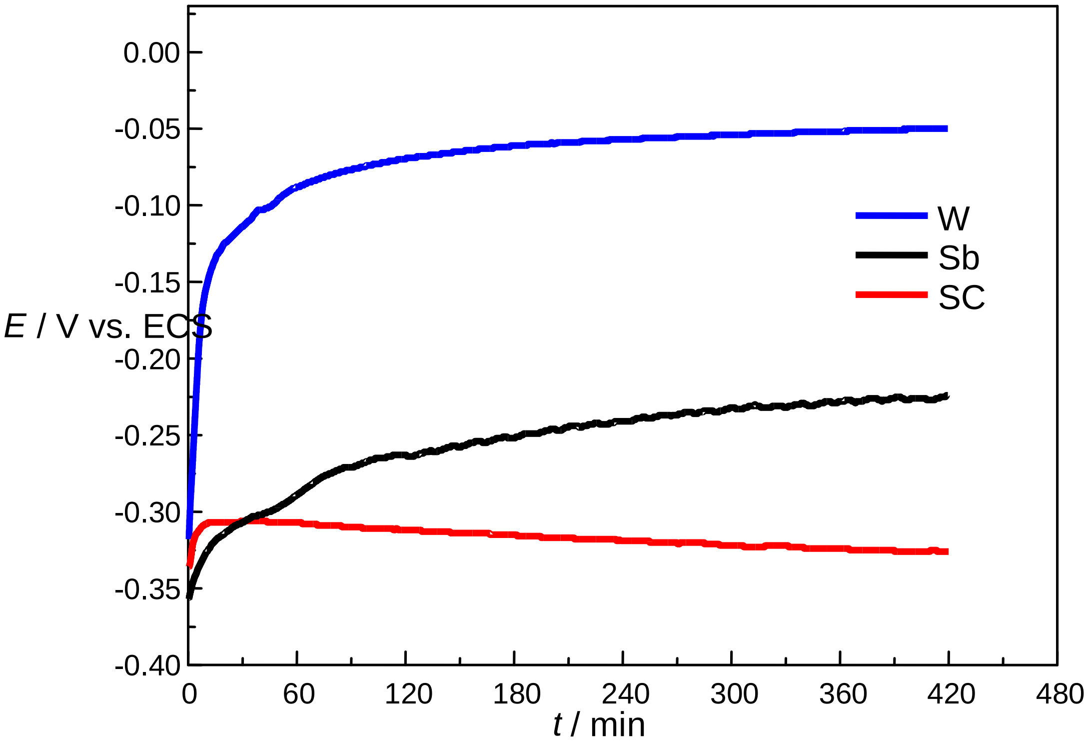


Figure 1 – Curves of open circuit potential with time of alloys W, SB and SC in 0.05% NaF.

Table 3 shows the stabilization potentials for the alloys W, SB and SC in all studied media, thus it can be see that the alloy SC has a lower stabilization potential for all media showing that it really has the lowest corrosion resistance.

Table 3 – Apparent stabilization potential (V/SCE) for alloys W, SB and SC.

Media used	W	SB	SC
NaF 0.05%	-0.08	-0.04	-0.30
NaCl 0.05%	-0.15	-0.15	-0.25
CP	-0.05	-0.14	-0.43
NS	-0.05	-0.20	-0.33
L	-0.12	-0.26	-0.30
C	-0.32	-0.20	-0.35

3.3 Potentiodynamic polarization curves.

The potentiodynamic polarization curves obtained with a scan rate of 20 mV/min in NaF 0.05%, 0.05% NaCl and different commercial mouthwashes, aerated at 37 °C for the alloys W, SB and SC are shown in Figure 2.

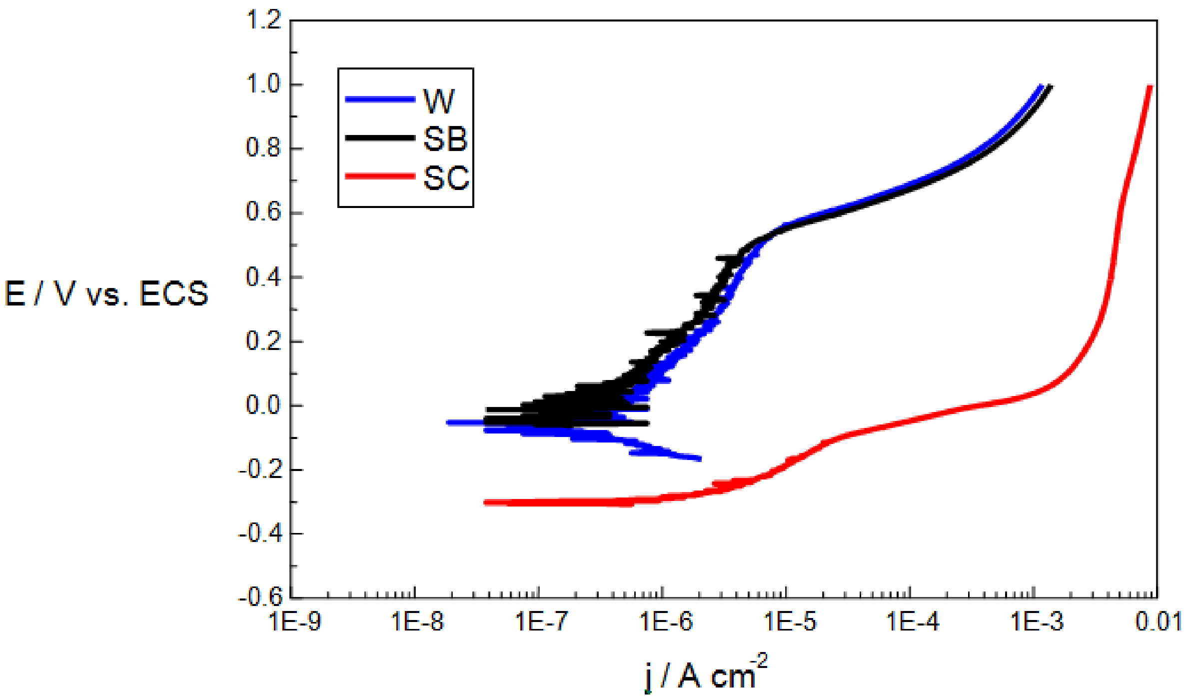


Figure 2 - Potentiodynamic polarization curves of alloys W, SB and SC in 0.05% NaF.

Table 4 shows the parameters, passivation current density (j_{pass}), passivation range ($E_{rupture}$-E_{corr}), rupture potential ($E_{rupture}$), obtained from potentiodynamic polarization curves of the alloys W, SB and SC in different media.

Table 4 - Parameters obtained from potentiodynamic polarization curves of the alloys W, SB and SC.

Used media	W			SB			SC		
	j_{pass} (A cm^{-2})	E_{rup}-E_{corr} (V)	E_{rup} (V)	j_{pass} (A cm^{-2})	E_{rup}-E_{corr} (V)	E_{rup} (V)	j_{pass} (A cm^{-2})	E_{rup}-E_{corr} (V)	E_{rup} (V)
NaF	2.0×10^{-6}	~0.6	~ 0.5	1.0×10^{-6}	~0.6	~0.5	10^{-5}	~0.2	
NaCl	2.0×10^{-6}	~0.8	~0.6	5.0×10^{-6}	~0.6	~0.8	$5x\ 10^{-6}$	~ 0.3	
CP	1.2×10^{-6}	~0.5	~ 0.5	1.0×10^{-6}	~0.7	~ 0.5	1.0×10^{-5}	~0.9	~0.5
NS	1.1×10^{-6}	~0.7	~0.6	1.0×10^{-6}	~0.8	~0.6	$5.0x\ 10^{-5}$	~0.5	~0.2
L	1.0×10^{-6}	~0.8	~0.6	1.0×10^{-6}	~0.8	~0.6	5.0×10^{-6}	~0.9	~0.6
C	1.1×10^{-6}	~0.7	~ 0.5	1.9×10^{-6}	~0.7	~ 0.5	5.0×10^{-5}	~0.9	~ 0.5

3.4 Cyclic voltammetry

In Figure 3, the cyclic voltammograms of alloys W, SB and SC in 0.05% NaF, pH 6.0 were obtained in potential region between -0.6 V and 0.6 V (SCE) with scan starting towards positive potentials at a speed of 10 m V s^{-1}. This potential range covers the main electrochemical processes, such as formation and increase of oxide in passive region. The scan reversal was taken in the material dissolution region. It is observed, according to this figure, alloys W and SB display a passive region in this medium, with the greatest passivity range for the alloy W, while the SC is not passive [16].

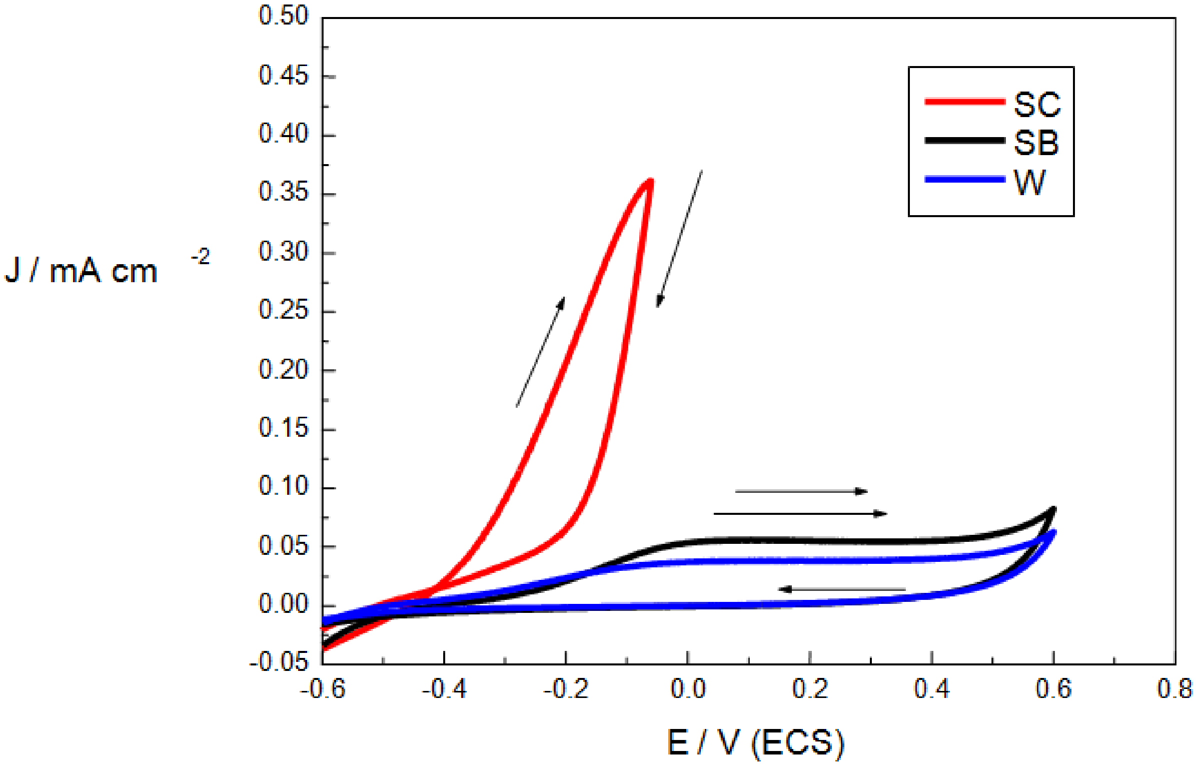


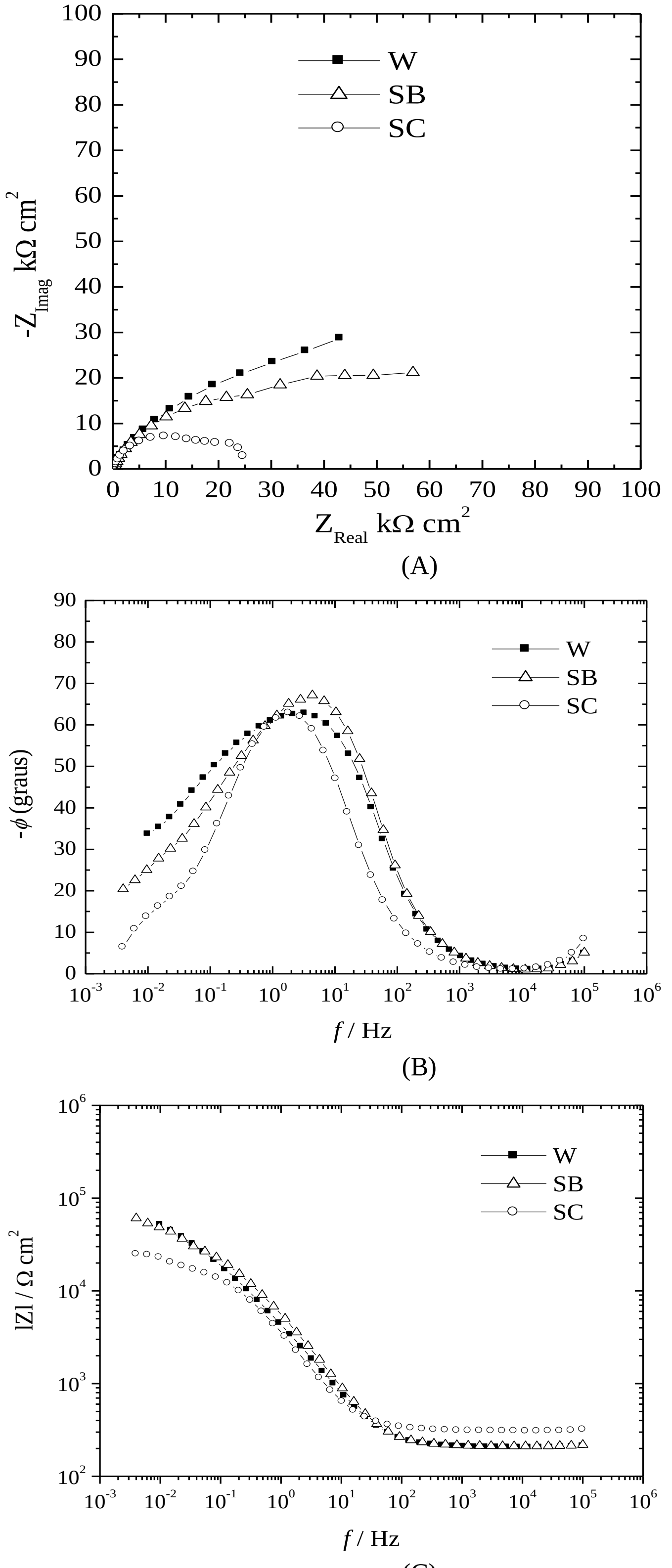
Figure 3 - Cyclic voltammograms of the alloys W, SB and SC, with v = 10 mV/s. in 0.05% NaF.

3.5 Electrochemical Impedance Spectroscopy (EIS)

The technique of electrochemical impedance spectroscopy is considered an important analysis technique for the investigation of electrochemical behavior of passive films. In this method, the polarization resistance, R_p, a parameter for estimating the corrosion resistance, is obtained by impedance measurements at low frequencies [17,18].

3.5.1 Concentration effect of fluoride and chloride ions on the electrochemical behavior:

The similarity in the electrochemical response between W and SB in medium NaF can be seen by the graphs of impedance, especially at the complex plane format (Nyquist graph), Figure 4 (A). The impedance limit at low frequencies intersects the real component and is considered the polarization resistance, that is, the sum of all resistances of electrochemical system, which in this case is higher for W and smaller for SC, as observed in j_{pass} reverse order (Table 4). It is noted, according to this figure, the appearance of a fuzzy semicircle in the three alloys. This result indicates the worst performance to SC in medium NaF. In Figure 4 (B) (graph Bode-phase), the larger maximums achieved for W and SB which include the range of medium at low frequencies appear to be the result of two coupled capacitive components. For SC alloy beyond this maximum, it is possible to observe the appearance of a new component at around 20 mHz, with phase angles less than 20° [19]. In general, such a low value as this may be associated with diffusion within pores or superficial imperfections resulting from the casting process of the alloy. According to change in impedance modulus as a function of frequency, Figure 4 (C) (Chart Bode-Module), it becomes easy to visualize the corrosion resistance relative to the three alloys, basically determined by the resistance of its films, with the best performance for W, followed by SB and last SC [20].



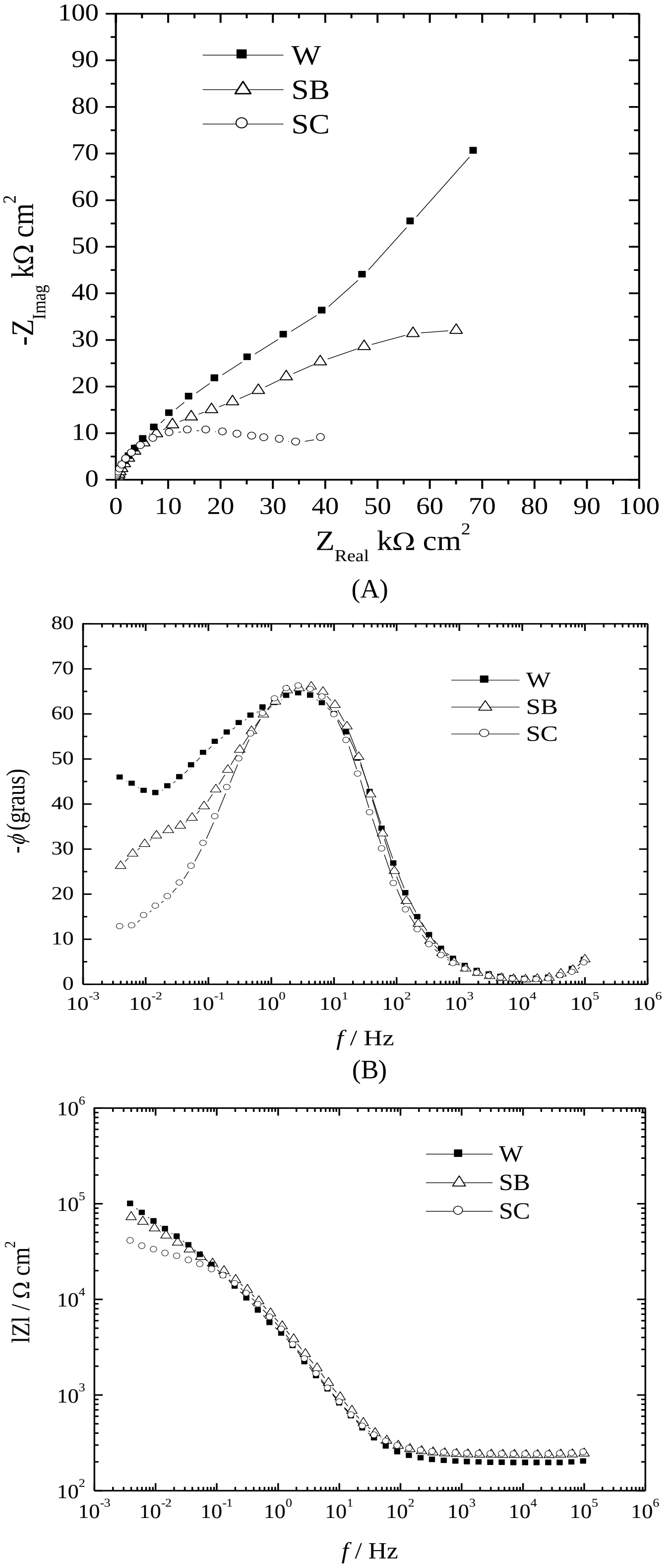
(A)

(B)

(C)

Figure 4 - Impedance graphs obtained for the different alloys studied in NaF medium: (A) Complex plane format, (B) Variation of phase angle with frequency, (C) Variation of the impedance modulus with frequency.

In Figure 5, impedance graphs are presented obtained for the three studied alloys in NaCl 0.05% of distortion with respect to the ideal semicircle, Figure 5 (A), W alloy showed greater capacitive arc being coupled to a diffusion component, and therefore higher value of R_p on the real axis at low frequencies, followed by SB and, finally, the SC with the worst performance. This result is in agreement to the study by [20], in which an alloy of concentration very near W showed a high corrosion resistance in NaCl solution even more concentrated than in question, showing the concentration of Cr is essential for the alloy protection. This result was also observed in medium containing 0.05% NaF, phase graph (Figure 5 B). The Z module graph (Figure 5 C) shows similar behavior of these alloys in the two investigated media. For SC, the increase in depression of distorted semicircle in region of low frequencies is characteristic of corrosive processes enhanced by presence of pores or pits on the electrode surface.



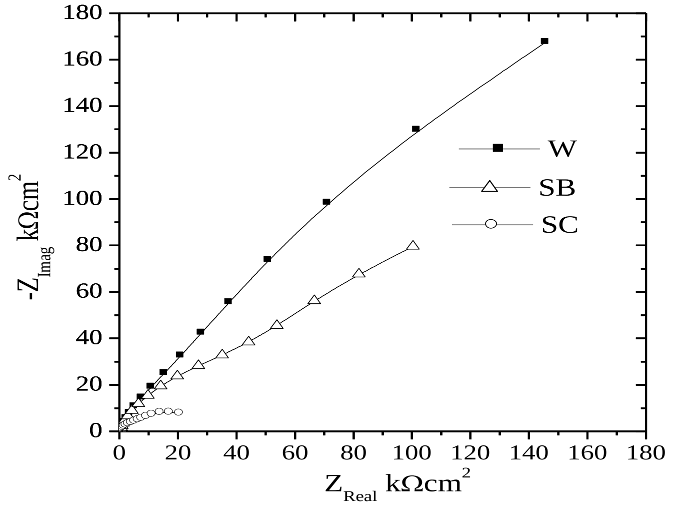
(A)

(B)

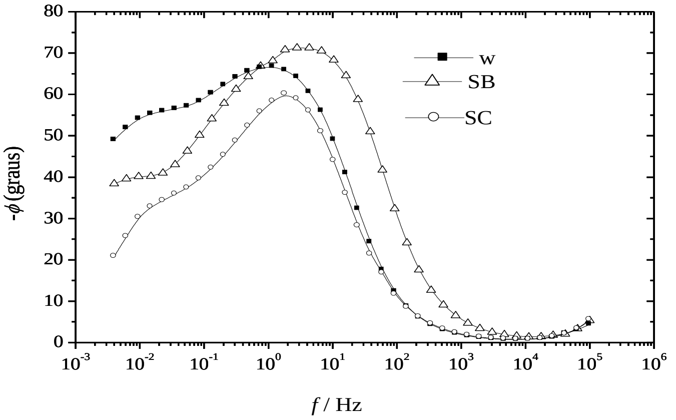
(C)

Figure 5 - Impedance Graphs obtained for the different alloys studied in NaCl medium: (A) Complex plane format, (B) Variation of phase angle with frequency, (C) Variation of impedance modulus with frequency.

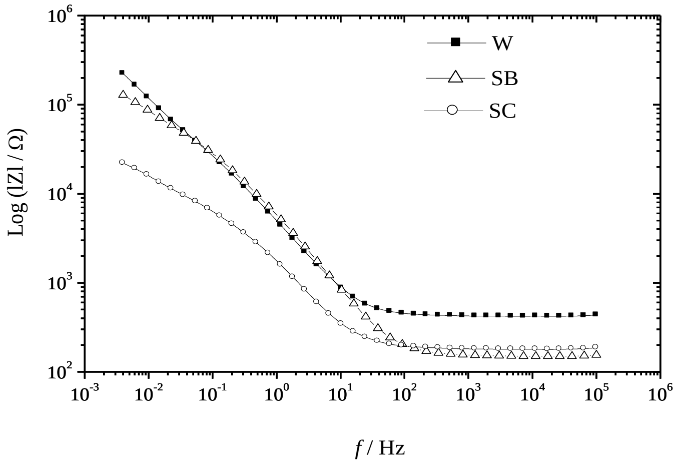
The sequence of resistance to relative corrosion in middle CP denoted by the magnitude of diameter of semicircle is the same as that observed for alloys in means NaCl and NaF, Figure 6 (A). The SC alloy had the worst performance; it was observed a higher R_p value for W followed by the SB and then SC, as in previous cases. In addition, the observed peak, the phase angle and amplitude in frequency are not common to the same frequency range, denoting processes apparently distinct for the three alloys studied. Modulus at low frequencies confirms greater protection of film formed on the alloy W.



(A)



(B)



(C)

Figure - 6 Graphs of impedance obtained for the different alloys studied in CP medium: (A) Complex plane format, (B) Variation of phase angle with frequency, (C) Variation of impedance modulus with frequency.

In medium C, the relative corrosion resistance, denoted by magnitude of diameter of semicircle, is similar to that observed for these alloys in NaF, NaCl and CP media Figure 7 (A). The W alloy showed the largest diameter of the capacitive arc, and therefore, greater resistance, followed by SB and SC with the worst performance, in agreement with the results obtained by other techniques. All tests were carried out in triplicate and the results in agreement with each other and with previous techniques prove that the worst result is for the SC alloy, due to its low chromium concentration.

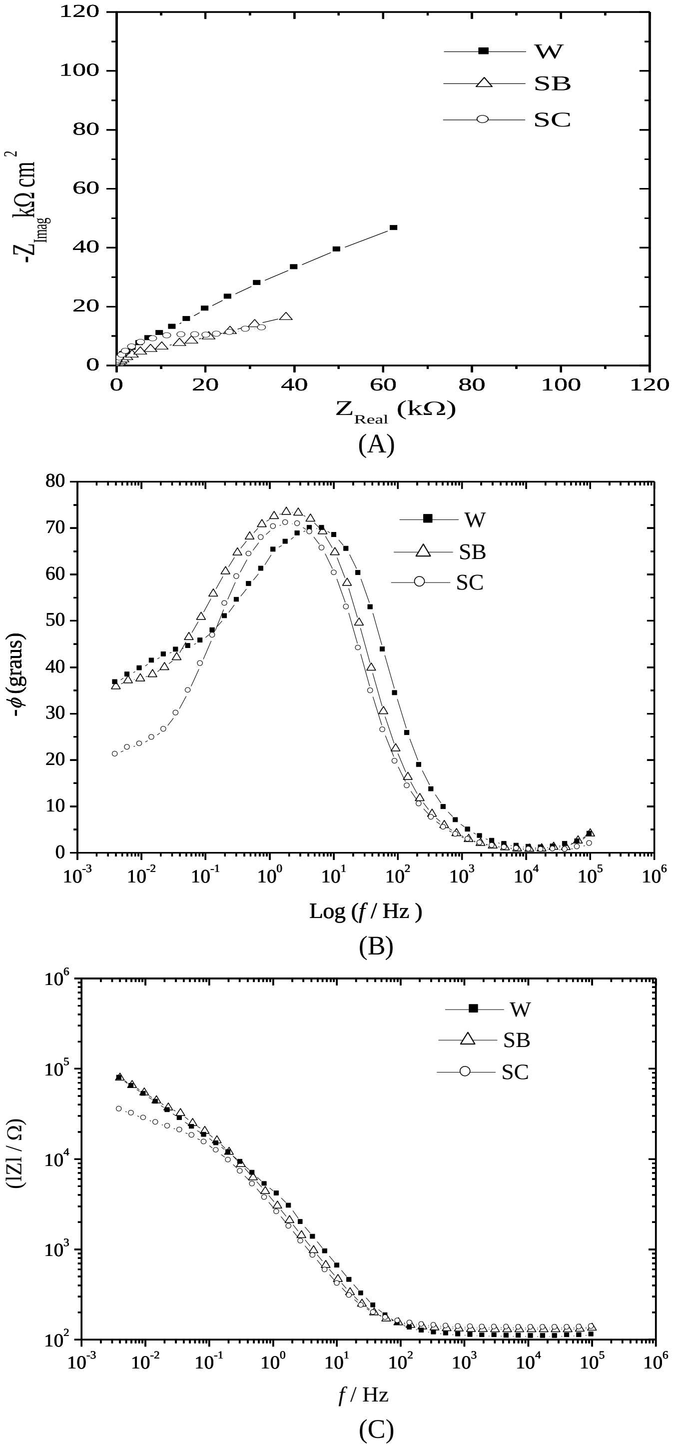


Figure 7 - Impedance graphs obtained for the different alloys studied in medium C: (A) Complex plane format, (B) Variation of phase angle with frequency, (C) Variation of impedance modulus with frequency.

IV. CONCLUSIONS

The alloys that have higher chromium concentration, as for instance the alloy W (22.5%) and SB (25%), have a higher corrosion resistance compared to the SC with lower Cr content but containing Be due to the passivation phenomenon attributed to formation of a protective layer of chromium oxide, thus inhibiting the corrosion process continuation.

The percentage mass loss of the alloys W, SB and SC after immersion was virtually zero, being the latter which the most weight lost (-0.05%), which suggests that this alloy is even the least corrosion resistant due to the lower chromium concentration.

Analyzing the measurements carried out in open circuit as well as the potentiodynamic profiles, there is a clear distinction among the electrochemical behaviors corresponding to the SC as compared to the two other alloys. In general, the alloys W and SB displayed very similar responses and good corrosion resistance, being the worst performance for the SC. Moreover, the results obtained by these electrochemical techniques traditionally carried out to evaluate corrosion resistance of metals and alloys in aqueous media are in agreement with those of immersion.

Given the results, it is evident that the best alloy for dentures fixed manufacture in terms of corrosion resistance is W and the worst is SC.

By analyzing the impedance modulus magnitude, it was found that in the means 0.05% NaF, NaCl 0.05%, CP and the C alloy SC showed the worst performance, and W had the best one.

With the results it is evident that the best alloy for the manufacture of fixed dental prostheses in terms of corrosion resistance and is W and the worst is SC.

The mouthwash to be used depends on the type of alloy that compounds the prosthesis in contact with it: if using the W alloy, it is better to use CP mouthwash (containing fluoride); if it is using the SB alloy, the best mouthwash is L and for the SC alloy the best mouthwash is NS.

The best combination alloy-mouthwash is W alloy with mouthwash CP and unless stated is the SC alloy and mouthwash L.

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