

The Corrosion of Dental Amalgam in some Commercial Mouthwash Solutions

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This study investigates the electrochemical behaviour of a Tytin[®] dental amalgam in two commercial mouthwash solutions and compares it with the behaviour of dental amalgam in artificial saliva. Open circuit potential measurement, potentiodynamic polarization curves and electrochemical impedance spectroscopy (EIS) are the electrochemical procedures of investigation. Our results have shown that Tytin[®] dental amalgam has a somewhat good corrosion resistance in artificial saliva. After immersion in mouthwash solutions the corrosion currents increase. The EIS results show that dental amalgam exhibited passivity at open circuit potential in artificial saliva whereas in mouthwash solutions the protectiveness of passive layer was no more present.

Keywords: dental amalgam, commercial mouthwash solutions, artificial saliva

Dental amalgam has been used as a restorative material for the replacement of the decayed tooth structure for more than 150 years [1]. An amalgam is a metallic alloy formed by reaction between mercury and an original alloy in powder form containing silver (40-70%), tin (15-30%), copper (10-30%), and with a very complex metallurgical structure. Mercury diffuses into the alloy particles and reacts with silver, tin and copper forming various chemical compounds. Actually, the nature of these compounds depends on the chemical composition of the powder and on the particle shape. The main formed phases of the system are Ag-Hg and Sn-Hg, whereas Ag-Cu and Ag-Sn old phases are remaining unchanged.

A number of factors may influence the corrosion rate. Among these factors are the acidity and temperature of the contacting medium which both can undergo sharp variations in a short period of time in the oral cavity, as well as the effective potential of the amalgam [2, 3].

Nevertheless, in recent years investigations of amalgam corrosion have become of great interest due to the questions about the toxic effects of the corrosion products in oral cavity. A further question results from the possible release of mercury and mercury vapor [4-7] as well as the effect that mercury can have antioxidant [8] and neurotoxicological activity [9].

Numerous investigations on the corrosion behavior of the dental amalgams have been carried out using various experimental tests and methods. The corrosion behaviour of 16 different commercial amalgams in artificial saliva using cyclic voltammetry was compared [10]. It was found that the electrochemical corrosion rates varied widely over several orders of magnitude in the corrosion medium. The effect of the composition of dental amalgams on their electrochemical behavior, including reactions occurring outside of oral conditions have been examined using cyclic voltammetry [11]. AgCl and Hg₂Cl₂ films were formed on all amalgams except that with 40 wt %. The effect of sliding wear on the corrosion behavior of two dental amalgams high-copper content was reported [12]. The results showed that sliding wear caused a shift in the corrosion potential, a significant increase in the corrosion rate and a decrease in

the repassivation rate of both amalgams. The importance of the organic component in the oral liquid environment on the rate of corrosion and adsorption phenomena on the amalgam surface have been demonstrated [13].

It was also examined the corrosion of individual dental amalgam phases under *in vitro* conditions. The electrochemical behavior of the dental phases in 0.9% NaCl solution is characteristic for passive system; the mechanism involves the formation of corrosion products which protect metallic surface [14]. Recently, it was observed a slower rate of corrosion potential ennoblement for admixed amalgam restorations than would be predicted from *in vitro* studies [15].

Electrochemical reactions are one of the most important interactions between amalgam restorations and oral fluids. The dental amalgam fillings corrode in the oral environment and often show extensive degradation, significant localized penetrations and marginal fractures [16, 17]. The modifications of the dental alloys properties could be determined using rapid electrochemical tests to estimate the corrosion resistance [18-21].

In this study the electrochemical methods have been used to investigate the behaviour in artificial saliva and in two commercial mouthwash of an amalgam consisting in a high-copper content Tytin[®] mixed intimately with liquid mercury (50 % w/w).

Experimental part

Tytin[®] dental amalgam (KerrDental, USA) was investigated. The composition of powder alloy Tytin[®] is (in wt %): 69.7% Ag, 17.7% Sn, 12% Cu and 0.9% Zn, with a mercury-to-alloy ratio of approximately 50%. After triturating it was casted into cylinder-shaped pieces of 5 mm diameter and 4 mm length. On one of the amalgam sample faces a cooper wire was attached using conductive paint to ensure electrical conductivity. The assembly was then embedded into an epoxy resin disk. Then the samples were polished with SiC abrasive paper up to 1000 grit, final polishing being done with 1 μm alumina suspension. The samples were degreased with ethyl alcohol followed by ultrasonic cleaning with deionised water and dried under

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a hot air stream. The exposed electrode area was 0.21 cm².

The electrochemical behaviour was studied in three types of solutions: artificial saliva (Carter-Brugirard AFNOR/NF) having the composition: NaCl – 0.7 g/L, KCl – 1.2 g/L, Na₂HPO₄·H₂O – 0.26 g/L, NaHCO₃ – 1.5 g/L, KSCN – 0.33 g/L, carbamide – 1.35 g/L (pH=8.0) and two commercial mouthwash formulations, Licor del Polo® (Schwarzkopf & Hankel, Spain) with 225 ppm F⁻ ions and Oral B® (Gillette, Spain) with 500 ppm F⁻ ions.

For all electrochemical tests, the experiments were performed at 25 ± 1°C. The assembled specimen was placed in a glass cell, which was filled with electrolyte. A saturated calomel electrode (SCE) was used as the reference electrode and a platinum coil was the counter electrode.

The measurements were performed with a Princeton Applied Research potentiostat (Model 263 A) controlled by a personal computer and a specific software (PowerCorr, Princeton Applied Research).

Open circuit potential (E_{OC}) was recorded for 24 h with the sample immersed in aerated artificial saliva. Then the dental amalgam was immersed in mouthwash solutions and the open circuits potential was further monitored 3 h.

Linear polarization was conducted at a scanning rate of 0.166 mV/s, to identify the b_a (Tafel slopes for the partial anodic processes) and b_c (Tafel slopes for the partial cathodic processes), i_{corr} (corrosion current density) and ZCP (zero current potential). It was then followed by the general polarization tests from -600 mV to +1200 mV at a scanning rate of 0.5 mV/s in order to evaluate the stability of passivation.

A coulometric zone analysis was also carried out. The method proposed here consists of dividing the polarization curves into two separate zones: zone I, from ZCP up to +300 mV and zone II, from +300 mV to +600 mV. The separation is somewhat arbitrary, but the potential span of zone I, is close to clinical conditions. The intensity of the degradation phenomenon may vary from one patient to another; zone I was considered as the high-risk zone and zone II a lower-risk zone barring exceptional situations [22]. The limits of zone I (ZCP to +300mV) was chosen because they cover potential ranges most frequently observed in the oral cavity [23].

The electrochemical impedance spectroscopy (EIS) was performed in aerated solutions at the open circuit potential using a Princeton Applied Research potentiostat (Model 263 A) connected with a Princeton Applied Research 5210 lock-in amplifier.

The spectra were recorded in the 10⁻² Hz to 10⁵ Hz frequency range. The applied alternating potential signal had amplitude of 10 mV.

The EIS spectra were interpreted using the ZSimpWin program. The soft uses a variety of electrical circuits to numerically fit the measured impedance data. This procedure allowed the construction of an equivalent circuit, whose simulated responses fit well the actually measured data.

Results and discussions

In figure 1 the variation of open circuit potential with time for the amalgam in artificial saliva and in mouthwash solutions is showed. These results are based on the dental amalgam studied after 24 h of immersion in artificial saliva and 3 h of immersion in mouthwash solutions.

The sharp variation toward more positive values of potential was recorded during the first minutes of

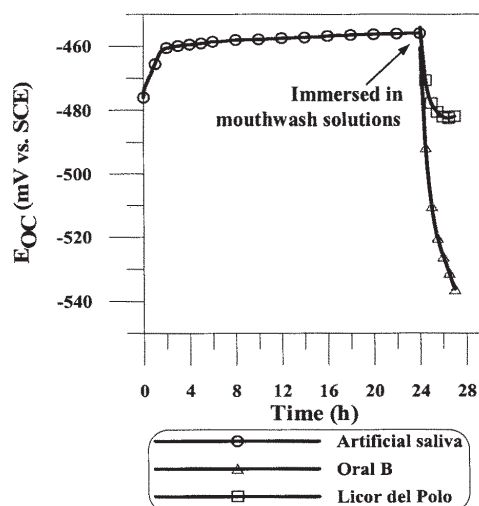


Fig. 1. Variation of open circuit potential with time for Tytin® dental amalgam maintained 24 h in artificial saliva and after 3 h immersed in mouthwash solutions

immersion in artificial saliva. Afterwards, the E_{OC} remained slowly increasing during the 24 h, which represent the period before immersion in the mouthwash solutions.

The change of open circuit potential with time is accompanied by the formation of a film of corrosion products seen by visual inspection and in agreement with other studies [13, 24]. The open circuit potential ranged from -476 mV to -456 mV. These potential values are consistent with the potentials of the electrochemical reactions corresponding to corrosion of Sn and formation of tin oxides and hydroxides [25, 26].

After immersion in mouthwash solutions a shift to more negative values in the electrode potential of dental amalgam is observed. In the Licor del Polo® mouthwash solution the potential drops from -456 mV to -471 mV. In the Oral B® mouthwash solution, the open circuit potential shows a significant change from -456 mV to -536 mV. This phenomenon may be interpreted as a decrease in the corrosion resistance of the dental amalgam in mouthwash solutions.

After recorded the open circuit potential, the sample was subjected to the polarization tests.

Standard procedures were used to extract “zero current potential” (ZCP) and i_{corr} values from the potentiodynamic polarization plots. The Tafel slopes (b_a and b_c) were estimated by fitting the theoretical polarization curve to the experimental polarization curve plotted in a range of ± 150 mV vs. E_{OC} . The two Tafel slopes are intersecting in the point of the coordinates (ZCP, i_{corr}). An alloy with tendency toward passivation will have the value of b_a greater than b_c , while an alloy that corrodes will have b_a smaller than b_c [27]. The ZCP value is the potential at which the current reaches a minimum during the forward potentiodynamic polarization scan.

The average values of b_a , b_c , ZCP and i_{corr} determined from polarization curves by using the PowerCorr program are presented in table 1.

The mean values of ZCP are approximately identical to E_{OC} . Under clinical conditions the corrosion may occur at elevated potentials with respect to the ZCP [28].

After 24 h of immersion in artificial saliva the higher value of b_a than the b_c value for the dental amalgam indicates an anodic control in the corrosion process. The control implies the existence of a passive film on the surface. In this case we found that the value of the corrosion current density is small: 170 nA/cm².

After immersion in mouthwash solutions the ZCP decreases (this behavior is in agreement with the variation of the open circuit potential) and the corrosion currents increase suggesting that the dental amalgam had retained its protective passive film. After 3 h immersion the behavior in Licor del Polo[®] mouthwash is similar with that in Oral B[®] mouthwash: the corrosion current is increasing slowly. Corrosion currents are also higher, as expected, in Oral B[®] mouthwash because of its higher fluoride concentration. Figure 2 shows the polarization curves recorded in a range of ± 150 mV vs. E_{OC} for Tytin[®] dental amalgam, after 24 h immersion in artificial saliva and after 1 min or 3 h immersion in Oral B[®] mouthwash solutions.

For Tytin[®] dental amalgam, the Oral B[®] mouthwash solutions caused a shift of the polarization curves to the

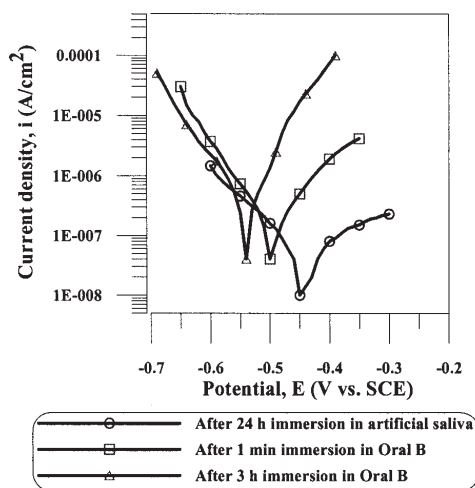


Fig. 2. Potentiodynamic polarization curves for Tytin[®] dental amalgam after 24 h immersion in artificial saliva and after 1 min or 3 h immersion in Oral B[®] mouthwash solutions; 0.166 mV/s, 25 °C temperature

active region corresponding to more negative potentials and significantly higher current density.

In order to explore all the potential range, the potentiodynamic polarization curves are plotted in a semi-logarithmic version between -600 mV and +1200 mV vs. SCE. Curves of the dental amalgam after 24 h in artificial saliva or after 1 min in mouthwash solutions are displayed in figure 3. In mouthwash solutions the potentiodynamic polarization curves were registered with the dental amalgam initially maintained 24 h in artificial saliva.

After 24 h immersion in artificial saliva a relative passivation beginning at -0.35 V and up to 0.2 V is evidenced. Oxidation of Tytin[®], leads to insoluble corrosion products: hydrated $\text{Sn}(\text{OH})_2$ and $\text{Sn}(\text{OH})_4$ [29]. However, the passive film is not considered to be an efficient barrier to amalgam dissolution [30]. As a result the anodic current shows a monotonically increase near the ZCP (diffusion controlled process). At more positive potentials than $+0.25$ V the passive film breakdown is produced. The potential range situated between the ZCP and the breakdown

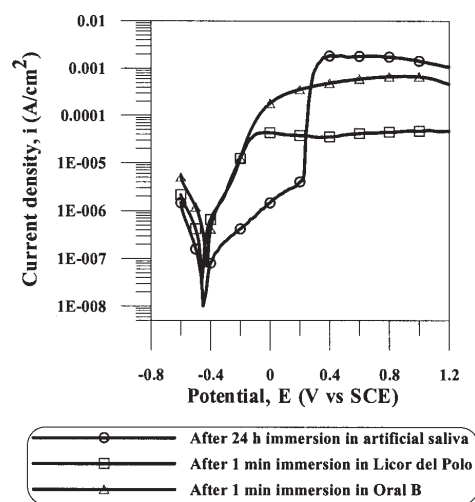


Fig. 3. Potentiodynamic polarization curves for Tytin[®] dental amalgam, after 24 h immersion in artificial saliva or after 1 min immersion in mouthwash solutions; 0.5 mV/s, 25°C temperature

potential represents the passive zone in which corrosion is weak.

In the polarization curves for dental amalgam after 1 minute in mouthwash solutions, the anodic current densities increasing with increasing potential indicate a continuous anodic dissolution of metal. Anodic current densities are also higher in Oral B[®] mouthwash solution. No passivation could be established with the dental amalgam when polarized in mouthwash solutions.

Coulometric analysis provides a simple way for a rough clinical risk analysis. It consists of dividing the anodic polarization curves into two distinct zones: a first zone (zone I) extending from ZCP to $+300$ mV and a second zone (zone II) from $+300$ mV to $+600$ mV. The amounts of electrical charge consumed by the corrosion phenomenon for each zone are shown in tables 1. The charges along high-risk zone for Tytin[®] dental amalgam immersed in both mouthwash solutions are about ten times higher than in the case for Tytin[®] dental amalgam immersed in artificial saliva. In mouthwash solutions the Tytin[®] dental amalgam has much less corrosion resistance than in artificial saliva. On the other hand, for dental amalgam in all three test solutions the charge amounts, in zone II are very substantial: 1020 mC/cm² in artificial saliva, approximately 700 mC/cm² in Licor del Polo[®] mouthwash and approximately 900 mC/cm² in Oral B[®] mouthwash.

The results are consistent with the observations above presented.

It is obviously that comparing results after immersion in three solutions, artificial saliva is the least corrosive medium, Licor de Polo[®] mouthwash is more corrosive and Oral B[®] mouthwash is slightly more corrosive, influenced by the higher fluoride concentration.

In a complementary study, electrochemical impedance spectroscopy (EIS) was used to investigate the corrosion resistance of Tytin[®] dental amalgam. EIS measurements

Table 1
ELECTROCHEMICAL DATA RESULTED FROM POLARIZATION CURVES FOR TYTIN[®] DENTAL AMALGAM IMMERSSED IN ARTIFICIAL SALIVA AND IN MOUTHWASH SOLUTIONS FOR VARIOUS PERIODS OF TIME

| Solution | Immersion time | ZCP (mV) | b_a (mV) | b_c (mV) | i_{corr} (nA/cm ²) | Coulometric analysis | |
|---------------------------------------|----------------|----------|------------|------------|----------------------------------|----------------------------------|----------------------------------|
| | | | | | | ZCP-300 mV (mC/cm ²) | 300-600 mV (mC/cm ²) |
| Artificial saliva | 24 hours | -448 | 180 | 110 | 170 | 32.5 | 1020 |
| Licor del Polo [®] mouthwash | 1 minute | -472 | 82 | 91 | 551 | 380 | 650 |
| | 3 hours | -497 | 91 | 95 | 715 | 390 | 670 |
| Oral B [®] mouthwash | 1 minute | -505 | 85 | 89 | 864 | 460 | 870 |
| | 3 hours | -535 | 92 | 91 | 1010 | 480 | 890 |

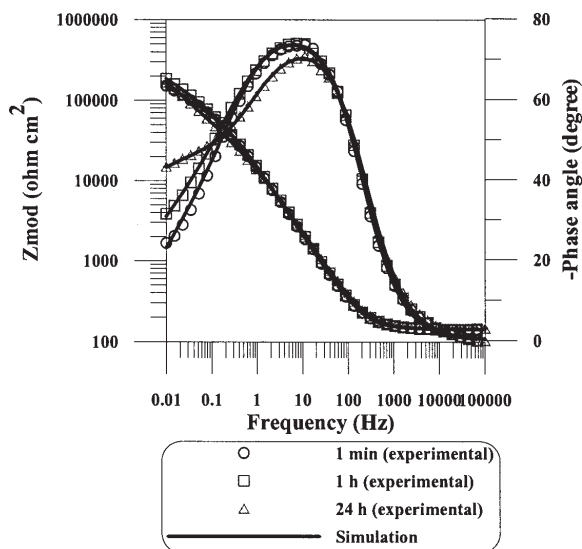


Fig. 4. Bode plots of the Tytin® dental amalgam for various exposure times into the artificial saliva; plots measured at E_{oc} , 25 °C temperature

are carried out for the dental amalgam before and after immersion in mouthwash solutions. The impedance data, obtained, at open circuit potential, for dental amalgam immersed in artificial saliva for 1 min, 1 h and 24 h are presented as Bode plots (impedance modulus vs. frequency and phase angle vs. frequency) in figure 4.

The Bode spectra indicate the presence of a compact passive film because: (a) the phase angle maxima are closer to -70° over a wide frequency range, and (b) the impedance modulus shows linear portions at intermediate frequency.

In the highest frequency region, $\log Z_{mod}$ tends to become constant with a phase angle values 0° ; this is a typical response for the resistive behaviour and corresponds to the solution resistance, R_{sol} .

Figure 4 shows at intermediate frequency, the phase angle shifted to -70° , indicating a near capacitive response for the dental amalgam. At 1 h of immersion, the shape of Bode plots was similar to that of 1 minute of immersion. However, with increase in time to 24 h of immersion the increase in the impedance value indicates an increase in corrosion resistance of the film.

For the interpretation EIS spectra, an appropriate physical model of the electrochemical reactions occurring at electrode/solution interface is necessary. An equivalent circuit (fig. 5) provides the most relevant corrosion parameters applicable to the substrate/electrolyte system.

Instead of pure capacitors, constant phase elements (CPE) were introduced in the fitting procedure to obtain good agreement between the simulated and experimental

data. The impedance of CPE is defined as $Z_{CPE} = \frac{1}{Q(j\omega)^n}$ where Q is the combination of properties related to both the surfaces and electroactive species independent of frequency; the exponent n is related to a slope of the $\log Z_{mod}$ vs. \lg Frequency (Bode plots); ω is the angular frequency and j is imaginary number ($j^2 = -1$). The exponent n is an adjustable parameter that usually lies between 0.5 and 1. When the value of n is equal to 1, the CPE describes an ideal capacitor with Q equal to the capacitance (C). For $0.5 < n < 1$ the CPE describes a distribution of dielectric relaxation times in frequency space, and when n is equal to 0.5 the CPE represents a process with diffusional character.

The impedance data for dental amalgam in artificial saliva were fitted with the EC presented in figure 5 and the resultant parameters values are given in table 2.

In the figure 4, the experimental data are shown as individual points, while the theoretical spectra resulting from the fits to a relevant EC model are shown as lines.

The physical meaning of the given circuit is the association of the passive layer/electrolyte interface R_1 - CPE_1 with the passive layer itself R_2 - CPE_2 . The time constant at high frequencies is originated from the R_1 - CPE_1 combination while that at low frequencies was initiated from the R_2 - CPE_2 combination. R_p representing the polarization resistance (R_p) and CPE_1 the double layer capacitance. The double layer capacitances are typical for the C_{dl} of the passive layers [31-33]. The characteristics of the passive layer are reflected in the values of R_2 .

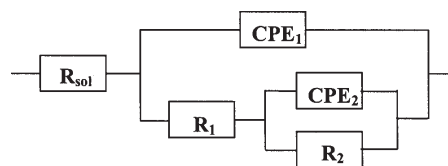


Fig. 5. Equivalent circuit (EC) used in simulated data for Tytin® dental amalgam immersed in artificial saliva

A high R_p value is an indication of the working electrode strongly resisting change from its equilibrium state and is representative of the degree of protection of the passivation layer of the dental amalgam surface. The more the value of R_p increases, the more the dental amalgam will resist corrosion. Returning to the data from potentiodynamic curves the corrosion current may be also calculated from Stern-Geary equation [34]:

$$i_{corr} = \frac{b_a b_c}{2.3 R_p (b_a + b_c)} = \frac{B}{R_p} \quad (1)$$

where: b_a and b_c are the Tafel slopes for the partial anodic and cathodic processes, respectively and B is a constant:

$$B = \frac{b_a b_c}{2.3(b_a + b_c)} \quad (2)$$

The resulted value of corrosion current for the Tytin® dental amalgam immersed in artificial saliva for 24 h is small: 206 nA/cm². The results obtained from this research clearly indicate a good agreement between the polarization and EIS data.

For the dental amalgam maintained in artificial saliva the values of n , are in the range of 0.73-0.76 indicating the presence of a diffusion process within the interfacial layer of the solution. The involving of a diffusion process means that the passive layer formation under open circuit condition proceeds through a dissolution-precipitation mechanism [35].

In mouthwash solutions the impedance data were registered with the dental amalgam initially maintained 24 h in artificial saliva. Figure 6 shows the Bode plots at ZCP for dental amalgam after 1 min and respectively 3 h in Licor del Polo® mouthwash formulation and figure 7 shows spectra for the same sample in the same time condition but immersed in Oral B® mouthwash formulation.

After initial immersion (1 minute) in both mouthwash solutions, the Bode phase spectra show a decrease in phase angle at medium frequency from -80° (after 24 h in artificial saliva) close to -55° . At 3 h of immersion in Licor

Table 2
RESULTS OF THE FITTING FOR THE IMPEDANCE MEASUREMENTS IN
ARTIFICIAL SALIVA AND MOUTHWASH SOLUTIONS

| | Immersion time | R_{sol} ($\Omega \text{ cm}^2$) | R_1 ($\Omega \text{ cm}^2$) | CPE_1 ($S \text{ cm}^{-2} s^b$) | n_1 | R_2 ($\Omega \text{ cm}^2$) | CPE_1 ($S \text{ cm}^{-2} s^b$) | n_2 | i_{corr} (nA/cm^2) |
|---------------------------------------|----------------|-------------------------------------|---------------------------------|-------------------------------------|-------|---------------------------------|-------------------------------------|-------|--------------------------|
| Artificial saliva | 1 minute | 32 | $2.9 \cdot 10^4$ | $1.3 \cdot 10^{-5}$ | 0.87 | $1 \cdot 10^5$ | $2.3 \cdot 10^{-5}$ | 0.73 | - |
| | 1 hour | 35 | $4.6 \cdot 10^4$ | $1.2 \cdot 10^{-5}$ | 0.87 | $1.1 \cdot 10^5$ | $2.1 \cdot 10^{-5}$ | 0.75 | - |
| | 24 hours | 38 | $4.5 \cdot 10^4$ | $1.2 \cdot 10^{-5}$ | 0.88 | $1.4 \cdot 10^5$ | $1.6 \cdot 10^{-5}$ | 0.76 | 206 |
| Licor del Polo [®] mouthwash | 1 minute | 85 | $3.9 \cdot 10^4$ | $1.3 \cdot 10^{-5}$ | 0.81 | - | - | - | 481 |
| | 3 hours | 79 | $3.5 \cdot 10^4$ | $1.5 \cdot 10^{-5}$ | 0.80 | - | - | - | 577 |
| Oral B [®] mouthwash | 1 minute | 105 | $2.9 \cdot 10^4$ | $1.5 \cdot 10^{-5}$ | 0.81 | - | - | - | 651 |
| | 3 hours | 98 | $2.3 \cdot 10^4$ | $1.7 \cdot 10^{-5}$ | 0.81 | - | - | - | 865 |

del Polo[®] mouthwash solution, the shape of Bode plots was similar to that of 1 min of immersion. As the immersion time increased (3 h) for dental amalgam in Oral B[®] mouthwash solution, the phase angle, at medium frequency, decreased to nearly -45° . This decrease in the impedance values indicated a decrease in a corrosion resistance.

The impedance data of Tytin[®] dental amalgam, immersed 1 min and 3 h in Licor del Polo[®] and Oral B[®] mouthwash formulations were fitted with the EC presented in figure 8, and the resultant circuit parameters are also given in table 2. In the figures 6 and 7, the experimental data are shown as individual points, while the theoretical spectra resulting from the fits to a relevant EC model (Figure 8) are shown as lines.

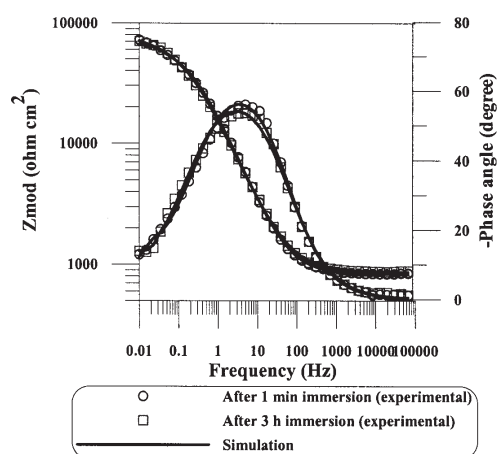


Fig. 6. Bode spectra for Tytin[®] dental amalgam after immersion for 1 min and, respectively, 3 h in Licor del Polo[®] mouthwash solution, at E_{OC} ; 25 °C temperature

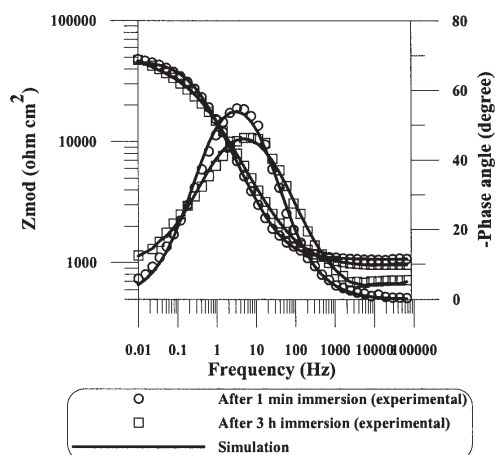


Fig. 7. Impedance spectra for Tytin[®] dental amalgam after immersion for 1 min and, respectively, 3 h in Oral B[®] mouthwash solution, at E_{OC} ; 25°C temperature

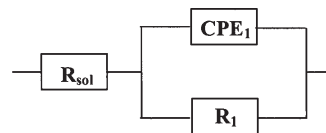


Fig. 8. The equivalent circuit used for fitting the impedance data for Tytin[®] dental amalgam immersed in both mouthwash solutions.

When the dental amalgam is immersed in mouthwash solutions the passive layer initially formed on dental amalgam immersed in artificial saliva was severely destroyed. In this corrosive medium the protectiveness of passive layer was no more present. This is much clearer than from other electrochemical techniques such as polarization curves. The corrosion currents increase suggested that the dental amalgam retained its protective passive layer and these results are in agreement with the results obtained by the polarization curves.

Comparing the results obtained by different electrochemical techniques, the data obtained suggests that the artificial saliva is the least corrosive medium and also that Oral B[®] mouthwash is slightly more corrosive than Licor del Polo[®] mouthwash, probably because of its higher fluoride concentration.

Conclusions

The electrochemical behavior of a dental amalgam, prepared with a high-copper content Tytin[®] mixed intimately with liquid mercury (50 % w/w) in artificial saliva and in two commercial mouthwash revealed a somewhat good corrosion resistance in artificial saliva. The corrosion rates of dental amalgam in mouthwash solutions were found to be larger; the corrosion currents are 3-5 times higher than in artificial saliva. No passivation could be established with the dental amalgam when it is polarized in mouthwash solutions. In artificial saliva, the high impedance was mainly ascribed to the presence of a protective passive layer formed on dental amalgam; the polarization resistance was around $10^5 \Omega \text{ cm}^2$, with one order of magnitude higher than that when was immersed in mouthwash solutions. The impedance mainly corresponded to the charge transfer resistance and was around $10^4 \Omega \text{ cm}^2$. In our opinion both mouthwash solutions (Licor del Polo[®] and Oral B[®] solutions) are contraindicated for Tytin[®] dental amalgam.

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