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Evaluation of the hexanoic Anise extract as inhibitor for dental amalgam in synthetic saliva

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Abstract
An electrochemical and gravimetric study of the corrosion of dental amalgam was carried out in electrolytes similar to artificial saliva with or without Anise extracts. Electrochemical study indicates that the Anise extract act as an anodic-type inhibitor. The corrosion rate of dental amalgam and the inhibition efficiencies of the extracts were calculated. The results obtained show that the extract solution of the Anise plant could serve as an effective inhibitor for the corrosion of amalgam in saliva media. The inhibition efficiency was found to increase with extract concentration until 0.33g/L. The maximal protection efficiency exceeded 90%.

Keywords: amalgam, saliva, anise extract, inhibitor, weight loss, electrochemical study.

1. Introduction
The corrosion and wear resistance of dental amalgams has been of much interest in recent years as a result of increased concern regarding toxicity arising from amalgam particles and corrosion products in the oral cavity. A further concern results from the possible release of mercury and mercury vapor [1-4].
Many factors can influence the rate of corrosion. These include acidity of the contacting medium and temperature, which can both undergo sharp variations in a short period of time in the oral cavity, as well as the effective potential of the amalgam [5, 6]. For these reasons, a full understanding of the electrochemical and corrosion behavior of the amalgams can be of much benefit in taking appropriate measures to reduce amalgam corrosion as well as ensuring complete amalgamation such that there is no free mercury that can be vaporized.
Dental amalgam is formed by the rapid reaction of liquid mercury with a powder alloy containing principally silver (40–70%), tin (15–30%) and copper (10– 30%). Mercury diffuses into the alloy particles and reacts with silver, tin and copper, forming various compounds. The exact compounds formed depend on the chemical composition of the powder and on particle shape (which can be spherical or irregular) but are mainly phases of the systems Sn–Hg, Ag–Hg, with Ag–Cu and Ag–Sn phases remaining from the reactants. For the currently used, high copper amalgams, the main reaction is as follow [7].

$$\gamma\text{-}Ag_3Sn + Ag\text{–}Cu + Hg \longrightarrow \gamma 1\text{-}Ag_2Hg_3 + \gamma\ 2\text{-}Sn_7Hg + \gamma\text{-}Ag_3Sn + Ag\text{–}Cu.$$

The Sn–Hg phase, which has a relatively low corrosion resistance, then undergoes further reaction, according to/

$$\gamma_2\text{-}Sn_7Hg + Ag\text{–}Cu \longrightarrow \acute{\eta}\text{-}Cu_6Sn_5 + \gamma\ 1\text{-}Ag_2Hg_3$$

A great number of scientific studies have been devoted to the subject of corrosion inhibitors for dental amalgam in artificial saliva [8–10]. The majority of well-known inhibitors are organic compounds containing heteroatom, such as O, N or S, and multiple bonds, which allow an adsorption on the metal surface [11-17].
Plant extracts have became important as an environmentally acceptable, readily available and renewable source of materials for wide range of corrosion prevention; therefore, finding naturally occurring substances as corrosion inhibitors is a subject of great practical significance [18–23].
The objective of this investigation was to examine the corrosion behavior of silver-tin-mercury dental amalgam alloy in artificial saliva, without and with the addition of anise extract, using a potentiostatic technique and gravimetric measurements at a constant temperature of 37 °C.

2. Experimental

2.1. Specimen Preparation

The electrode specimen was prepared by mixing in 1:1mercury to alloy (Ag 35.05%, Sn 9%, Cu 5.95%) ratio. The specimen was put into a resin split mould with 1cm in diameter and 12 mm in length and kept at ambient temperature until solidification. Products of amalgamation were presented inTable1. Prior to each experiment, the working electrode was successively polished with finest- grade emery papers, washed by bidistilled water and then dried.

Table 1: XRD analysis of dental amalgam alloy

Chemical formula
Ag_2Hg_3
Ag_3Hg_2
Ag_4Sn
CuSn

2.2. Preparation of Anise Extract

The Anise Extract was obtained by Soxhlet extraction of 100 g of seed for 24 h in about 750 mL of hexane. The extract was concentrated to dryness and the residue was kept at - 4°C. This extract was used to study its corrosion inhibition properties on the dental amalgam.

2.3. Solution Preparation

The synthetic saliva media was prepared by adding 1.12g of KCl and 1.24g of $NaHCO_3$ in one Liter of distilled water. The concentration range of anise extract employed was varied from 0.11g/L (11µL/L) to 0.33g/L (33µL/L) and the volume of electrolyte used was 100 mL for each experiment.

2.4. Weight loss measurements

Gravimetric experiments were carried out in a double glass cell. The solution volume was 20 mL and experiments were performed with concentration of 0.33g/L of Anise extract. The maximum duration of tests was 1 week (168h) at 37°C in non-de aerated solutions. At the end of the tests, the specimens were carefully washed in distilled water and dried in hot air and then weighted. Duplicate experiments were performed in each case and the mean value of the weight loss is reported.
Weight loss allowed calculation of inhibition efficiency of our extract according to the following equation:

$$E\% = \left(\frac{W^\circ - W}{W^\circ}\right) \times 100 \qquad (1)$$

Where W and W° are the weight loss of dental amalgam samples obtained in artificial saliva solution in the presence and in the absence of inhibitor, respectively.

2.5. Polarization measurements

Electrochemical measurements were carried out in a conventional three electrode cylindrical glass cell, containing 100 mL of electrolyte at 37°C in which working electrode was dental amalgam, calomel electrode was the reference electrode and platinum was the counter electrode. All potentials are reported vs. SCE. The Potentiodynamic curves of dental amalgam in artificial saliva solution in the absence and in the presence of Anise extract were obtained in the potential range from -1 to +1 V. For polarization measurements, a potentiostat Voltalab 301 PGZ monitored by a PC computer and Voltamaster 4.0 software were used for run the tests, collect and evaluate the experimental data. During each experiment, the test solution was mixed with a magnetic stirrer.
The inhibition efficiency (E%) was calculated using the equation:

$$E\% = \left(\frac{I^0 - I}{I^0}\right) \times 100 \qquad (2)$$

Where I° and I are, respectively, the corrosion current densities obtained in artificial saliva solution in the absence and the presence of inhibitor.

2.5. *Spectroscopy induction coupled plasma*

ICP method was used to analyze the solution. The sample was ionised by injecting it in plasma of argon. Analyses were performed on a Jobin Yron ULTIMA 2 with radical aim. The calibration is made in the same solution of artificial saliva to eliminate the effect of matrix.

3. Result and discussion

3.1. Gravimetric Study

Table 2 gives values of the inhibition efficiency E %, and the corrosion rate obtained with the addition of 0.33 g/L of anise extract for the corrosion of dental amalgam in synthetic saliva after 1 week of immersion at 25°C. It is evident from these results that anise extract has an inhibition affect of amalgam in saliva solution containing 0.33 g/L. At room temperature, the inhibition efficiency is about 85%.

Table 2: The gravimetric measurements of amalgam

Medium	rate of corrosion $mg.cm^{-2}.day^{-1}$	E%
In absence of inhibitor	0.155	
With anise extract (0.33g/L)	0.023	85

3-2- Spectroscopic of induction coupled plasma analysis (ICP)

To determinate the effect of anise extract on the corrosion of amalgam, the solutions of immersion were analysed by ICP (Table 3). These results showed that in the absence of the inhibitors, the dissolved metallic ions quantities in solution are very high with regard to the quantities of it in the presence of the inhibitors (hexanoic anise extract).

Table 3: Results of ICP analysis

	Zn	**Hg**	**Sn**	**Cu**	**Ag**	medium
mg/L	0.589	0.01652	1.12	1.6036	0.00882	In the absence of inhibitor
mg/L	0.1644	0.00096	0.2124	0.3786	0.00238	In presence of inhibitor

The analysis of these results shows that there is a preferential dissolution of copper and tin in the absence and in presence of inhibitor. Based on the quantities of elements dissolved in solution, we tried to calculate the efficiency of our extract as corrosion inhibitor. The hexanoic extract has inhibitor efficiency about 78% at concentration of 0.33 g / L. For the mercury, the inhibition efficiency is up to 96%. These results are in good agreement with the obtained result by gravimetric method.

3.3- Electrochemical study

Fig. 1 shows the cathodic and anodic polarization plots of amalgam in saliva medium at 37°C in the absence and presence of different concentrations of anise extract.

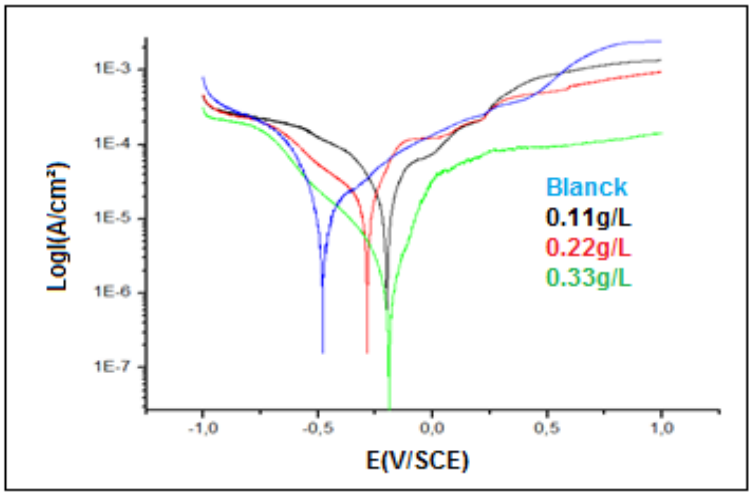

Figure 1: Anodic and cathodic polarization curves for amalgam in synthetic saliva without and with various concentration of inhibitor at 37°C**.**

The electrochemical parameters for the different anise extract concentrations are given in Table 4. The data show that increasing inhibitor concentration decreases the corrosion current density (I_{corr}) and increases the inhibition efficiency of anise extract. The presence of inhibitor resulted in a shift of the corrosion potential

(E_{corr}) toward more positive values in comparison to the result obtained in the absence of inhibitor. The inhibition efficiency was estimated to be 90%for anise extract at 0.33g/L.

Table 4: Electrochemical parameters of amalgam at various concentrations of anise extract in synthetic saliva at 37°C.

Concentration of anise extract (g/L)	E_{corr}(mV/SCE)	i_{corr} (mA/cm²)	E%
0	-480.8	33.9	°°°°
0.22	-286.8	19.7	41
0.33	-191	3.3	90

Conclusion

Corrosion problems have received considerable attention because of their economic and security consequences. Indeed, among the methods of protection, the use of corrosion inhibitors consists of an essential technique to limit the attack of materials in various environments. However, these inhibitory molecules must be non-toxic and does not represent any risk for the environment. The objective of this work is to study the effect of the medicinal plant extracts (the anise) on the corrosion of dental amalgam in synthetic saliva.

Electrochemical and gravimetric studies combined with solution analysis show that the addition of the anise extract in the medium results in a reduction of the corrosion rate of dental amalgam in saliva. This action depends on the concentration of inhibitor. The inhibitory efficiency increases with increasing concentration. A good agreement is observed between the gravimetric results and those from electrochemical measurements. Also, the analyses by ICP highlight the preferential dissolution of copper from other metals forming amalgam and show the important inhibitor effect of anise extract on the dissolution of mercury.

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