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Inhibitive action of anise extract on the corrosion of dental amalgam alloy in artificial saliva media

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ABSTRACT

The effect of natural extract of anise plants on the corrosion of dental amalgam in synthetic saliva was investigated by potentiodynamic polarisation techniques, weight loss measurements and surface analysis (SEM). Electrochemical study indicate that the plant extract behave 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 93%.

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KEYWORDS

Anise extract;
Dental alloy;
Saliva;
Inhibition;
Corrosion.

INTRODUCTION

Even though new materials such as ceramics and resins have been developed for dental prosthesis, dental alloys are still necessary for restorative dentistry. Therefore, the safety of dental alloys in the oral cavity of the human body has been extensively investigated^[1-6]. The corrosion resistance is the most important property for dental materials because of biocompatibility and cytotoxicity of the products of the corrosion process. The human mouth presents a very aggressive environment for metals used. To minimize biologic risks, dental alloys showed have the best corrosion resistance and the lowest release of elements. Therefore, it is necessary to improve the corrosion resistance of alloys for dental usage. The interaction of dental alloys with

saliva medium or drugs within the mouth has been studied^[1-6]. One of the most important methods to minimize the corrosion problem is the use of inhibitors. However, the majority of synthetic inhibitors have hazard effects. The need to develop environmentally friendly processes is widely welcomed by the scientific community. Researcher is reoriented to the use of natural products, which are known for their acceptable environmental and ecological properties. Also, naturally occurring antioxidants are cheap, readily available and renewable sources of materials^[7-10].

Silver-tin-mercury alloys are of a considerable importance of their commercial usage as dental restorative materials. The objective of this investigation was to examine the corrosion behavior of silver-tin-mercury dental amalgam alloy in artificial saliva, without and with

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the addition of anise extract, using a potentiostatic technique and gravimetric measurements at a constant temperature of 37 °C.

EXPERIMENTAL SECTION

Preparation of anise extract

100 g of dried plant sample was introduced into a glass fiber filter and extracted in Soxhlet equipment with 750mL of hexane as a solvent during 24 h. The extract was concentrated to dryness and the residue was kept at - 4°C^[11]. This extract was used to study its corrosion inhibition properties on the dental amalgam in artificial saliva media.

Gas chromatography-mass spectrometry (GC-MS)

Anise extracts, sonicated with a mixture of methanol, chloroform and n-hexane, were analyzed using Ultra Trace GC series gas chromatography and a Thermo-Fisher Scientific mass spectrometer. VP-5 capillary fused silica column (30 m x 0.25 mm, 0.25 µm film thickness) was used. Oven temperature was kept at 60°C for 2 min and programmed to 280°C at a rate of 16 °C/min and kept constant at 300°C for 20 min. The carrier gas was He (99.99%) at a rate of 1.4mL/min. The injection temperature and detector temperature were 220°C and 300°C respectively. The injection volume was 1mL with a split ration 1:25. EI/MS were taken at 70eV ionisation energy.

Specimen preparation

The electrode specimen was prepared by mixing in 1:1 mercury 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 are presented in TABLE 1. 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 ₂ Hg ₃
Ag ₃ Hg ₂
Ag ₄ Sn
CuSn

Solutions preparation

Artificial saliva was prepared by 1.12 and 1.24 grams per litter of KCl and NaHCO₃ respectively in aqueous solution with a pH of 8.2. The concentration range of anise extract employed was varied from 0.11g/L (10µL/L) to 0.33g/L (33µL/L). The solutions were not deaerated.

Weight loss measurements

Gravimetric experiments were carried out in a double glass cell. The solution volume was 30mL and experiments were performed with concentration of 0.33g/L of anise extract. The maximum duration of tests was 1 week (168h) at room temperature 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:

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

Where W and W⁰ are the weight loss of dental alloy samples obtained in artificial saliva in the presence and in the absence of inhibitor, respectively.

Polarization measurements

Electrochemical measurements were carried out in a conventional three electrode cylindrical glass cell, containing 100mL of electrolyte at the temperature of 37 ± 1°C. A standard three-electrode cell was used with Platinum electrode as a counter electrode and a saturated calomel electrode (SCE) as a reference electrode. All potentials are reported vs. SCE. The Potentiodynamic curves of dental amalgam specimens in saliva media in the absence and in the presence of anise extract were obtained in the potential range from -1 to +1 V. For polarisation measurements, a potentiostat Voltalab 301 PGZ monitored by a PC computer and Voltmaster 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 following equation:

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

Where I^0 and I are, respectively, the corrosion current densities obtained in saliva media in the absence and the presence of inhibitor.

Surface analysis

The surface morphology and chemical analysis of dental amalgam specimens after polarization measurements in saliva media in the absence and the presence of anise extract were studied using a scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS).

RESULTS AND DISCUSSION

Extraction of the anise

Gas chromatography (GC)-mass spectroscopy (MS) analysis of plant extract showed that the plant extract contains 4 different compounds (TABLE 2). The majority in the extract of both anise of Yemen and Morocco is the 1-methoxy-4-propenyl benzene (Figure 1).

TABLE 2 : GC-MS data of anise extract

Retentiontime (min)	Compound
4.17	Fenchone
5.11	1-methoxy-4-(2-propenyl) Benzene (CAS)
11.73	11-dodecen-1-al
18.02	Oxirane, hexadecyl

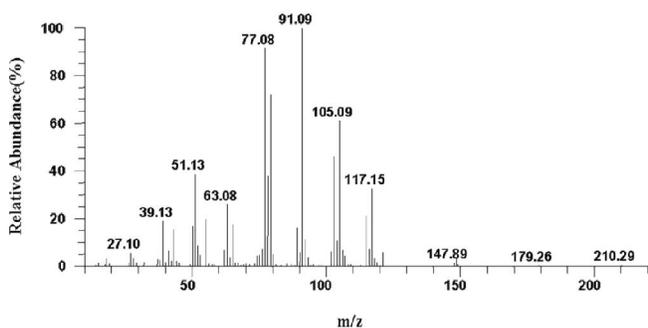


Figure 1 : Mass spectra of anise extract

Weight loss measurements

The corrosion rate and inhibition efficiency for dental amalgam alloy in artificial saliva media at 37°C in the absence and presence of anise extract of Yemen and Morocco are given in TABLE 3. It is evident from these

results that inhibition efficiency for dental amalgam alloy in saliva containing 0.33g/L of plant extract at 37°C is 100 percent.

TABLE 3 : Corrosion parameters obtained from weight loss measurements of dental amalgam in saliva media containing 0.33g/L of anise extract of Yemen and Morocco at 37°C.

Saliva Medium	0.00 g/L	0.33g/L (Anise of Yemen)	0.33g/L (Anise of Morocco)
Time (h)	168	168	168
W(mg cm ² h ⁻¹)	1.03 10 ⁻²	0	0
E%	-	100%	100%

Potentiodynamic polarisation results

Potentiodynamic polarization curves for dental alloy in saliva media in the absence and presence of different of anise extracts concentrations are shown in Figure 2 and 3. As seen, addition of plant extracts affects anodic dissolution of alloy indicating that the extracts could be classified as anodic inhibitors. The corrosion current density was calculated from the intersection of cathodic and anodic Tafel line. The values of the electrochemical parameters for the different plant extract concentrations are given in TABLES 4 and 5. The data show that increasing plant extract 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 negative values by 60-80 mV in comparison to the result obtained in the absence of inhibitor. The inhibition efficiency was estimated to be

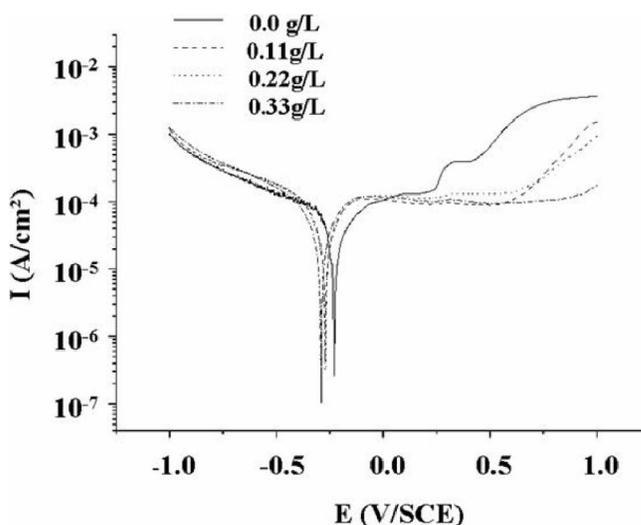


Figure 2 : Polarization curves of dental amalgam alloy in synthetic saliva media at 37 ± 1 °C containing different concentration of anise extract of Yemen.

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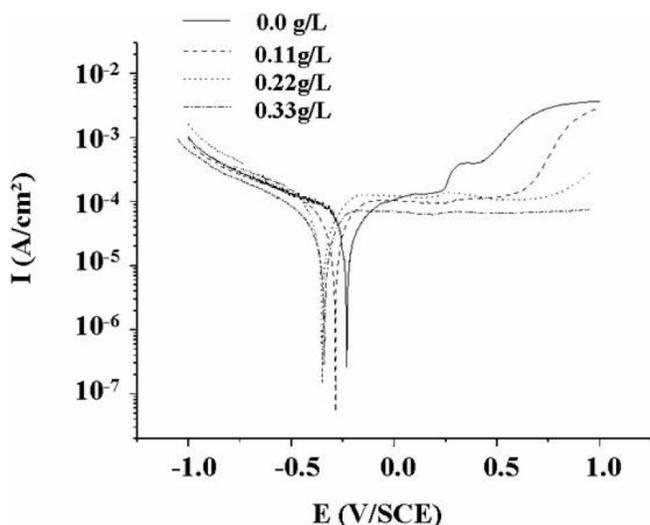


Figure 3 : Polarization curves of dental amalgam alloy in synthetic saliva media at 37 ± 1 °C containing different concentration of anise extract of Morocco.

93.53% for Anise extract of Yemen and 94.52% for Anise extract of Morocco.

SEM examination

To investigate the performance of the inhibitor, an SEM study of dental amalgam was carried out after 168h exposure test at free potential in artificial saliva solution with and without plant extract. Before immersion, dental amalgam alloy was characterised by

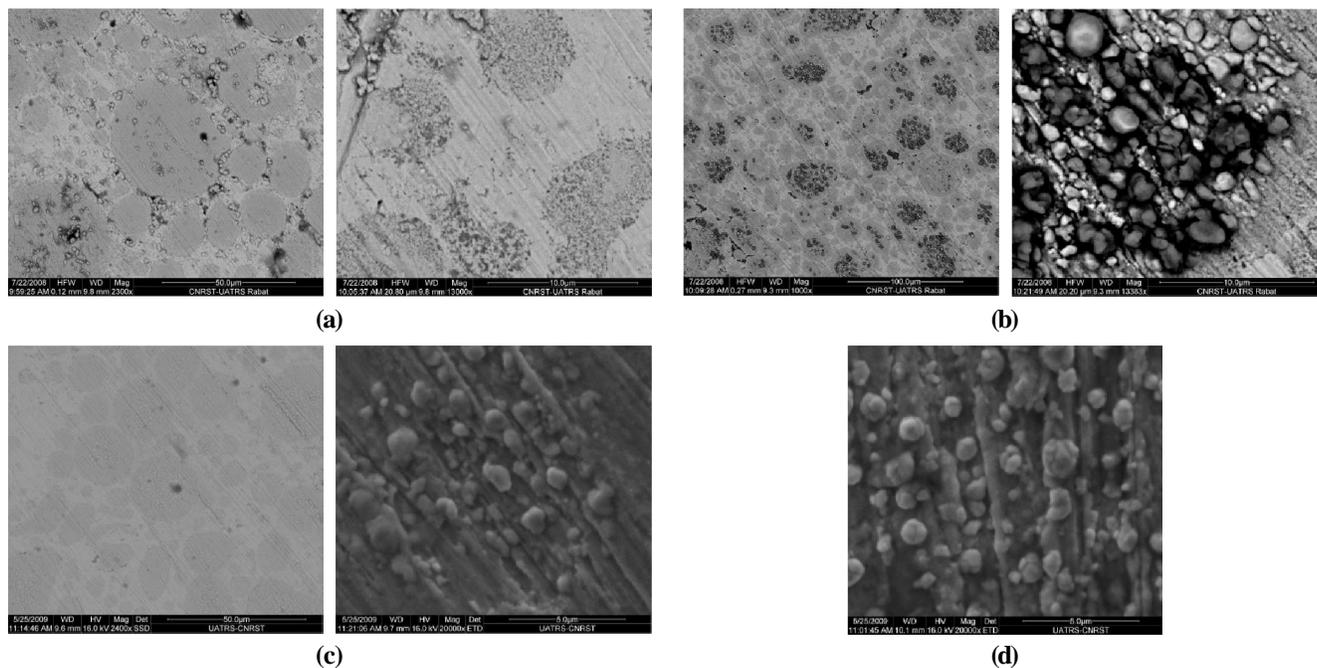


Figure 4 : SEM micrographs of the surface of dental amalgam alloy before and after immersion in synthetic saliva media : (a) before immersion; after immersion (b) in the absence of inhibitor (c) in presence of "anise extract of Yemen (d) in presence of "anise extract of Morocco.

TABLE 4 : Electrochemical parameters (E_{corr} and I_{corr}) and inhibitor efficiency ($E\%$) of the dental amalgam alloy in artificial saliva media at 37°C containing different concentrations of anise extract of Yemen.

Concentration of plant extract (g.L^{-1})	E_{corr} (mV/ECS)	I_{corr} (mA.cm^2)	E (%)
0	-220	1.39	-
0.11	-298	0.106	92.37
0.22	-285	0.112	90.94
0.33	-292	0.090	93.52

TABLE 5 : Electrochemical parameters (E_{corr} and I_{corr}) and inhibitor efficiency ($E\%$) of the dental amalgam alloy in artificial saliva media at 37°C containing different concentrations of anise extract of Morocco.

Concentration of plant extract (g.L^{-1})	E_{corr} (mV/ECS)	I_{corr} (mA.cm^2)	E (%)
0	-220	1.39	-
0.11	-286	0.101	92.37
0.22	-292	0.130	90.64
0.33	-292	0.076	94.53

SEM (Figure 4a). There is thus a complex metallurgical structure, containing different phases, as can be seen in the micrograph of figure 4.

After immersion in artificial saliva media in the

absence and presence of plant extract, the presence of corrosion products on the surface was noticed. In the absence of inhibitor (Figure 4b), the structure of the alloy is still observed and a thick and discontinuous corrosion product layer was observed. EDS analysis shows the presence of oxygen, Ag, Sn and Hg. In the presence of inhibitors (anise extract of Yemen or anise extract of Morocco), the surface was covered by a continuous and uniform layer. The presence of precipitate spheres on the surface was noticed (Figure 4c and 4d).

CONCLUSION

In this study, a new inhibitor molecule was extracted from anise plant and tested for dental amalgam alloy in synthetic saliva media at 37°C. Electrochemical study showed that plant extract is a good corrosion inhibitor and the inhibiting effect results in a reduction of anodic currents. The inhibition efficiency of these extract depends on its concentration and increase as the concentration of anise extract increases.

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