Deposition process of the Platinum/Gold nanoparticle system on inorganic surface, with potential application in bacteria removal

Proceso de deposición del sistema de nanopartículas de Platino/Oro sobre superficie Inorgánica, con potencial aplicación en la eliminación de bacterias

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Abstract

In the present work is presented the procedure to deposit Platinum (Pt) and gold (Au) nanoparticles on an inorganic surface, through the direct current sputtering technique, and using a sputtering system for surface coating. The deposit of nanoparticles on inorganic surfaces has the objective that they can potentially be used for antibacterial action tests. The process of preparing the sheets (surfaces) where the nanoparticles will be deposited is described, establishing a methodology in which the organic impurities must be removed first, and, subsequently, the possible native oxide on the surfaces. For comparison, the depositions were carried out under the following scheme: deposition of Pt nanoparticles, deposition of Au nanoparticles and deposition of the Pt/Au nanoparticle system. In visual terms, the sheets where the Pt/Au nanoparticle system was obtained showed a characteristic coloration. Representatively, the characterization by electron dispersive spectroscopy of a surface with Pt nanoparticles is included, where the presence of this element is observed in the analyzed spectrum.

Nanoparticles, Platinum, Gold, Sputtering

Resumen

En el presente trabajo se presenta el procedimiento para depositar el sistema de nanopartículas de Platino (Pt)/ oro (Au) sobre una superficie inorgánica, a través de la técnica de pulverización catódica de corriente directa, y empleando un sistema de sputtering para recubrimiento de superficies. El deposito de nanopartículas sobre las superficies inorgánicas tiene el objetivo de que puedan potencialmente usarse para pruebas de acción antibacterial. Se describe el proceso de preparación de las láminas (superficies) donde se depositarán las nanopartículas, estableciendo una metodología en la que se deben remover primero las impurezas orgánicas, y, posteriormente, el posible óxido nativo sobre las superficies. A modo comparativo, se realizaron los depósitos bajo el siguiente esquema: depósito de nanopartículas de Pt, depósito de nanopartículas de Au y depósito del sistema de nanopartículas de Pt/Au. En términos visuales, las láminas donde se obtuvo el sistema de nanopartículas de Pt/Au mostraron una coloración característica. De forma representativa, se incluye la caracterización por espectroscopía por dispersión de electrones de una superficie con nanopartículas de Pt, donde se observa la presencia de este elemento en el espectro analizado.

Nanoparticulas, Platino, Oro, Pulverización catódica

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Introduction

The technological advance that has occurred in recent years has become an important factor for the development of new technologies. In some of the sectors in which changes and innovations have been made, they are the health and manufacturing sectors. Innovations are due to different factors, among them, the development of new materials with properties or characteristics different from those currently available. A particular case is the use of materials for surface coating. In one of its forms, this type of coating is made with nanoparticles or nanostructures, and for this, different techniques are used such as: spray pyrolysis, sol-gel, electrodeposition, sputtering, thermal evaporation, ion implantation, chemical deposition in vapor phase, photochemical and radiolytic reduction, microwave irradiation, solvothermal synthesis, among others [1-5].

Nanotechnology is one of the disciplines with a strong potential for the development of products in different branches, such as the sector health, environment, information technologies, energy, agriculture and industry 6, 7]. The synthesis of nanomaterials and nanoparticles for medical uses has led to the generation of nanomedicine, a branch of nanotechnology that allows us to diagnose, treat and prevent diseases and trauma, relieve pain, preserve and improve human health using tools and molecular knowledge of the body human [8]. Metallic nanoparticles such as Au and Ag have been shown to be very efficient in terms of biological applications [9]. In this research, experimental exploration is carried out for the deposition of nanoparticles with metallic materials with 99.99% purity such as Pt and Au. The purpose of this work is to obtain a nanoparticle system through the sputtering technique, which can be evaluated for a potential antibacterial action on inorganic surfaces.

Methodology

The potential application of nanoparticles involves the general methodology presented in Figure 1. At this stage, the proposal focuses on obtaining the Pt/Au nanoparticle system by means of the sputtering technique, using equipment for coating samples to be characterized in scanning electron microscopy. As seen in the diagram, samples were obtained with deposition of Pt nanoparticles, Au nanoparticles and the Pt/Au nanoparticle system.

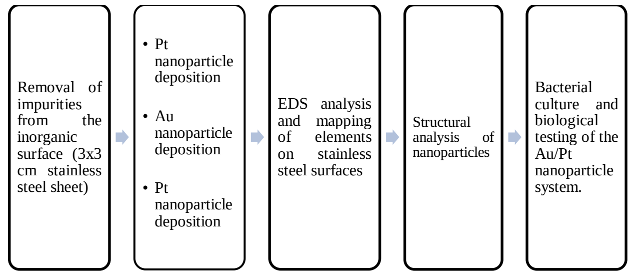

Figure 1 Sequential diagram that presents the main stages for obtaining the Pt/Ag nanoparticle system

Preparation of surfaces (sheets)

To deposit nanoparticles on the inorganic surface, organic and inorganic impurities must be removed. The surfaces were stainless steel plates or copper oxide sheets, with dimensions of 3x3 cm (30 x 30 mm) that were obtained from commercial 22 gauge (0.323 in) plates, as seen in figure 2. For the oxide copper, the copper plates were oxidized in a muffle furnace.

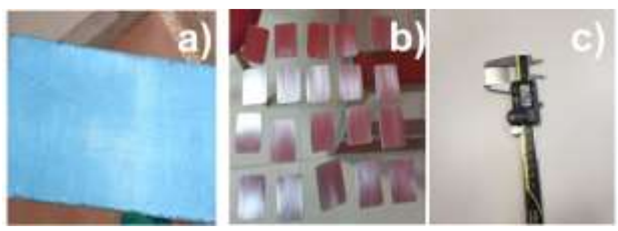

Figure 2 Surfaces where the Pt and Au nanoparticles and the Pt/Au nanoparticle system were deposited. a) Stainless steel sheet in the original dimensions, b) appearance once Cu sheets have been sized, and c) reading the size of the substrates

The steps taken to prepare the surfaces are as follows:

- The first step consists of removing general impurities from the sheets using soap and water (50 ml of water, 10 ml of soap are placed in a beaker). In the basin that has the sonicator, 150 ml of tap water is placed and the beaker is placed. The sonicator is programmed at 50 W for a working time of 10 minutes. At the end of the time, the solution is removed from the beaker and the surfaces are rinsed in running water.
- The second step involves three stages of solvent in the ultrasound system. The first is with Xylene, the second with Acetone, and the third with isopropyl alcohol. Each of these steps is through the sonication process used in the previous stage, with a power of 50 W for 10 minutes. In this way, fats most adhered to the surfaces are removed. Upon completion of degreasing, the sheets are rinsed under running water.

– The third step is the removal of a possible native oxide (only from stainless steel sheets) through a 10% pickling solution of hydrofluoric acid and water, placing it in the sonicator with 50 W for 10 minutes. Finally, the stripping solution is removed, the sheets are rinsed in running water and the moisture is removed with a commercial dryer. These slides are kept in a beaker. Before depositing nanoparticles on the sheets, a label must be marked or placed on one of the two sides, to identify it as the side on which the deposit should not be made, taking into account that it is not always possible. Locate the surface of the sheet where the deposit has been made.

Deposition of Pt and Au nanoparticles

The deposition of the Pt and Au nanoparticles was carried out using the sputtering method, through an AGAR AUTO SPUTTER COATER coating equipment, Model 108a (Figure 3). The targets used have a diameter of 57 mm, with 0.1 mm thickness. For both the Pt and Au blanks, the purity was 99.99%.

The maximum operating time of each target spray cycle on the equipment is 300 seconds. The operation can be manual or automatic. The equipment works by setting the current and vacuum pressure parameters in the reaction chamber (mA/mbar). In the case of Pt nanoparticles, the target was sprayed for 180 s, and for Au nanoparticles, it was sprayed for 60 s.

Figure 3 Surface coating equipment by sputtering, AGAR AUTO SPUTTER COATER, Model 108a

Figure 4 shows the view from above of the sputtering equipment, where important elements can be located such as the cover of the sputtering system, where the metal targets (Pt or Au) are placed, the base, which is the location of the sheets where the nanoparticles are deposited; and the vacuum pump, which during plasma generation, allows increasing the average trajectory that the particles travel before colliding with others. You can also see the cylinder that serves as a reactor and some target supports.

Figure 4 View from above of the sputtering system used for the deposition of the nanoparticles

Characterization

Representative samples were characterized by element analysis, using the energy dispersive spectroscopy (EDS) probe of a Jeol brand scanning electron microscope, model J100.

Results

Figure 5 presents images of the samples obtained from Pt nanoparticles, Au nanoparticles, and the Pt/Au nanoparticle system. In the first column it is observed that the stainless steel sheets have their characteristic light gray color. For the run where the Pt nanoparticles were deposited, the surface exhibits a darker color, indicating the presence of the new metal. In the case of the sheets where Au nanoparticles were deposited, a yellowish coloration is observed, characteristic of bulk Au. Finally, for the samples where the Pt/Au nanoparticle system was deposited, the coloration turns coppery, which shows the combination of both metal nanoparticles. The samples were obtained in three runs as part of the request for subsequent antibacterial tests.

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It should be noted that even under this different coloration in the three types of samples, the electrical continuity recorded by means of a commercial multimeter (STEREN-MUL600) as a function of ohmmeter, indicated a resistance above 10 ohms, which is an indicative that no continuous layers were formed on the surface of the stainless steel sheets, since if this were the case, the resistance measurement would be below 1 ohm. Furthermore, the two metals (Pt and Au) are resistant to oxidation, which also rules out the possible formation of a metal oxide.

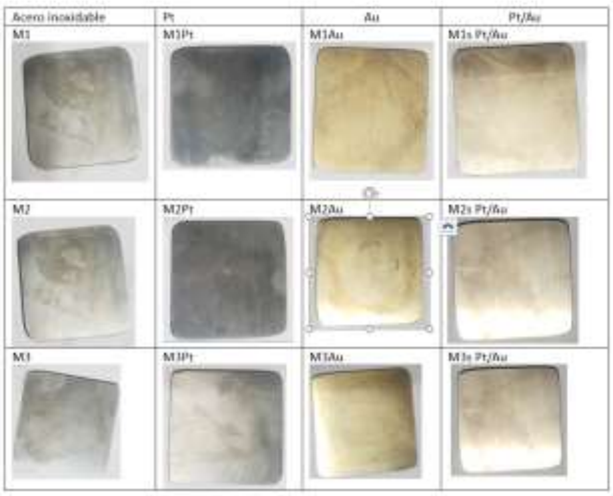

Figure 5 Images showing the stainless steel sheets where the Pt nanoparticles, or the Au nanoparticles, or the Pt/Au nanoparticle system were deposited

Figure 6 shows a scanning electron microscopy micrograph of a representative sample (copper oxide sheet), to which Pt nanoparticles were deposited, and from which the EDS spectrum was obtained. Depending on the magnification of the image, only a homogeneous surface is observed, where no clusters are found that could indicate agglomeration of the Pt nanoparticles.

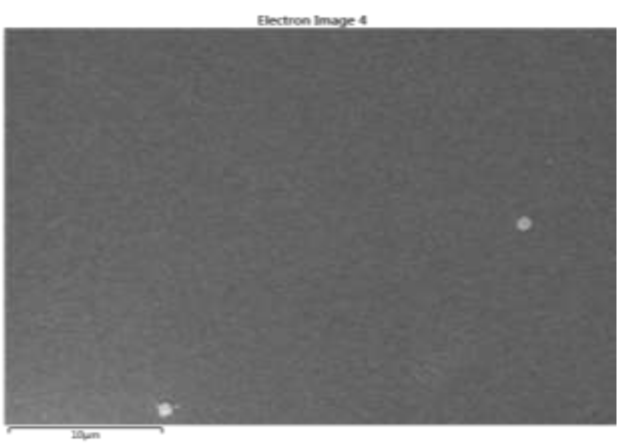

Figure 6 Scanning electron microscopy image, showing the surface of a copper oxide sheet to which Pt nanoparticles were deposited

Figure 7 shows a representative EDS spectrum of a copper oxide sheet, to which Pt nanoparticles were deposited. It is found that the elements present in the sample are Pt, Cu, O and Si. In terms of weight percentage (Wt%), it is observed that Pt appears at 62.9%, which is indicative that the nanoparticles have been deposited on the sheet.

The 17.6 and 10.6% found for copper and oxygen, respectively, correspond to the surface of the copper oxide sheet. Considering that EDS analysis provides superficial information; This result suggests that the electron beam partially hits the surface of the sheet, which again rules out the deposition of a continuous layer of metals. When performing electrical continuity measurements on the surface of this sample, it was found that the electrical resistance is in values of around megaohms, which suggests that the reading is obtained from the copper oxide sheet, and not from the Pt metal. which, if obtained uniformly over the surface, would give a resistance reading of about 0.4 ohms.

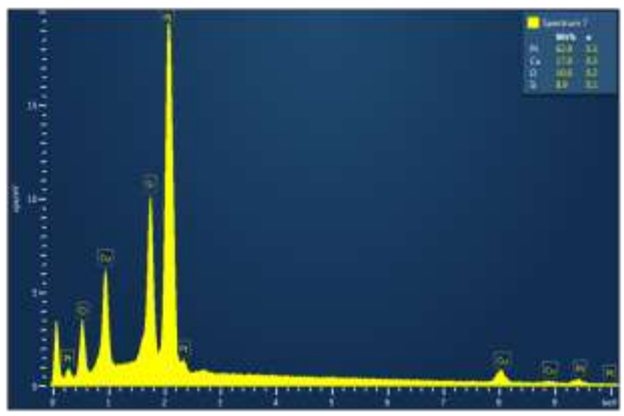

Figure 7 EDS spectrum of a copper oxide sheet used as a surface for the deposition of Pt nanoparticles

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Conclusions

Pt/Au nanoparticle systems were deposited using the sputtering technique. Based on the observation, it was found that the Pt/Au system presents a characteristic color in relation to the deposited Pt and Au nanoparticles individually on the same surface. EDS spectroscopy and electrical continuity measurements allowed us to indirectly determine that the deposition of both metals using the experimental conditions reported here occurs in a dispersed manner rather than promoting the growth of a continuous layer.

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