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Research Article

The corrosion resistance effect of nanocomposites containing AuNPs coated onto titanium surfaces

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ABSTRACT

The purpose of this work was to evaluate the corrosion of commercially pure titanium (cpTi) and polymeric AuNPs nanocomposite thin film coatings on cpTi *in vitro* in artificial saliva by using different electrochemical tests and surface analysis techniques. Electrochemical impedance spectroscopy (EIS), open circuit potential (OCP) and inductively coupled plasma mass spectrometry (ICP-MS) were associated with FESEM for surface characterization. OCP tests revealed higher potential values for AuNPs-Coated surfaces. Increase of the corrosion resistance for AuNPs-Coated surface was presented by EIS in artificial saliva. Average size of 20 - 30 nm AuNPs in spherical morphology forming agglomerations were observed on AuNPs-Coated surfaces. Compared to cpTi surfaces, a lower release tendency of Ti ions from the AuNPs-Coated Ti surfaces after immersion into artificial saliva was confirmed by ICP-MS. Results suggest that AuNPs-Coated Ti surfaces exhibit an enhanced corrosion resistance.

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INTRODUCTION

Titanium (Ti) implants are widely applied in oral and maxillofacial challenges in modern dentistry [1]. Biocompatibility, mechanical properties and corrosion resistance are utmost important factors specifying the choice of dental implant metals and alloys used in oral implantology [2]. Interactions between adjacent tissues and dental implants are determined at the cellular and molecular levels by the cell functions in contact with surface of the dental implants [3].

Titanium is a biocompatible material used as a dental implant material [1]. Its corrosion behavior depends on its stable and adherent oxide film. Chemical proprieties of the oxide film determine the biocompatibility of the titanium implants at the surrounding living tissues [4]. Despite the high corrosion resistance of the oxide film, under certain circumstances oral environment acts as a media for the corrosion of dental implants [1].

Presence of Ti in a biological milieu indicates external contamination in the human body. The presence of

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titanium particles in intra-oral tissues as well as in regional lymph nodes and pulmonary tissue were reported [5]. Mechanical, chemical and microbiological events in oral cavity adversely affect dental implants leading to release of metal ions and particles into the surrounding tissues [4]. This accumulation of metallic ions and particles in a neighboring biological milieu can trigger a variety of inflammatory responses [1]. Composition, electrode potentials, applied stress, adjacent oral environment, surface roughness and chemistry determine the corrosion properties of metals and alloys [2].

The dissolution of the surface oxide by corrosion leads to an increase at the releasing metallic ions from the surface. Corrosion of metal dental implants within the adjacent milieu create adverse effects on biocompatibility and material integrity with the body [6]. This biological response to ionic corrosion products is a concern for implant survival at the living environment [7].

Corrosive (chemical and/or electro-chemical) events may adversely affect the biocompatibility of the dental implants leading to toxicity, allergic reactions or tissue inflammation, which could result in early failure of the dental implant [4].

Inflammation changes the pH conditions at the adjacent milieu in terms of acidification leading to the active dissolution of metal ions around the dental implants. Inflammatory reaction produced by these ions initiate peri-implantitis [8].

In addition to Ti, there are numerous materials available for abutments, cover screws and gingiva formers fabrication in contact with peri-implant tissues that are subject to corrosion. Noble gold (Au)–based alloys may have comparatively reduced strength and hardness but they are more resistant to tribocorrosion. Rather than their mass production, they can be used as coatings on durable biocompatible base materials like titanium to take advantage of their strength against corrosion [9].

The incorporation of AuNPs into nanocomposites for corrosion protection is essential since they enhance corrosion resistance, customize material properties, increase durability, enable self-healing features, and provide a cost-effective solution for maintaining material integrity in harsh environments in long term. There are different methods in the literature for the synthesis of metal/polymer nanocomposites, including chemical, thermal, and photochemical methods. Among these methods, photochemical synthesis is easy to apply, contains little or no solvent, uses low energy, and it is a low-costing fast method for obtaining nanocomposite materials with desired properties due to the photoreduction of the metal salt to the nanoscale with simultaneous polymerization [10,11].

There are several reports on the photochemical in-situ synthesis of some metallic nanoparticles (Ag, Au, Cu, Pt) in polymer matrix. In these studies, various photoinitiators of type I or type II were used to synthesize nanoparticles by the photochemical reduction method [12-15]. Irgacure-2959 is one of these photoinitiators and it is suitable for photocured biomaterials due to its high reactivity, biocompatibility, and water solubility. When exposed to UV light, this photo initiator undergoes Norrish type I cleavage, resulting in the formation of ketyl radicals. These ketyl radicals then act as reducing agents, reducing Au^{3+} to Au^{0}, and following the nucleation reaction the formation of nanoparticles is observed. Photochemical synthesis provides high spatial and temporal resolution of nanoparticles. A slight excess of Irgacure-2959 is required to generate enough ketyl radicals, which will reduce tetrachloroaurate to form Au^{2+}. Ketyl radicals are responsible for the reduction of Au^{3} to Au^{1} and then to Au^{0}, leading to nanoparticle formation (Fig. 1) [15]. In this study, this method was applied to photochemically synthesize AuNPs coating onto cpTi surfaces forming a thin film to enhance the corrosion resistance of titanium.

Therefore, the purpose of this study was to find out the corrosion resistance of uncoated cpTi and cpTi specimens coated with polymeric nanocomposite thin films containing in-situ photochemically prepared AuNPs as well as to determine the level of ICP-MS detection, and quantification of Ti ions collected from two different dental implants surfaces immersed into artificial saliva for avoiding inflammatory responses of the adjacent host tissues. Authors

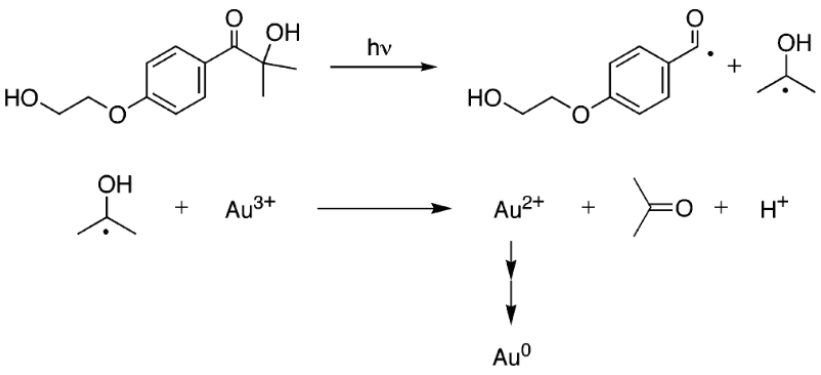
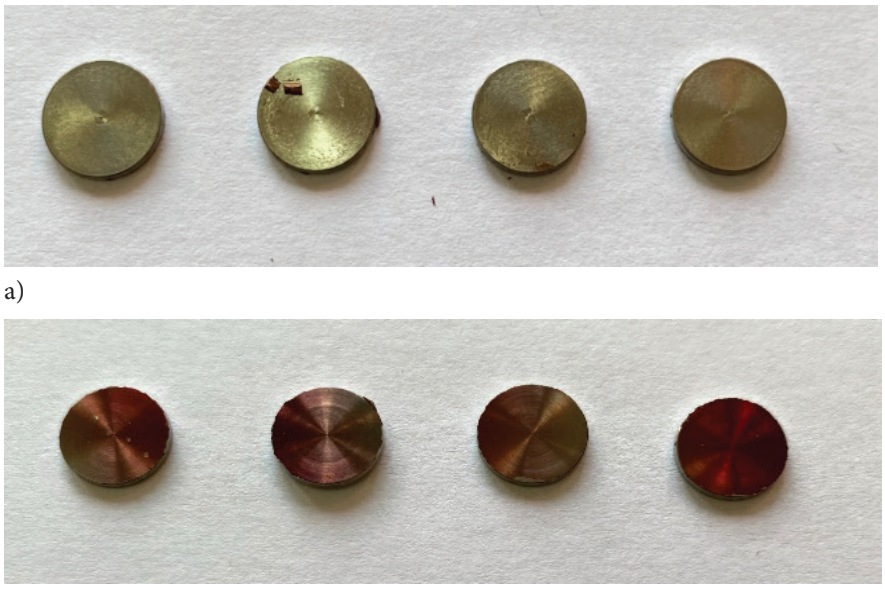


Figure 1. Photochemical preparation of Gold Nanoparticles in the presence of Irg-2959 as photo initiator.



a)

b)

Figure 2. a) Uncoated Ti and b) AuNPs-Coated Ti samples.

hypothesized that nanocomposite coatings containing AuNPs of a dental implant surface may influence the metallic corrosion levels.

MATERIALS AND METHODS

Materials

Poly (ethylene glycol) methyl ether methacrylate (PEGMEA, Mn=480) and Poly (ethylene glycol) diacrylate (PEGDA, Mn=700), were obtained from Sigma-Aldrich. 2-Hydroxy-4′ - (2-hydroxyethoxy)-2-methylpropiophenone (Irgacure 2959 Mw=224.25) was obtained from Ciba Specialty Chemicals. $HAuCl_4$ and dimethyl sulfoxide (DMSO) were obtained from Sigma-Aldrich and used as received. Epoxy resin (Epikote™ Resin MGS LR 135) and hardener (Epikote™ Resin MGS LR 137) were obtained from Hexion Inc. and used in a 10:3 weight ratio.

Preparation of Specimens

The materials used in this study included two groups where the composition of these specimens were commercially pure Ti (cpTi) discs (American Society for Testing and Materials grade 2 titanium, 12 mm in diameter and 5 mm in height) and Au-Coated cpTi discs (12 mm in diameter and 5 mm in height) (Fig. 2).

For the fabrication of Au-Coated cpTi discs, 2-Hydroxy-4′-(2-hydroxyethoxy)-2-methylpropiophenone (Irgacure-2959) (0.5% w/w) as a photoinitiator and $HAuCl_4$ (1 w%) were dissolved in 0.05 ml DMSO. This mixture was then added to a monomer mixture of PEGMEA/PEGDA (19.7%/78.8%) and thoroughly mixed. After preparing the formulation, the mixture was coated onto titanium discs using a spin coater and cured by Mini UV-Cure unit. The next procedure was the annihilation of the cured nanocomposite film on the hot plate for a few minutes.

Four specimens were prepared for each group. All specimens were ultrasonically cleaned with deionized water and 70% propanol and dried with air. Consecutively, specimens of both groups were embedded in epoxy resin.

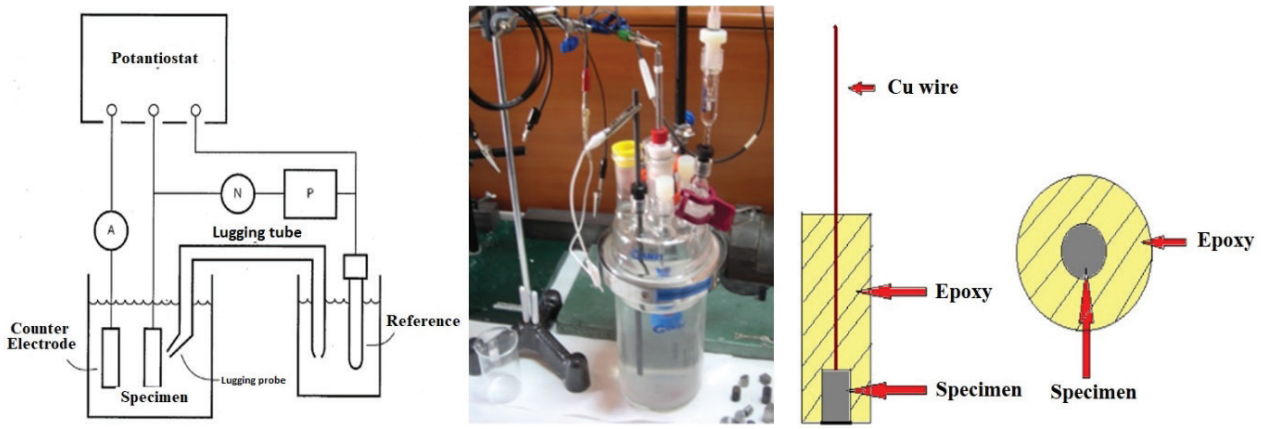
SCANNING ELECTRON MICROSCOPY

Surface characterization of Au-Coated cpTi discs were qualitatively examined by using field emission scanning electron microscopy (FESEM, Thermo Scientific Apreo 2 S LoVac) at a 5 kV accelerating voltage.

Open Circuit Potential (OCP) Analysis

To evaluate the influence of nanocomposite thin films containing AuNPs coating on the corrosion inhibition performance of Ti, uncoated Ti and Au-Coated Ti specimens were examined. After measurement of open circuit potential (E_{OCP}) and its stabilization, potentiodynamic polarization measurements were carried out. The electrochemical tests were performed in an electrochemical cell made of artificial saliva. For each electrochemical corrosion test, 1 L of artificial saliva (electrolyte) solution was used. The composition of the artificial saliva composition was 40 g/L NaCl, 0,80 g/L $CaCl_2$, 0,40 g/L KCl, 0,005 g/L Na_2S, 0,80 g/L NaH_2P04, 1 g/L urea. During the test, the electrolyte temperature was maintained at room temperature.

All measurements were performed by a standardized method of 3-cell electrodes. The potentials were measured relative to that of the saturated calomel electrode (reference electrode; Gamry Instruments), and a graphite rod was used as the counter electrode (graphite rod; Gamry



a) b c)

Figure 3. a) Electrochemical corrosion test set-up, b) photograph of the electrochemical corrosion cell and c) corrosion test specimens.

Instruments). The exposed area (1.131 cm^2; other parts of the discs were isolated by the epoxy resin from the electrolyte) of the cpTi discs and AuNPs-Coated Ti discs were used as a working electrode. The OCP was monitored for a period of 2000 seconds for evaluation of the free corrosion potential of each material in electrolyte solution. For the electrochemical corrosion measurements, a potentiostat (Interface 1000; Gamry Instruments) connected to a computer was used (Fig. 3).

The *Ecor* and *Icor* parameters are obtained by extrapolating the Tafel slopes. The Tafel slopes are also used to obtain the Tafel coefficients: anodic (β_a) and cathodic (β_c). These values are then used to calculate the corrosion rate (CR in mm/year).

Electrochemical Impedance Spectroscopy (EIS)

To investigate the formation, growth, and properties of the oxide layer on the surfaces of the cpTi discs and AuNPs-coated Ti discs, electrochemical impedance spectroscopy (EIS) was used. The electrochemical corrosion measurements were performed using electrochemical impedance spectroscopy (EIS) at the open circuit potential (E_{OCP}) in the frequency range from 104 to 10−3 Hz with an ac amplitude of ±5 mV. Electrochemical impedance spectroscopy data were used to determine the real (Zreal) and imaginary (Zimag) components of the impedance. A potentiostat (Interface 1000; Gamry Instruments) was used for data acquisition.

Static Immersion Test

Four samples from each group have been used for the metal ion recovery test. The 4 samples of each group were placed in the same Eppendorf with 50 mL of artificial saliva and stored at 37 °C. Artificial saliva should be extracted and stored in sterile recipients at 4°C in the refrigerator after 1, 3, 7, 14, 21 and 28 days.

After 28 days, the concentration of released titanium ions was measured by inductively coupled plasma mass spectrometry at the test times indicated above (ICP-MS; Thermo Fisher Scientific, Bremen, Germany). Artificial saliva solution without Ti immersion was used as a reference (blank).

RESULTS AND DISCUSSION

Scanning Electron Microscopy

FESEM was used to examine the morphology and size distribution of AuNPs synthesized as nanocomposite thin films on cpTi discs. The synthesized Au nanoparticles are presented in the Figure 4. The images represent that AuNPs nanoparticles had a spherical morphology. The average size of Au NPs nanoparticles was between 20 - 30 nm and agglomeration was observed.

Open Circuit Potential (OCP)

The open-circuit potentials (OCP; potential measured when no current is flowing) over time of the uncoated Ti and Au-Coated Ti specimens in artificial saliva are depicted in Figure 5. Au coating was found to increase the corrosion inhibition performance of titanium.

The corrosion potentials (*Ecor*) and the corrosion current density were evaluated by the Tafel curves and the obtained data for two groups are presented in Figure 6. The corrosion properties were also evaluated by the potentiostatic polarization curves and the obtained data for two groups are presented in Figure 7.

The potentiodynamic analysis confirmed that AuNPs-coated Ti surfaces with the best corrosion resistance showed the lowest values of corrosion current density (*Icor*) and corrosion rate (Vc) (Table 1). AuNPs coating produced a loss of the corrosion rate with respect to uncoated Ti specimens. The higher the current density at a given potential the more prone was the material to corrode. Among all,

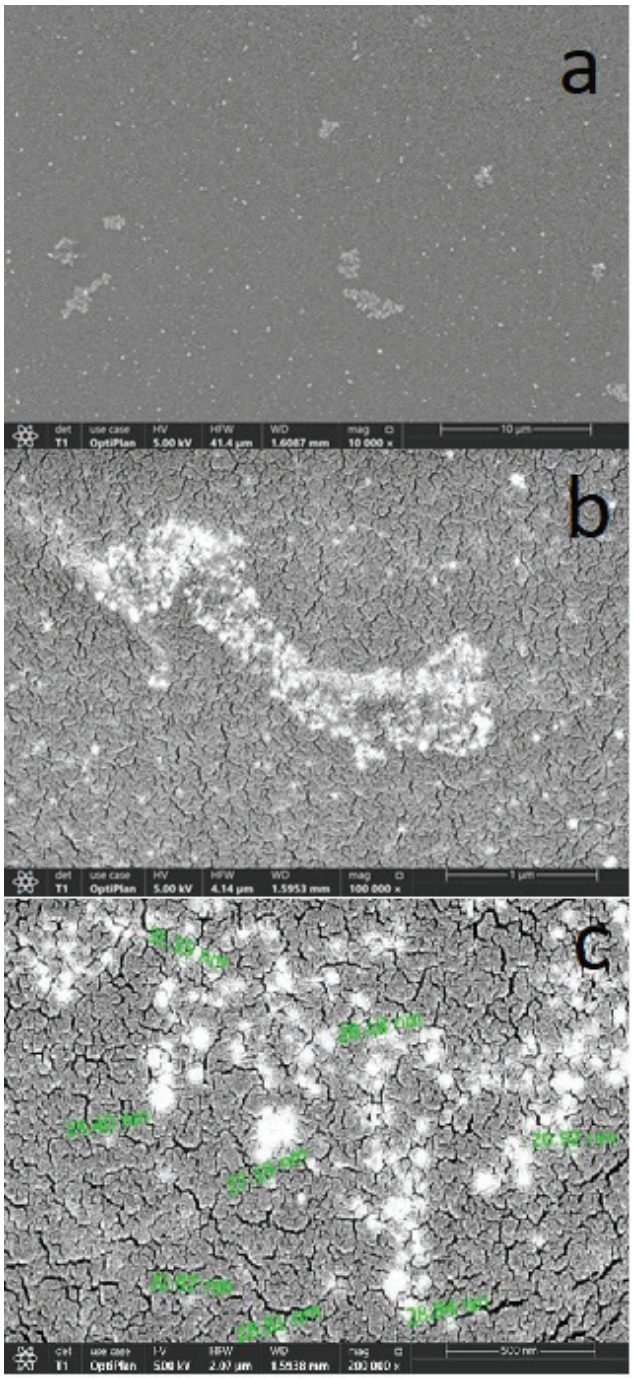


Figure 4. Representative top-view SEM micrographs of AuNPs in nanocomposites (a) original magnification x 10000; bar= 10 µm; (b) original magnification x 100000; bar= 1 µm; (c) original magnification x 200000; bar= 500 nm.

uncoated Ti specimens had the most active, AuNPs-Coated Ti discs had the most noble current density values.

Electrochemical Impedance Spectroscopy (EIS)

The evolution of resultant impedance as a function of Z_{real} and Z_{img} components is represented in Figure 8. EIS results of the nanocomposite thin films on AuNPs-Coated

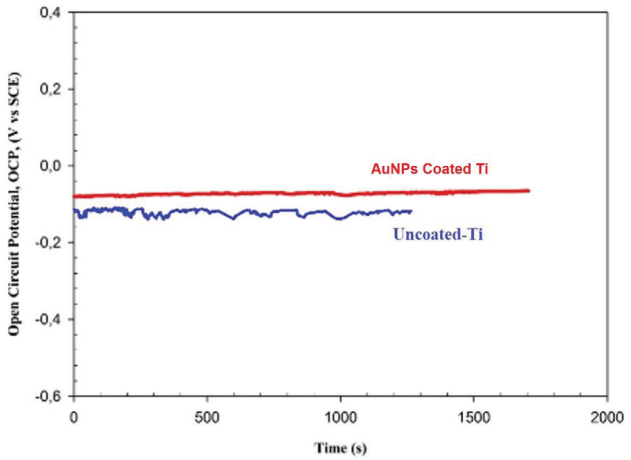


Figure 5. Open-circuit corrosion potential versus time of immersion in artificial saliva of the a) uncoated Ti, b) AuNPs-Coated Ti discs.

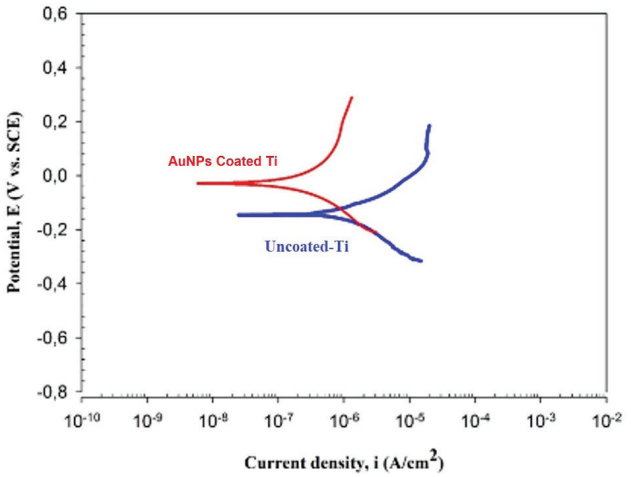


Figure 6. Tafel curves of the uncoated Ti and AuNPs-Coated Ti discs.

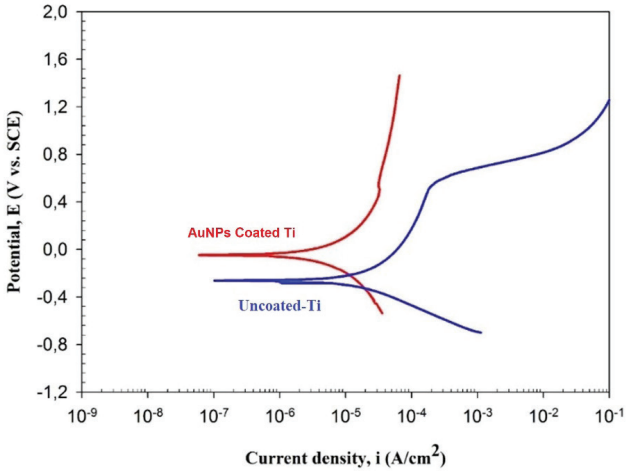


Figure 7. Potentiodynamic polarization curves of the uncoated Ti and AuNPs-Coated Ti discs.

Table 1. The electrochemical corrosion parameters obtained from the Tafel curves

Specimen	$\beta_{anode\ (V/decade)}$	$\beta_{cathode\ (V/decade)}$	E_{cor} (V vs SCE)	I_{cor} (A/cm²)	Corrosion Rate (Vc) mm year^{-1}
Uncoated Ti	0.17	0.15	-0.16	2.10^{-6}	2.11±0.20
AuNPs-coated Ti	0.15	0.16	-0.04	3.10^{-7}	0.21±0.05

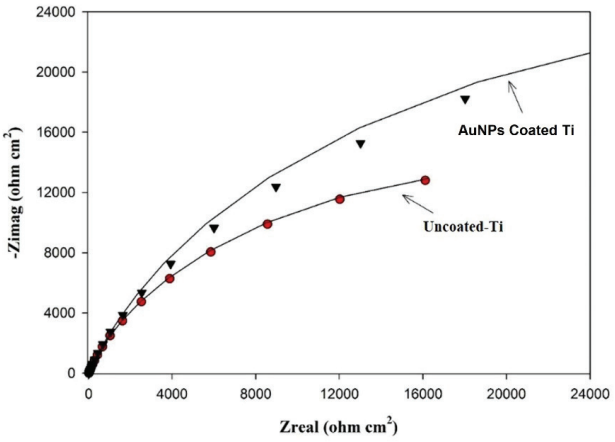


Figure 8. EIS curves of the uncoated Ti and AuNPs-Coated Ti discs.

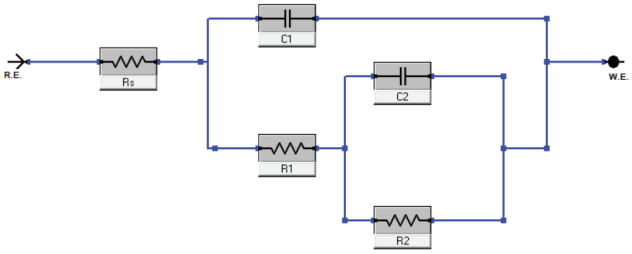


Figure 9. Equivalent electrical circuit for EIS data.

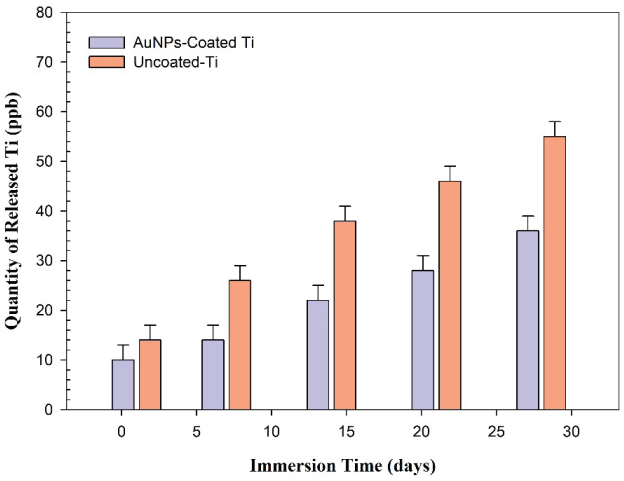


Figure 10. Ti ion release values of the uncoated Ti and AuNPs-Coated Ti discs.

Ti discs clearly showed a positive influence on the electrochemical corrosion resistance in comparison to the uncoated Ti surface revealing structural finesses of AuNPs coated on Ti.

For simulation of the oxide layer properties, equivalent electrical circuit were adapted for electrochemical impedance spectroscopy (EIS) data, which showed resistance of solution (Rsol) in combination of constant phase element (C) and polarization resistance (R) of the surface layer of the titanium in Figure 9.

R_s represents solution resistance, and constant phase element (C) representing shift from ideal capacitor is used instead of capacitance. Fitting quality of the EIS data was judged by chi-squared (χ^2) values of about 10^{-6}, indicating a good fitting to the proposed equivalent electrical circuit (Table 2).

Static Immersion Test

Figure 10 shows the cumulative Ti ion release in parts per billion (ppb) from the different samples studied in artificial saliva after increasing days of incubation. Analogous to the highest electrochemical stability, Ti ion release was the lowest from AuNPs-Coated Ti surfaces, with a total cumulative concentration for all immersion groups. Differences are statistically significant when comparing Ti ion release from AuNPs-Coated Ti surfaces with respect to the uncoated Ti surfaces.

This study investigated the effect of the polymeric nanocomposites containing in-situ photochemically prepared

Table 2. Electrochemical impedance spectroscopy parameters of the uncoated Ti and AuNPs-Coated Ti discs obtained by fitting the equivalent electrical circuit model

Specimen	R_s (Ω cm2)	R_1 (Ω cm²)	C_{pore} (S s^a/cm²)	R_2 (Ω cm²)	C_{barr} (S s^a/cm²)	χ^2
Uncoated Ti	14	7.02	000.4	691	0.016	$6x10^{-6}$
AuNPs-coated Ti	12	16.1	000.3	989	0.021	$3x10^{-6}$

AuNPs coating of cpTi implant surfaces for corrosion resistance. Variety of biomaterials for dental implantation are expected to induce osseointegration of the bone as well as to prevent adverse inflammatory and allergic reactions.

Bacterial biofilm formation, mediated by anaerobic and aerobic bacteria, is followed by microbially-induced corrosion on dental implant metals and alloys. Underneath the biofilm, the anodic sites are created where corrosion can occur by the releasing metal ions into the saliva. Saliva's chloride ions or the bacteria's end-products generate more reactive species causing the metal being corroded further [16].

Significantly increased levels of dissolved titanium in submucosal plaque of implants with peri-implantitis, indicated an association between titanium dissolution and peri-implantitis [17]. The dissolution of non-bioinert Ti ions were found to play a role as secondary stimuli for inflammatory activation in human macrophages leading to IL-1β release in peri-implantitis [18].

Metal particles and ions in the peri-implant area were found accumulated in both soft tissue and bone next to implant bulk material [19]. In addition to Ti and its alloys, there are numerous materials in contact with peri-implant tissues fabricated as of abutments, gingiva formers and cover screws that are subject to corrosion. The study by Flatebø et al. [7] demonstrated the presence of corrosion particles in the tissue adjacent to Ti cover-screws. In addition to their statement, it was reported that these Ti particles may be released during insertion of the fixture and be retained in the covering soft tissue [7]. It was stated that modification of the dental implant surfaces with noble metal coatings such as palladium, gold and silver could prevent bacterial adhesion and surface corrosion leading to stimulation of osseointegration. Strategies to prevent Ti ion release by surface corrosion include coating the Ti surfaces with Au on and in close contact with the adjacent bone and soft tissues thereby facilitating the host defense and reducing the presence of corrosion particles nearby [20].

Zainali et al. [21] documented that AuTi coating on experimental porous implants showed no negative results in early mechanical strength, implant osseointegration, periimplant bone density, and bone remodeling activity. Abutment composition coated with Au were found to influence ion release increasing short-term mechanical strength and osseointegration [9].

The immobilized double layers of gold nanoparticles at the surface of dental implants were found to promote the proliferation of cells without cytotoxicity both in *in vitro* tests and *in vivo* models in osteoporotic patients as osteogenic agents. Gold nanoparticles were found to induce osteogenic differentiation of mesenchymal stem cells and activation of p38 mitogen-activated protein kinase signaling pathway causing the up-regulation of runt-related transcription factor 2 responsible for the essential transcription factor for osteoblast differentiation [22].

In our study, as a photoinitiator, Irgacure-2959 was used for the in-situ synthesis of Gold nanoparticles in polymer matrix by the photochemical reduction as an alternative method for Gold coating. AuNPs morphology were revealed as agglomerated spheres on cpTi substrates by FESEM. On the other hand, the high OCP values for AuNPs-Coated surfaces showed higher corrosion resistance in artificial saliva. Tafel curves presented high potential and low current values for AuNPs-Coated surfaces whereas cpTi surfaces presented lower potential and high current density. Potantiodynamic polarization curves showed passivation for both specimens, while AuNPs-Coated surface showed a decrease in corrosion rate. EIS data for AuNPs also correlates with these data presenting higher corrosion resistance compared to uncoated-Ti. These findings on corrosion were confirmed by detecting the release of Ti ions in the artificial saliva induced during the potentiodynamic polarization analysis. Uncoated-Ti released more Ti ions for all the immersion times. In-situ synthesis of Gold nanoparticles in polymer matrix by the photochemical reduction for the coating of cpTi surfaces increased the corrosion resistance of cpTi surfaces and lowered the Ti ion release in the artificial saliva. This coating can be used at titanium dental implants for the coating of special parts of the implant such as cover screws, gingiva formers and abutments. These parts of the implant are in contact with the gingiva and the saliva. Increasing the corrosion rate of the titanium with AuNPs would lower the Ti ion release into the adjacent gingival soft tissues and the surrounding salivary fluid. This AuNPs coating in our study is pink in color, which makes it a candidate for good pink esthetics matching with the neighboring gingival tissues rather than the titanium parts grey in color.

CONCLUSION

Surface features of dental implants can be potentially modified to add further corrosion resistance for Ti ion release. The polymeric nanocomposites containing in-situ photochemically prepared AuNPs coating could add extra corrosion resistance to lower the accumulation of Ti ions in the soft tissue and bone adjacent to dental implants and would be a candidate for pink esthetics. As a major limitation of this primary study, additional data from *in vitro* biocompatibility tests and *in vivo* human trials are required to validate this assumption for a better understanding of the long-term clinical performance of this in-situ photochemically prepared AuNPs coating.

AUTHORSHIP CONTRIBUTIONS

Authors equally contributed to this work.

DATA AVAILABILITY STATEMENT

The authors confirm that the data that supports the findings of this study are available within the article. Raw data that support the finding of this study are available from the corresponding author, upon reasonable request.

CONFLICT OF INTEREST

The author declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

ETHICS

There are no ethical issues with the publication of this manuscript.

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