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Fabrication and Characterization of Anti Corrosion Titnia-Nanocompsite Coatings for Biomedical Applications

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Abstract

Titanium dioxide nanoparticles merged with chitosan biopolymer as coatings were deposited on 316L SS substrate via pulsed-cathodic electrophoretic deposition mode (PCEPD) technique with different duty cycle and deposition time to enhance implant surface and electrochemical performance of biomedical implant in ringer solution. A homogeneous distribution of the Titania nanoparticles in coatings with crack free were obtained for utilizing in medical field. Surface, morphological and structural characterization of the composite coatings were investigated by using Field Emission Scan Electron Microscope (FE-SEM), Fourier transform infrared spectroscopy (FT-IR) and X-RAY diffraction technique. The corrosion behavior of the coated substrate was evaluated using potentiodynamic polarization technique (PDP) in Ringer's solution at 37 ºC. The results showed that the chitosan-TiO_2 coated 316L SS substrate exhibited better corrosion resistance compared to the uncoated one.

Keyword: Titanium oxide, Chitosan, FE-SEM, PCEPD and PDP

Introduction

The metallic implants using in various medical applications has significantly increased in recent years. In the biological conditions, however the metallic implants can cause fail as a result of corrosion that posing a serious health problems. To overcome this challenge, the researchers have been focused on creating as well as developing anti-corrosion coatings for resisting corrosion reaction in the biomedical implants[1,2]. The anti-corrosion coatings can inhibit, slow down or minimize the deterioration of implant materials. These coatings can reduce the implant exposure to the corrosive agents by acting as a barrier against corrosive agents in the biological environment [2-4]. Several coating techniques have been developed for depositing different coatings onto the surface of implants, including electroplating technique, physical vapor deposition (PVD), chemical vapor deposition (CVD), and electrophoretic deposition (EPD) [5]. Each technique has its limitations and advantages addressed as coating quality, processing cost and adhesion to the substrates. Electrophoretic deposition (EPD) technique is a versatile technique that used for thin films of different materials to deposit onto the various substrates [3 , 4]. EPD can be utilized to apply coatings onto the biomedical implant surfaces to enhance their

functionality and longevity. The EPD process involves applying an electric field to a suspension of charged particles, which then migrate towards and deposit onto the substrate surface [6].

High and uniform deposition efficiency, control the film thickness, reduced waste and not requirement for post-treatment processes, making it more environmentally friendly, cost-effective can be performed at room temperature and atmospheric pressure, requiring relatively simple equipment all these advantages making the EPD process a versatile deposition process used widely in various field including the production of film, coatings and composites. Organic/inorganic composite coatings produced by EPD offer the advantage of a better adhesion between bone tissue and implant by providing a "soft" interface essential for the success of orthopedic and dental implants as well as avoided need to sintering steps after deposition [7-12].

Chitosan CS is a versatile and widely used type of natural polysaccharide derived from alkaline de-acetylation of chitin which extracted from the exoskeletons of crustaceans found in crab, shrimp, as well as fungi cell walls composed of -1, 4-N-acetyl- D-glucosamine units in different degree of de-acetylation exhibited a biological activity make its suitable for applicator in different fields as in materials engineering and biomedical engineering [13-15].

Besara, et.al, prepared a dense, bubble free Alumina films deposited by pulsed electrophoretic deposition technique with a 50% duty cycle compared to DC-EPD [16 and 17]. Electrophoretic deposition in acidic solution of chitosan (CS) at (0.5-10) g/l of TiO_2 nanoparticles were investigated by Cordero-Arias et. al [18], the best deposition parameters were 25volt, 1min that provides film coatings with (2-6)µm. Anticorrosion resistance in Dulbecco's MEM (DMEM) solution at 37 ^{o}c were enhance with increasing ceramic content added to improvement of contact angles to be 25º to 54º .

Cathodo-phoretic deposition of chitosan (nc-TiO2) composite coatings on X_2CrNiMo17-12-2 from 50:50 water to ethanol suspension containing 1% acetic acid with the optimal deposition parameter as 14 and 16 V, pH in the range between 3.0 and 4.2, a uniform and crack-free deposits were obtained with a lower roughness in a suspension containing 0.5 g/l and 3 g/l TiO2 and enhancing corrosion resistance of the coatings in Ringer's solution compared that the uncoated X2CrNiMo17-12-2 steel [19]. Jos´e Oliveira and co-authors studied the effect of applied voltage on deposition of chitosan-molybdenum Cs-Mo composite coatings on 1020 carbon steel substrate. Results show a uniform, homogeneous distributed films at 5 volt and 5.5 pH with enhancing anticorrosion behavior in saline solution [9].

The goal of the present work was the fabrication of organic–inorganic nanocomposite coatings containing TiO_2 and chitosan as coating used for biomedical applications and investigate its corrosion resistance in simulated body fluid (SBF). The targeted was the fabrication of uniform, crack free, homogeneous distribution with high coverage area of

composite coatings with optimal deposition parameters through application of pulsed voltage mode cathodic electrophoretic deposition (PCEPD).

Materials and Methods

1. Materials

Chitosan has medium molecular weight (M_{Wt} = 80 kDa) with 85% degree of de-acetylation soluble in (1vol % acetic acid) purchased from Sigma Aldrich was used to deposit on 316L stainless steel substrates surfaces having an area of (10×10×20) mm and a chemical composition listed in Table1. TiO_2 Nano powder has a particles' size of about 30 to100 4nm with 99% purity supplied by (Sigma Aldrich) was used to deposit it as coating layers. Solvents of absolute ethanol, deionized water (DI) and acetic acid were used to prepare deposition solution.

Table.1 Chemical Composition of 316L SS Alloy.

Elements	C	P	Mn	Si	Al	Cu	Cr	Ni	Fe
wt.%	0.0255	0.0405	1.45	0.330	0.0021	0.481	17.63	10.85	Bal.

2. PCEPD of prepared suspension

First of all, the 316L stainless steel surfaces were grinding and polishing using a silicon carbide (SiC) grit abrasive paper up 2000 mesh, then after polishing, ultrasonically in alcohol (ethanol) for about 15 min and washed with distilled water then finally rinsed with acetone and drying with hot air for keeping in the vacuum desiccators in order to protective them from air oxidation before the deposition step.

The most important step in the PCEPD is the preparation of a stable aqueous suspension starting with dissolved 0.5g/L of bio chitosan polymer in 1wt% acetic acid solution until complete dissolving then 79% absolute ethanol was added, stirring the solution with magnetic stirrer for about 15 minutes before the addition of 6g/l TiO_2 nano-powder passed on previous studies [20]. All the prepared suspensions having 4 ∓ 0.2 as a pH were de-agglomerated via magnetically stirrer assisted by high-energy (Ultrasonic Processor, MIXSONIX Incorporated N.Y, USA) sonicator for 30 min.

The cathodic pulsed PCEPD cell configuration consists of a beaker containing two electrodes: 316L stainless steel electrodes with the same surface area immersed in the suspension as a working and a counter electrodes at 1cm separating distance to ensure the reproducibility of EPD. All coated Samples with $1cm^2$ surface area were weighted using (DENVER ISO9001 type T.P214) instrument before and after deposition to calculate the weight gain [21].

For the selection of the appropriate deposition conditions of CS matrix were reinforced with TiO_2 cracked free and homogenous distributed thin films, several trials were done via testing the applied voltage (10-20), solution concentration 6 g/l at deposition time (1, 3 and 5) min and duty cycle (DC) of about (30-80)% to select the best conditions controlled via optical observation and weight gain "deposition rate".

3. Characterization of the Organic/Inorganic Composite Coats

Characterization of the deposited films structure were done by using FTIR spectrometer, X-ray diffraction (XRD), whereas FE-SEM tests the morphological and cross section analysis of the surface at deposition conditions as well as EDX analysis of the deposited films. Electrochemical technique for Vertex potentiostat, Netherlands via potentiodynamic polarization test was examine the corrosion behavior of the bare 316L SS and coated substrate at the optimized conditions in ringer solution at 37 ºC.

Results and discussion

1. Deposition kinetics

Two groups were investigated in this research; the first one deals with duty cycle range as (30, 50 and 80) %, while the second one deals with time deposition change as 1, 3 and 5 min at a constant concentration of CS and TiO_2 nano particles.

The CS macromolecules is considered as interconnection among the ceramic nano particles (TiO_2) under Vander Waals force resulting in improving in the distribution of particles thus allowing the cathodic depositions forming films [9]. So for improvement of the effective deposition of the coatings, the deposition yield was calculated as shown in Fig. 1 for varying duty cycle at 5 min (300sec) of deposition time.

The weights for deposited coatings from alcoholic/water suspensions containing 6 g/L of TiO_2 shown in Figure.2 which illustrates that the deposition yield shows increasing with increasing duty cycle from 30-80 % for the 0.5 g/l of chitosan.

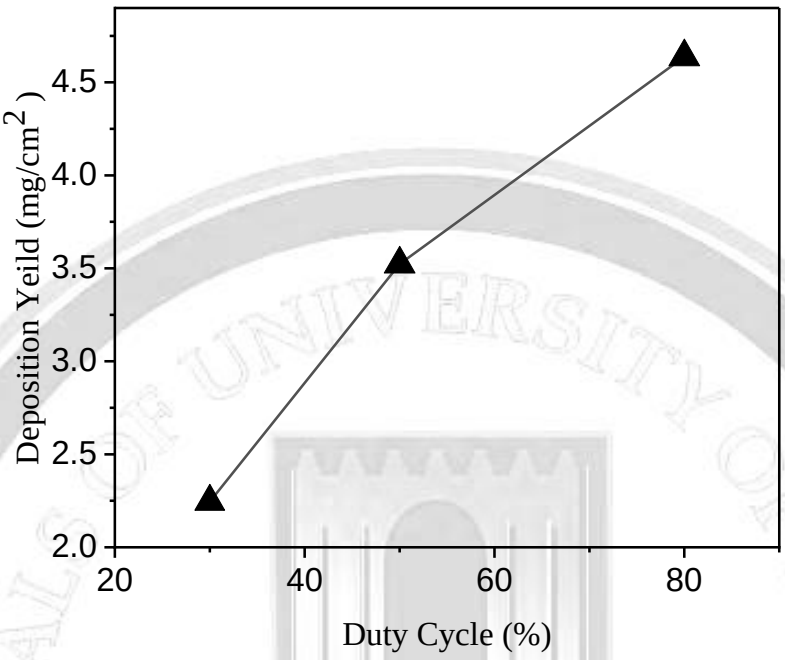

Fig.1. Deposited weight gain vs. duty cycle % of TiO_2-CS composite coatings.

Fig.1 shows a linear increase in deposition yield against Duty cycle varying in the range of (30-80) %, the optical observation shows that increasing duty cycle a crack is obtained while duty cycle at 50% and 30% shows a thin film and crack free deposits this is due to increasing duty cycle means increasing pulse oN time (T_{on})and decreasing relaxation time pulse off time (T_{off})this result in agreement with L.besra, et.al[13, 21].

As the electrolysis reaction of water is generally considered as a slow electrochemical reaction, and with applied a short pulse duration, the slow reaction kinetics could prevent the products from formation. Furthermore, when the relaxation time between pulses was long enough, the products formed by electrolysis or half reaction's should be able to diffuse away from the electrodes, thus hampering the bubbles nucleation [21].

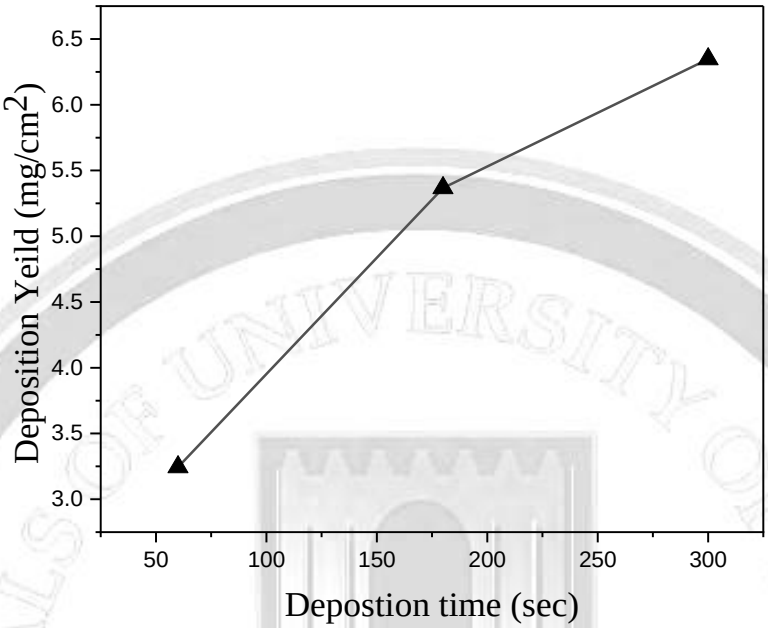

Fig.2 Deposited time effect on weight gain.

2.Characterization of deposited films

The FTIR spectrum of the powders and deposited composite coatings containing chitosan as adhesive to the nano-titania particles are shown in Fig.3. The results confirms the chemical structure of the deposited film with the overlap absorption bands of O-H and N-H appeared in the range of 3000-3500 cm-1 in pure CS powder compared to less intensive FTIR spectra of the deposited CS-TiO2 film [22] which may be attributed to the molecular interaction between CS functional groups and titania nanoparticles . The band detected at 1000 Cm^{-1} in CS-TiO_2 film related to O–Ti–C bond confirmed that a chemical bonding occurs as well as adsorption into the chitosan matrix [23-25].

The bands range 1150-1026 cm^{-1} related to the glycoside- linkages of the saccharine units in CS matrix. 1262-1380 cm^{-1} corresponding to amine groups I and II in chitosan and C-N bonds [9,26].The peaks between 1380 and 1419 cm^{-1} were assigned to the –CH symmetrical band. The peaks ranging from 1593 cm^{-1} may be attributed to the vibrational bending of –NH group.

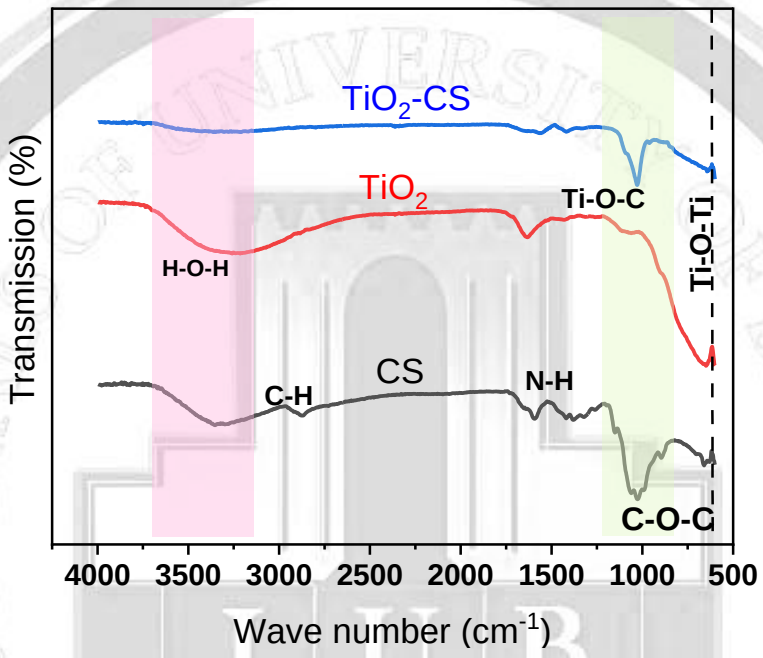

Fig.3 the FTIR spectrum of CS, TiO2 and Composite coating.

For further characterization of the deposited film, the XRD results of the TiO_2 and TiO_2-CS film were examined at 2Θ º of (20-85) are shown in Fig.4. The diffraction patterns and phases of the TiO_2 with its composite that indexed in JCPDS 00-021-1272 for anatase as (25.6, 38, 44, 49, 51 and 55)º related to (101, 103, 004, 200, 212) miller indices respectively. Therefore, the results concluded that the TiO_2-CS coating was successfully deposited on the 316L SS surface by one step pulses EPD technique at room temperature without needing for heat treatment in case of deposition of Titania nanoparticles in alcoholic solution, this behavior related to using of biopolymer CS as a binder for coatings constituents [26].

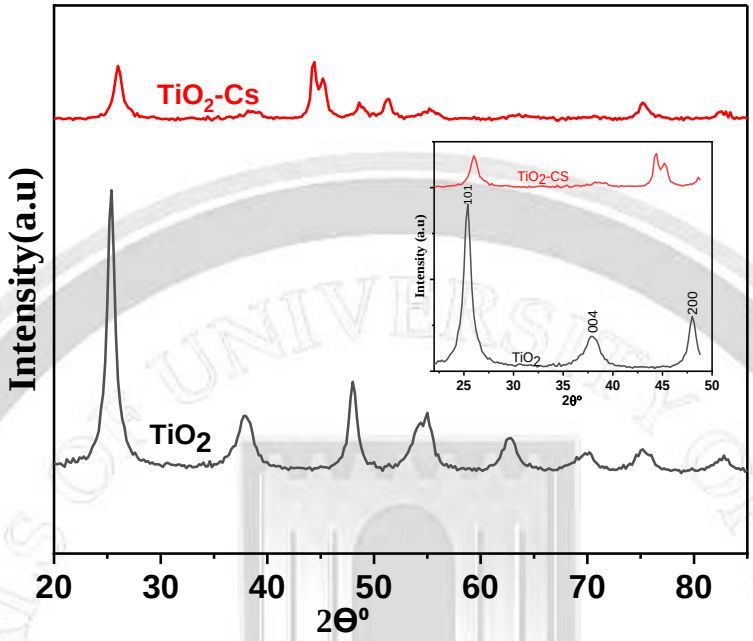

Fig.4 The XRD patterns of TiO_2 powder and TiO_2-CS film.

Morphologies of TiO2-CS coatings by EPD at 300 sec was studied via field emission scanning electron microscopy (FE-SEM) associated with Energy Dispersive X-ray (EDX) microanalysis technique. The micrographs of the deposited films are presented in Fig.5a with different magnifications, which confirms a uniform, successful homogeneous deposition with some agglomerations of TiO2-CS on 316L SS. Furthermore, the cross-sectional view in Fig.5b of the coating measured the thickness of about 20µm ± 1µm. Hence, the findings provide strongly supported evidence of the optimized parameters of PCEPD technique for suitable, homogeneous deposited crack-free coatings. Additionally, the study demonstrated that the coatings adhere firmly to the substrate without any underlying cracks as a result of chitosan's incorporation in the coatings resulted in decreased degradability due to its functions as a dispersant, charging additive, and enhancer of interfacial bonding, allowing the coatings to adhere well at room temperature[27,28].

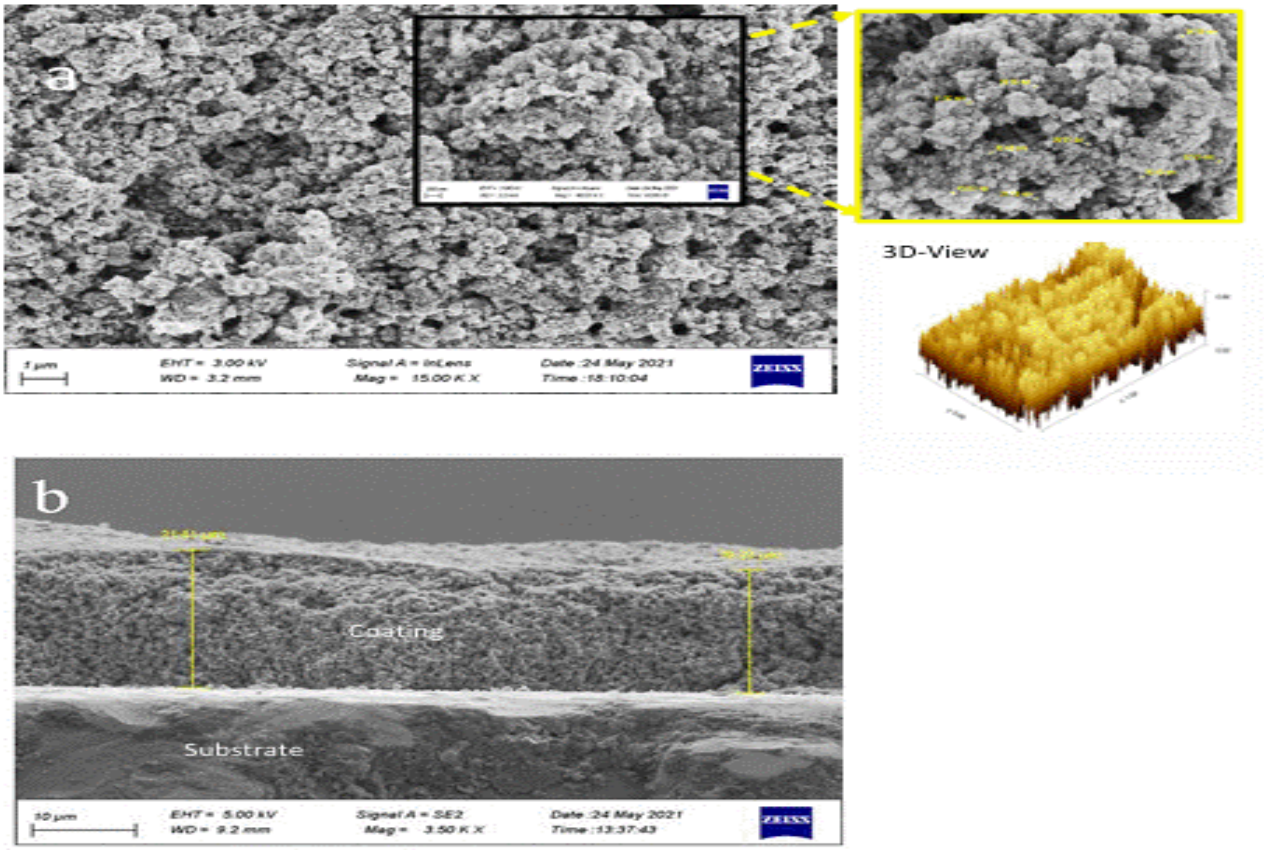

Fig.5 FE-SEM micrograph (a) and (b) cross sectional views of TiO_2-CS deposited at 300 sec at 50% DC.

The chemical elemental analysis of the EPD composite coatings were investigated by using energy-dispersive X-ray (EDX) analysis. EDX spectra of the TiO_2-CS is shown in Fig. 6. The EDX spectra present clear and distinct peaks of carbon and oxygen elements that related to CS matrix. Moreover, the peaks of Ti in the (EDX) spectra corresponded to the TiO_2 element in the composite coatings. The scanning across the coated samples provide clear view of the Ti homogeneity in the coatings without contamination present in the synthesis of the TiO_2-CS film via EPD technique.

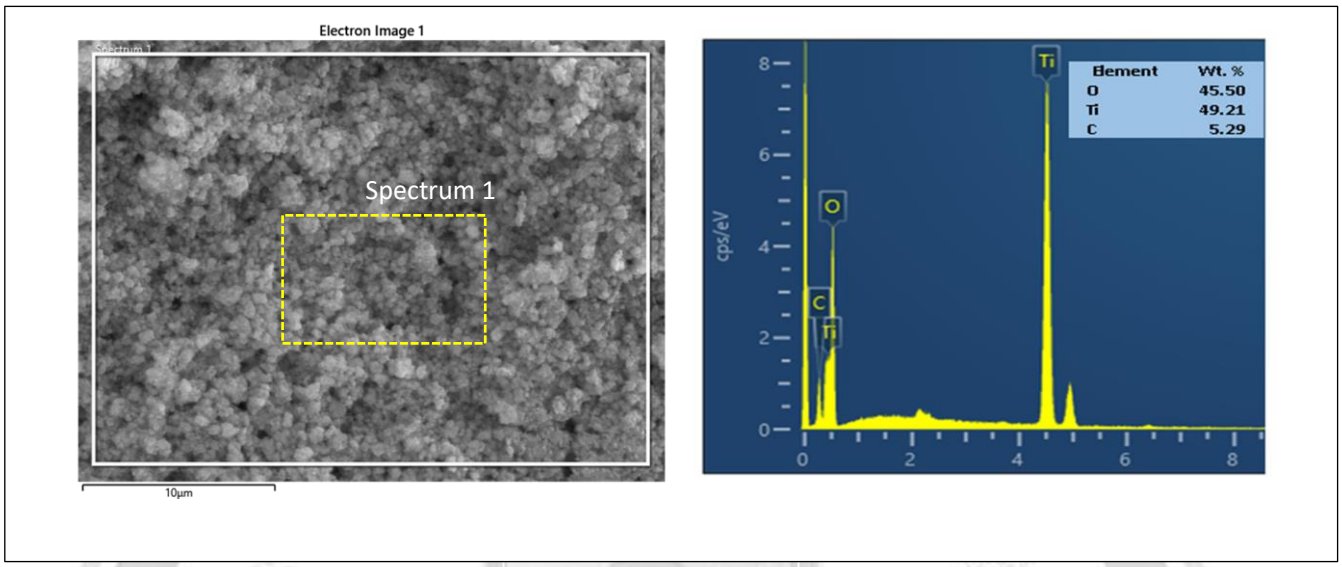

Fig.6. The EDX spectra of the TiO_2-CS composite coating.

3.Electrochemical Characterization

Corrosion resistance results in the un-coated and coated 316L SS specimens with 6g/L TiO_2-CS coatings deposited at 300 sec for 50% duty cycle were investigated by potentiodynamic polarization curves (PDP) in Ringer's solution at 37 ºC. The results are shown Fig. 7 and listed in Table 2. The curve related to composite coated stainless steel revealed a higher corrosion potential E_{corr} in the passivation direction than uncoated substrate associated with lower corrosion current density. Therefore, it can be concluded that a higher corrosion resistance was obtained through coating of the TiO_2-CS than the uncoated 316L SS substrate in aggressive solution, the same results were confirmed in another studies [28-32].

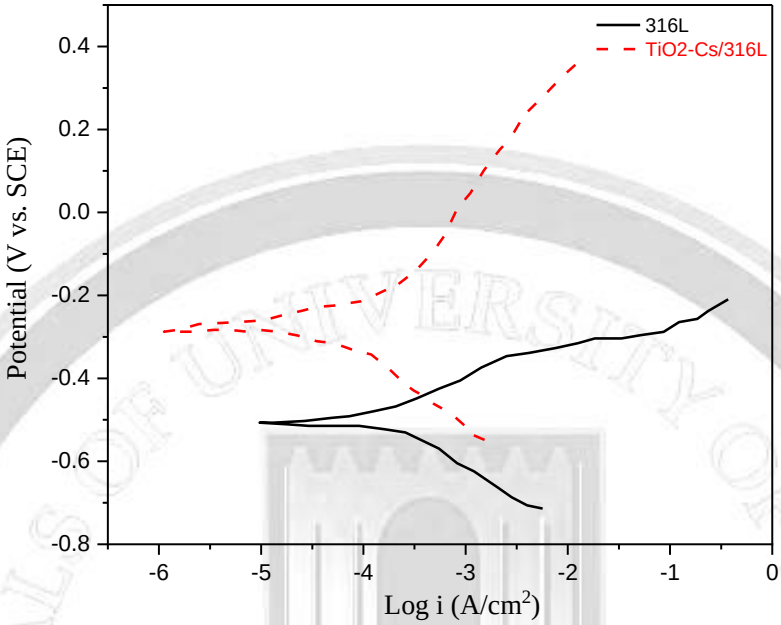

Fig.7 Potentiodynamic polarization curve of the un-coated and 6g/l TiO_2-CS coated substrate in Ringer solution at 37 ºC.

Table 2. Potentiodynamic polarization extracted data.

Sample	i_{corr} (mA/cm^2)	E_{corr} (mV_{SCE})
316L	1.35*10-1	-506
TiO_2-Cs/316L	3.16*10-2	-268

5.1 Conclusions

Pulse-mode electrophoretic deposition of nano TiO_2 particles in chitosan matrix were successfully deposited on 316L SS substrate with smooth, higher yields, uniform and crack free distribution at optimized deposited conditions of 50% DC and 300sec.

A finer matrix was observed and this behavior increased with decreasing duty cycle due to more nucleation and diffusion dominated kinetics. Corrosion parameter extracted from potentiodynamic polarization curves for uncoated and coated 316L SS concluded a lower corrosion rate with higher corrosion resistance of coated substrate vs. un-coated sample.

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الجامعة التكنولوجيا/ قسم هندسة المواد

الخلاصة

تم في هذا البحث ترسيب طلاءات محضرة من البوليمر الحيوي الشيتوسان مع الجسميات النانوية لثاني أوكسيد التيتانيوم بطريقة الترسيب الكهربي النبضي الكاثودي لتحسين سطع المزروعات الطبية لمعدن الفولاذ المقاوم للصدا 316l والاداء الكهروكيميائي في محلول رنجر. اظهرت النتائج الحصول على طلاءات ذات توزيع متجانس لجسيمات التيتانيا النانوية وخالية من الشقوق لاستخدامها في المجال الطبي. تم دراسة الخصائص السطحية والمورفولوجية والهيكلية للطلاءات المتراكبة باستخدام المجهر الإلكتروني الماسح للانبعاث الميداني (FE–SEM)، ومطيافية الأشعة تحت الحمراء (FT–IR)، وتقنية حيود الأشعة السينية(X–RA) . تم تقييم سلوك التآكل للركيزة المطلية باستخدام تقنية الاستقطاب الديناميكي (PDP) في محلول رينجر عند ٣٧ درجة مئوية. أظهرت النتائج أن الركيزة المطلية SS(316l) بالشيتوسان–TiO2ذات مقاومة أفضل للتآكل مقارنة بالركيزة غير المطلية.

الكلمات الدالة: أوكسيد التيتانيوم, الشيتوسان, المجهر الالكتروني الماسح للانبعاثات الميدانيFE–SEM , الترسيب الكهربي النبضي الكاثودي وتقنيه الاستقطاب الديناميكيPCEPD and PDP .