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14:00-14:15	F. Kurşun Baysak, M. Arslan, C. Özcan: "REJECTION OF SOME METALS WITH DIFFERENT MEMBRANES by PERVAPORATION METHOD"
14:15-14:30	Ç. Kurşun, E. Hasanoğlu Özkan, M. Akkurt, N. Kurnaz Yetim, C. Özcan: "DETERMINATION OF Cr(III) AND Cr(VI) IONS USING MAGNETIC DENDRIMER AS ADSORBENT FOR SOLID PHASE EXTRACTION BY ATOMIC ABSORPTION SPECTROSCOPY"
14:30-14:45	O. Özcan, Ö. Taşpınar: "LEAD ADSORPTION MECHANISM ON NANO-STRUCTURED CALCIUM SILICATE, ACTIVATED CARBON, AND RICE HULL ASH"
14:45-15:00	A. Abourajab, S. Koyuncu, H. İcil: "A WATER-SOLUBLE NDI DERIVATIVE AS A DETECTOR FOR HEAVY METAL CATIONS"
15:00-15:15	E. Doğru, B. Kızılduman, H. İcil: "XRD AND SEM CHARACTERIZATION OF ARCHAEOLOGICAL LATE BRONZE AGE FINDINGS
15:15-15:30	E. Dagasan Bulucu, M. E. Palaz: "MORPHOLOGICAL CHARACTERIZATION OF AL BASED HYBRID COMPOSITE POWDERS VIA MECHANICAL ALLOYING"
15:30-15:45	**Coffee Break**
15:45-18:30	**9th session (oral talks) Chair: Prof. Dr. Hatice Öğütcü**
15:45-16:00	A Z. Macit, H. Öğütcü, E. Hasanoğlu Özkan, D. Nartop: "ANTIMICROBIAL EVALUATION OF SOME NOVEL POTENT BIOACTIVE COMPOUNDS"
16:00-16:15	Ö. Cesur, S. Erdoğan, H. B. Özalp: "EXTRACTION AND CHARACTERIZATION OF ANIMAL-DERIVED NANOCRYSTALLINE CELLULOSE FROM TUNICATE Phallusia mammillata (Cuvier, 1815)"
16:15-16:30	A. Z. Macit, D. Nartop, H. Öğütcü: "SYNTHESIS OF NOVEL THIAZOLE-BASED THIOSEMICARBAZONES AS POTENTIAL ANTIBACTERIAL AND ANTIFUNGAL AGENTS"
16:30-16:45	M. Dönmez, M. Türkyılmaz: "SYNTHESIS OF PINCER TYPE CARBENES AND THEIR SILVER(I) COMPLEXES WITH CYCLOPHANE STRUCTURE, ELUCIDATION OF THEIR STRUCTURES, AND ANTIMICROBIAL AGENT PROPERTIES"
16:45-17:00	N. Kurnaz Yetim, E. Hasanoğlu Özkan, H. Öğütcü: "ANTIMICROBIAL ACTIVITIES AND CATALYTIC DEGRADATION OF METHYLENE BLUE BY Co_3O_4 NANOPARTICLES"
17:00-17:15	S. Duman: "ECO-FRIENDLY DEHYDROGENATION OF DIMETHYLAMINE-BORANE CATALYZED BY MNPs@CELLULOSE"
17:15-17:30	S. Duman: "GREEN APPROACHES TO DEHYDROGENATION OF DMAB CATALYZED BY STARCH STABILIZED Ru(0), Cu(0) AND Ni(0) NANOPARTICLES"
17:30-17:45	S. Adıgüzel, S. Altuntaş: "FABRICATION OF PS: PANI SERS SUBSTRATES FOR DETECTION OF HUMAN SERUM COMPONENTS"
17:45-18:00	G.Karabulut, N. Beköz Üllen: "SYNTHESIS OF ULTRASOUND ASSISTED GREEN SILVER NANOPARTICLES AND COATING ON 316L STAINLESS STEEL WITH AIRBRUSH SPRAY TECHNIQUE"
18:00-18:15	G. Karabulut, N. Beköz Üllen, S. Karakuş: "CHEMICAL, MORPHOLOGICAL, AND STRUCTURAL CHARACTERISTICS OF PEGYLATED MATCHA COPPER OXIDE NANOPARTICLES"
18:15-18:30	Y. Azizian-Kalandaragh, J. Farazin and G. Pirgholi-givi: "PREPARATION AND OPTICAL CHARACTERIZATION OF CdS/ZnS-POLYMER SUPERLATTICE NANOCOMPOSITES"
18:30	**Award Ceremony**

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SYNTHESIS OF ULTRASOUND ASSISTED GREEN SILVER NANOPARTICLES AND COATING ON 316L STAINLESS STEEL WITH AIRBRUSH SPRAY TECHNIQUE

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Abstract

In this study, novel PEGylated maca root extract-silver nanoparticles (PEGylated Mac-Ag NPs) were prepared using a green sonochemical method. The 316L stainless steel (SS316L) was coated with the prepared PEGylated Mac-Ag NPs using a low-cost and easy-to-use airbrush spray technique. The prepared PEGylated Mac-Ag NPs and coated SS316L with PEGylated Mac-Ag NPs were characterized to determine the surface morphology, crystal structure, and chemical functional groups using different methods such as scanning electron microscopy, transmission electron microscopy, ultraviolet-visible spectrophotometry, X-ray diffraction, Fourier transform infrared spectroscopy, X-ray photospectrometry, surface roughness, and profilometry. According to the surface characterization results, it was clear that the sono-synthesized PEGylated Mac-Ag NPs had a spherical-agglomerated aggregate structure in the particle range of 30 to 50 nm. Furthermore, the surface of the coated 316L stainless steel had a uniform particle distribution with spherical and particle diameters of less than 50 nm. Consequently, it was observed that surface of the SS316L was covered with nano-sized PEGylated Mac-Ag NPs using a green chemistry approach. The proposed synthesis and coating process were promise to use a simple and green process to fabricate advanced nanomaterials in biomedical applications.

Keywords: Silver nanoparticle, green chemistry, airbrush spray coating, sonochemical synthesis

1. INTRODUCTION

In recent years, M/MO nanoparticles have been widely used in coating applications of metallic surfaces in order to improve the surface properties and provide various functional properties such as mechanical, corrosion resistance[1], and antipathogenic properties [1]. Silver nanoparticles (Ag NPs) based coatings have been widely used in recent years due to the antipathogenic properties of silver [2,3], as well as its superior physicochemical and biological properties such as conductivity and catalytic properties [4]. There are many studies in the literature where Ag NPs-based coatings are coated on the metal surface [5,6] and other substrates such as glass [7], polymer [8], etc. In these studies, coating methods such as dip [5,6], sol-gel [9], atomic layer deposition [10], physical vapor deposition [11], and electro-deposition [12] were used. The methods used in these studies in the literature are difficult, expensive, and long-term processes. In this study, the goal is to ensure that biocompatible and synthesized with green chemistry Ag NPs were coated homogeneously to the metallic surface in a cheap, fast, and greener way. For this purpose, Ag NPs were synthesized using the green sonochemical method in this study. In this method, nanoparticle formation is carried out by acoustic cavitation using ultrasonic sound waves [13]. It is a green synthesis method to reduce Ag ions to form Ag NPs without the use of chemical reducing or hazardous stabilizing agents [14]. However, Ag NPs tend to agglomerate in the aqueous solutions. This causes them to be growing over time, reduce their surface area, and thus reduce their effectiveness. In order to prevent this, stabilizer agents should be used and nanoparticles should be control the size range [15]. In the literature, various natural polymers such as chitosan, pectin, starch, and alginate and various polymers such as poly (methyl methacrylate) (PMMA), polyvinyl alcohol (PVA), and PEG (Polyethylene Glycol) are used in the synthesis of Ag NPs. In recent years, the use of plant extracts as stabilizing and reducing agents has become a widespread trend due to their being natural and biocompatible [16]. In this study, fully biocompatible green maca root (*Lepidium Meyenii*) and Polyethylene Glycol (PEG) were used as stabilizers. Maca is a type of plant that has been used by people for about 2000 years and is called a medicinal plant [17]. PEG is a highly water-soluble, non-toxic, biochemically stable, low-cost, and biocompatible polymeric material [18]. For the first time in the literature, this study deals with the coating of a plant extract-based nanobiocomposite on SS316L surface by airbrush spray technique. The surface characterization and functional groups of PEGylated Mac-Ag NPs and coated surface were determined by various techniques Scanning electron microscopy (SEM), transmission electron microscopy (TEM), Fourier

transform infrared spectrophotometry (FT-IR), UV-Visible spectrophotometry (UV-Vis), X-ray photoelectron spectrophotometry (XPS), X-Ray diffraction (XRD), and profilometer.

2. MATERIAL AND METHODS

2.1. Materials

Maca root powder (*Lepidium meyenii*) was purchased from Arifoğlu Company (Turkey). Sodium hydroxide (NaOH) was purchased from Merck Company (Germany). The filter paper (Whatman® qualitative paper, diameter: 25 mm) and silver nitrate ($AgNO_3$, purity: ≥99.0%) were purchased from Sigma Aldrich Company (Germany). All chemicals and reagents were used without further purification. SS316L disks were obtained from a bar with a length of 1 m in 27 mm diameter (Birçelik Company, Istanbul, Turkey). The chemical composition of the SS316L was 0.014 w. %C, 0.437 w. %Si, %, 10.091 w.% Ni, 16.581 w.% Cr, 2.048 w.% Mo, 0.326 2.% Cu, 1.317 w.% Mn, 0.024 w.% S, 0.025 w.% P, 0.041 w.% N, and balance-Fe.

2.2. Preparation of PEGylated Mac-Ag NPs and PEGylated Mac-Ag NPs coated SS316L

Maca root powders were washed by several times to remove dust and impurities, and then dried in a vacuum oven at 70 °C for 5 h. 5 g of the maca root powder was added to 100 mL of distilled water and mixed at 500 rpm for 10 min. Then, it was kept in a closed sterile container in a dark environment at 27 °C for 3 days. Finally, the sample was decanted and filtered with 0.45-µm retention of sterile syringe filter, so that maca extract was obtained. 0.1 g of PEG was added to 20 mL of prepared maca root extract and mixed at 500 rpm for 10 minutes. 0.1 g of $AgNO_3$ was dissolved in 50 mL of distilled water and stirred for 10 minutes at 27 °C. 0.2 g of NaOH was dissolved in 50 mL of distilled water and stirred for 10 minutes at 27 °C. $AgNO_3$ and NaOH solutions were added drop by drop into the PEGylated maca solution and sonicated at %50 amplitude for 30 min at 27 °C. Finally, the prepared PEGylated Mac-Ag NPs was filtered using a 0.22- µm retention of sterile syringe filter.

The stainless steel bar was sliced with a computer numerical control (CNC) machine to obtain 2 mm in thickness discs. The cutting was performed using DNMG 150608 PM 1525 insert while a cutting speed 10 m/min, a feed rate of 0.25 mm/rev and a cutting depth of 1 mm. After the cutting process, all samples were ultrasonicated for 10 min in ethanol-acetone solution, and dried for 10 min at 60 °C in the vacuum oven. Then, an airbrush compressor kit (piston type/ Model Number: AS186) was used for the coating process. The

schematic view of the coating process is given in Figure 1. During spraying, a plastic pipe of 100 mm long and 25 diameter was used in order to spray from an equal distance and to prevent it from spreading around. After the spray gun was placed with the plastic pipe, the distance of the nozzle to the surface was measured as 75 mm. The spraying was carried out at 3 bar pressure for 4 seconds. The process was carried out only once and the surfaces were completely coated.

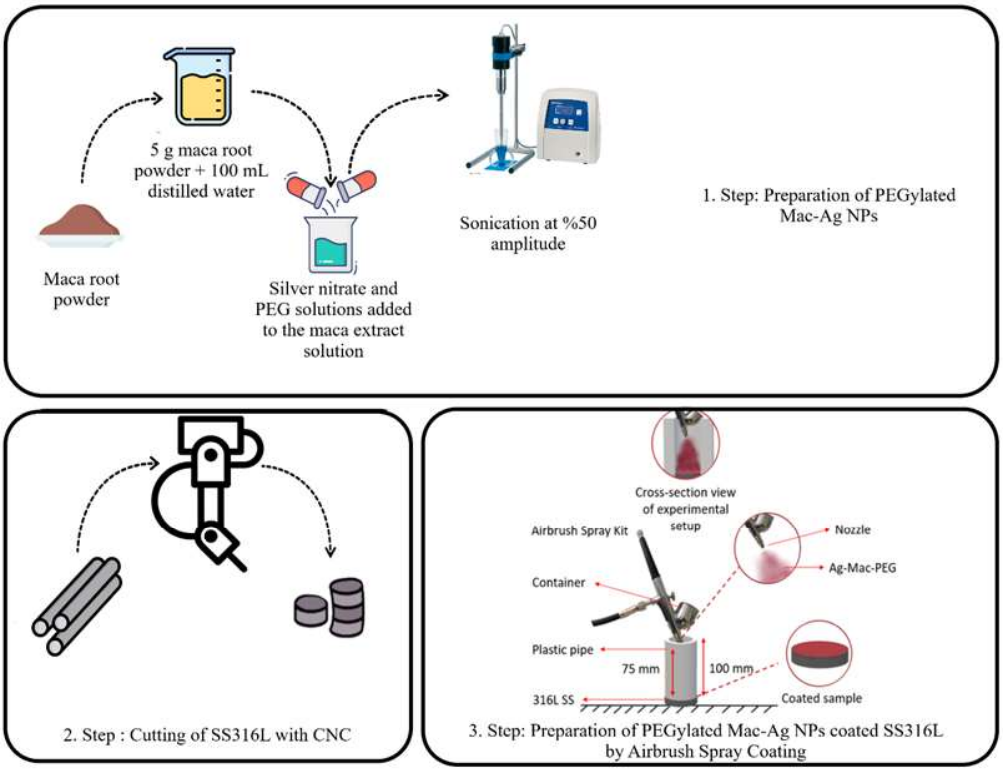

Figure 1. Schematic diagram of the process stages of the preparation of PEGylated Mac-Ag NPs coated SS316L

2.4. Characterization of PEGylated Mac-Ag NPs and PEGylated Mac-Ag NPs coated SS316L

The surface morphology and topography of synthesized PEGylated Mac-Ag NPs and coated samples were characterized by SEM (Zeiss-Sigma 300 device). Particle size and morphology of the PEGylated Mac-Ag NPs were characterized by TEM in a high vacuum mode at 100 kV (Hitachi High Tech HT7700). The absorbances measurement of the PEGylated Mac-Ag NPs was made by a double-beam UV–Vis spectrophotometer with a UVWin 5 Software (T + 80 model, PG Instrument). Functional groups of the PEGylated Mac-Ag NPs were obtained with FT-IR analysis (Perkin Elmer, Spectrum Two model) in the region

of 4000–400 cm^{-1} frequency range with a resolution of 4 cm^{-1} and 8 scans. XRD analysis was performed to determine crystalline structure of the PEGylated Mac-Ag NPs by Rigaku D/Max2200/PC device with Cu-Kα radiation from 10° to 80 at 40 kV and 15 mA. XPS analysis was performed to determine the elemental composition and chemical properties of the PEGylated Mac-Ag NPs as a coating solution by Specs-Flex device with Al-Kα radiation. The surface roughness, distribution, and thickness of the PEGylated Mac-Ag NPs coating on the surface were examined by Kla Tencor Stylus Profiler P7 profilometer device.

3. RESULTS AND DISCUSSION

3.1. Characterization of PEGylated Mac-Ag NPs

The surface morphology of the synthesized PEGylated Mac-Ag NPs was characterized by SEM and TEM techniques. In Figure 2, SEM (a) and TEM (b) images of the PEGylated Mac-Ag NPs were presented. According to the SEM image, PEGylated Mac-Ag NPs have homogeneous distribution with a spherical shape and in the particle size range of 10-40 nm. As a result of TEM, it was observed that the Mac-PEG structure, which is the matrix phase, showed a heterogeneous distribution, and the Ag NPs were dispersed in small sized and acceptable forms on the Mac-PEG matrix. The results show that the nanobiocomposite structure of PEGylated Mac-Ag NPs has been successfully formed. The results are consistent with previous studies on the sonochemical synthesis of the plant-derived Ag NPs [19].

UV-Vis analysis was performed to obtain surface plasmon resonance behavior of the PEGylated Mac-Ag NPs. UV-Vis spectra of the synthesized PEGylated Mac-Ag NPs were given in Figure 3(a). According to the literature, the formation of Ag NPs with surface plasmon resonance behavior forms a peak region of 400-460 nm [20]. As seen in Figure 3(a), Ag^0 in the nanobiocomposite were formed a peak at 422 nm. This proves the presence of Ag NPs in the structure. The FTIR analysis of PEGylated Mac-Ag NPs was performed determine the functional groups of nanobiocomposite. FTIR spectra of the PEGylated Mac-Ag NPs was given in Figure 3 (b). The characteristics peaks were found at 3303,46 cm^{-1} (O-H stretching), 2971,77 cm^{-1} (C-H stretching), 1557,24 cm^{-1} (C=O), 1396,21 cm^{-1} (O-H bending), and 1054,87 cm^{-1} (C-O stretching), respectively. The functional groups of nanobiocomposite containing polyphenol compounds are compatible with the literature [21]. XRD analysis was performed to examine the crystalline structure of the nanobiocomposite. XRD pattern graph of the PEGylated Mac-Ag NPs was given in Figure 3(c). As shown in the graph, the peaks were formed 2θ= 38°, 44.1°, 64.5°, and 77.4°. These peaks are corresponding to the plane of (1 1 1), (2 0 0),

(2 2 0), and (3 1 1), respectively. According to this result, the silver crystal has a face-centered cubic (FCC) structure. The peaks were formed at 2θ=27.7°, 29.2°, and 32.2° originate from the organic phases in the nanobiocomposite structure [19].

To obtain elemental composition and oxidation states of the synthesized PEGylated Mac-Ag NPs XPS analysis was performed. The binding energies of the PEGylated Mac-Ag NPs at 283.50 eV for C_{1s}, 531.10 eV for O_{1s}, and 399.00 eV for $Ag_{3d/2}$ are corresponded to in Figure 4(a-d). In Figure 4(b) the O_{1s} spectra observed at 531.10 eV was corresponded to carboxylate group. In C_{1s} spectrum (Figure 5(c)) was observed a single peak at 283.50 eV, this peak indicates the nonoxygenated ring carbon (C-C) and arises from compounds of phytochemical of maca root extract [19].

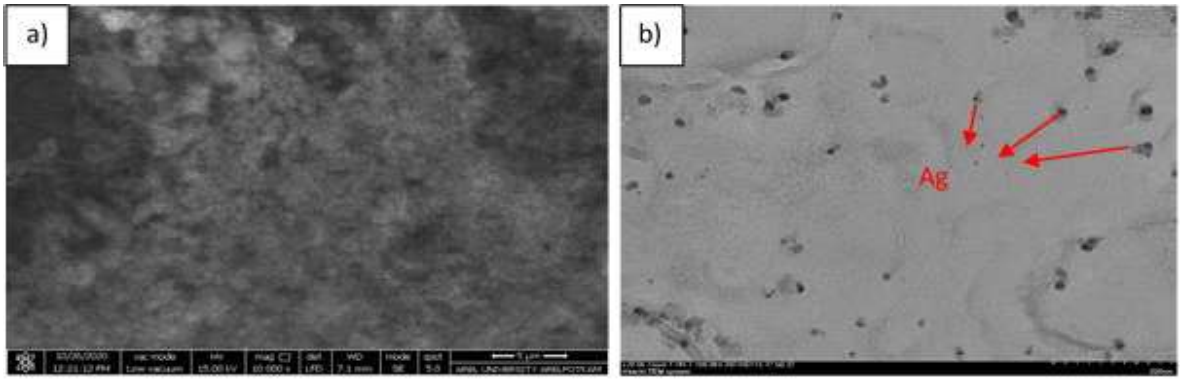

Figure 2. (a) SEM and (b) TEM images of the PEGylated Mac-Ag NPs

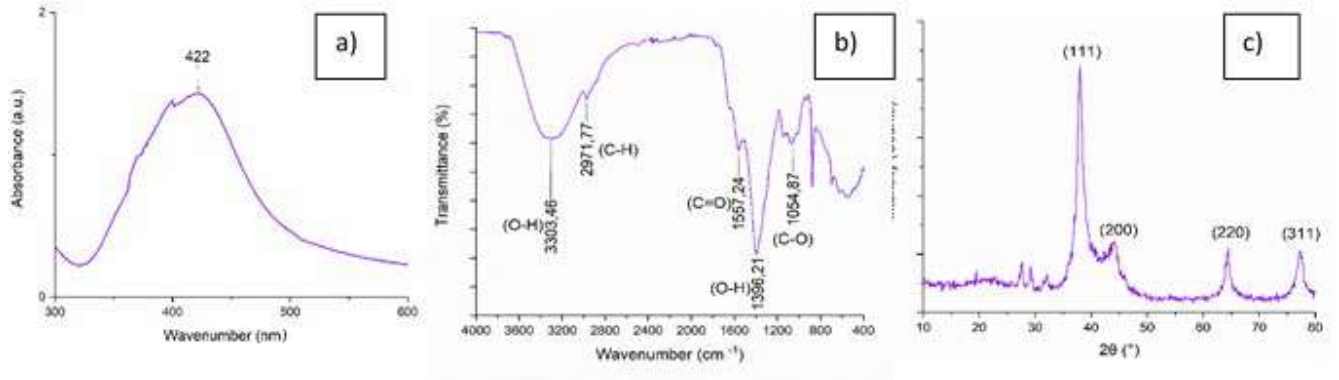

Figure 3. (a) UV-Vis, (b) FT-IR, and (c) XRD spectrum of the PEGylated Mac-Ag NPs

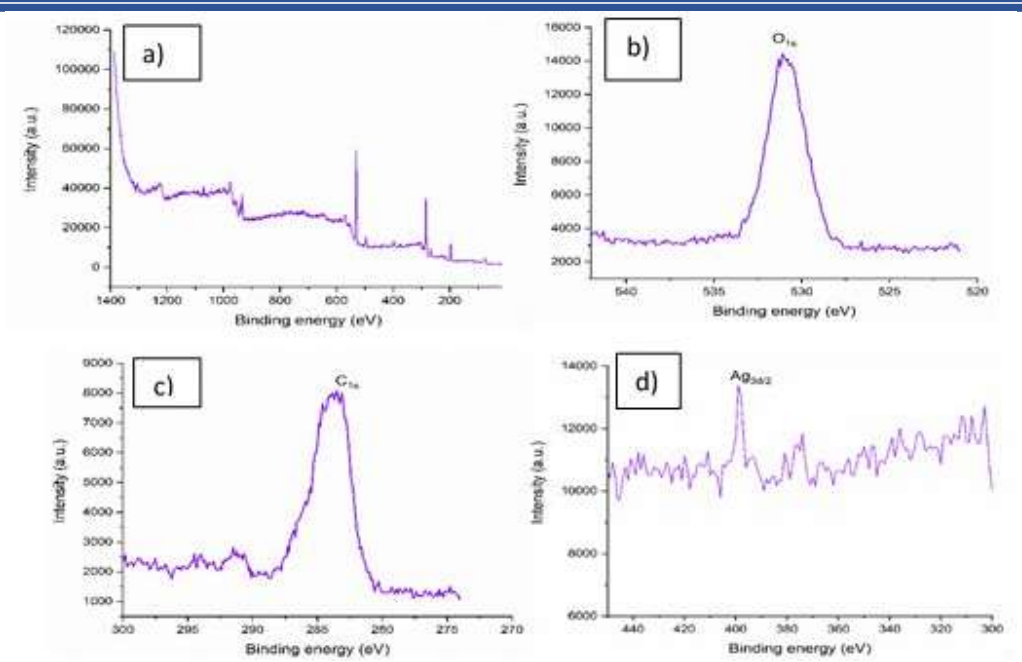

Figure 4. XPS spectrum of PEGylated Mac-Ag NPs (a) XPS survey spectrum, (b) binding energy spectrum for O_{1s}, (c) binding energy spectrum for C_{1s}, and (d) binding energy spectrum for Ag_{3d}

3.2. Characterization of PEGylated Mac-Ag NPs coated 316L stainless steel

SEM images of the coated samples with Airbrush spray coating technique are given in Figure 5(a-d) at different magnifications. According to the SEM results, PEGylated Mac-Ag NPs coating was done successfully. In Figure 5(a), the layers seen on the coating surface are the cutting lines formed during the cutting process on the steel surface. At lower magnifications (Figure 5(a-b)), the PEGylated Mac-Ag NPs coating has a branched structure. At larger magnifications (Figure 5(c-d)), the structural form of the coating material is fully visible. It is also seen that the PEGylated Mac-Ag NPs are homogeneously dispersed on the substrate surface, and Ag NPs sizes are in the range of 30-40 nm. The distribution and near-spherical shape of the Ag NPs on the SS316L substrate are consistent with the works on metallic surfaces coated with Ag NPs [23,25]. In terms of improving the resistance properties of the coating, the homogeneous distribution of Ag NPs on the SS316L substrate is important. Jothi et al. reported that the homogeneous distribution of nanoparticles on the stainless steel surface coated with Ag NPs increases the resistivity properties of the substrate [9].

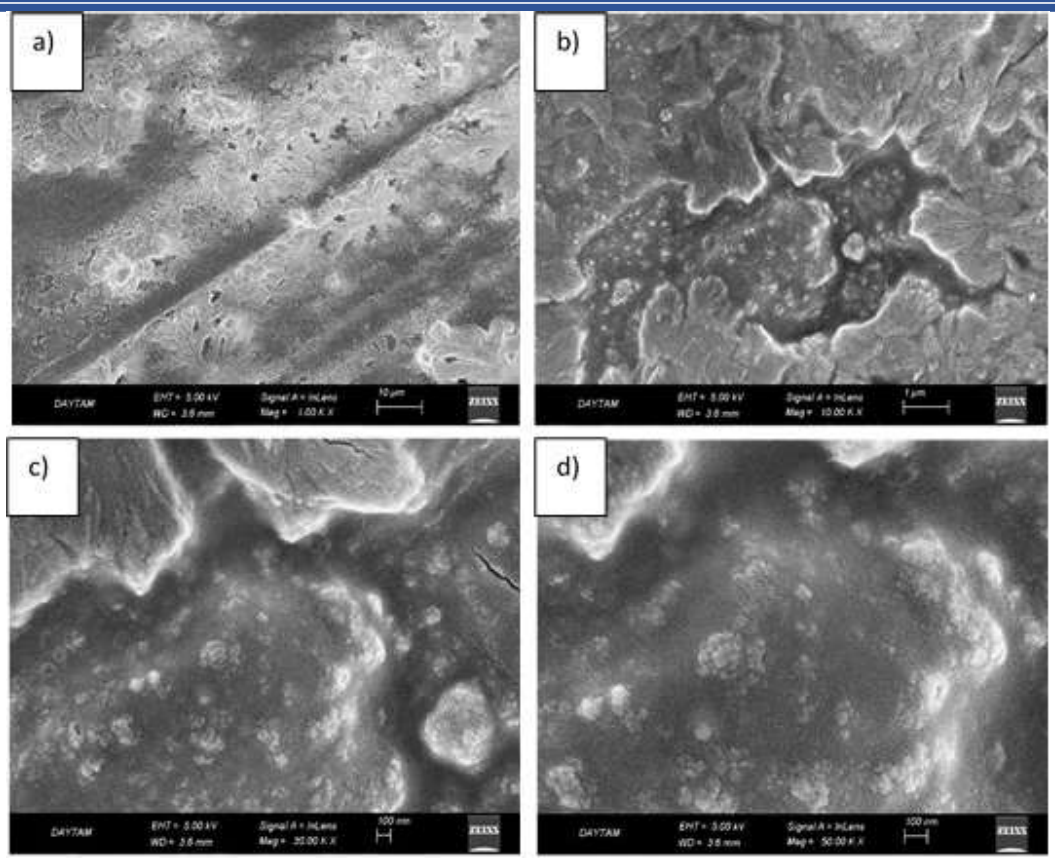

Figure 5. SEM images of the PEGylated Mac-Ag NPs coated 316L stainless steel at different magnifications (a) x100, (b) x10.000, (c) x30.000, and (d) x50.000

The surface roughness, distribution, and thickness of the PEGylated Mac-Ag NPs coating on the steel surface were examined by a profilometer. The 3D view of the coating morphology was given in Figure 6. As can be seen, the PEGylated Mac-Ag NPs coating is homogeneously distributed on the surface of SS316L. Similar to the SEM image (Figure 5 (a)), the cutting lines are also seen in the 3D images as a black region. The coating thickness was determined approximately 7.7 µm. Average surface roughness value of the coating was obtained approximately 9.021 nm. It can be seen from the figure that Ag NPs are coated on the SS316L surface as a single-layer and homogeneous structure. In the literature, this coating structure is defined as a "single-layer structure" [11]. Since the coating is carried out with nanoparticles, it is usual for the surface roughness to be at nanoscales.

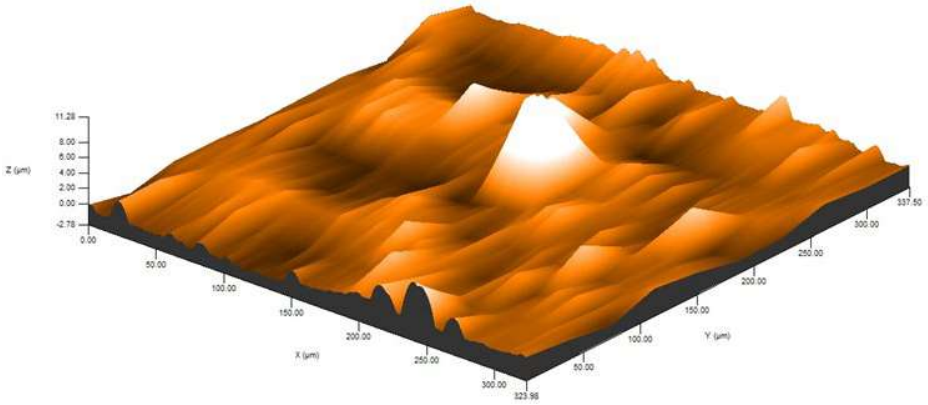

Figure 6. 3D view of the PEGylated Mac-Ag NPs coated 316L stainless steel

4. CONCLUSION

In this study, a novel green PEGylated Mac-Ag NPs coating was performed on the SS316L. The synthesized PEGylated Mac-Ag NPs are in FCC structure, covered by the organic phase, and have a size range of 10-40 nm. The coated PEGylated Mac-Ag NPs on the steel by airbrush spray method are homogeneously dispersed on the surface and their sizes are in the range of 30-40 nm. After coating, the roughness of the steel surface decreased. All of the manufacturing processes consist of green processes and products, so the synthesized nanostructure is suitable for biomedical applications. The coating technique used in the study is a preferable method to improve surface properties with nanoparticles. The surface modification of the SS316L was positively affected.

ACKNOWLEDGMENTS

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