EVALUATING ACETONE AND METHANOL FOR ELECTROPHORETIC DEPOSITION OF SS 316L COATED WITH HYDROXYAPATITE/MULTIWALLED CARBON NANOTUBES DENTAL IMPLANTS: A FOCUS ON CORROSION RESISTANCE

Ersyi Arini Valmelina¹, Praswasti Pembangun Dyah Kencana Wulan^{1*}, Yudan Whulanza², Mehmer Sükrü Adin³

¹Chemical Engineering Department, Faculty of Engineering, Universitas Indonesia, Depok 16424, West Java, Indonesia ²Mechanical Engineering Department, Faculty of Engineering, Universitas Indonesia, Depok 16424,

West Java, Indonesia

³Besiri OSB Vocational School, Batman University, 72060, Turkey

* Email: praswasti.pembangun@ui.ac.id

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Abstract

Dental implants offer a reliable solution for replacing damaged tooth roots. This research investigates the comparative performance of acetone and methanol as suspension media in the fabrication of stainless steel type 316L-based dental implants using the Electrophoretic Deposition (EPD) method, a technique known for its simplicity and cost-effectiveness. Voltage variations of 20V, 30V, and 40V were applied to both acetone and methanol suspensions for a duration of 20 minutes. The morphology of the Hydroxyapatite/Multiwalled Carbon Nanotube (HA/MWCNT) coatings was meticulously characterized using Scanning Electron Microscopy (SEM). Corrosion resistance was evaluated through Potentiodynamic Polarization (PDP) and Electrochemical Impedance Spectroscopy (EIS) techniques. Remarkably, at 30V, a homogeneous and crack-free coating was achieved, demonstrating superior corrosion resistance. This was further corroborated by the resistance values of 23.891 Ω and 114.990 Ω for the acetone and methanol samples, respectively. Additionally, the corrosion rates of 0.075 (mmpy) and 0.0004 (mmpy) for the acetone and methanol samples further emphasized the superiority of methanol as a suspension medium. These findings unequivocally establish methanol as the optimal choice for achieving superior deposition quality and corrosion resistance in the context of the EPD method for stainless steel type 316L-based dental implants.

Keywords: acetone, electrophoretic deposition, methanol, stainless steel

Introduction

The lack of public attention to oral health has contributed to the increasing use of dental implants (Nasar, 2018). Dental implants serve as functional and biocompatible devices, placed in the alveolar bone and anchored for several months to support fixed or removable prostheses. They function as replacements for lost tooth roots (Chaturvedi and Upadhayay, 2010).

The selection of metal-based dental implants must meet the requirement that the metal is biologically inert and does not release metal ions into the body tissues (Manivasagam *et al*., 2010).

The use of stainless steel as a dental implant material has attracted researchers' attention in recent years, as titanium implants are relatively expensive. The mechanical properties of stainless steel type 316L (SS 316L) closely resemble human bone, exhibiting high biocompatibility and corrosion resistance. The potential failure of SS 316L-based implants due to the release of ions from the SS 316L surface containing Ni, Cr, and Fe can be addressed by coating the SS 316L surface with Hydroxyapatite (HA) (Bruschi *et al*., 2015).

Hydroxyapatite (HA) has long been used as a coating for dental implants. HA helps dental implants achieve good stability due to its bioactive and biocompatible nature, as well as its bioresorbable properties, aiding in bone integration with the implant (Akbar *et al*., 2021). The main drawback of HA is its inherent brittleness (Sivaraj and Vijayalakshmi, 2018; Ranamanggala *et al*., 2020). This limitation can be overcome by incorporating Multiwalled Carbon Nanotubes (MWCNT). MWCNT are commonly used to enhance the mechanical properties of the HA matrix in metal implants (Rehman *et al*., 2020). HA integrated with MWCNT forms the HA/MWCNT nanocomposite ready for coating the surface of SS 316L.

One commonly used method for coating dental implants is electrophoretic deposition (EPD). The EPD technique is widely accepted for coating medical implants based on SS 316L with HA/MWCNT because it offers a relatively simple and cost-effective process (Liu *et al*., 2018).

Previous research by (Thom *et al*., 2017) utilized a water suspension, which has a higher viscosity than methanol and the highest dielectric constant among popular suspension choices (Amrollahi *et al*., 2015). This resulted in a crack-free and homogeneous coating morphology. However, an optimal suspension medium specifically for electrophoretic deposition (EPD) fabrication of SS 316L dental implants has yet to be identified.

Among the list of popular solvents for EPD, acetone and methanol stand out as top contenders for ideal suspensions. This is because they meet the key parameters for successful EPD: low viscosity, high

dielectric constant, and low conductivity (Rehman *et al*., 2020). Acetone exhibits a viscosity half that of methanol, with values of 0.3087 cP and 0.557 cP, respectively. In contrast, methanol boasts a higher dielectric constant compared to acetone, reaching 32.63 while acetone stands at 20.7 (Amrollahi *et al*., 2015). While the viscosity and dielectric constant data suggest that acetone and methanol are promising suspension media for EPD dental implant fabrication, further experimental validation is required to confirm their suitability.

This research aims to evaluate the efficacy of acetone and methanol as suspension media in the fabrication of SS 316L dental implants using the EPD method, ultimately determining the superior choice between the two.

Research Methods

Materials

The f-MWCNT (functionalized Multiwalled Carbon Nanotubes with COOHfunctional groups) were purchased from Chengdu Organic Chemicals with specifications of OD: 10– 20 nm, purity: >98%, and length: 10–30 μ m. Calcium chloride (CaCl₂), diammonium hydrogen phosphate $((NH_4)_2HPO_4)$, ammonium hydroxide $(NH₃,H₂O)$ 25%, acetone, and methanol were procured from Merck, while SS 316L was obtained from a distributor in Jakarta.

Substrate preparation

Stainless steel type 316L (SS 316L) was cut into 15 mm \times 4 mm dimensions using a CNC Wire Cut Machine, ensuring precise sizing without compromising the Substratee's composition. SS 316L was then smoothed with silicon carbide (SiC) based material with grit #400. Subsequently, the samples were cleaned using an ultrasonic cleanser (KLS 303363) in an acetone solution to completely remove impurities. Following

this, the samples were rinsed with deionized water.

HA/MWCNT synthesis

Two hundred (200) mg of f-MWCNT was stirred for 1 hour in a 5 ml solution of calcium chloride $(CaCl₂)$ 0.01 M. After 1 hour of stirring, ammonium hydroxide $(NH₃.H₂O)$ was added drop by drop to adjust the pH of the mixture to 10. Five (5) ml of diammonium hydrogen phosphate ((NH4)2HPO4) 0.01 M solution was then added dropwise with constant stirring into this mixture. To eliminate unreacted reagents and by-products, the product was filtered and washed several times with distilled water. HA/MWCNT was air-dried for 24 hours at room temperature.

Preparation of suspension

The preparation of the suspension for the EPD process began with air-drying the HA/MWCNT nanocomposite for 24 hours at room temperature. The ratio of HA/MWCNT to acetone was 3:1, while the ratio of HA/MWCNT to methanol was 2:1. The difference in these ratios is due to the physical and chemical properties of the EPD suspension. The boiling points of acetone and methanol are 82 °C and -64 °C, respectively (Chaudhary *et al*., 2021).

The polarity of acetone is 1.329, while methanol is 1.358. The solubility of acetone and methanol at 20 °C is approximately 730 g/L and 1.243 g/L, respectively (Henkel *et al*., 2018). The mixture was then sonicated for 60 minutes to achieve a homogeneous dispersion of HA/MWCNT particles. The pH of the suspension was adjusted to 4 by adding nitric acid (Kang *et al*., 2021). Adjusting the suspension pH to 4 can make the zeta potential and charge of the suspension positive by absorbing H^+ ions (Rodrı´guez-Lugo & Karthik, 2018).

Electrophoretic deposition

The EPD process was carried out at room temperature with DC voltage, illustrated in Figure 1. SS 316L was utilized as both the cathode and anode. The EPD process was conducted for 20 minutes in potentiostatic mode using a DC power supply YIHUA 605D with variations of 20V, 30V, and 40V. The coated samples were carefully removed from the EPD cell to minimize the resistance between the wet layer and the remaining suspension. The samples were horizontally air-dried at room temperature for 24 hours and stored in a desiccator at room temperature.

Figure 1. Scheme of electrophoretic deposition method

Instrumentation

Characterization of FTIR was performed to identify the HA functional groups formed on the surface of MWCNTs (Chaudhary *et al*., 2021). 1 mg of the HA/MWCNT nanocomposite was placed on the ATR crystal. Then, the spectrum of the sample was recorded with FTIR over a wavenumber range of 569– 3500 cm-1 . The working principle of the FTIR ATR instrument is shown in Figure 2.

Figure 2. Scheme of FTIR-ATR

The surface morphology of the HA/f-MWCNT-coated SS 316L was examined through Scanning Electron Microscopy (SEM) using a Hitachi S-4800 Scanning Electron Microscope. Corrosion testing of the SS 316L dental implant with an HA/MWCNT coating was conducted using the Potentiodynamic Polarization (PDP) and Electrochemical Impedance Spectroscopy (EIS) methods. Simulated Body Fluid (SBF) was employed as the electrolyte in this study. The Nyquist and polarization curves were obtained using the CorrTest CS310 Potentiostat

Results and Discussion

HA/MWCNT Synthesis

The suspension preparation initiates with the fabrication of the HA/MWCNT nanocomposite. The proposed synthesis route of HA/MWCNT composite is depicted in Figure 3. The MWCNTs employed in this study are functionalized with COOH⁻ functional groups. These functional groups impart a negative charge, activating the MWCNT surface to act as nucleation sites. The negatively charged surface sites of MWCNT attract Ca^{2+} , and the presence of HPO₄²⁻ in the $Ca²⁺$ accumulation region results in the formation of HA on the MWCNT surface (Ivvala *et al*., 2019). Additionally, adjusting the solution pH to 10 enhances solubility, accelerating the deposition and agglomeration of Ca^{2+} and $HPO₄²⁻$ ions (Nasar, 2018).

Figure 3. Proposed synthesis route of HA/MWCNT

The success of HA/MWCNT synthesis is confirmed by FTIR measurement. Figure 4 showed the presence of f-MWCNT with characteristic functional groups such as OH ; -C=O; and -C-OH, observed at wavenumbers of 3445 cm^{-1} ; 1637 cm⁻¹; and 1389 cm⁻¹, respectively. The functional groups of f-MWCNT

(MWCNT functionalized with COOH groups), namely OH⁻ and C-OH, were observed at peaks of 3455 cm⁻¹ and 1389 cm⁻¹, respectively. The 1389 cm⁻¹ peak was identified as a strong absorption peak associated with the interaction of Ca^{2+} (in HA) with the COO⁻ group of f-MWCNT (Chaudhary *et al*., 2020).

Figure 4. FTIR characterization results of HA/MWCNT nanocomposites

These negatively charged functional groups activate the MWCNT surface, which attracts Ca^{2+} ions and assists in the formation of the HA layer with the arrival of $HPO₄²$ (Nguyen, et al., Electrodeposition of Hydroxyapatite-Multiwalled Carbon Nanotube Nanocomposite on Ti6Al4V, 2020). The 1637 cm-1 range shows a characteristic of carbon material associated with the C-C stretching bond characteristic of nanotubes (Gopi *et al*., 2013; Tucureanu *et al*., 2016). The successful formation of HA on the nanocomposite is marked by the appearance of peaks in the range of 560 cm⁻¹, 610 cm⁻¹ and 1110 cm⁻¹, which are associated with P-O bending from phosphate groups and phosphate ions from the O-P-O bond of hydroxyl sites. These results are in accordance with the research (Chaudhary *et al*., 2021) and

(Nguyen *et al*., 2020). Thus, all of these peaks confirm the successful formation of the HA/MWCNT nanocomposite.

Scanning Electron Microscopy (SEM)

The SS 316L Substratee coated with HA/MWCNT was examined using scanning electron microscopy (SEM). SEM morphology indicates the absence of detected impurities. SEM images reveal a uniform HA/MWCNT layer, with no particle segregation observed at any detected points (Tsosie *et al*., 2017). The circular structure of the cluster morphology indicates that HA and MWCNT are well-mixed, as illustrated in Figures 5. These images also confirm the proper mixing of HA and MWCNT due to the well-dispersed structural distribution (Sivaraj and Vijayalakshmi, 2018).

Figure 5. SEM characterization of HA/MWCNT layers in acetone suspension (A) 20V; (B) 30V; (C) 40V and methanol suspension (D) 20V; (E) 30V; (F) 40V

The Acetone 40V sample, as shown in Figure 5, exhibits agglomeration within the layer. Agglomeration is a condition where layer particles gather in a specific area, induced by high voltage and prolonged deposition time during the EPD process. This phenomenon occurs because the HA/f-MWCNT nanocomposite moves too rapidly due to the high voltage, and insufficient time is provided for the particles to reorganize into an optimal arrangement (Rehman *et al*., 2020). Furthermore, cracks were found in the methanol 40V sample in Figure 5. This phenomenon occurs due to the evolution of hydrogen gas at the cathode during EPD. At higher voltages, the amount of hydrogen gas formed will increase according to the Eq. (1) (Thom *et al*., 2017).

$$
2H^{+}(aq) + 2e^{-} \rightarrow H_{2}(g) \quad (1)
$$

The number of bubbles formed in the cracks will increase when the EPD voltage is raised. The pressure from this hydrogen gas will create cracks that will widen as more hydrogen gas is formed (Zhang *et al*., 2014; Farokhi *et al*., 2019). Based on Figure 5, the methanol sample produces a smoother, more uniform layer with a denser structure. This is because the polarity of methanol as a solvent is

more promising than that of acetone. The polarity of methanol, which is compatible with the substrate, will form a better layer due to optimal interactions, such as strong adhesion and even spreading on the substrate surface (Alaska & Bartmanski, 2020). In this study, the suspended particles possess COOH⁻ functional groups originating from acidfunctionalized multiwalled carbon nanotubes (MWCNTs). These functional groups impart polarity to the particles, enabling them to interact with methanol molecules. This strong solvent-particle interaction enhances the stability of the suspended particles. In contrast, acetone is less effective in stabilizing polar particles (Rodrı´guez-Lugo and Karthik, 2018). Methanol is an ideal dispersant for use in EPDs and is preferred due to its good polarity as a solvent. Methanol's high viscosity can stabilize the dispersion by reducing the sedimentation rate. (Amrollahi *et al*., 2015).

Corrosion test

The corrosion rate of the materials was assessed through Potentiodynamic Polarization and Electrochemical Impedance Spectroscopy (EIS) methods (Garbacz and Kro´likowski, 2019). A comparative analysis was conducted between the uncoated SS 316L sample and SS 316L samples utilizing acetone and methanol as suspensions, as illustrated in Figure 6.

The parameters in the Potentiodynamic Polarization (PDP) method are characterized by the values of I_{corr} , E_{corr} , and the corrosion rate. A leftward shift in the polarization curve corresponds to a decrease in the Icorr value, while an

upward movement indicates an increase in the Ecorr value (Magar *et al*., 2021). The Tafel extrapolation, marked by the intersection point between the anodic and cathodic currents as shown in Figure 6, is valuable for extracting results from the corrosion tests, which are tabulated in Table 1.

Figure 6. Potentiodynamic polarization curves in SBF solution with scan rate 0.5 mV/s for acetone and methanol

	Acetone Suspense			Methanol Suspense			
Voltage (V)	E _{corr} (V vs Ref)	L corr $(\mu A/cm^2)$	Corrosion Rate (mmpy)	E _{corr} (V vs Ref)	<i>icorr</i> $(\mu A/cm^2)$	Corrosion Rate (mmpy)	
SS 316L	-0.244	1.070	0.960	-0.225	0.981	0.022	
$SS 316L - 20V$	-0.057	0.123	0.144	-0.192	0.408	0.009	
$SS 316L - 30V$	-0.053	0.087	0.075	-0.111	0.017	0.0004	
$SS 316L - 40V$	-0.022	0.108	0.084	-0.157	0.040	0.0009	

Table 1. PDP measurement results

The increase in deposition voltage applied during the EPD process led to a leftward shift in the curve trend. The I_{corr} value for the acetone 30V sample (0.087 µA/cm2) is higher than that of the methanol 30V sample (0.017 µA/cm2). A low I_{corr} value indicates a low corrosion rate. Therefore, the methanol 30V sample demonstrates favorable corrosion resistance, suggesting a lower corrosion rate (Sivaraj and Vijayalakshmi, 2018).

84 *Online ISSN: 2528-0422* The HA/f-MWCNT layer effectively hinders electrolyte diffusion to the SS 316L surface, functioning as a barrier to

the ionic transport of Ni, Cr, and Fe cations within the substrate. This restricted ionic movement impedes the corrosion process, thereby offering enhanced protection against corrosion (Kang *et al*., 2021).

Electrochemical corrosion testing revealed a more positive Ecorr value of - 0.053 V for the acetone 30V sample compared to -0.111 V for the methanol 30V sample. This indicates superior corrosion resistance for the methanol 30V sample due to its higher E_{corr} value. A less negative Ecorr value corresponds to lower

susceptibility to corrosion (Magar *et al*., 2021).

The methanol-treated sample at 30V exhibited superior corrosion resistance compared to the acetone-treated sample at 30V, as evidenced by lower I_{corr} and E_{corr} values and a decreased corrosion rate. This enhanced performance can be attributed to the presence of the HA/MWCNT layer, which effectively restricts the ingress of SBF solution into the SS 316L implant, thereby mitigating corrosive attacks (Zhang *et al*., 2014).

The employment of acetone and methanol suspensions yielded significantly distinct I_{corr} and E_{corr} values during PDP testing. This disparity could be attributed to the differing zeta potential of the two suspensions. Zeta potential is a crucial parameter in the EPD method (Amrollahi *et al*., 2015). The variance in zeta potential of the suspensions in this study is evident from the deposition sites.

The deposition of acetone HA/MWCNT on the anode in the sample indicates that acetone's zeta potential is negative (Rehman, *et al*., 2020). In contrast, the deposition of HA/MWCNT

on the cathode suggests that methanol's zeta potential is positive (Rehman, *et al*., 2020). In EPD, anode deposition is uncommon due to the ease of oxidation (Nguyen, et al., 2020). Adjusting the pH to 4 using nitric acid can render the zeta potential positive. HA/MWCNT nanocomposites exhibit stability under alkaline conditions but exhibit higher solubility under acidic conditions (Kang *et al*., 2021).

The Nyquist plot was obtained through fitting. The equivalent circuit based on Figure 7 consists of several electrical elements reflecting the surface conditions between the metal and the electrolyte. These electrical elements include solution resistance (Rs), polarization resistance (Rp), and charge transfer resistance from the Substratee-coating interface (Rb). Higher polarization resistance (Rp) values indicate better passive layer characteristics, resulting in a slower corrosion process (Garbacz and Kro´likowski, 2019). Figure 8 depicts the Nyquist plot generated by the equivalent circuit.

Figure 7. Equivalent circuit of (A) uncoated SS 316L and (B) SS 316L-HA/MWCNT

Figure 8. Nyquist plot of SS 316L and SS 316L-HA/f-MWCNT

The values of the fitting elements can be observed in Table 2. A higher polarization resistance (Rp) implies a more challenging flow of electrical current on the metal surface. This will decrease the charge transfer rate, electron release, and metal ion release, leading to a lower corrosion rate (Magar *et al*., 2021).

The methanol 30V sample exhibits the highest Rp value (114.990 Ω), significantly exceeding that of the acetone 30V sample (23.891 Ω). This suggests that the formed HA/f-MWCNT layer possesses superior corrosion resistance. As the applied voltage during EPD increases, a greater number of

nanocomposites adhere to the SS 316L surface, resulting in a denser layer structure with smaller pores (Boccaccini *et al*., 2020).

High voltages, as shown in Figure 5, promote crack formation, thereby compromising the layer's corrosion resistance. This is further confirmed by Electrochemical Impedance Spectroscopy (EIS) testing, which corroborates the findings of Potentiodynamic Polarization (PDP). The EIS results indicate that the sample treated with methanol at 30V exhibits the lowest corrosion rate, reflecting its superior corrosion resistance.

	Acetone Suspense			Methanol Suspense			
Voltage (V)	$\operatorname{Rs}(Ohm)$	Rb (Ohm)	Rp(Ohm)	\mathbf{Rs} (Ohm)	Rb (Ohm)	Rp(Ohm)	
SS 316L Sample	9.91	$\overline{}$	5.127	9.91	$\overline{}$	5.130	
$SS 316L - 20V$	14.15	13.8	20.909	13.68	13.9	16.380	
$SS 316L - 30V$	17.81	14.3	23.891	16.76	15.8	114.990	
$SS 316L - 40V$	13.68	13.2	18.529	14.15	14.5	37.620	

Table 2. EIS measurements result of uncoated SS 316 and SS 316L-HA/MWCNT

Suspensions containing different functional groups resulted in significantly disparate Rp values during experimentation (Hassan *et al*., 2020). These variations can likely be attributed to the distinct chemical properties, ion solubility, electron availability, and electrolyte concentration of the suspensions employed (Magar *et al*., 2021). Amrollahi *et al*. (2015) identified methanol as an ideal suspension medium due to its high dielectric constant, leading to the formation of high-quality coatings via Electrophoretic Deposition (EPD). This aligns with our findings, suggesting that methanol is superior for producing HA/f-MWCNT layers that effectively protect SS 316L-based dental implants from corrosion.

The presence of a highly electronegative oxygen atom bonded to a hydrogen atom in the hydroxyl group (O-H) of methanol (CH₃OH) creates a significant polarity due to the unequal sharing of electrons. This results in a larger dipole moment for methanol compared to acetone (C_3H_6O) , which has a carbonyl group (C=O) with a less pronounced polarity (Gopi *et al*., 2013; Alaska and Bartmanski, 2020).

Methanol exhibits a greater ability to dissolve specific ions compared to acetone. This facilitates the electrochemical reactions at the electrodeelectrolyte interface. The difference in molecular polarity between acetone and methanol likely plays a crucial role in explaining the observed disparity in the Rp values (Henkel *et al*., 2018).

Conclusions

This study reports the successful fabrication of SS 316L-HA/MWCNT dental implants via the EPD method using acetone and methanol suspensions. Voltage variations of 20V, 30V, and 40V were employed. The 30V methanol sample exhibited superior layer quality, demonstrating greater homogeneity and smoothness compared to the 30V acetone sample. Furthermore, the corrosion rate of the 30V methanol sample (0.0004 mmpy) was significantly lower than that of the 30V acetone sample (0.075 mmpy). The preference for methanol as a suspension medium in the EPD process is attributed to its higher polarity, which facilitates the effective dissolution of specific ions compared to acetone. The HA/MWCNT layer not only enhances corrosion resistance but also functions as a barrier, reducing electrolyte diffusion to the SS 316L surface and hindering the ionic transport of Ni, Cr, and Fe from the substrate.

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Conflict of Interest

All authors declare no conflicts of interest and concur with the manuscript's contents.

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