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ORIGINAL RESEARCH

DEPOSITION SILVER BASED THIN FILM ON STAINLESS STEEL 316L AS ANTIMICROBIAL AGENT USING ELECTROPHORETIC DEPOSITION METHOD

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Abstract

SUS316L stainless steel has been widely used in medical applications. However, some germs frequently adhere to the device surface, resulting in infections following implantation surgery. Unfortunately, the material lacks antibacterial characteristics that prevent microorganisms from adhering to the surface. This study aims to use electrophoretic deposition to deposit chitosan/silver (Ag) as an antibacterial agent on stainless steel 316L. The antimicrobial effects of chitosan and silver are well established. During the deposition, the rectifier voltage was adjusted to a constant 10 volts with a suspension pH range of 2.7 to 5.1. The effect of varying the pH of the suspension on the physical, mechanical, and antibacterial properties of chitosan/Ag thin films was investigated. The materials' structure and morphology were studied using X-ray diffraction (XRD), scanning electron microscopy (SEM), and Fourier-transform infrared spectroscopy (FTIR). The antimicrobial inhibition was examined using the Kirby-Bauer antimicrobial test. The results reveal that increasing the pH of the suspension causes an increase in the thickness, size, and aggregation of the chitosan/Ag thin film. The highest thickness achieved during deposition with a pH 5.1 suspension is 5.265 μm. The best antibacterial agent is achieved at a pH 3.5 suspension sample with an inhibitory zone diameter of 4 mm.

KEYWORDS:

Antimicrobial, Chitosan/Ag, Electrophoretic Deposition, SUS316L

1 | INTRODUCTION

Materials selected for use as implants must have high mechanical qualities as well as corrosion resistance. Because of its outstanding mechanical qualities and corrosion resistance, stainless steel 316L (SUS316L) is commonly utilized as an implant material. However, bacterial infections on SUS316L surface implants are common [1, 2]. The infection begins with the formation

of biofilm on the surface. Antimicrobial agent coatings can be used to inhibit biofilm formation. Silver is a typical antibacterial agent that can be put as a thin layer on surface implants. Chitosan is also recognized to have antibacterial characteristics that are beneficial to many microbes. The mechanism of antimicrobial action has been explored using the antibacterial ability of chitosan due to ionic contact with bacterial cell walls[3].

In the current study, a mixture of chitosan and silver was used. However, the composition of the combination must not produce toxicity in the body[4]. Ag^+ ions are responsible for Ag's antibacterial properties. The Ag^+ ion emitted from the silver layer interacts with microorganisms by attracting amino groups from microorganisms such as proteins, as well as purine nitrogen and pyrimidine from DNA and RNA. In this study, an electrophoresis deposition method was used to deposit a chitosan/Ag thin layer on the SUS316L substrate with modifications in the acidity level of the chitosan/Ag suspension solution. The combination of chitosan and Ag is expected to create a synergy between the two antimicrobial materials.

2 | PREVIOUS RESEARCHES

Hans et al.[5] did previous research using a combination of materials, including silver (Ag) and copper (Cu). According to the findings of this study, adding a silver layer to the porosity of the copper layer can boost resistance to Escherichia coli germs. The effectiveness of antibacterial capabilities is also affected by environmental factors. When exposed to arid environments, copper has a higher rate of destroying microorganisms. Because silver possesses antibacterial qualities that are particularly effective in moist or wet settings, it is a popular choice for covering bone implants[5].

Pishbin et al.[6] conducted another study on coating SS316L with various materials 2013. They investigated the antimicrobial properties and biocompatibility of SS316L without coating, SS316L coated with chitosan, chitosan/bioglass composite, and chitosan/bioglass/AgNPs composite with coated coating—antimicrobial testing with Staphylococcus aureus bacteria utilizing the electrophoretic deposition method. The addition of AgNPs to the thin film reduced the quantity of Staphylococcus aureus bacterial cells, according to the findings of this study. Meanwhile, regarding biocompatibility, adding Ag might potentially render the coating harmful, as the biocompatibility test findings showed that the osteoblast cells decreased with increasing Ag concentration[6].

3 | MATERIAL AND METHOD

In this experiment, stainless steel 316L was used as the substrate. The diameter of the substrate is 10 mm, and the thickness is 5 mm. The wire-cut procedure was used to prepare the sample. It was then polished to grade 600 and washed with distilled water to remove contaminants. The solution was created by combining 0.5 g of chitosan powder and one mM silver nitrate ($AgNO_3$) in 20 ml of acetic acid (0.35 Molar). The solution was agitated with a magnetic stirrer at 400 rpm for 24 hours to disseminate the chitosan and silver nitrate.

The pH of the solution was adjusted to 2.7, 3.5, 4.3, and 5.1 by adding HCl and NaOH to the solution. In this electrophoretic deposition process, SS 316L was used as the cathode (negative electrode) and graphite as the anode (positive electrode) with a power of 10 Volts for a deposition time of 10 minutes. The electrochemical reactions that occur during the deposition of chitosan/Ag on the substrate surface can be approximated as follows:

Cathode:

$$2H_2O + 2e^- \rightarrow H_2 + 2OH^- \tag{1}$$

$$O_2 + 2H_2O + 4e \rightarrow 4OH^- \tag{2}$$

Anode:

$$Chit - NH_3^+ + OH \rightarrow Chit - NH_2 + H_2O \tag{3}$$

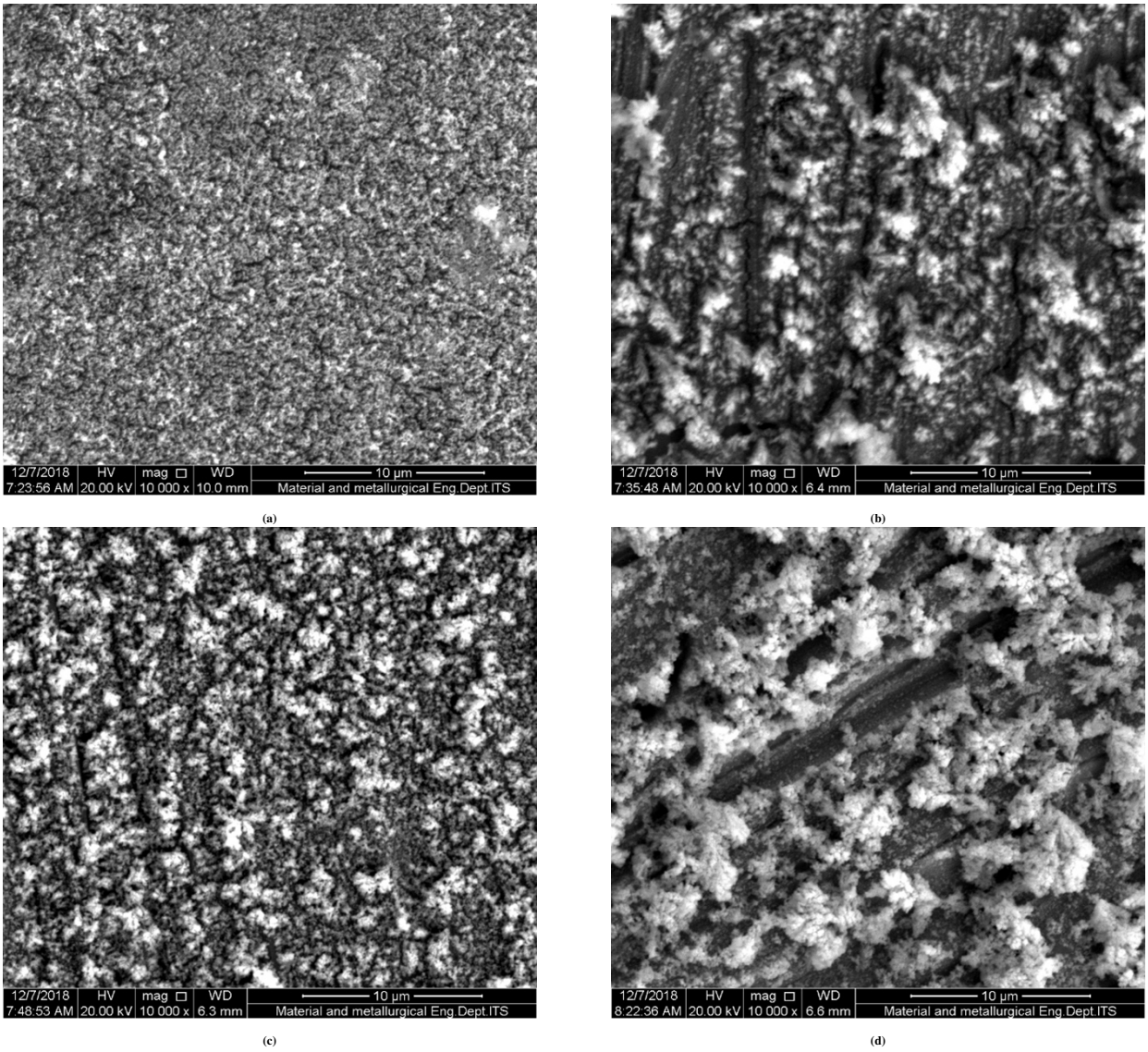

FIGURE 1 The SEM micrograph of chitosan/Ag thin film surface morphology with variation pH (a) 2.7 (b) pH 3.5 (c) pH 4.3 (d) pH 5.1.

$$Ag^{+}_{(aq)} + e \rightarrow Ag \tag{4}$$

4 | RESULTS AND DISCUSSION

Fig. 1 shows a scanning electron microscope (SEM) image of the surface morphology following deposition. White agglomerates can be seen spread across the layers. The white dots represent Ag particles dispersed in terms of location and size[7]. Furthermore, the higher the pH, the more chitosan and Ag are distributed in the chitosan/Ag thin layer. The greater the pH of the electrophoretic deposition suspension, the greater the macromolecular mobility toward the substrate and the greater the number of deposited macromolecules on the surface[8]. The particle size of Ag is affected by the suspension solution and pH. The structure morphology of Ag particles is formed in small size to agglomeration as a function of pH.

X-ray diffraction (XRD) testing was utilized to identify chemicals and elements produced in thin layers of chitosan/Ag. Fig. 2 depicts the XRD test findings. Some discovered elements and compounds, such as Ag, FeNi, and Cr, can be recognized using the

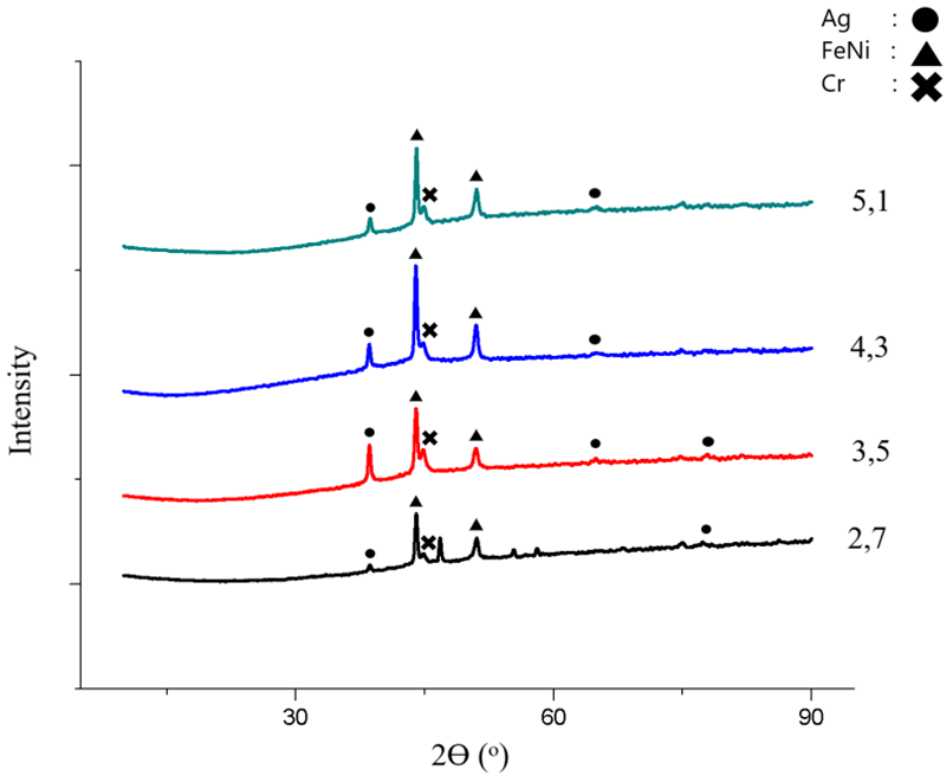

FIGURE 2 The XRD characterization results with pH variation.

XRD test findings. SUS316L is mostly composed of FeNi and Cr. According to the XRD results, the pH fluctuation during the deposition process does not affect the structure of the thin films. The peak of chitosan does not appear in the XRD test results; the concentration of chitosan is probably too low.

Fig. 3 presents FTIR characterization results at various pH levels. Chitosan's functional groups are O-H, N-H (amine), C=O, and C-O. According to the FTIR characterization data, raising pH increases the absorbance area of O-H and N-H stretching. This is accomplished by forming intermolecular hydrogen bonds with chitosan molecules in approximately 3450 cm-1 [9].

The bound hydrogen between -OH from additives and -OH or -NH2 from chitosan influences the shifting of the peak location at various wavelengths. It causes the amine group peaks at 1590 cm-1 and the amide peak at 1650 cm-1 to move or even disappear due to interactions between NH3+ and additions to adjust the pH. When samples with pH 2.7 are compared to samples with higher pH and chitosan without Ag doping, the O-H and N-H stretching peaks are observed. There is no intermolecular hydrogen bond because there are no contacts between chitosan molecules and -OH from additives in the chitosan/Ag thin layer created on a pH 2.7 sample. Adding Ag to chitosan allows the absorbance peak of N-H stretching primer and O-H stretching to occur. This phenomenon is induced by the reduction process of Ag+ by amino groups and alcohol, which causes the O-H peak and primary N-H to slope. The addition of Ag also causes the absorbance of C = O and N-H bending to change and new peaks to develop around 1558–1559 cm-1 [10].

Fig. 4 shows how altering pH alters the antibacterial property using the Kirby-Bauer antibacterial test in the medium bacterium Staphylococcus aureus. The graph illustrates the trend of adding pH to the chitosan/Ag suspension solution in the electrophoretic deposition method to acquire the best antibacterial capabilities of the chitosan/Ag at pH 3.5 of the solution. These findings are consistent with the results of XRD testing, which revealed the maximum Ag intensity and wt% at pH 3.5. The mechanism by which the Ag+ ion released from the silver layer interacts with microorganisms by attracting electrons from sulfur and nitrogen atoms in sulfhydryl and amino groups from microorganisms such as proteins, as well as nitrogen and pyrimidine from DNA and RNA, is the way the Ag element carries out its microbial activity.

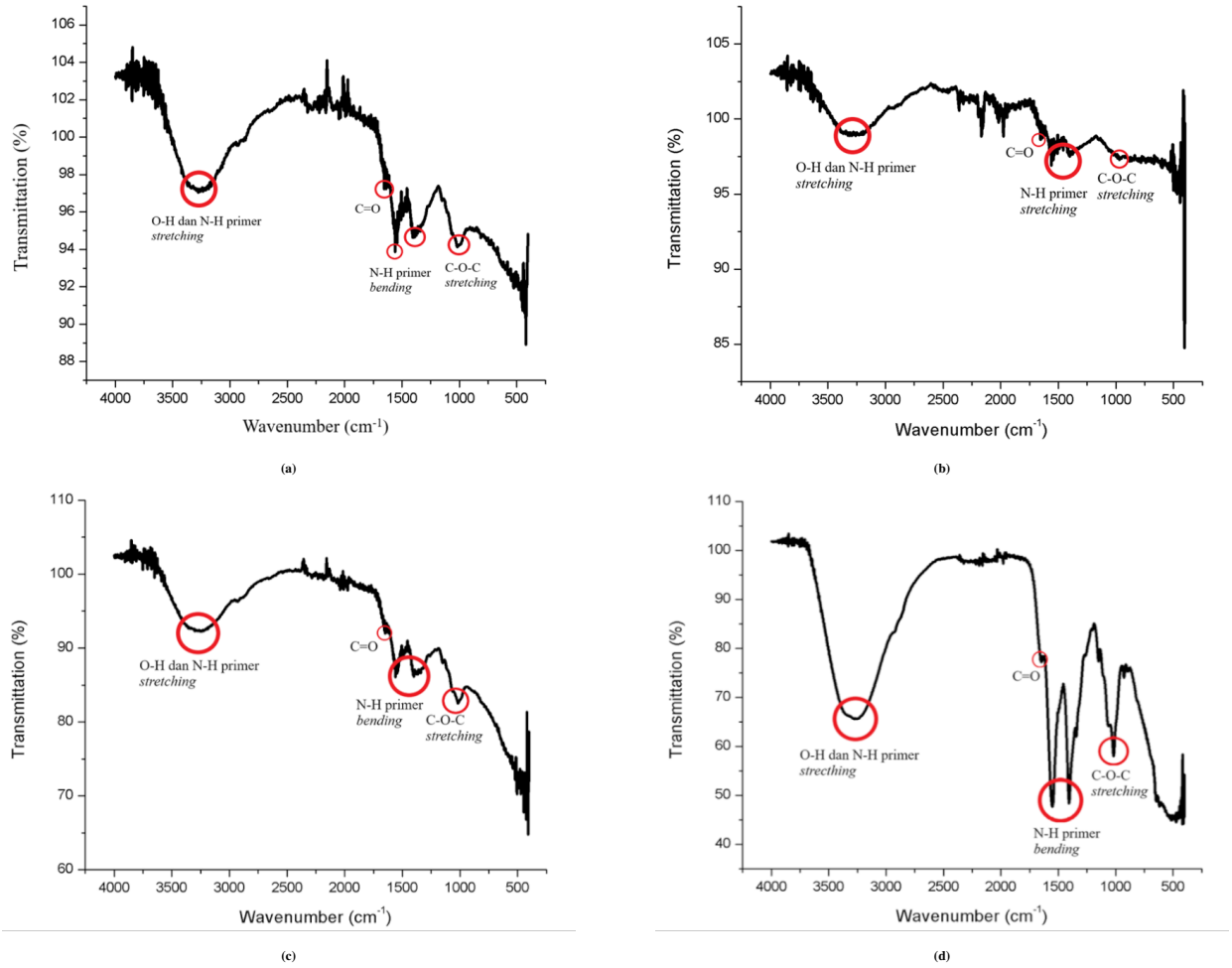

FIGURE 3 The FTIR analysis of a chitosan/Ag thin film surface with pH variations (a) 2,7, (b) 3,5, (c) 4,3, and (d) 5,1.

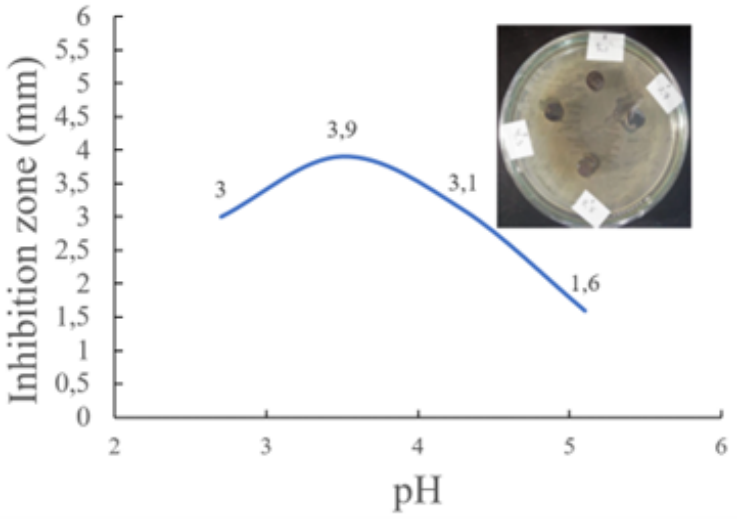

FIGURE 4 The effect of pH variation during the deposition process on the antimicrobial properties shown by inhibition zone .

Chitosan also has antibacterial activity by releasing ions that produce inter-ion interactions where the amine group is protonated, and the electron pair of nitrogen amine is available as an ion donation, resulting in hydrolysis and damage to microorganism cell walls. These mechanisms are controlled by environmental pH[11], followed by a positive charge from chitosan ions, which interact with bacteria and impede DNA and protein production. The decrease in antimicrobial characteristics produced by raising pH at the cathode surface will reduce the charge possessed by chitosan[12], hence diminishing antimicrobial properties because the charged ion is the core of chitosan's antibacterial action.

5 | CONCLUSION

The results and discussion show that adding NaOH to the chitosan/Ag suspension solution influences the thickness of the chitosan/Ag layer on the SUS316L substrate. When using pH 2.7, the lowest layer thickness is 2.3 μm, while the largest layer thickness is 5.3 μm when using pH 2.7 and 5.1, respectively. According to the inhibition test, adding a pH level of acidity to the chitosan/Ag suspension solution has little impact on the inhibitory zone in medium levels of Staphylococcus aureus bacteria. The most optimal pH value used during a deposition in this study was pH 3.5, which resulted in an inhibition zone of 3.9 mm.

CREDIT

Agung Purniawan: Conceptualization, Methodology, Writing - original draft preparation, Formal analysis and investigation, and Supervision. **Lukman Noerochim:** Formal analysis and investigation. **Laurentius Aditya Widagdo:** Writing - review and editing, Funding acquisition. **Ditta Gabriella Sinaga:** Writing - review and editing, Resources.

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