



Effect of corrosion rate and surface energy of silver coatings on bacterial adhesion

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ABSTRACT

Many studies suggest a strong antimicrobial activity of silver coatings. The biocidal activity of silver is related to the biologically active silver ion released from silver coatings. However, no studies have been reported on the effect of surface energy of silver coatings on antibacterial performance. In this paper, three silver coatings with various corrosion rates and surface energies were prepared on stainless steel plates using AgNO_3 based electroless plating solutions. The corrosion rate and surface energy of the silver coatings were characterized with CorrTest Electrochemistry Workstation and Dataphysics OCA-20 contact angle analyzer, respectively. The antibacterial performance of the silver coatings was evaluated with *Pseudomonas aeruginosa* PA01, which frequently causes medical device-associated infections. The experimental results showed that surface energy had significant influence on initial bacterial adhesion at low corrosion rate. The extended DLVO theory was used to explain the bacterial adhesion behavior.

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1. Introduction

The high incidence of infections caused by the use of implanted biomedical devices, including bone fracture fixation pins, catheters and heart valves etc. has a severe impact on human health and health care costs. For example, pin tract infection is the most common complications associated with the use of external fixation with infection rate as high as 33% [1]. Bacterial adhesion to medical devices is the first step in the development of such infections. Once bacteria attach to a medical device, a multistep process starts leading to the formation of a complex, adhering microbial community that is termed a biofilm. Once a biofilm has formed, it is very difficult to treat clinically because the bacteria on the interior of the biofilm are protected from phagocytosis and antibiotics. Medical devices-related infections often require device removal and replacement in order to avoid the development of severe and even lethal complications. The removal of infected devices and the additional treatment greatly increase patients' discomfort, treatment costs and mortality [2–4]. Thereby, an alternative strategy is required to control infections. Since bacterial adhesion to biomaterial surfaces is the essential step in the process of infections [5], modifications to biomaterial surfaces are considered to diminish infections by inhibiting initial bacterial adhesion [5].

In view of increasing bacterial resistance to antibiotics and antiseptics, silver coating may be an effective strategy to prevent device-related infections due to its good antimicrobial activity and

low toxicity [6]. The biocidal activity of silver is related to the biologically active silver ion released from silver coatings [6]. In general metallic silver appears to be inert and have no antibacterial action. Silver ions (Ag^+), however, bind to and react with proteins and enzymes, thereby causing structural changes in the bacterial cell wall and membranes, leading to membrane permeability damage, cellular disintegration and death of the bacterium [6,7]. It has been reported that the surface energy of coatings also has significant influence on initial bacterial adhesion [8–10]. However, no studies have been reported on the effect of surface energy of silver coatings on bacterial adhesion. In this paper, three types of silver coatings with various surface energies were prepared on stainless steel plates using AgNO_3 based electroless plating solutions. The effect of corrosion rate and surface energy of the coatings on bacterial adhesion was investigated with *Pseudomonas aeruginosa* PA01, which frequently causes medical device-associated infections.

2. Experimental

2.1. Electroless silver coatings

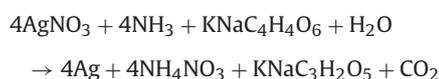
Silver were coated on 25 mm × 12 mm × 0.5 mm stainless steel (SS) 316L plates by electroless plating method. The stainless steel plates were first cleaned in alkaline solution at 80–90 °C for 10 min. The plates were electropolished in alkaline solution at 60–80 °C with current density of 5–10 A/dm² for 2 min. They were then dipped into a solution of HCl (30%):HNO₃ (65%):H₂O = 3:2:5 at 52–66 °C for 5 min. The plates were rinsed with de-ionized water after each step. The details of the pretreatment procedures are given in Table 1.

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Table 1
Pretreatment procedures of stainless steel 316L plates.

Alkaline cleaning	NaOH: 60–80 g/L; Na ₂ CO ₃ : 20–60 g/L; Na ₃ PO ₄ : 15–30 g/L; Na ₂ SiO ₃ : 5–10 g/L; 80–90 °C; 10 min.
Rinsing	De-ionized water at room temperature.
Electropolishing	NaOH: 10–20 g/L; Na ₂ CO ₃ : 50–60 g/L; Na ₃ PO ₄ : 50–60 g/L; 60–80 °C; 2 min; current density: 5–10 A/dm ² .
Rinsing	Hot de-ionized water, then de-ionized water at room temperature.
Pickling	HCl(30%):HNO ₃ (65%):H ₂ O = 3:2:5; 52–66 °C, 5 min.
Rinsing	De-ionized water at room temperature.

The electroless deposition of silver were carried out by immersing SS 316L plates into electroless plating solutions at room temperature. Three electroless silver coatings under three coating time (5 min, 15 min and 30 min) were prepared from AgNO₃–NH₃·H₂O based solutions. For electroless deposition of silver, the color of solution changed by dropping ammonia into the silver nitrate solution from clear to dark until clear again. In this experiment, the potassium sodium tartrate tetrahydrate was applied as the reducer. The reaction equation is as follows:



The ratio of silver–ammonia salt solution to the reducing agent solution was 1:1. The compositions of the electroless silver solution are shown in Table 2.

2.2. Thickness of silver coatings

In this investigation the standard direct Volhard titration method was used to determine coating mass. Silver coating was first dissolved in nitric acid, and then 0.015 M NH₄Fe(SO₄)₂·12H₂O was added into the solution. 0.01 M NH₄SCN was dropped into the solution until the endpoint of titration when the color changed to pink. The silver mass M_{Ag} in mg was calculated using the following equation:

$$M_{\text{Ag}} = V \times 1.08 \quad (1)$$

where V is the volume (ml) of 0.01 M NH₄SCN used.

The mass of silver coating can be calculated using the equation:

$$M_{\text{Ag}} = \frac{S \times h \times \rho}{10000} \quad (2)$$

where h is the thickness of the coatings in μm ; ρ is the density of the coatings in mg/cm^3 and S is the area of the coatings in cm^2 . So the thickness of silver coating can be calculated as follows:

$$h = \frac{V \times 1.08 \times 10000}{S \times \rho} \quad (3)$$

2.3. Corrosion test

The corrosion rate was measured with a CorrTest Electrochemistry Workstation which consists of a CS300UA potentiostat with a computer and a CorrTest software. Potentiodynamic polarization measurements were carried out in a 250 ml cell with three standard electrodes: saturated calomel electrode (SCE) as a reference, a platinum electrode as the counter and a coating sample as the working

Table 2
Compositions of electroless silver solution.

Silver–ammonia salt solution		Reducing agent solution	
AgNO ₃	15 g/L	KNaC ₄ H ₄ O ₆ ·4H ₂ O	150 g/L
NH ₃ ·H ₂ O	Suitable	C ₂ H ₅ OH	10 ml/L

Table 3
Component of artificial urine.

Chemicals	Concentration (g/L)
KH ₂ PO ₄	1.4
Urea	2.5
Creatinine	1.1
pH	6.0
Autoclave	121 °C, 15 min

electrode. All the potentials were reported with respect to SCE. Prior to Tafel polarization, the specimens were allowed to immerse in the corrosion solution for 30 min to obtain a stable open-circuit potential (OCP). The surface area exposed to the test solution was 6 cm². All the specimens were washed with distilled water and acetone prior to experiment. The experiments were performed in 0.9% NaCl solution and artificial urine at room temperature (25 °C), respectively. The composition of artificial urine is given in Table 3. Potentiodynamic polarization curves were recorded from a starting potential 50 mV below the open-circuit potential and scanned towards the positive direction at a scan rate of 0.5 mV s⁻¹, until the potential reached 50 mV.

2.4. Contact angle and surface energy

Contact angles were measured using the sessile drop method with a Dataphysics OCA-20 contact angle analyser. Three to five contact angle measurements were made on each sample for all probe liquids including distilled water and ethylene glycol (Sigma), and diiodomethane (Sigma). The surface tension components of the test liquids are shown in Table 4 [11]. All measurements were made at 25 °C. The contact angles of bacterial cells were measured by producing the bacteria lawns deposited on membrane filters with a pore diameter of 0.45 μm [12,13] and applying negative pressure. Prior to contact angle measurement, the bacterial lawns were allowed to air-dry until a certain state, indicated by stable water contact angles [12,13]. The surface energy of the coatings was calculated using van Oss acid–base approach [9]. The surface energy is seen as the sum of a Lifshitz–van der Waals apolar component γ_i^{LW} and a Lewis acid–base polar component γ_i^{AB} :

$$\gamma_i^{\text{TOT}} = \gamma_i^{\text{LW}} + \gamma_i^{\text{AB}} \quad (4)$$

The acid–base polar component γ_i^{AB} can be further subdivided by using specific terms for an electron donor (γ_i^-) and an electron acceptor (γ_i^+) subcomponent:

$$\gamma_i^{\text{AB}} = 2\sqrt{\gamma_i^+ \gamma_i^-} \quad (5)$$

The relation between the measured contact angle and the solid and liquid surface energy terms are described as:

$$\gamma_L \cdot (1 + \cos \theta) = 2(\sqrt{\gamma_S^{\text{LW}} \cdot \gamma_L^{\text{LW}}} + \sqrt{\gamma_S^+ \cdot \gamma_L^-} + \sqrt{\gamma_S^- \cdot \gamma_L^+}) \quad (6)$$

In order to determine the surface energy components (γ_S^{LW}) and parameters γ_S^+ and γ_S^- of a solid, the contact angles of at least three liquids with known surface tension components (γ_L^{LW} , γ_L^+ , γ_L^-), two of which must be polar, have to be determined [14].

Table 4
Test liquids and their surface tension components.

Surface tension data (mN/m)	γ_L	γ_L^{LW}	γ_L^{AB}	γ_L^+	γ_L^-
Water (W), H ₂ O	72.8	21.8	51.0	25.5	25.5
Diiodomethane (D), CH ₂ I ₂	50.8	50.8	0	0	0
Ethylene glycol (E), C ₂ H ₆ O ₂	48.0	29.0	19.0	1.92	47.0

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