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## Electrodeposition, characterization, and antibacterial activity of zinc/silver particle composite coatings



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#### ABSTRACT

Composite coatings consisting of zinc and silver particles (Zn/AgPs) with antibacterial activity were prepared using an electrodeposition technique. The morphology, composition, and structure of the Zn/AgPs composite coatings were analyzed using scanning electron microscopy (SEM) coupled with energy-dispersive spectroscopy (EDS), inductively coupled plasma (ICP) spectrometry, and X-ray diffraction (XRD). The antibacterial properties of the coatings against the microorganisms *Escherichia coli* as a model Gram-negative bacterium and *Staphylococcus aureus* as a model Gram-positive bacterium were studied quantitatively and qualitatively. The results revealed that the dispersant cetyltrimethylammonium bromide (CTAB) assisted in the formation of a stable suspension of Ag particles in the electrolytic bath for 24 h. Likewise, a high concentration of CTAB in the electrolytic bath promoted an increase in the number of Ag particles occluded in the Zn/AgPs coatings. The Zn/AgPs coatings that were obtained were compact, smooth, and shiny materials. Antimicrobial tests performed on the Zn/AgPs coatings revealed that the inhibition of bacterial growth after 30 min of contact time was between 91% and 98% when the AgPs content ranged from 4.3 to 14.0 mg cm<sup>-3</sup>.

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#### 1. Introduction

Epidemiological data from Europe, North America, and Australia indicate that bacteria and viruses cause the spread of a significant percentage of diseases from one person to another [1,2]. One route via which bacteria or viruses are transmitted is directly through contact with contaminated metallic surfaces (e.g., supermarket shopping carts, handrails on buses, and metallic structures in recreational parks). In fact, supermarket shopping carts and bus railings are among the five most heterotrophic-bacteria-contaminated sites. These structures predominantly consist of steel protected with a metallic coating, such as chromium (Cr), nickel (Ni), or zinc (Zn). Zn coatings are widely used to protect steel surfaces because they are more resistant to environmental corrosion than is the steel substrate [3,4].

In recent years, inorganic antibacterial agents have attracted the attention of researchers because of their thermal resistance and the persistence of their antibacterial effects compared with organic antibacterial agents [5]. Since the nineteenth century, silver (Ag)-based compounds have been used in many antimicrobial applications; colloidal silver was used in wound antisepsis and in combination with citrate salts for the treatment of skin infections, and silver nitrate was employed almost 100 years ago for the treatment of ophthalmia neonatorum [6]. Currently, nanoparticles are used in numerous physical, biological, and pharmaceutical applications. The antibacterial properties of silver particles against Gram-negative and Gram-positive bacteria have been widely demonstrated [7-11]; for this reason, silver particles (AgPs) are used in a large variety of applications, such as athletic apparel, washing machines, food packaging materials [12,13], and, most importantly, in the medical field as bactericidal and therapeutic agents. AgPs are used in the fabrication of dental devices, as bactericidal coatings in water filters, and as antimicrobial agents in air sanitizer sprays, pillows, respirators, socks, wet wipes, detergents, soaps, shampoos, toothpastes, and many other consumer products.

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AgPs are also used as antimicrobial agents in many public places such as railway stations and elevators in China, and they are said to demonstrate good antimicrobial activity. Although silver particles offer various benefits, primarily as a result of their beneficial antibacterial properties [14,15], there is also the problem of the nanotoxicity of silver. Various studies in the literature suggest that nanoparticles can cause various environmental and health-related problems [16,17]. Nevertheless, there is a need for more studies to be conducted before it can be unequivocally concluded that the use of silver nanoparticles poses a significant problem.

In this work, the antimicrobial properties of silver particles (AgPs) are combined with the protective capacity of Zn coatings to form Zn/AgPs composite coatings. The advantage of such Zn/AgPs composite coatings is as follows: during the zinc electrodeposition process, the silver particles become occluded in the zinc metal coating matrix without losing their antibacterial properties, thus forming part of the coating and not merely a surface film; in this manner, hygienic metal coatings are formed. The combination of the antimicrobial properties of silver particles (AgPs) with the protective capacity of Zn coatings enables the possibility of a number of novel applications for Zn/AgPs composite coatings, in the biomedical field or in food processing, for example. More broadly, this technology can be applied in any situation in which metallic objects are in constant contact with people's hands and in which the surfaces involved must be durable, safe, readily cleanable, and resistant to microbial contamination.

The objective of the present work was to investigate the effects of the dispersant (surfactant) concentration in the electrolytic bath on the stability of the AgPs suspension as well as the chemical composition, morphology, structure, and antibacterial activity of the Zn/AgPs coatings obtained using an electrolytic bath containing suspended Ag nanoparticles.

The morphologies, elemental composition profiles, and structures of the Zn/AgPs coatings were analyzed using scanning electron microscopy coupled with energy-dispersive spectroscopy (SEM–EDS), inductively coupled plasma (ICP) spectrometry, and X-ray diffraction (XRD). The antimicrobial activities of the Zn/AgPs composite coatings against *Escherichia coli* (*E. coli*) as a model Gramnegative bacterium and *Staphylococcus aureus* (*S. aureus*) as a model Gram-positive bacterium were investigated.

#### 2. Materials and methods

#### 2.1. Turbiscan Lab Expert stability analyses

To achieve the occlusion of the Ag particles (AgPs) during the zinc electrodeposition process, a stable dispersion of the AgPs in the aqueous solution is required. The modification of a solid surface via the adsorption of a water-soluble polymer is an efficient and widely employed method of enhancing the dispersion stability of a suspension [18,19]. Electrostatic and steric mechanisms are typically considered to be responsible for the stabilization of suspensions. Adding a dispersant, because of its spatial structure and hydrophilic functional groups, can enhance the electrostatic repulsion and steric hindrance between AgPs. In addition, because of the adsorption of the cationic surfactant onto the surfaces of the silver particles, the AgPs develop a positive surface charge (see Fig. 1), which facilitates their migration toward the surface of the cathode (negatively charged electrode), where they are occluded during the formation of the Zn coating.

The following procedure was used to analyze the influence of the dispersant (surfactant) on the stability of AgPs in an electrolytic bath. First, 0.0625 g of AgPs (99.9%, 50–60 nm, SkySpring Nanomaterials, Inc.) was weighed and added to 25 mL of a base solution  $S_0$  (electrolytic bath) at pH 5.0 that contained the following: 81.0 g  $L^{-1}$ 

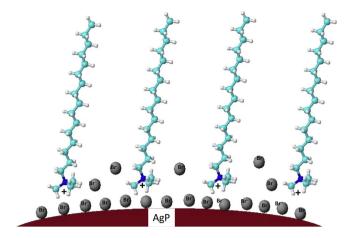


Fig. 1. Schematic model of the absorption of CTAB on charged electrode surfaces.

 $ZnCl_2.6H_2O + 208.80 gL^{-1} KCl + 25 gL^{-1} H_3BO_3 + 0.75 gL^{-1} sodium$ benzoate  $+0.2 \,\mathrm{g}\,\mathrm{L}^{-1}$  benzylideneacetone  $+1.5 \,\mathrm{g}\,\mathrm{L}^{-1}$  PEG  $+2.8 \,\mathrm{g}\,\mathrm{L}^{-1}$ triethanolamine. Next, the dispersant, cetyltrimethylammonium bromide (CTAB) (98%, Spectrum Labs, USA), was added to the solution in the desired concentration (mM) at a given pH value for the evaluation of the long-term dispersion stability. Several concentrations of CTAB in the electrolytic bath were tested. The aqueous suspension of AgPs was placed into cylindrical glass tubes and set into a Turbiscan Lab Expert analyzer (Formulation Co., L'Unión, France). The Turbiscan Lab Expert not only allows for the measurement of the long-term stability of opaque and concentrated colloidal dispersions using a single instrument but also is capable of detecting instability much earlier and more easily than can be accomplished with the naked eye [20]. The sensors for transmitted and backscattered light in this optical analyzer scanned the entire height (53 mm) of the aqueous AgPs suspension (25 mL) for 24 h. The stability analysis of the aqueous AgPs suspension was performed based on the transmission ( $\Delta T$ ) and backscattering ( $\Delta BS$ ) profiles shown in Fig. 2. The thickness of the clarifying layer ( $\Delta H$ ) in the clarifying zone can be calculated from the transmittance graph using the TLAB EXPERT software. In this software, the user defines the lower and upper limits of transmittance ( $H_{inf}$  and  $H_{sup}$ , respectively); the software then determines the positions of  $H_{inf}$ and  $H_{\text{sup}}$  with respect to the length of the vial ( $H_{\text{d-sup}}$  and  $H_{\text{d-inf}}$ ) (see Fig. 2a). The distance  $\Delta H$  (mm) with respect to the length of the vial between the two values  $(\Delta H = H_{d-sup} - H_{d-inf})$  defines the extent of the precipitation of the AgPs. Small values of  $\Delta H$  indicate a stable suspension, whereas large values indicate an unstable suspension.

#### 2.2. Zn/AgPs coatings

Zn/AgPs composite coatings were formed via electrodeposition, using a methacrylate parallel-plate cell with an interelectrode distance of 5 cm. The temperature of the electrolytic bath was maintained at 25 °C. A Zn plate (99%, Atotech) was used as the anode, and a plate of AlSI 1018 steel with an exposed area of  $10 \times 15 \text{ cm}^2$  was used as the cathode. The Zn/AgP composite was electrodeposited from a base solution ( $S_0$ ) composed of the following:  $81.0 \text{ g L}^{-1}$  ZnCl<sub>2</sub>.6H<sub>2</sub>O+208.80 g L<sup>-1</sup> KCl+25 g L<sup>-1</sup> H<sub>3</sub>BO<sub>3</sub>+0.75 g L<sup>-1</sup> sodium benzoate+0.2 g L<sup>-1</sup> benzylideneacetone+1.5 g L<sup>-1</sup> PEG+2.8 g L<sup>-1</sup> triethanolamine+2.5 g L<sup>-1</sup> AgNPs+x mM CTAB, where x = 0.0, 0.05, 0.1, 0.5, 1.0, 10, or 50. All reagents were of analytical grade, and the corresponding solutions were prepared using deionized water (18 MΩ cm). In all cases, the pH of the working solution was 5.0. The composition of the bath was formulated and optimized in our laboratory. The electrodeposition current density (0.021 A cm<sup>-2</sup> over

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