Morphology, Structural and Electrical Properties of Ag–Cu Alloy Nanoparticles Embedded in PVA Matrix and Its Performance as E. coli Monitoring Sensor

Huda Abdullah · Norshafadzila Mohammad Naim · Aisyah Bolhan · Noor Azwen Noor Azmy · Aidil Abdul Hamid

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Abstract A new low-cost, simple and clean method for microbial monitoring sensor based on bimetallic thin film is fabricated in this research. The nanocomposite of Ag–Cu doped with PVA has been synthesized via sol–gel method and deposited on glass substrate. Ag–Cu alloy thin films with various concentrations were characterized using XRD, FTIR, UV–Vis, AFM, TEM and I–V measurement. The peaks in XRD pattern confirm the presence of Ag, Cu and Ag–Cu alloy nanoparticles in FCC structure. FTIR spectra found the O–H stretching of hydroxyl group and C–H stretching of PVA. The resonance UV–Vis absorption peaks are all sharp and lie between the wavelengths of 296–299 nm. The surface of the films has been found to be smoother as the Cu concentrations increased. The sensor performance was tested by measuring the changes of current of the film using I–V measurement. The Ag0.2–Cu0.8 alloy film shows obvious changes in current when it was incubated with E. coli. The sensitivity measurement, S, shows that the high sensitivity can be observed at higher concentrations of Cu.

Keywords Ag–Cu alloy · PVA · Morphology · Structural properties · Electrical properties · E. coli · Sensor

1 Introduction

Escherichia coli (E. coli) are microorganism that can cause food poisoning in humans. E. coli O157:H7 is rare strain of E. coli that is considered to be one of the most dangerous foodborne and waterborne pathogens, which can cause food poisoning in humans and can become life threatening [1]. This O157:H7 strain produces large quantitative of a potent toxin, in the lining of the intestine and causes severe damage resulting in hemorrhagic colitis or hemolytic uremic syndrome which may lead to death, especially in children. There have been a lot of cases of food poisoning outbreak causes by E. coli [2–5]. E. coli can be transmitted and spread not only through the food and water, but also people. Therefore, it is necessary and important to control and detect E. coli which is the life-threatening microbial species. So, a biosensor which has high sensitivity and fast identification is important to be produced in order to prevent the infection of E. coli. Many tests have been developed for the detection of E. coli from the other researchers by using various methods such as surface plasma resonance (SPR) [6], electronic nose [7], potentiometric alternating biosensing (PAB) [8], electrochemical impedance spectroscopy (EIS) [9] and differential pulse voltammetry (DPV) [10]. Our research is to fabricate a new low-cost, simple and clean method to detect E. coli, and it was found that the use of Ag–Cu doped with the polyvinyl alcohol (PVA) in nanocomposite particle is suitable for detecting E. coli. However, specific ratio of concentration of metal can detect specific type of microorganism [11–13]. Silver and copper are two metals which have antibacterial properties. According to Guo et al. [14], silver exhibited much greater antibacterial effects, and it has the highest electrical conductor among all metal according to periodic table. Guzman et al. [15] also reported that the high reactivity of silver nanoparticles due to the large surface to volume ratio,
nanoparticles play a crucial role in inhibiting bacterial growth in aqueous and solid media. Silver-containing materials can be employed to eliminate microorganisms on textile fabrics. The nanoparticles of Ag and Cu are combined to increase the sensitivity of the sensor. PVA have been frequently used as a size-controlling agent and particle stabilizers in chemical synthesis of metal colloids, since they prevent agglomeration and precipitation of the particles [16–19]. PVA is also widely known as crystalline polymer [20]. When the metal nanoparticles are embedded or encapsulated in polymer, the polymer can acts as surface-capping agent. In addition, film preparation become easier using PVA and the particle size can be controlled well within the desired regime when a thin film is formed.

In this paper, the synthesis of Ag–Cu alloy in PVA nanocomposite using spin-coating technique to be applied as E. coli monitoring sensor was proposed. The thin films were characterized using a few analytical methods such as X-ray diffraction (XRD), atomic force microscopy (AFM), Fourier transform infrared spectroscopy (FTIR), UV–Vis spectroscopy and transmittance electron microscopy (TEM). The sensitivity and the effectiveness of the sensor on E. coli were tested by measuring the current–voltage (I–V) of the thin film before and after incubation with E. coli bacteria in solution.

2 Experimental

2.1 Preparation of PVA Doped Ag–Cu Nanocomposite Thin Film

Silver nitrate (AgNO₃, 99.99 % purity) as the precursor, copper acetate (Cu(CH₃COO)₂, 97 %) and polyvinyl alcohol (PVA, 99 % hydrolysis, molecular weight = 85,000–124,000 g/mol) as the size-controlling agent were purchased from Sigma-Aldrich Chemicals. 2.5 g of PVA was dissolved in 40 mL of deionized water and stirred at 8–90 °C. Ag–Cu solution was synthesized by dissolving 0.5 g of silver nitrate and copper acetate in deionized water following the formula of Ag₁₋ₓCuₓ with x represents the weight ratios of Ag and Cu (x = 0.8, 0.6, 0.5, 0.4 and 0.2). When the PVA becomes completely dissolved, the solution transforms into a transparent liquid, and then Ag–Cu solution was added drop by drop into PVA solution by using pipette. In this state, the temperature was maintained between 60 and 70 °C, and the solution was continuously stirred until it became a yellowish brown liquid. At hot condition, the hydrogen de-bonds from the OH of the dispersed PVA and this hydrogen de-bonded PVA serves as a template to form Ag⁺ and Cu⁺ reactions, surface stabilizer and a protective agent for silver and copper nanoparticles [17]. Spin-coating technique has been used to deposit Ag–Cu alloy-doped PVA nanocomposite on the glass substrate. The mixture was spin-coated onto the glass substrate using Laurell Technologies Corporation photoresist spinner, with the speed of 3,000 rpm within 30 s. The nanocomposite thin films were dried on the hot plate for 1 h. Then, the thin films were cooled down to a room temperature by turning off the hot plate temperature. A comb-type structure of silver electrode was sputtered on the nanocomposite thin films using RF magnetron sputtering. 1,000 Å of thickness has been fixed by the sputtering equipment during the deposition operation. Copper wires were soldered to the silver electrodes as the connection between thin film and the measuring device. The silver electrode on the thin film surface has been designed with the measurement of 3 mm width and 2 mm length of separation between two electrodes. The layers of the thin film sensor and the image of fabricated thin film sensor are shown in Fig. 1a, b.

2.2 Characterization

X-ray diffraction analysis was conducted on Bruker model D8 Advanced X-ray diffractometer using CuKα radiation (λ = 1.5406 Å), and the measurement were performed in 2θ range from 20 to 60°. AFM analysis has been done on the Ag–Cu thin film to observe the surface roughness of the thin film. The bonding groups in the thin film were identified by FTIR spectroscopy and the type of transition by observing the characteristic of absorption peak can be measured using UV–Vis. TEM analysis was conducted to study the morphology. The sensor performance was conducted using GAMRY Physical Electrochemistry by measuring I–V characteristic of the thin film. E. coli concentration used in this experiment is constant (3.57 × 10⁶ CFU/mL) for each films sensor.