CHAPTER III

EXPERIMENTAL

This chapter describes the experimental systems and procedures used in this study. It is divided into four sections. Section 3.1 is materials, used in this study. Section 3.2 describes procedures for gold colloids synthesis. Then, the fabrication of gold nanoparticles thin film is presented in section 3.3. Finally, characterizations of the products are presented in the last section, in section 3.4.

3.1 Materials

Hydrogen tetrachloroaurate (III) trihydrate ($HAuCl_4$, purity:99.9+%) and sodium citrate ($Na_3C_6H_5O_7$, purity:99%) were purchased from Sigma and Aldrich. Glass slide use microscope slide, made in China and (3-aminopropyl)-trimethoxysilane (APTMS, purity: 97%), (3-mercaptopropyl)-trimethoxysilane (MPTMS, purity: 95%) and polyethylenimine (PEI, Mw=25,000, water-free) were purchased from Aldrich Chemicals. Methanol (CH_3OH, purity: 99.9%) purchased from Merck Germany was employed as solvent for the solution of all modified surface reagent. Hydrogen peroxide (H_2O_2, purity: 30%), sulfuric acid (H_2SO_4, purity: 96.9%), Ethyl Acohol (C_2H_5OH, purity: 99.8%) were purchased from Merck Germany, J, T. Backer USA, Carlo Erba Reactifs SA, respectively and used for substrate treatment. Water was deionized by Milli-Q water system.

3.2 Gold Nanoparticles Synthesis

Gold nanoparticles were prepared by citrate-reduction method [91]. Gold colloids were prepared by adding 32 ml of 5mM $HAuCl_4$ aqueous solution and 100 ml of 25 mM sodium citrate aqueous solution into 800 ml of boiling water. The citrate ion served as both reducing agent for AuNPs formation and as stabilizer preventing agglomeration of AuNPs.

3.3 Fabrication of AuNPs Thin Film

Surfaces used in this work are glass slide and polyimide surfaces. Prior to the deposition, substrates were thoroughly cleaned in ethanol and deionized water for 15 min under ultrasonic agitation. Then, they were pretreated in a 30:70 (v/v) mixture of H_2O_2 (30%) and H_2SO_4 (concentrated) at 60-80°C for 45 min, washed with deionized water for 15 min and dried in an oven at 110°C for 45 min [92]. The resulting SiO_2 surface was considered to have about five OH group per nm^2 [93, 94].

Cleaned surfaces of the glass and polyimide substrates were modified by putting the substrates in a solution of modifying agent, i.e. MPTMS, APTMS and PEI, in methanol for 12 hours. Then, the modified surfaces were washed with methanol and deionized water respectively, in an ultrasonic bath. Subsequently, the modified substrates were immersed in gold colloidal solution prepared earlier. The deposition time for AuNPs was generally 12 hours, after which the glass slide was extensively rinsed with deionized water. Finally, monolayer of AuNPs thin film was fabricated.

3.4 Surface Characterization

The obtained products were characterized by using various techniques, as following:

3.4.1 Atomic force microscope (AFM)

The surface morphology was characterized with atomic force microscopy (AFM measurements), using a MultiMode (Digital Instruments) microscope operated in the tapping mode, in air, and room temperature.

3.6.2 X-ray photoelectron spectroscopy (XPS)

The relative elemental composition of gold nanoparticles on modified surface was measured with a Amicus X-ray photoelectron spectroscopy generated at 20 mA and 12 kV.

3.6.3 UV/Visible spectroscopy (UV-Vis)

The detection of gold nanoparticles thin film was also conducted by UV/Visible Spectroscopy (Lambda 650 of PerinElmer).