

CHAPTER III

EXPERIMENTAL

3.1 Materials

3.1.1 Chemicals

3.1.1.1 *Aniline (ANI)*

3.1.1.2 *Ammonium Persulfate (APS)*

3.1.1.3 *Polystyrene Sulfonate (PSS)*

3.1.1.4 *Poly(sodium 4-styrenesulfonic Acid-co-maleic acid) (COPSS)*

3.1.1.5 *Chloroform (CHCl₃)*

3.1.1.6 *Polydiallyldimethylammonium Chloride (PDADMAC)*

3.1.1.7 *Sodium Chloride (NaCl)*

3.1.1.8 *Sodium Hydroxide (NaOH)*

3.1.1.9 *Hydrochloric Acid (HCl)*

3.1.1.10 *Sulfuric Acid (H₂SO₄)*

3.1.1.11 *Silver Nitrate (AgNO₃)*

3.1.1.12 *Sodium Borohydride (NaBH₄)*

3.1.1.13 *Ammonia (NH₃)*

3.1.1.14 *Hydrogen Peroxide (H₂O₂)*

3.1.1.15 *Ethanol (EtOH)*

3.2 Equipments

3.2.1 Ultraviolet -Visible Spectrophotometer (UV-VIS)

3.2.2 Fourier Transform Infrared Spectroscopy (FTIR)

3.2.3 Scanning Electron Microscope (SEM)

3.2.4 Atomic Force Microscopy (AFM)

3.2.5 Four Point Probe Resistivity Measurements

3.2.6 X-Ray Diffraction (XRD)

3.2.7 pH Meter

3.3 Methodology

3.3.1 Glass Slides Cleaning

Glass slides are immersed in hot ammonia that contains DI water: $\text{NH}_4\text{OH}:\text{H}_2\text{O}_2$ (5:1:1 ml) for 20 minutes then glass slides are rinsed with EtOH and dried.

3.3.2 Apply Primer on the Glass Slides

5 layers of PDADMAC/PSS are coated on the glass slides. PDADMAC solution contains 10 mM PDADMAC and 1 M NaCl. PSS solution contains 10 mM PSS and 1 M NaCl. The dipping time is 1 min/layer.

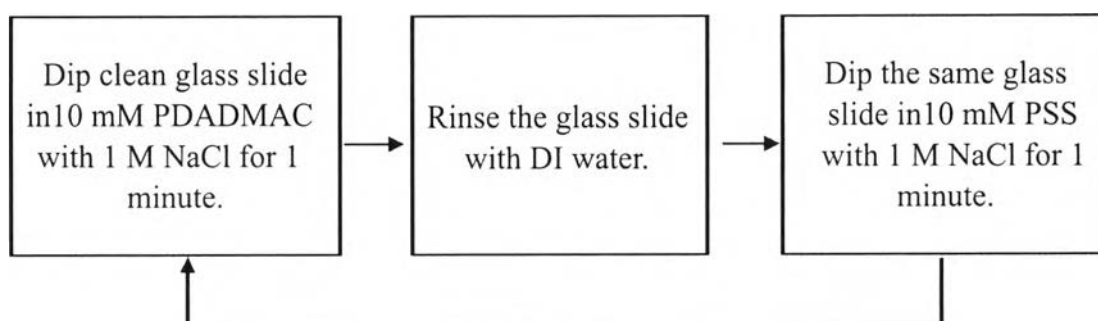


Figure 3.1 Primer applying diagram.

3.3.3 PANI-PSS Synthesis

3.3.3.1 *Interfacial Polymerization*

Adding PSS with different concentration as a template and 2 mM APS with 0.1 M H₂SO₄ in upper aqueous phase and 10 mM aniline monomer in CHCl₃ lower phase then the reaction was kept in 4 °C for 24 h.

3.3.3.2 *Bulk Polymerization*

Adding PSS with different concentration as a template, 2 mM APS and 10 mM aniline monomer with 0.1 M H₂SO₄ together then the reaction was kept in 4 °C for 24 h.

3.3.4 Monolayer Film Assembly

Dipping the glass slide with primer in PANI-PSS solutions from Bulk and interfacial polymerization and vary pH of PANI-PSS solutions for 2 h then rinse with pH adjusted DI water.

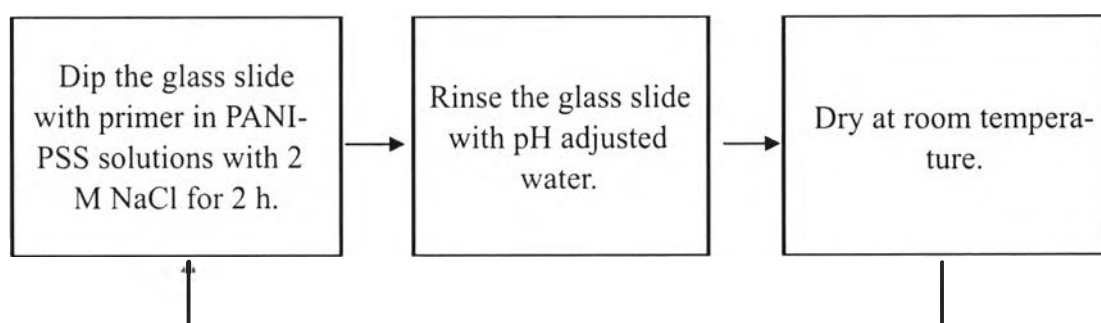


Figure 3.2 Monolayer film assembly diagram.

3.3.5 Layer-by-Layer Film Assembly

Dipping the glass slide with primer in best PANI-PSS solution that contains 2 M NaCl at best pH condition for 10 min then rinse the glass slide with pH adjusted DI water. Dip the same glass slide in 10 mM PDADMAC that contains 0.2 M NaCl at best pH condition for 5 min and rinse the glass slide with pH adjusted DI water then continually dip in PANI-PSS and PDADMAC alternately and respectively.

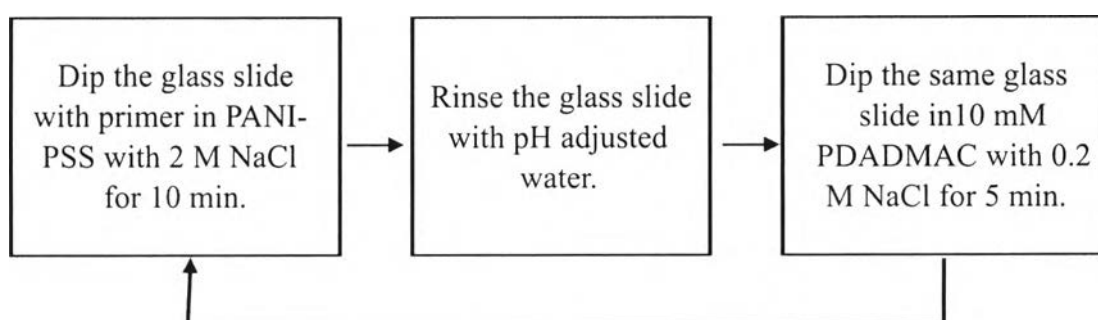


Figure 3.3 Layer-by-layer film assembly diagram.

3.3.6 Preparation of In Situ Ag Nanoparticles in PANI Layer-by-layer

Film

Immerse PANI film into 20 mM of AgNO_3 solution for 20 min. rinse the film with stirred DI water then immersed the film into 1 mM NaBH_4 for 5 min. to reduce Ag^+ to Ag^0 .

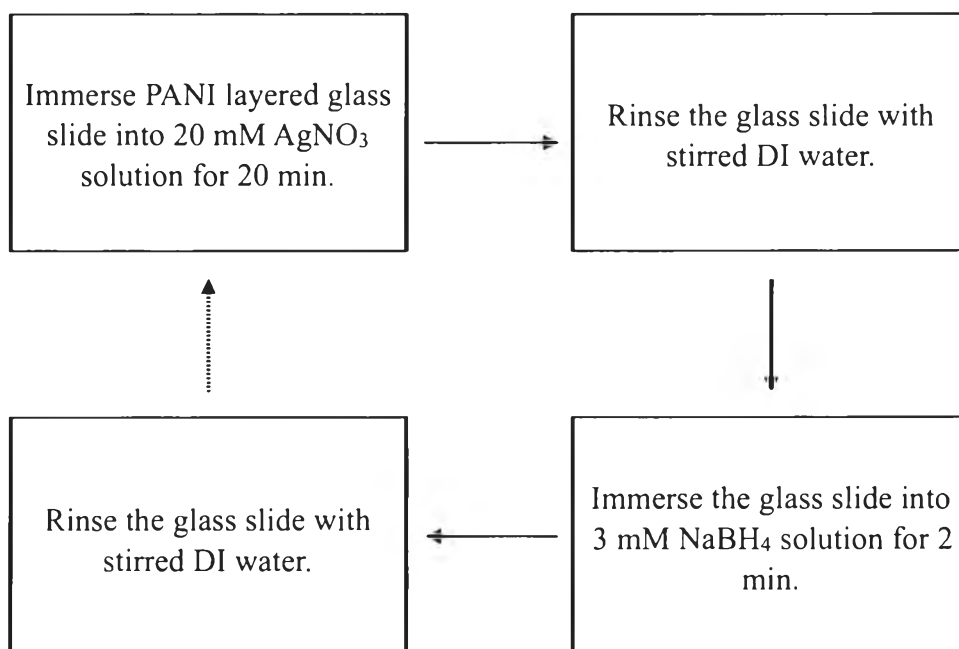


Figure 3.4 Preparation of in situ Ag nanoparticles in PANI Layer-by-layer film diagram.

3.4 Characterizations

3.4.1 UV-VIS is used to measure a optical property of the film.

3.4.2 FTIR is used to determine functional groups of the film.

3.4.3 AFM is used to measure thickness and surface morphology of the film.

3.4.4 4-point Probe is used to measure electrical properties of the film.

3.4.5 SEM is used to determine surface morphology of the film.

3.4.6 XRD is used to characterize silver on the film.

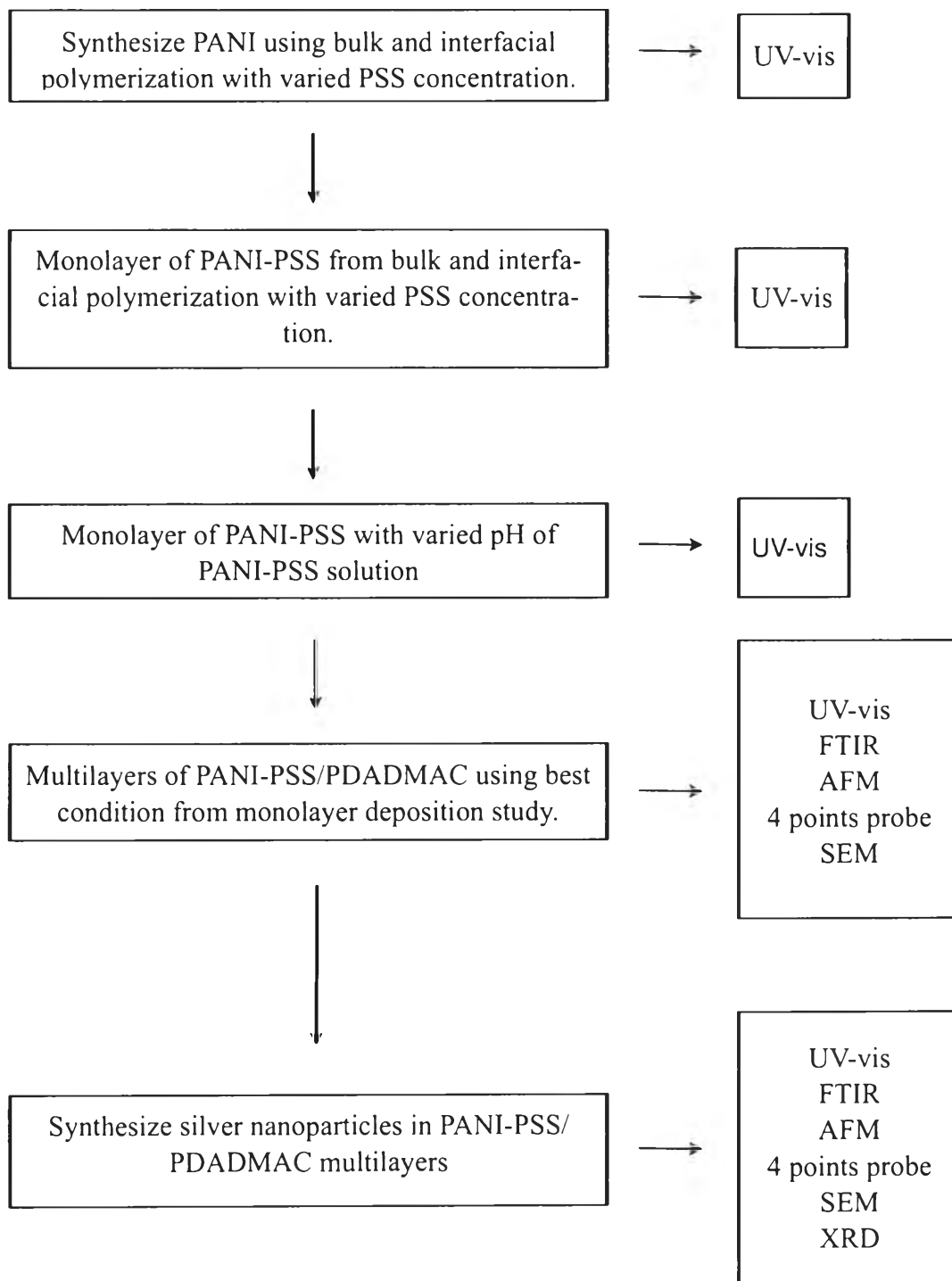


Figure 3.5 Methodology diagram.