CHAPTER 2

EXPERIMENTAL PROCEDURES

2.1 Chemical reagents, equipments and instruments

2.1.1 Chemical reagents (All chemical reagents were analytical grade)

1) Zinc metal plate**,** 15 mm ×15 mm ×0.25 mm
2) Copper metal plate**,** 15 mm ×15 mm ×0.25 mm
3) Sodium hydroxide, NaOH, MW = 40.00, minimum assay 97.0% , RCI Lab-scan, Thailand
4) Lithium hydroxide**,** LiOH, MW = 41.96, minimum assay 98.0%, Carlo Erba reagent, USA.
5) Ammonium hydroxide**,** NH_4OH, MW = 25.03, minimum assay 25.0%, QREC, Thailand
6) Ethanol**,** C_2H_5OH**,** MW = 46.07, 95%, Merck, Germany
7) Sodium chloride**,** NaCl, MW = 58.44, 99.5%, Fisher Scientific, UK
8) Yeast extract, Lab-scan, Thailand
9) Peptone, Scharlau, Germany
10) Agar, Scharlau, Germany
11) Deionized water
12) Bacterial, Escherichia coli and Staphylococcus aureus

2.1.2 Equipments

1) Analytical balance, Model PA-SERIES, Pioneer , Ohaus, USA
2) Oven, Model UE-400, Memmert, Germany
3) Hotplate and magnetic stirrer, Fisher Scienctific, U.S.A.
4) Ultrasonic bath, Bandelin, Sonorex, Germany
5) X-ray Diffractometer (XRD) model D-500, Siemens, Germany
6) Scanning Electron Microscope (SEM) model JEM-6335, JEOL, Japan
7) Transmission Electron Microscope (TEM) model JEM-2010, JEOL, Japan
8) Raman Spectrophotometer, Model T64000 HORIBA, Germany
9) Photoluminescence (PL) Spectrometer, Model Perkin-Elmer LS50B, Germany

Figure 2.1 Equipments for used

2.2 Experimental procedure

2.2.1 Synthesis of zinc oxide with NaOH by a hydrothermal method

The ZnO nanorods were fabricated by the following procedure. Firstly, the zinc metal plates (15 mm ×15 mm ×0.25 mm) were carefully cleaned with ethanol and deionized water, respectively, in an ultrasonic bath. Second, adjust pH of DI water (20.0 ml) to 9-12 by 3M NaOH. Then the cleaned Zn plate was putted in to precursor solutions and transferred into a Teflon-lined stainless steel autoclave with a capacity of 50.0 ml. Finally, sealed and heated the Teflon lined stainless steel autoclave at 80-120^{o}C for 24 h in an electric laboratory oven. Then the autoclave was cooled to room temperature. After thoroughly washed by deionized water several times and dried at 70^{o}C under air atmosphere, and kept for further characterization.

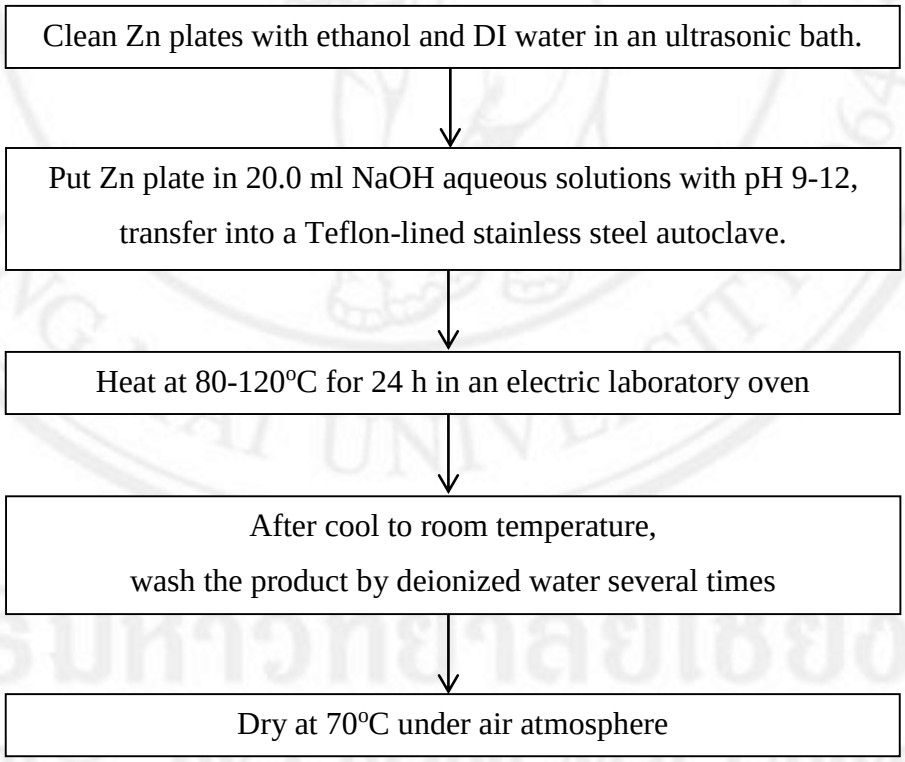

Figure 2.2 Schematic diagram used for synthesized zinc oxide on Zn plate with NaOH by hydrothermal process.

2.2.2. Synthesis of zinc oxide with LiOH by a hydrothermal method

The ZnO nanorods were fabricated by the following procedure. The zinc foils (15 mm ×15 mm ×0.25 mm) were carefully cleaned with ethanol and deionized water, respectively, in an ultrasonic bath. The Zn foil was putted in 0-0.4 g LiOH, 20.0 ml aqueous solutions and transferred into a Teflon-lined stainless steel autoclave with a capacity of 50.0 ml. Finally, sealed and heated the Teflon lined stainless steel autoclave at 120^{o}C for 1-24 h in an electric laboratory oven. Then the autoclave was cooled to room temperature. After thoroughly washed by deionized water several times and dried at 70^{o}C under air atmosphere, and kept for further characterization.

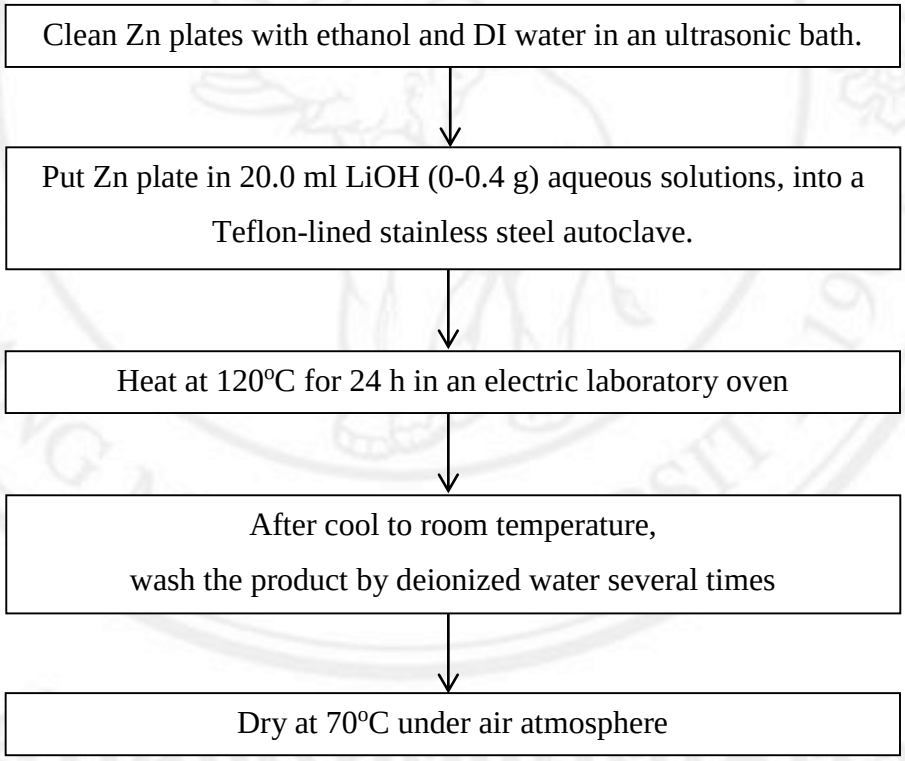

Figure 2.3 Schematic diagram used for synthesized zinc oxide on Zn plate with LiOH by hydrothermal process.

2.2.3 Synthesis of zinc oxide with NH_4OH by a hydrothermal method

The ZnO nanorods were fabricated by the following procedure. The zinc plates (15 mm ×15 mm ×0.25 mm) were carefully cleaned with ethanol and deionized water, respectively, in an ultrasonic bath. The Zn plate was putted in NH_4OH solutions with pH 8-10 and transferred into a Teflon-lined stainless steel autoclave with a capacity of 50 ml. Finally, sealed and heated the Teflon lined stainless steel autoclave at 120^oC for 24 h in an electric laboratory oven. Then the autoclave was cooled to room temperature. After thoroughly washed by deionized water several times and dried at 70^oC under air atmosphere, and kept for further characterization.

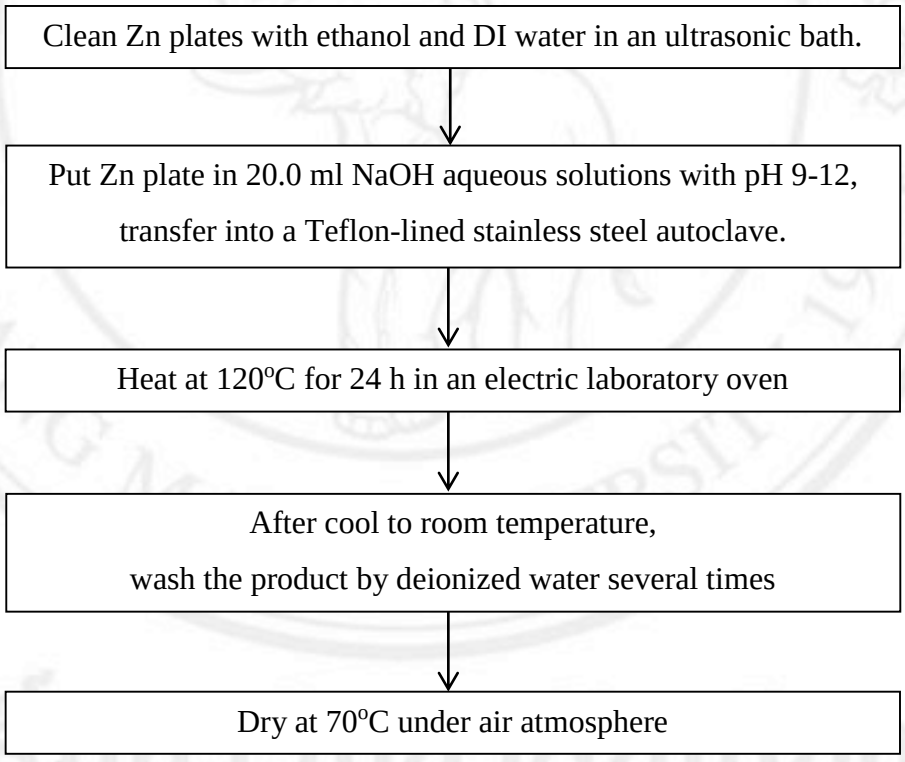

Figure 2.4 Schematic diagram used for synthesized zinc oxide on Zn plate with NH_4OH by hydrothermal process.

2.2.4 Synthesis of copper oxide by solution chemistry at room temperature

All reagents in this experiment were of analytical grade and were used without further purification. CuO thin films were grown on Cu foils by the following sequence. A large piece of Cu foil 0.25 mm thick was cut into several 15 × 15 mm^2 foil samples, which were carefully cleaned with deionized water and absolute alcohol in an ultrasonic bath to remove surface impurities and oxide layers. They were then separately immersed in 10 ml NaOH aqueous solutions with pH 13 at room temperature for 3–21 days. During immersion, the color of the solutions changed from clear to light blue, which was attributed to dissolution of the copper foils. At the conclusion of the process, the copper foils were thoroughly washed several times with deionized water and dried at 70^{o}C in an electric oven for 12 h.

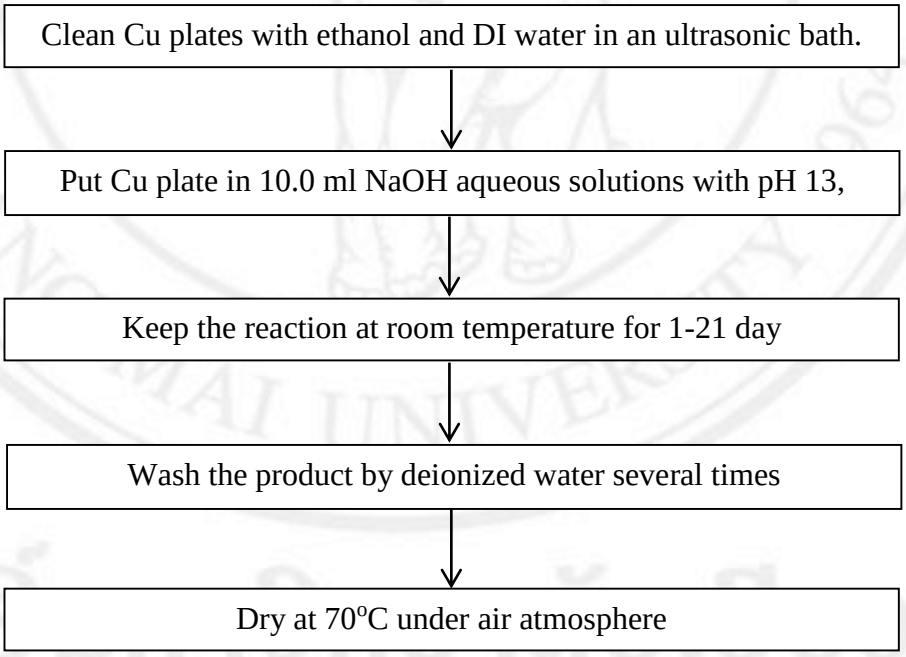

Figure 2.5 Schematic diagram used for synthesized copper oxide on Cu plate with NaOH by solution chemistry method.

2.3 Characterizations

2.3.1 X-ray Diffraction (XRD)

Crystallinity and phase purity of the products were analyzed by using X-ray diffraction (XRD) with Cu K_α radiation (λ = 1.5418 Å) operating at 20 kV-15mA, at a scanning rate of 5°/min in the 2θ range of 10°- 60°. The products were interpreted by Philips X'pert Highscore Computer Software (search-match program) on the database of JCPDS software.

Figure 2.6 X-ray Diffractometer

2.3.2 Raman Scattering Spectroscopy

The Raman Scattering technique is a vibrational molecular spectroscopy which derives from an inelastic light scattering process. With Raman Spectroscopy, a laser photon is scattered by a sample molecule and loses (or gains) energy during the process. The amount of energy lost is seen as a change in energy (wavelength) of the irradiating photon. This energy loss is characteristic for a particular bond in the molecule. Raman can best be thought of as producing a precise spectral fingerprint, unique to a molecule or indeed and individual molecular structure. In this respect it is similar to the more commonly found FT-IR Spectroscopy. However, unlike FT-IR, there are a distinct number of advantages when using Raman Spectroscopy.

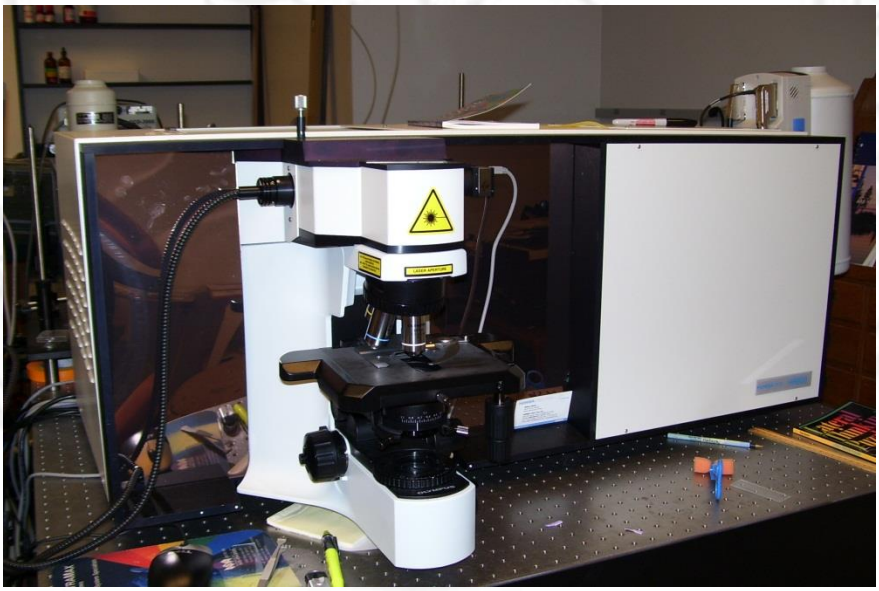

Figure 2.7 Raman Scattering Spectroscopy

2.3.3 Field Emission Scanning Electron Microscopy (FE-SEM)

The morphology of the products were analyzed by Field Emission Scanning Electron Microscope, JEOL model SEM, JSM-6335F operating at 15 kV as accelerating voltage. The products were dispersed in absolute ethanol using an ultrasonic bath. The dispersed samples were dropped on conductive gold tapes which were attached to the SEM stubs. The stubs were then coated with gold particles by sputtering under argon atmosphere in order to increase conductivity of the samples.

Figure 2.8 Field Emission Scanning Electron Microscope

2.3.4 Transmission Electron Microscopy (TEM)

The morphology and structure of the products were characterized by Transmission Electron Microscope, JEOL model JEM-2010 operating at 20 kV. The samples for TEM analysis were prepared by dispersing their small amount in absolute ethanol and put a drop of the solution onto copper grids coated with holey carbon films and letting the ethanol evaporate solely in ambient atmosphere.

Figure 2.9 Transmission Electron Microscope

2.3.5 Photoluminescence (PL) Spectroscopy

The Photoluminescence properties of the products were investigated by Perkin Elmer Luminescence spectrometer LS50B at room temperature using an excitation wavelength of 250 nm. The appropriate amount of powder samples were dispersed in absolute ethanol using ultrasonic bath, and tested for emission.

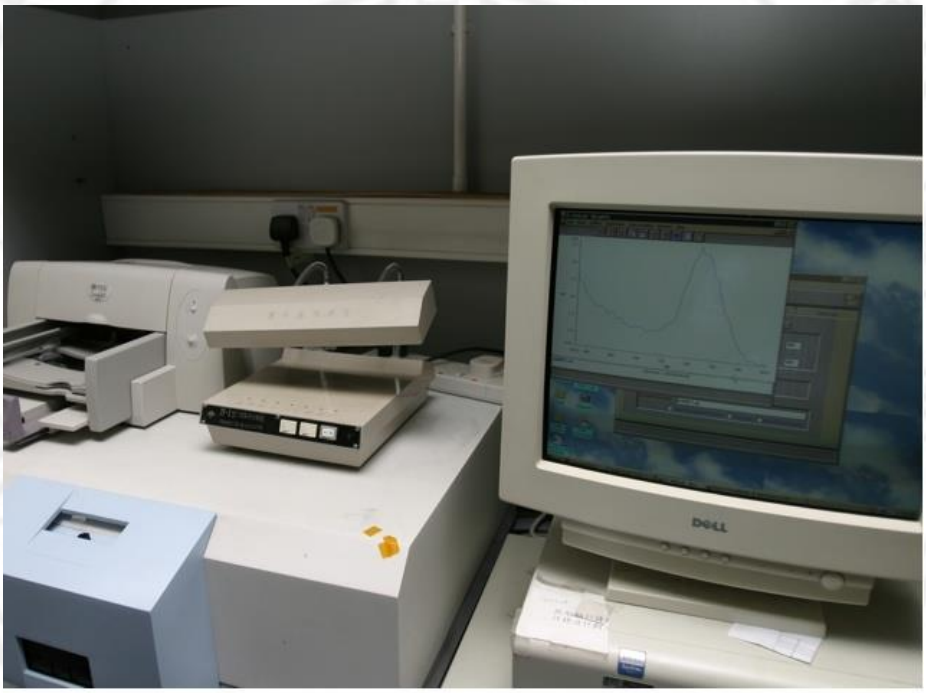

Figure 2.10 Photoluminescence Spectrophotometer

2.4 Antibacterial Activity

In this research, two kinds of bacteria – Gram-positive (Staphylococcus aureus) and Gram-negative (Escherichia coli) – were used to study the antibacterial activity of CuO thin films by an inhibition zone method. Both strains were transferred into flasks containing nutrient broth (NB) with an initial optical density (OD) of 0.1 at a 660 nm orange wavelength; and the bacteria were cultured at 37^{o}C in aerated conditions until reaching an OD of 0.3. Agar was then added to the flasks. Modified agar diffusion assays (testing disks) were used to determine the antibacterial activity of CuO thin films after 24 h incubation at 37^{o}C by the formation of clear zones around the zinc/copper foils.

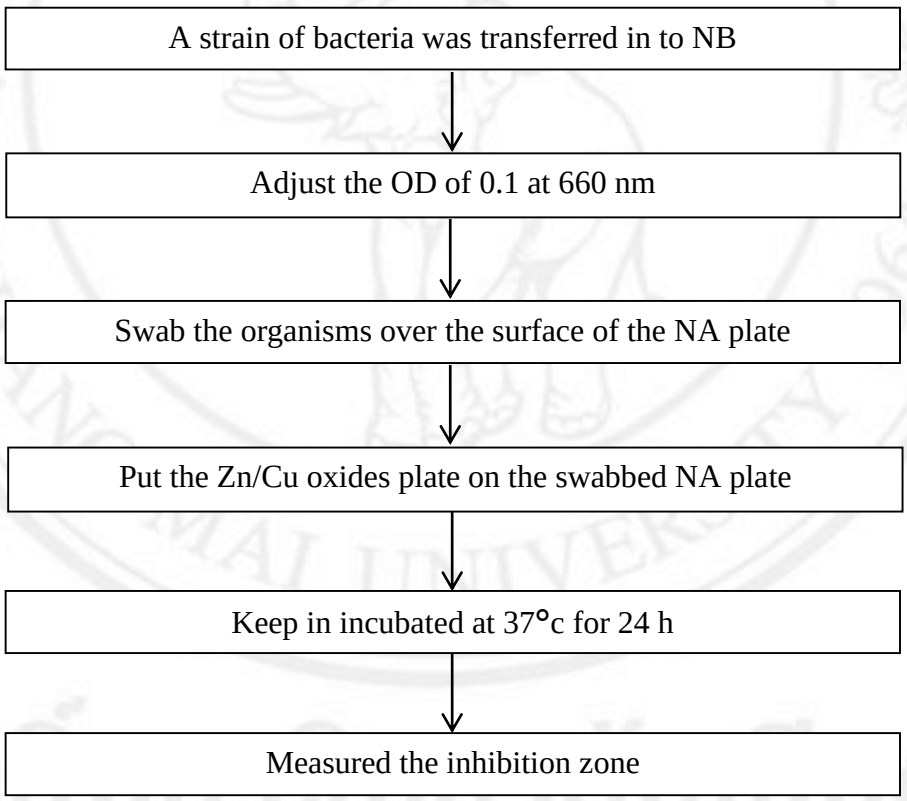

Figure 2.11 Schematic diagram used for test the antibacterial activity.