CHAPTER 2

EXPERIMENTAL PROCEDURE

2.1 Chemical reagents and equipments

2.1.1 Chemical reagents

1) Zinc nitrate hexahydrate, $Zn(NO_3)_2.6H_2O$, M_W = 297.47, 99%, Carlo Erba, Italy

2) Sodium hydroxide, NaOH, M_W = 40.00, 99%, Lab scan

3) Polyethylene glycol 200, PEG200, Mr = 190-200, Fluka

4) Polyethylene glycol 400, PEG400, Mr = 380-420, Fluka

5) Polyethylene glycol 600, PEG600, Mr = 570-630, Fluka

6) Polyethylene glycol 6000, PEG6000, Mr = 5000-7000, Fluka

7) Polyethylene glycol 10000, PEG10000, Mr = 8500-11500, Fluka

8) Polyethylene glycol 20000, PEG20000, Mr = 16000-24000, Fluka

9) Deionized water

10) Ethanol, C_2H_5OH, 95%, Merck, Germany

11) Absolute ethanol, C_2H_5OH, 99.0-100.0% AR, Merck, Germany

2.1.2 Equipments

1) Hotplate and magnetic stirrer, model 502P-2, PMC Industries, Inc., San Diego, U.S.A.

2) Analytical balance, Model BP-210S, Satorius AG. Goettingen, Germany

3) Oven, Model UE-400, Memmert, Germany

4) Microwave Oven, Electrolux model 2820S, China

5) Ultrasonic bath, Bandelin Sonorex, Germany

6) X-ray Diffractometer, model D500/501, Siemens, Germany

7) Fourier Transform Infrared Spectrophotometer, FT-IR 510 Nicolet, U.S.A.

8) Raman spectroscopy, model T64000 JY, Horiba Jobin Yvon, France

9) Perkin Elmer Luminescence spectrometer, model LS50B

10) Scanning Electron Microscope, model JSM-6335, JEOL, Japan

11) Transmission Electron Microscope, model JEM-2010, JEOL, Japan

12) Agate mortar

2.2 Synthesis method

2.2.1 Synthesis of zinc oxide via a microwave radiation method

2.2.1.1 Effect of liquid media

$Zn(NO_3)_2.6H_2O$ and NaOH were dissolved in different liquid media (H_2O, PEG200, PEG400 and PEG600). The mixture was vigorously stirred for 15 min. The reactions were proceeded in a microwave oven for 30 min. The precipitates were separated by filtering, washed with distilled water and 95 % ethanol, and dried in an oven at 70 ^oC for 24 h.

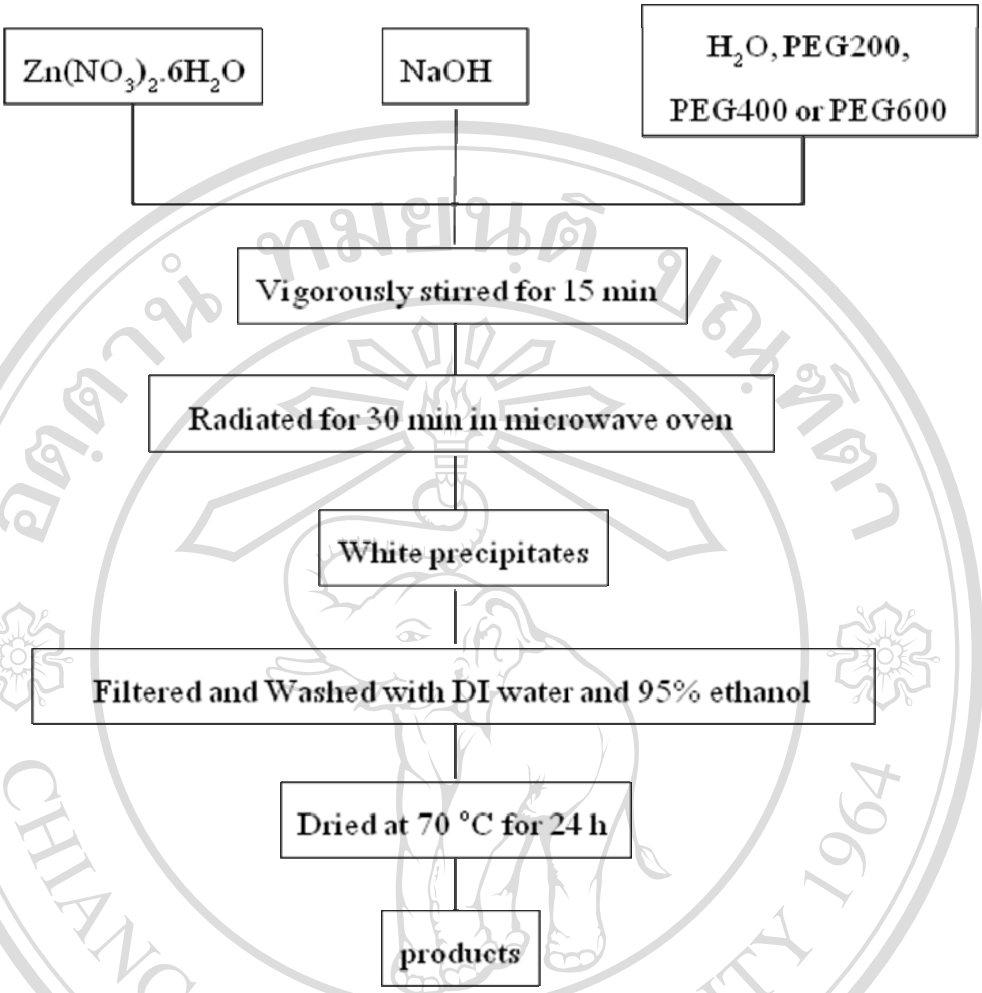

Figure 2.1 Schematic diagram used for preparing ZnO using H_2O, PEG200, PEG400 and PEG600 as liquid media.

2.2.1.2 Effect of surfactants

$Zn(NO_3)_2.6H_2O$, NaOH and different surfactants (PEG6000, PEG10000, PEG20000) were dissolved in H_2O. The mixture was vigorously stirred for 15 min. The reactions proceeded in a microwave oven for 30 min. The precipitates were separated by filtering, washed with distilled water and 95 % ethanol, and dried in an oven at 70 ^{o}C for 24 h. Conditions of various water volume

2.2.1.3 Effect of the amount of PEG20000

$Zn(NO_3)_2.6H_2O$, NaOH and different amount of PEG20000 (6 g, 10 g and 20 g) were dissolved in H_2O. The mixture was vigorously stirred for 15 min. The reactions were proceeded in a microwave oven for 30 min. The precipitates were separated by filtering, washed with distilled water and 95 % ethanol, and dried in an oven at 70 ^oC for 24 h.

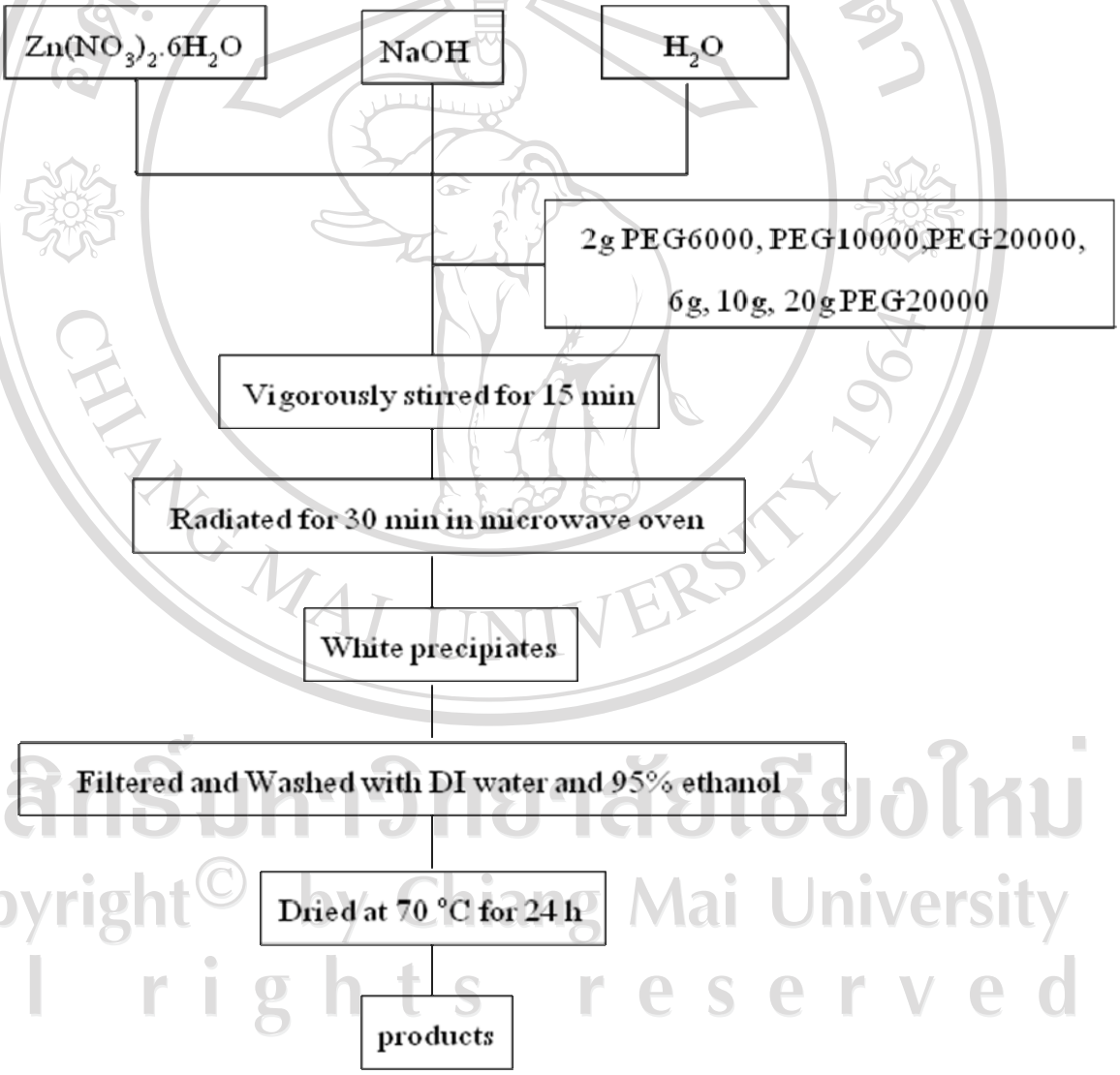

Figure 2.2 Schematic diagram used for preparing ZnO using 2 g PEG6000, PEG10000, PEG20000, 6 g, 10 g and 20 g PEG20000 as surfactant.

2.2.2 Synthesis of zinc oxide via ultrasonic radiation method

2.2.2.1 Effect of $Zn(NO_3)_2$ to NaOH ratios

$Zn(NO_3)_2.6H_2O$ and 25, 50, 75, 100 mmol NaOH were dissolved in H_2O. The mixture was vigorously stirred for 15 min. The reactions were proceeded in ultrasonic bath at 80 ^oC for 5 h. The precipitates were separated by filtering, washed with distilled water and 95 % ethanol, and dried in an oven at 70 ^oC for 24 h.

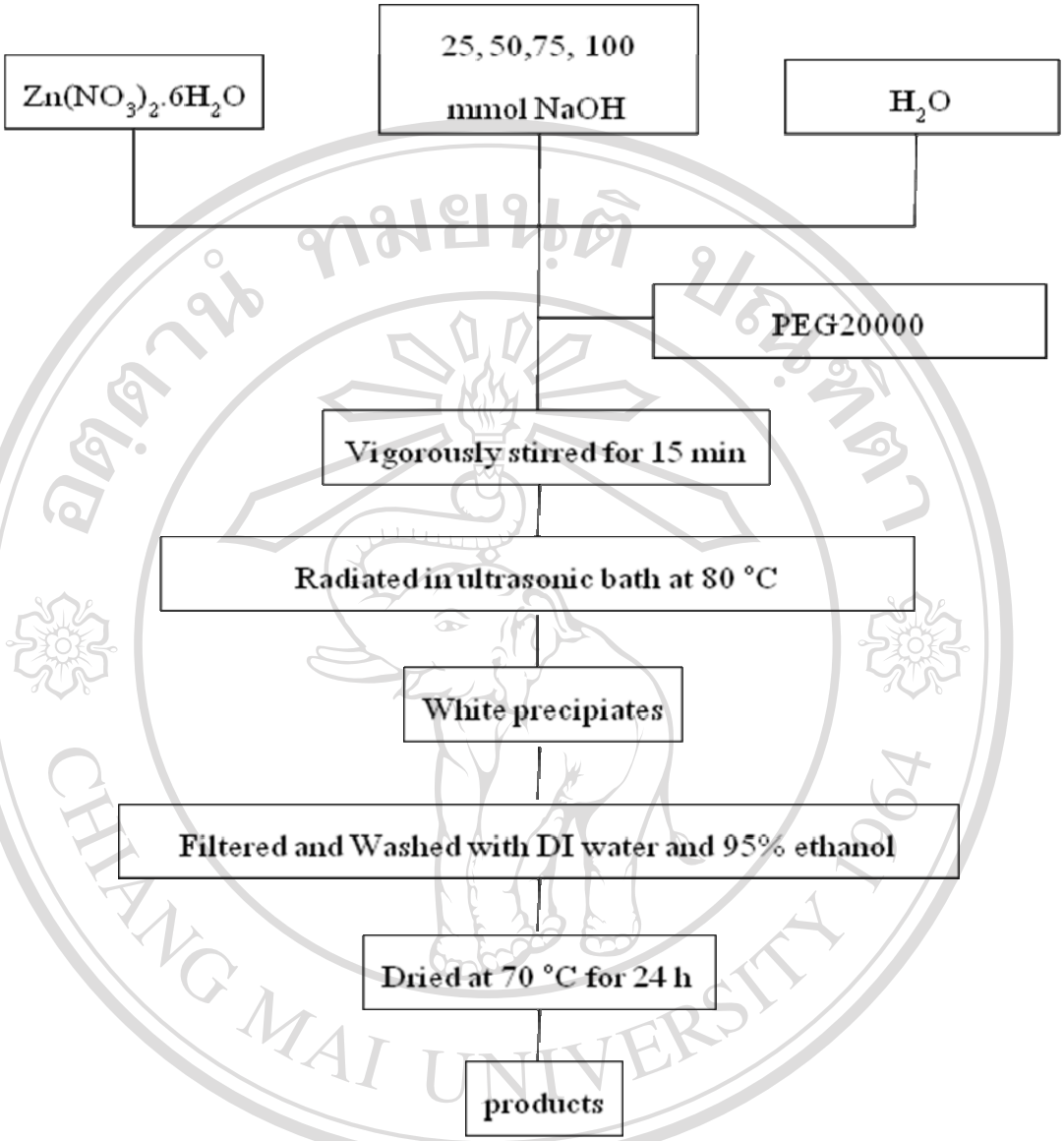

Figure 2.3 Schematic diagram used for preparing ZnO using different $Zn(NO_3)_2$ to NaOH ratios.

2.2.2.2 Effect of reaction time

$Zn(NO_3)_2.6H_2O$ and NaOH were dissolved in H_2O. The mixture was vigorously stirred for 15 min. The reactions were proceeded in an ultrasonic bath at 80 ^oC for 1, 3 and 5 h. The precipitates were separated by filtering, washed with distilled water and 95 % ethanol, and dried in an oven at 70 ^oC for 24 h.

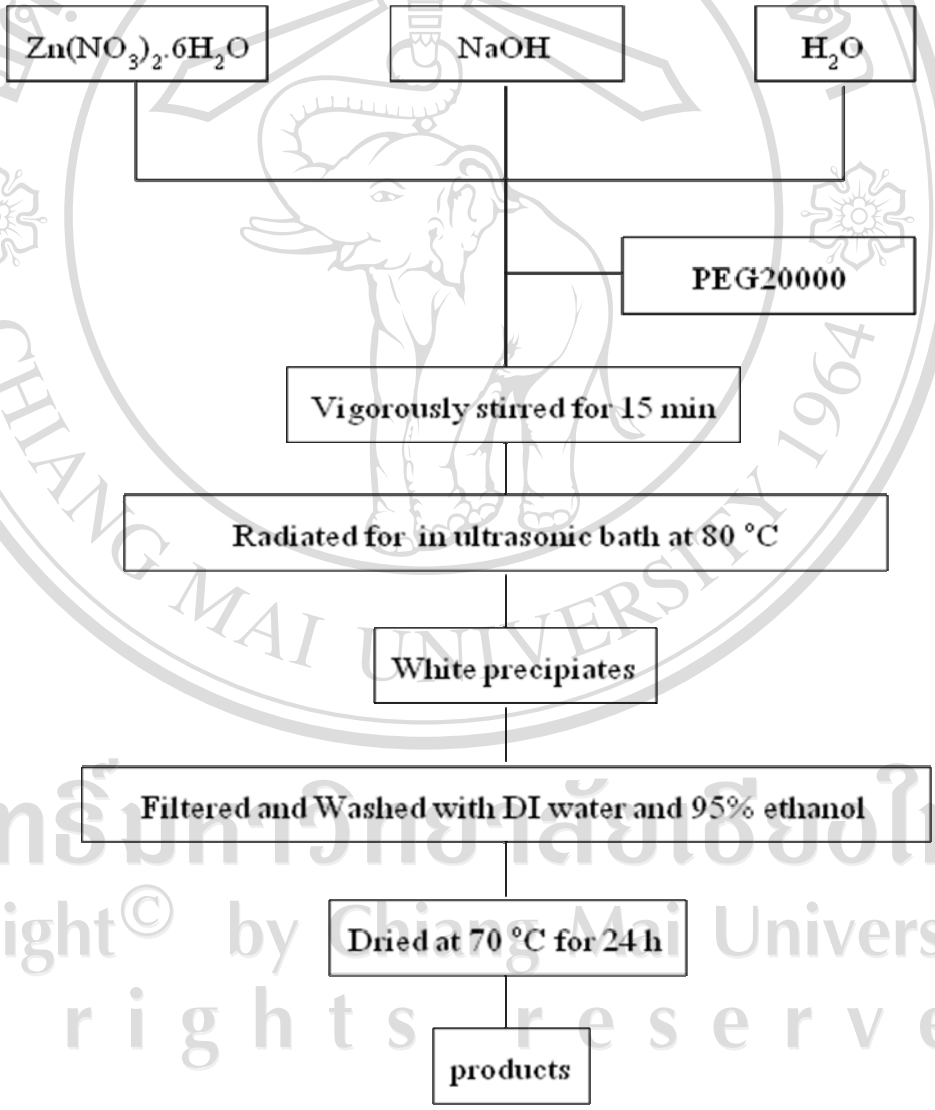

Figure 2.4 Schematic diagram used for preparing ZnO using different prolonged times.

2.2.2.3 Effect of the amount of PEG20000

$Zn(NO_3)_2.6H_2O$, NaOH and different amount of PEG20000 were dissolved in H_2O. The mixture was vigorously stirred for 15 min. The reactions were proceeded in ultrasonic bath at 80 ^oC for 5 h. The precipitates were separated by filtering, washed with distilled water and 95 % ethanol, and dried in an oven at 70 ^oC for 24 h.

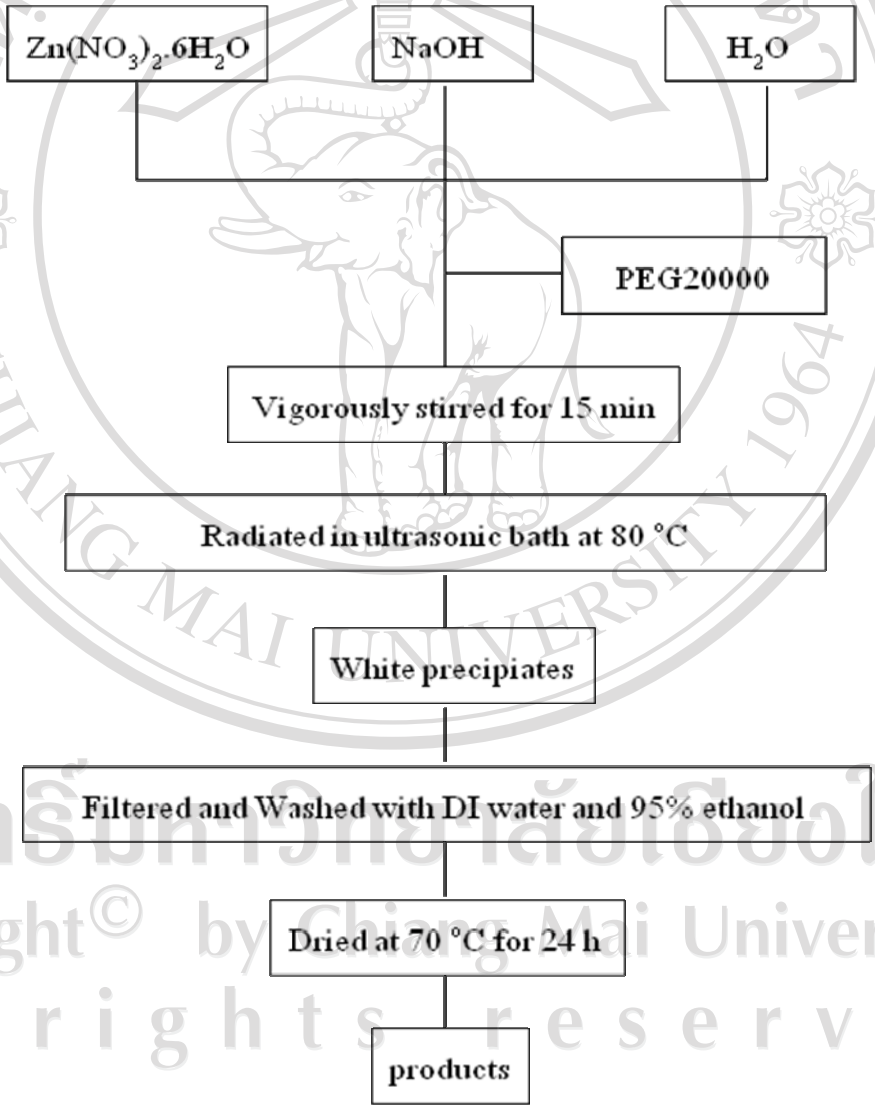

Figure 2.5 Schematic diagram used for preparing ZnO using different amount of PEG20000 as surfactant.

2.3 Charecterization

2.3.1 X-ray Diffraction (XRD)

The crystalline structure of ZnO powders were characterized by X-ray diffractometry.

The powder samples were finely grinded in agate mortar and then put on in sample holder.

The crystallinity and phase purity of the products were analyzed by using X-ray diffractometry (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 identification samples were assisted 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 Scanning Electron Microscopy (SEM)

The morphologies of ZnO were investigated using scanning electron microscopy.

The powder samples were dispersed in absolute ethanol using an ultrasonic bath. The dispersed sample was dropped on conductive gold tape which attached to the SEM stub. The stub was then coated with gold particle in order to increase conductivity under argon atmosphere by plasma sputtering.

The morphology and particle sizes of as-obtained samples were determined by a Field emission-scanning electron microscope (SEM, JSM-6335F) operated at 15 kV accelerating voltage.

Figure 2.7 Scanning electron microscope

2.3.3 Transmission Electron Microscopy (TEM)

The morphology and electron diffraction of the samples were studied using transmission electron microscopy.

The TEM samples were deposited on thin amorphous carbon films supported by cupper grid from ultrasonically processed absolute ethanol solution of the sample powder.

The particle size and morphology was also observed by transmission electron microscope (TEM, JEOL JEM-2010) operating at 20 kV. The samples for TEM analysis were prepared by dispersing small amount of the powder in absolute ethanol and placing a drop of the solution onto a copper grid coated with holey carbon film and letting the ethanol evaporate slowly in air.

Figure 2.8 Transmission electron microscope

2.3.4 Photoluminescence Spectroscopy

Optical properties of the products were analyzed by photoluminescence spectroscopy

The appropriated amount of powder samples were dispersed in absolute ethanol using ultrasonic bath. Photoluminescence (PL) spectrometer was operated using a 210 nm exiting wavelength for CdS and 205 nm for CdSe.

The luminescence emission spectra of the samples were investigated using Perkin Elmer Luminescence spectrometer LS50B at room temperature.

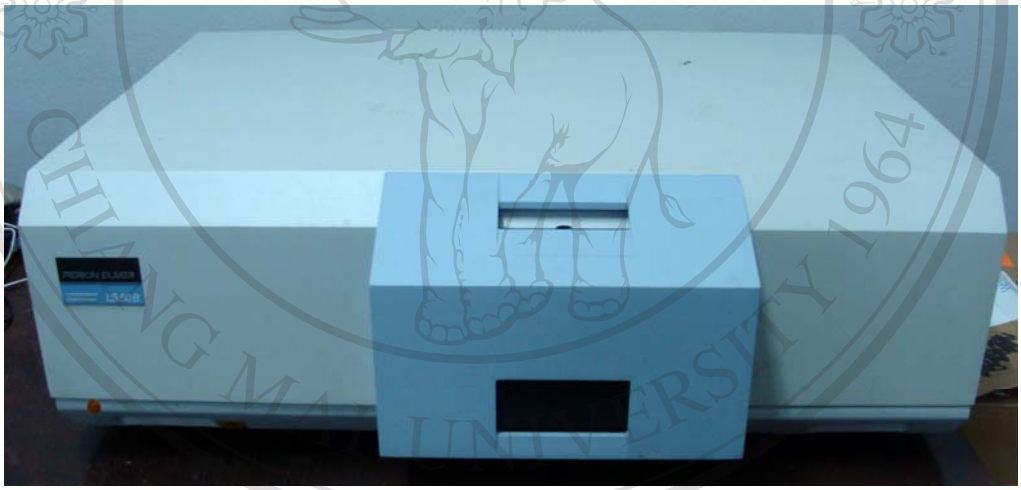

Figure 2.9 Luminescence spectrometer