# CHAPTER 2

# EXPERIMENTAL PROCEDURE

## 2.1 Chemical reagents, equipments and instruments

### 2.1.1 Chemical reagents

1) Lanthanum (III) Chloride heptahydrate ($LaCl_3 \cdot 7H_2O$), MW = 371.38 g/mol, minimum assay 99.50%, Merck
2) Cerium (III) nitrate hexahydrate ($Ce(NO_3)_3 \cdot 6H_2O$), MW = 434.22g/mol, minimum assay 99.50%, Acros organic
3) Tri-sodium phosphate ($Na_3PO_4 \cdot 12H_2O$), MW = 380.12 g/mol, Fluka
4) Nitric acid ($HNO_3$), MW = 63.09, minimum assay 65.0%, Carlo Erba
5) Ethanol ($C_2H_5OH$), MW = 46.07, minimum assay 99.8%, Merck

### 2.1.2 Equipments and instruments

1) Microwave oven, Electrolux, EMS 2820, 2.45 GHz, China
2) X-ray diffractometer (XRD) model D-500, Siemens, Germany
3) Scanning electron microscope (SEM) model JEM-6335, JEOL, Japan

4) Transmission electron microscope (TEM) model JEM-2010, JEOL, Japan
5) Fourier transform infrared spectrophotometer (FTIR) model TENSOR 27, Bruker Optrik, Germany
6) Photoluminescence (PL) spectrometer model Perkin-Elmer LS50B
7) Ultraviolet- Visible near-Infrared Spectroscopy (UV-vis NIR) model Perkin Elmer Lambda 25, U.S.A.
8) Hotplate and magnetic stirrer, model 502P-2, PMC Industries, Inc., San Diego, U.S.A.
9) Analytical balance, Model BP-210S, Satorius AG. Goettingen, Germany
10) Oven, Model UE-400, Memmert, Germany

## 2.2 Experimental procedure

### 2.2.1 Synthesis of $LaPO_4$ (and $CePO_4$) using microwave irradiation method

Each 0.003 mole of $LaCl_3 \cdot 7H_2O$ (or $Ce(NO_3)_3 \cdot 7H_2O$) and $Na_3PO_4 \cdot 12H_2O$ were dissolved in 80 ml deionized water with 15 min vigorously stirring, and followed by 37% $HNO_3$ adding to the precursor solutions for adjusting the pH to be 1 - 6. Each of these solutions was continuously heated by a microwave oven at 180 W for 60 min, then naturally cooled down to room temperature. The white products were filtered, washed by deionized (DI) water and 95% ethanol several times, and dried at 80 $^{o}$C for 12 h, and further characterized and analyzed.

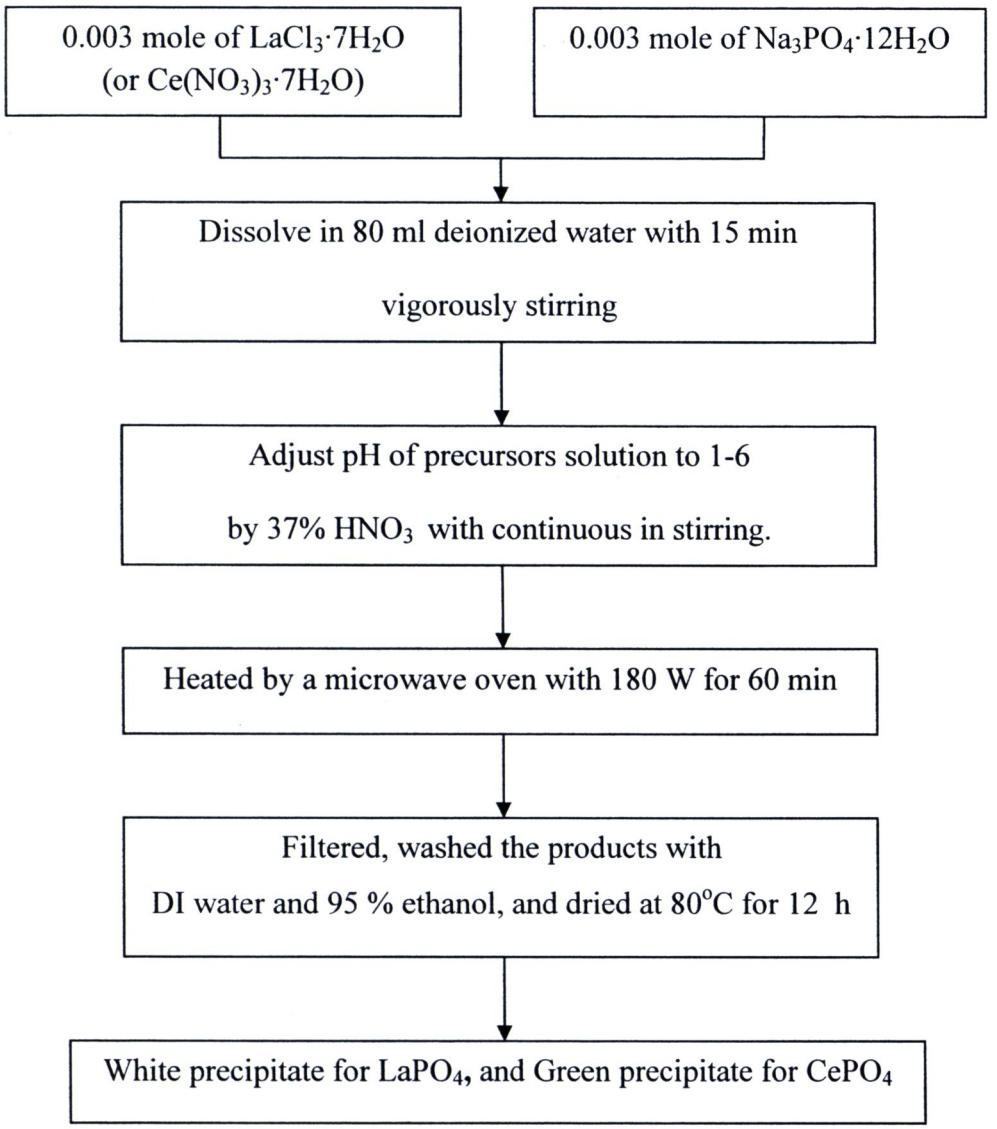


Figure 2.1 Schematic diagram used for preparation of $LaPO_4$ (and $CePO_4$).

## 2.3 Characterization

### 2.3.1 X-ray diffraction (XRD)

Crystallinity and phase purity of the products were analyzed by using X-ray diffraction (XRD) with Cu $K_\alpha$ radiation ($\lambda$ = 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 2.2 X-ray diffractometer.

### 2.3.2 Fourier transform infrared (FTIR) spectrometer

The functional groups of the products were analyzed by Fourier transform infrared spectroscopy (FTIR, BRUKER TENSOR27). The samples were diluted 40 times by KBr pellet and operated in the range 400–4000 $cm^{-1}$ for FTIR analysis.

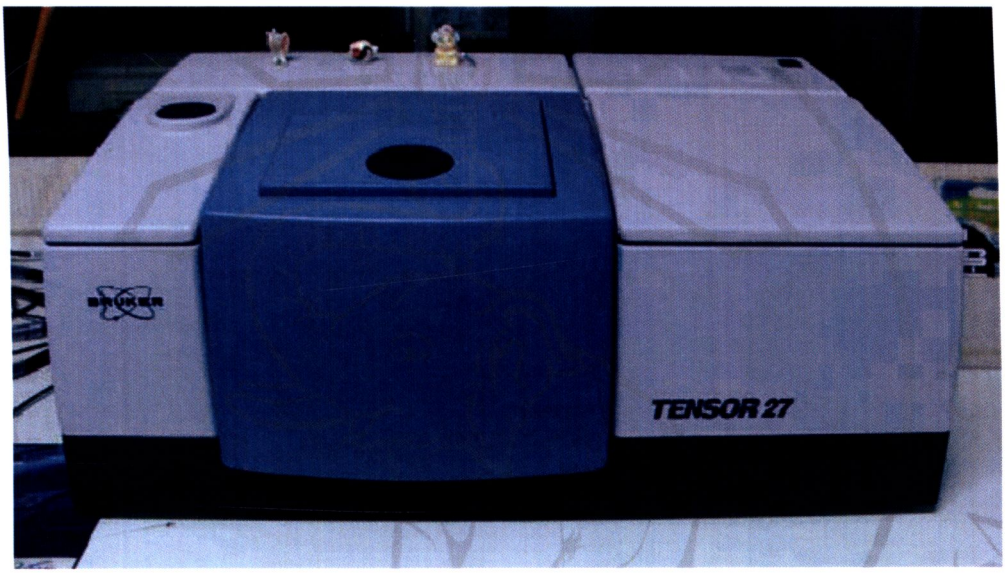


Figure 2.3 Fourier transform infrared spectrometer.

### 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 a accelerating voltage. The products were dispersed in absolute ethanol using an ultrasonic bath. The dispersed samples were dropped on conductive gold tape 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 2.4 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 2.5 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 2.6 Photoluminescence Spectrophotometer.

### 2.3.6 Ultraviolet- Visible Near-Infrared Spectroscopy (UV-vis NIR)

The optical properties of the products were analyses by Perkin Elmer Lambda 25 UV-vis spectrometer by at room temperature. The appropriate amount of powder samples were dissolve in absolute ethanol using ultrasonic bath, and tested for optical properties.

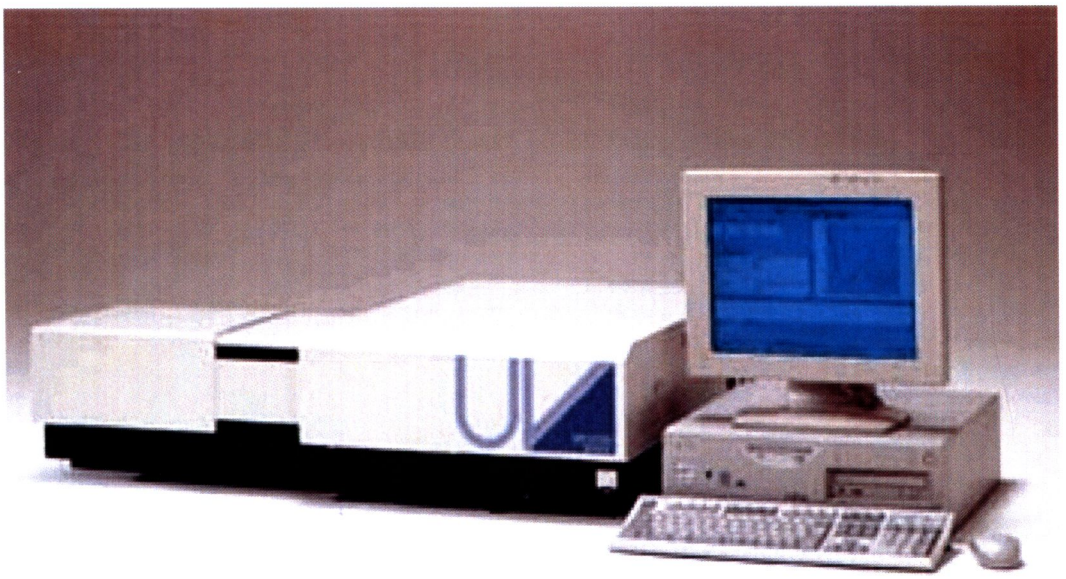

Figure 2.7 Ultraviolet- Visible Near-Infrared Spectrometer.