CHAPTER IV

EXPERIMENTS AND ANALYTICAL TECNIQUES

This chapter is donated for explaining the preparation of the experiments and analytical techniques. As mentioned above, synthesizing nanoparticles by using microemulsion technique is very simple and do not use any expensive equipment. In this work, the main objective is to study the effect of cosurfactants and anions on synthesizing ZnS nanoparticles in microemulsion. However, other important factor such as the molar ratio of water to surfactant (w_o), reactant concentration, and temperature are also investigated. So in this section, we will complain the main experimental procedure in three parts: **study the effect of cosurfactants, study the effect of anions and study the effect of temperature.**

4.1 Experimental

In this research, chemicals were used as follows:

1. Zinc sulfate ($ZnSO_4.7H_2O$)
2. Sodium sulfide ($Na_2S.9H_2O$)
3. Cyclohexane
4. Cosurfactant (use n-butanol, n-pentanol and n-hexanol)
5. Nonionic octyl phenyl ether (Triton x-100, x=9.5)

4.1.1 Effect of cosurfactants

All of the reactants and solvents used in our reaction system are analytical grade and used without any further purification. Triton X-100 (13.8 ml) was added into two 100 ml beakers containing 50 ml cyclohexane and stirred by magnetic stirrer. Next, the aqueous solution of $ZnSO_4$ or Na_2S was slowly dropped into these beakers and vigorously agitate. Then the cosurfactant (n-butanol, n-pentanol, or n-hexanol) was added and continuously agitated for about 15 min, then we will mix two separate microemulsion solutions together (Figure 4.1). The resulting mixture is then aged for 2 days at room temperature. Finally, one drop of the resulting solution diluted by ethanol will be dropped on a copper grid and dried at room temperature until suitable for SEM or TEM analysis.

In this section,

1. Studied the influence of the molar ratio of water to surfactant (w_o) at 7, 11, 15 and 20. In these cases we used the amount of reactant at 2.95, 4.63, 6.32 and 8.42 ml, respectively.
2. Studied the effect of reactant concentration at 0.1 and 0.05 mol/dm^3.

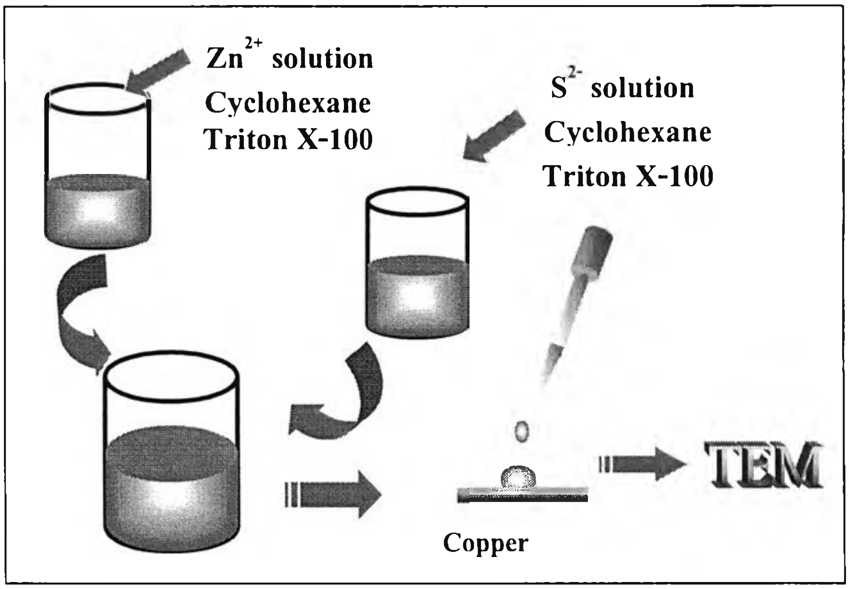

Figure 4.1 Synthesizing ZnS nanoparticles in microemulsion

Figure 4.2 the sample picture of two microemulsions in this experiment

4.1.2 Effect of anions

To investigate the effect of anions on synthesizing ZnS nanoparticles in quarternary W/O microemulsion, the main experimental method was the same. But before dropping the reactant solutions, we added 0.001 mol of anion salts (NaCl or NaBr) and used the reactant concentration only at 0.05 mol/dm^3.

4.1.3 Effect of temperature

For studying the effect of temperature, the reaction temperature in the experiment was changed by using at 10 °C and 60 °C. The concentration of reactants in this part was used only at 0.1 mol/dm^3. However, this part was out of the main scope of this thesis. So the factors such as w_o and cosurfactant types were investigated at only interesting conditions (depended on the results from part 4.2.1)

4.2 Analytical technique

The morphology and size of the samples were analyzed by SEM (JEOL JSM 5410 LV) and TEM (JEOL JEM-1230), type of element and crystalline phase of samples were analyzed by EDS and XRD respectively. Moreover DLS technique was used to investigate the size distribution.

4.2.1 Scanning electron microscopy (SEM)

The scanning electron microscopy (SEM) is extremely useful for imaging surface and subsurface microstructure[21]. SEM (JEOL JSM 5410) was used to investigate the samples in this experiment as shown in Figure 4.3. The samples were prepared by diluting them in ethanol and disperse in ultrasonic bath for 20 minutes and then drop on the copper stub and dry at room temperature until suitable to cover it with gold. The specimens were loaded into the sample chamber and used resolution at 15 or 20 kV. Then, the observation was conducted immediately with using image catcher scanner for taking the photo.

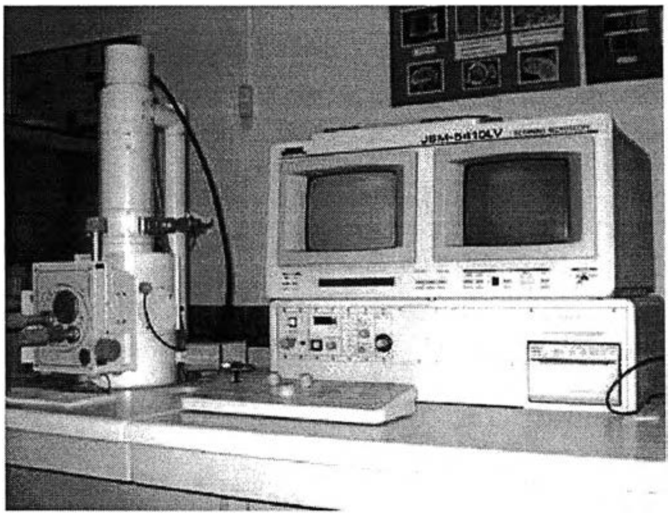

Figure 4.3. Scanning Electron Microscope (JEOL JSM 5410)

4.2.2 Transmission electron microscopy (TEM)

The conventional transmission electron microscope is a key tool for imaging the internal microstructure of ultrathin specimens[21]. In this thesis, the samples were analyzed by using TEM (JEOL JEM-1230) as shown in Figure 4.4. The samples of microemulsion were dilute by ethanol at 1:40 by volumn and then disperse by using ultrasonic bath for 15 minutes for uniform dispersion. One drop of the final solution was poured onto a grid covered with carbon thin film. The specimen was loaded into sample chamber and waiting for the vacuum condition and steady state inside the chamber for 30 minutes.

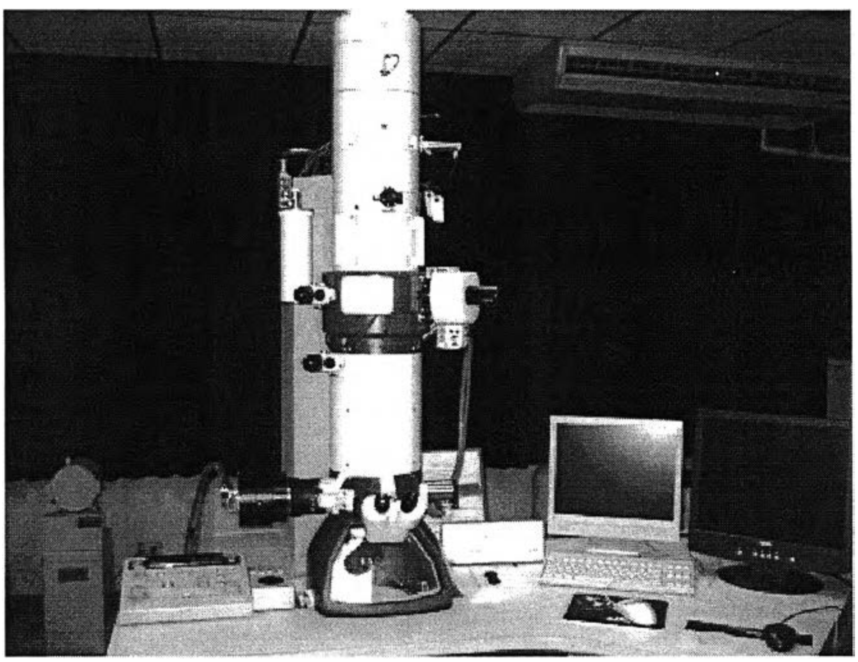

Figure 4.4. Transmission Electron Microscopy (JEOL JEM-1230)

4.2.3 X-ray diffraction (XRD)

X-ray diffraction is of course of paramount importance in determining the structures of crystals. The application of XRD to nanocrystalline solids, powders, single-crystal thin films or multilayers may be less spectacular[21]. In this experiment, The samples were analyzed by XRD (JEOL JDX-8030) as shown in Figure 4.5., to find their crystalline phase. The ZnS samples were spread on the glass slide and then set in the equipment which provide x-ray beam for the analysis.

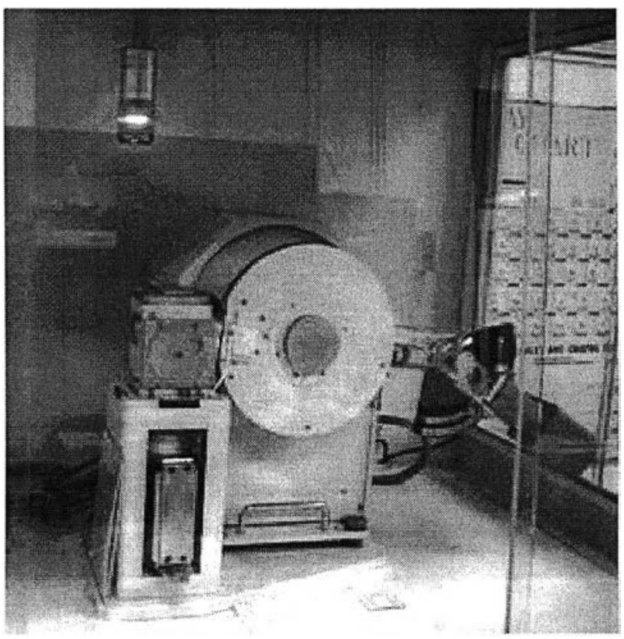

Figure 4.5. X-ray diffraction (JEOL JDX-8030) used in this thesis

4.2.4 Dynamic Light Scattering (DLS)

Dynamic Light Scattering (DLS) is used for particle size analysis. This equipment is based on the measurement of the dispersion of light scattering by particles motion in a static solvent such as ethanol, water or toluene, the measured particle should correspond to hydrodynamic diameter, not the real diameter of complex structures of particles. However, DLS results are expected to give at least the qualitative trend in particle sizes distribution. In this work DLS (ZETA SIZER Nano-ZS) shown in Figure 4.6 were used for analyzing the resulting nanoparticles. The sample from the final microemulsion was diluted by ethanol. Then, this solution was mixed by homogenizer for 5 minutes and dispersed in ultrasonic bath until ensuring its uniform dispersion. Then loaded it to the sample cell for analysis.

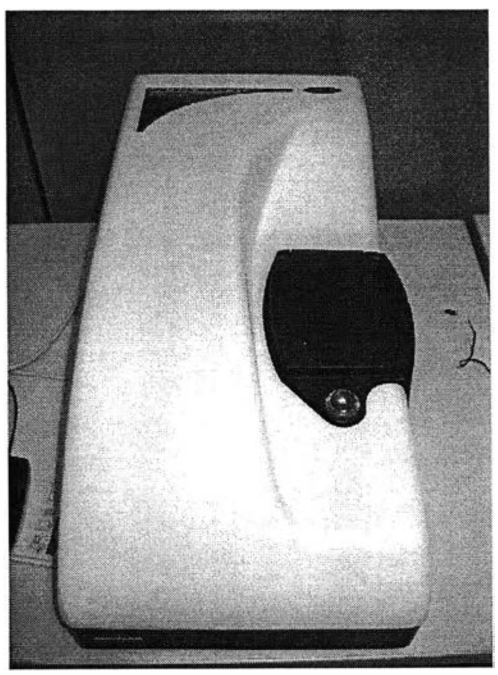

Figure 4.6 Dynamic light scattering (ZETA SIZER Nano-ZS) used in this research