

CHAPTER 4

CONCLUSIONS

4.1 Cyclic microwave-assisted synthesis of Cu_3BiS_3 dendrites using L-cysteine as a sulfur source and complexing agent

Cu_3BiS_3 dendritic structures were successfully synthesized using a facile cyclic microwave radiation-assisted synthesis. The products were characterized by XRD, FESEM, TEM, SAED, HRTEM, PL and UV-Vis spectroscopy. The XRD patterns indicate that the samples have orthorhombic phase with lattice parameter $a = 7.696 \text{ \AA}$, $b = 10.388 \text{ \AA}$ and $c = 6.712 \text{ \AA}$, respectively. FESEM and TEM images revealed that the as-prepared Cu_3BiS_3 composed of well-developed dendrites. The SAED patterns were used to identify crystal structures which similar to the acquired structures from XRD technique. Microwave powers play a significant role in the crystalline and shape of the products, with increasing microwave power, nanoparticles were gradually transformed into dendritic single crystals composed of a number of (011) parallel crystallographic planes. The growths of Cu_3BiS_3 dendrites have been studied. PL emission of the Cu_3BiS_3 dendrites was blue emission centered at 367 nm.

The optical properties of samples were analyzed by UV-Vis-NIR spectroscopy and the optical band gap was determined as 1.56 eV. This convenient and effective method is expected to be extended to synthesize other important metal sulfide compounds.

4.2 Characterization of Cu₃SbS₄ microflowers produced by a cyclic microwave radiation

Pure tetragonal Cu₃SbS₄ microflowers were produced from 2:2:4 molar ratio Cu:Sb:S in 40 ml ethylene glycol, by a 300W cyclic microwave radiation for 40 cycles. Crystalline phase, composition, morphology and atomic vibrations were specified by XRD, EDX, SEM, TEM, SAED, and Raman analyses. Their UV-visible absorption was studied to determine the energy gap (E_g). The molar ratios of reactant such as Cu:Sb:S is a crucial parameter for the preparation of Cu₃SbS₄ by microwave radiation synthesis. In addition, the concentration of the solution of sample is also affected the morphology of the product by the dilute solution of the sample will allow the crystal of the product to grow into larger size and shape. A formation mechanism was also proposed to relate with the experimental results.

4.3 Cyclic microwave-assisted synthesis of CuFeS₂ nanoparticles using biomolecules as sources of sulfur and complexing agent

A cyclic microwave radiation (CMR) has been developed to prepare CuFeS₂ chalcopyrite at 300 W with 40 cycles of each interval (40 sec power on- 40 sec power off). The hollow particle and agglomerate nanoparticle were produced depend upon iron source precursor. A suitable precursor of the reaction was needed in order to obtain a CuFeS₂ hollow particle with a higher crystallinity. The XRD, EDX, SAED and Raman analysis confirm the phase purity of products. The SEM and TEM images show the morphology of CuFeS₂ powder. The XPS spectra reveal the valence state of Cu⁺, Fe³⁺ and S²⁻ in CuFeS₂ products. A possible formation mechanism of CuFeS₂ was also proposed in accordance with the experimental results.