

Effects of temperature, ligands, and solvent on the size distribution of copper thioantimonide and copper thioantimonate nanoparticles synthesized by hot injection

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Cu₃SbS₄ and CuSbS₂ nanoparticles were synthesized by hot injection method, using CuCl and SbCl₃ as metal precursors, colloidal sulfur as chalcogenide precursor, oleylamine or dodecanethiol as capping-ligand, and oleylamine, diphenyl-ether and octadecene as solvents. The temperature of the reaction media at the moment of the injection was kept at 200, 230 and 250 °C. The reaction was allowed for 1 and 5 minutes. The nanoparticles were characterized by UV-VIS-NIR absorption spectroscopy, transmission electron microscopy (TEM), Raman spectroscopy, and X-ray diffraction (XRD). At 200 °C, using 1.35 mmol of CuCl and SbCl₃ in 10 mL of oleylamine the formation of anisotropic shaped nanoparticles was observed after 1 minute of reaction and quasi-spherical nanoparticles with average diameter of 11.2 nm ($\sigma=30\%$) were obtained after 5 minutes. At 230 °C, quasi-spherical nanoparticles with average diameter of 2.8 nm ($\sigma=30\%$) were obtained after 1 minute of reaction and 27.7 nm ($\sigma=26\%$) particles were obtained after 5 minutes. At same conditions, but using 5.7 mmol of oleylamine in 10 mL of diphenyl-ether, nanoparticles with average diameter of 25.3 nm were obtained after 1 minute and 25.1 nm nanoparticles were obtained after 5 minutes. Analyzing the partial results it is possible to associate the smaller size distribution of the particles formed with the smaller solubility of the oleylamine-capped Cu₃SbS₄ monomers in diphenyl-ether.

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