**Abstract**

The mixed phase CdS and NiS nanoparticles are prepared by adopting ultrasonic wave irradiation method under different doping concentration of Ni in CdS. The well defined nano spheres are obtained during this synthesis process. The predicted particle sizes from X-ray diffraction (XRD) analysis are found to lie in the range between 37 and 49 nm. The effective doping of Ni lead to form the mixture of two phases such as CdS and NiS. The respective change due to the formation of mixture of CdS and NiS is reflected well in the band gap energy which is measured in Diffused Reflectance Spectra (DRS). It is predicted in the range of 2.41–2.23 eV respectively. Consistency of particle size with XRD are confirmed from Transmission Electron Microscope (TEM) images and also identified the presence of Nickel Sulfide and Cadmium Sulfide in nanostate with average particle size as 54 nm. The Energy Dispersive X-ray (EDAX) analysis confirmed the existence of Ni, Cd and S the doping levels. The optical absorption analysis of samples are performed in UV–vis range 400–600 nm. The synthesized samples are further characterized Fourier Transform Infrared (FT-IR) spectroscopy, Thermogravimetric (TGA) analysis, I-V characteristic and conductivity measurements.