

PREPARATION OF CdS NANOPARTICLES IN THE [P_{6,6,6,14}][NTf₂] IONIC LIQUID

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Keywords: Cadmium sulfide, Nanoparticles, Ionic liquid, Preparation, characterization.

Introduction: Physicochemical properties of nanomaterials directly depend on the size of their particles [1], being different from those of bulk materials. Recently, a great variety of approaches have been developed for the synthesis of nanoparticles, enhancing the applicability of many materials. Semiconductor nanoparticles are widely used for biological labeling and diagnostics, light-emitting diodes, electroluminescent devices, photovoltaic devices, lasers and single-electron transistors [2,3].

Many different techniques have been developed to synthesize nanoparticles, being the most commonly used the liquid-phase methods. Room-temperature ionic liquids (RTILs) are usually non-volatile, non flammable and thermally stable, what confers them a great potential as a “green” recyclable alternative to the traditional organic solvents. Many combinations of cations and anions exist and they have, depending on the ion-combination, adjustable solvent properties. So, ionic liquids have become a perfect media for synthesis, as well as for separation processes, photochemical solar cells and electrochemical devices [4]. The use of RTILs for the preparation of nanoparticles is being studied [5-7].

Experimental: In this work we have prepared cadmium sulfide (CdS) nanoparticles using the ionic liquid trihexyl(tetradecyl) phosphonium bis(trifluoromethylsulfonil) imide, [P_{6,6,6,14}][NTf₂]. For its preparation no organic solvents or precursors have been used. The bulk solid was added to the ionic liquid in a round bottom flask at room temperature and the mixture was vigorously stirred for 4 hours and heated up to 120 °C, using a source of argon in order to have an inert atmosphere. To ensure maximum dispersion of nanoparticles an excess of bulk material was added to the ionic liquid. The resulting dispersion acquired the characteristic orange colour of the formed nanoparticles, indicating their presence in the ionic liquid. The dispersion was left to cool at room temperature and centrifuged at 4000 rpm for 15 minutes to withdraw the remaining solid in excess. Afterwards the precipitation of the nanoparticles is carried out in order to obtain the isolated solid nanoparticles. Dodecanethiol was added as a capping agent to avoid the agglomeration of the solid nanoparticles. Then, it was left to stand for 6 hours and ethanol was added to enhance the precipitation of nanoparticles. The precipitated was retrieved by centrifugation at 3000 rpm for about 10 minutes. The nanoparticles can be redispersed in many organic solvents such as toluene or isooctane.

Once the nanoparticles were prepared, they were characterized by means of UV/vis absorption spectroscopy, transmission electron microscopy (TEM) and X-Ray powder diffraction.

Results and discussion: Cadmium sulfide nanoparticles were formed by a dissolution method using an ionic liquid as only liquid medium and the employed precursor was the corresponding bulk powder. In contrast to other liquid-media methods of synthesis, this new route avoids additional solvents or solids.

The characterization of the nanoparticles started with the analysis of the UV/vis absorption. Then, the TEM images and the XRPD pattern were also obtained. As it can be seen in Figure 1, cadmium sulfide nanoparticles show a maximum absorption peak at 432 nm, consistent with previous works and which differs from the wavelength associated to bulk cadmium sulfide with absorption peak at 515 nm [8].

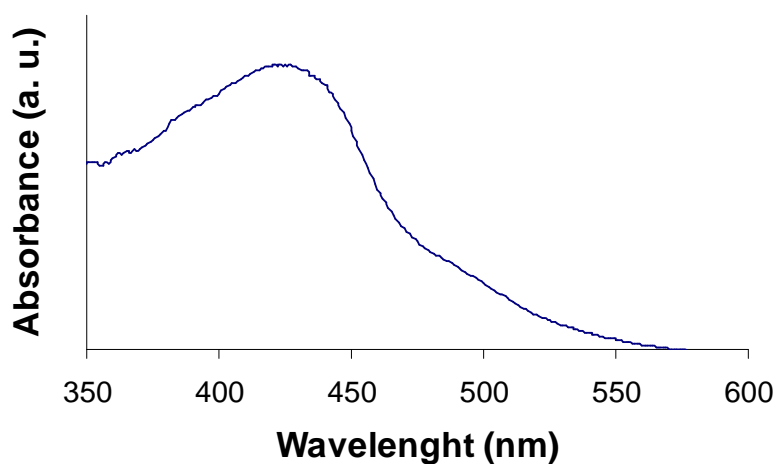


Figure 1. UV/vis absorption spectrum of the cadmium sulfide nanoparticles.

In the micrographs taken from different regions of the grid at the TEM, nanoparticles with a narrow size distribution (2-10 nm) are found. They do not form agglomerates and have a spherical shape, as it can be seen in Figure 2.

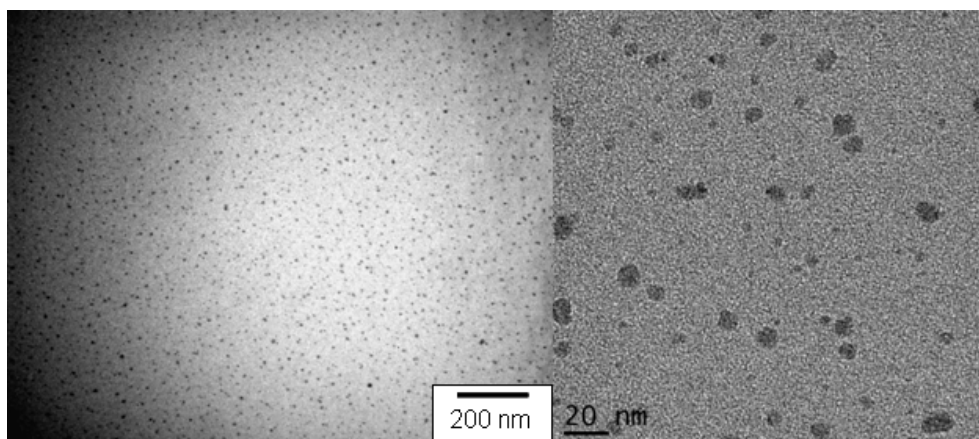


Figure 2. TEM images of the as-prepared CdS nanoparticles.

The X-Ray pattern corroborates the mixture of hexagonal (greenockite) and cubic (hawleyite) structures of the cadmium sulfide precipitated nanoparticles, the same of the bulk powder, pointing out that any structural transformation of the solid took place during the formation of the nanoparticles (Figure 3).

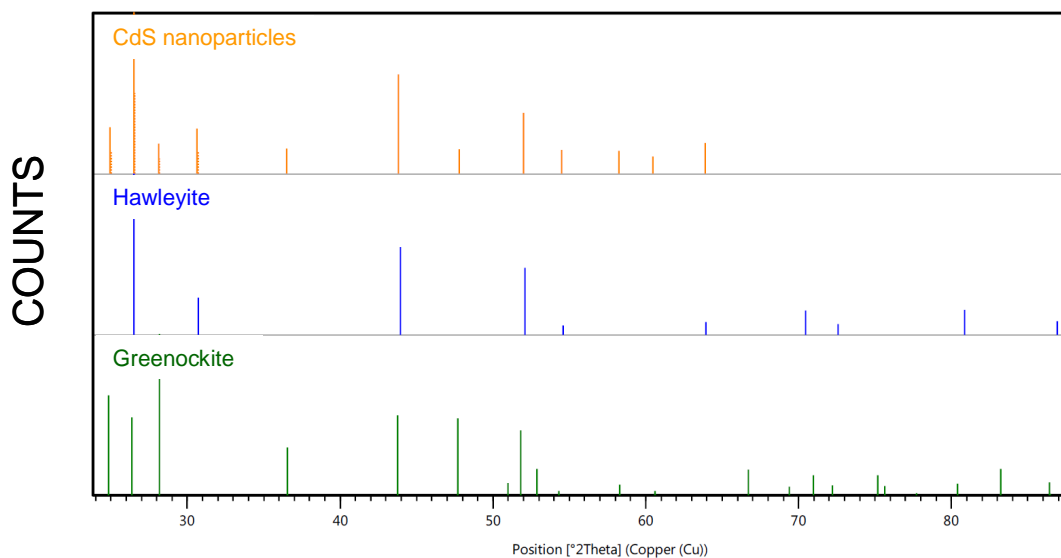


Figure 3. XRPD of the nanoparticles.

Conclusions: Nanoparticles of cadmium were formed using the bulky powder material as only precursor within an ionic liquid. This method allows a fabrication without atmospheric pollution problems since ionic liquids are non-volatile. Nanoparticles of CdS were fabricated using trihexyl(tetradecyl)phosphonium bis(trifluoromethylsulfonyl)imide, $[P_{6,6,6,14}][NTf_2]$. Uniform distributed spherical-shaped, well-defined and non-agglomerated nanoparticles, with sizes in the range of 2-10 nm were formed. The crystallographic structure of the solid did not suffer any alteration during the formation of the nanomaterial.

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