

Research Article

Synthesis and Spectral Studies of Ni(II) Dithiocarbamate Complexes and Their Use as Precursors for Nickel Sulphides Nanocrystals

Azile Nqombolo and Peter A. Ajibade

Department of Chemistry, University of Fort Hare, Private Bag Box X1314, Alice 5700, South Africa

Correspondence should be addressed to Peter A. Ajibade; pajibade@ufh.ac.za

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Ni(II) dithiocarbamate complexes have been synthesized and characterized by UV-Vis, FTIR, and NMR spectroscopic techniques. Electronic spectra measurements indicate that the complexes are four-coordinate square planar geometry while the FTIR confirmed that the dithiocarbamates act as bidentate chelating ligands. The compounds were used as single source precursors and thermolysed at 220°C to prepare HDA-capped NiS nanocrystals which were characterized by absorption and photoluminescence (PL) spectra measurements, powder X-ray diffraction (PXRD), transmission electron microscopy (TEM), scanning electron microscopy (SEM), and energy dispersive spectroscopy (EDS). Absorption spectra studies showed that the synthesized NiS nanoparticles are blue-shifted relative to the bulk material and PL studies showed emission maxima that are red-shifted compared to the absorption band edges. The XRD patterns of the as-prepared NiS nanoparticles revealed cubic crystalline phases. TEM images showed spherical and close-to-spherical nanocrystals with the size in the range 12–38 nm for NiS1, 8–11 nm for NiS2, and 9–16 nm for NiS3. SEM images showed homogeneous surface morphology and EDS confirmed the presence of Ni and S and the formation of NiS nanoparticles.

1. Introduction

In recent years, nanomaterials have received considerable attention and are being widely studied because of their size dependent properties that give rise to application in fields such as catalysis [1, 2], agriculture [3], or solar cells [4]. Different methods such as sputtering [5, 6], coprecipitation [7], sol-gel method [8], microemulsion [9], hydrothermal technique [10], and single source precursors [11] have been used for the preparation of metal sulphide nanoparticles. In spite of the numerous methods, the challenge in the development of materials with novel optical and structural properties remains getting uniform shapes and sizes. Thus, single source precursors are being used to prepare metal sulphide nanoparticles and thin films since they offer the advantage of one-pot synthesis with uniform size distributions [12–16].

Single source precursors have been employed to prepare NiS nanocrystals and this has resulted in particles with

varying crystalline phases and sizes [17–20]. Salavati-Niasari et al. reported the synthesis of nickel sulphide nanoparticles by the hydrothermal method in the presence of thioglycolic acid. The XRD patterns showed β -NiS and the optical studies showed that the as-prepared nanoparticles were blue-shifted compared to the bulk nickel sulphide (~ 2.1 eV) [17]. β -NiS nanoparticles have also been reported by Nagaveena et al. with peaks indexed to rhombohedral structure [18] while Wang et al. reported pure orthorhombic phase of nickel sulphide nanoparticles using the sonochemical method, and the TEM images revealed spherically shaped nanoparticles [19]. It has also been reported that NiS nanoparticles prepared by Yuan and Luan [20] using the solvothermal method showed nanoparticles with hexagonal NiS, cubic NiS₂, orthorhombic Ni₇S₆, and trigonal Ni₃S₂ crystalline phases [20]. In other studies, patch-like structures of NiS nanoparticles were obtained based on the morphological studies from SEM images [21]. The variation in size and shapes of the nanocrystals obtained indicates that there is still

a need to develop robust methods that might give nanocrystals with the desired shapes and sizes. In this study, we present the synthesis and spectroscopic characterization of Ni(II) dithiocarbamate complexes and their thermal decomposition to prepare NiS nanocrystals. The optical, structural, and morphological properties of the as-prepared nanocrystals were studied with absorption and emission spectroscopy, powder X-ray diffraction (PXRD), transmission electron microscopy (TEM), scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS).

2. Material and Methods

2.1. Materials. Materials used included anisidine, dibenzylamine, butylamine, carbon disulphide, potassium hydroxide, ethanol, methanol, diethyl ether, nickel(II) chloride, and acetone. All chemicals and reagents used were of analytical grade and were used as obtained without further purification.

2.2. Characterization. A Perkin Elmer Lambda 25 UV-Vis spectrophotometer was employed to carry out optical measurements at room temperature. The photoluminescence of the nanoparticles was measured using Perkin Elmer LS 45 fluorimeter. Powder X-ray diffraction patterns were recorded on Bruker-D8 Advance powder X-ray diffractometer instrument operating at a voltage of 40 kV and a current of 30 mA with Cu K α radiation. The X-ray diffraction data were analysed using EVA (evaluation curve fitting) software. Phase identification was carried out with the help of standard JCPDS database. The transmission electron microscopy (TEM) images were obtained using a Zeiss Libra 120 electron microscope operated at 120 kV. The samples were prepared by placing a drop of sample solution in toluene on a carbon coated copper grid (300-mesh, agar). Images were recorded on a MegaView G2 camera using iTEM Olympus software. The scanning electron microscopy (SEM) images were obtained on a Jeol JSM-6390 LV apparatus, using an accelerating voltage between 15 and 20 kV at different magnifications. Energy dispersive spectra were processed using energy dispersive spectroscopy (EDS) attached to a Jeol JSM-6390 LV SEM with Noran System SIX software.

2.3. Syntheses

2.3.1. Synthesis of Potassium Salt of the Ligands. The ligands, potassium salt of anisidine dithiocarbamate (KL¹), dibenzyl dithiocarbamate (KL²), and butyl dithiocarbamate (KL³), were prepared as follows.

5 mmol of anisidine (6.16 g), dibenzylamine (10 mL), and butylamine (5 mL) reacted with 5 mmol of potassium hydroxide (2.18 g) in a conical flask placed in ice water and stirred, after which 5 mmol (3 mL) of cold carbon disulphide was added dropwise. The temperature of the reaction mixture was maintained below 4 °C for about 3-4 hours. The resulting product was filtered, washed with diethyl ether, and then recrystallized in acetonitrile and dried.

KL¹: Yield: 3.49 g, 97%, M.p. 94 °C, IR (KBr, cm⁻¹): 1504 (ν C-N), 1296 (ν C=S), 1035 (ν C-S) ¹H-NMR

(dmsO-d₆) δ (ppm) = 2.5 (s, 1H) for -CH- proton of the ring, 9.80 (s, 1H) N-H proton, 6.76–7.38 (d, 2H) for =CH-CH= protons of aromatic ring.

KL²: Yield: 3.91 g, 91%, M.p. 98 °C, IR (KBr, cm⁻¹): 1516 (ν C-N), 1252 (ν C=S), 1000 (ν C-S) ¹H-NMR (dmsO-d₆) δ = 7.23–7.21 (m, 5H) for -C₆H₅ protons from the aromatic ring, 5.30 (d, 2H) for ethyl protons.

KL³: Yield: 8.32 g, 90%, M.p. 67 °C, IR (KBr, cm⁻¹): 1470 (ν C-N), 1159 (ν C=S), 1015 (ν C-S) ¹H-NMR (dmsO-d₆) δ = 0.83 (t, 3H) -CH₃, 0.92–1.26 (m, 5H) -CH₂, 7.8 (s, 1H) N-H protons.

2.3.2. Synthesis of Ni(II) Dithiocarbamate Complexes. 5 mmol (0.45 g) of potassium salts of anisidine dithiocarbamate, dibenzyl dithiocarbamate (0.57 g), and butyl dithiocarbamate (0.43 g) ligands was dissolved in 10 mL of methanol in a beaker. 2.5 mmol (0.59 g) of NiCl₂ was dissolved in methanol in a separate beaker. The two solutions were mixed and stirred for 1–3 hours. The resulting product was filtered and washed with water followed by diethyl ether and dried. The complexes are labelled as [Ni(L¹)₂] from anisidine dithiocarbamate, [Ni(L²)₂] from dibenzyl dithiocarbamate, and [Ni(L³)₂] from butyl dithiocarbamate ligand.

[Ni(L¹)₂]: Yield: 3.22 g, 49%, M.p. 201 °C, IR (KBr, cm⁻¹): 1571 (ν C-N), 1131 (ν C-S), 460 (Ni-S).

[Ni(L²)₂]: Yield: 1.35 g, 60%, M.p. 200 °C, IR (KBr, cm⁻¹): 1593 (ν C-N), 1234 (ν C-S), 495 (Ni-S).

[Ni(L³)₂]: Yield: 1.48 g, 49%, M.p. 139 °C, IR (KBr, cm⁻¹): 1470 (ν C-N), 1004 (ν C-S), 383 (Ni-S).

2.4. Synthesis of NiS Nanoparticles. The nickel complexes (0.2 g) were dissolved in 2 mL tri-n-octylphosphine (TOP) and injected into 2 g of hot hexadecylamine (HDA) at 220 °C. The temperature was maintained for 1 hour at 220 °C. After this, the mixture was allowed to cool to 70 °C and cold methanol was added to precipitate the NiS nanoparticles. The HDA-capped NiS nanoparticles were separated by centrifugation and washed three times with cold methanol and dried. The nanoparticles are labelled as NiS1 from nickel(II) anisidine dithiocarbamate complex ([Ni(L¹)₂]), NiS2 from nickel(II) dibenzyl dithiocarbamate complex ([Ni(L²)₂]), and NiS3 from nickel(II) butyl dithiocarbamate complex ([Ni(L³)₂]).

3. Results and Discussion

3.1. FTIR Spectra Studies of the Ligands and Metal Complexes. FTIR spectra of the dithiocarbamate ligands and their corresponding complexes are presented in Figure 1. In the ligands, the ν C-N appeared at 1501 cm⁻¹, for KL² at 1515 cm⁻¹ and for KL³ at 1470 cm⁻¹. The observed shift in the ν C-N stretching frequency of complexes to higher frequency compared to that of ligands is due to the increase in the single bond character of ν C-N [22] due to the dominant contribution of the thioureide resonance form of the dithiocarbamate. Also, the shift in ν C-S to lower frequency in the metal complexes in comparison

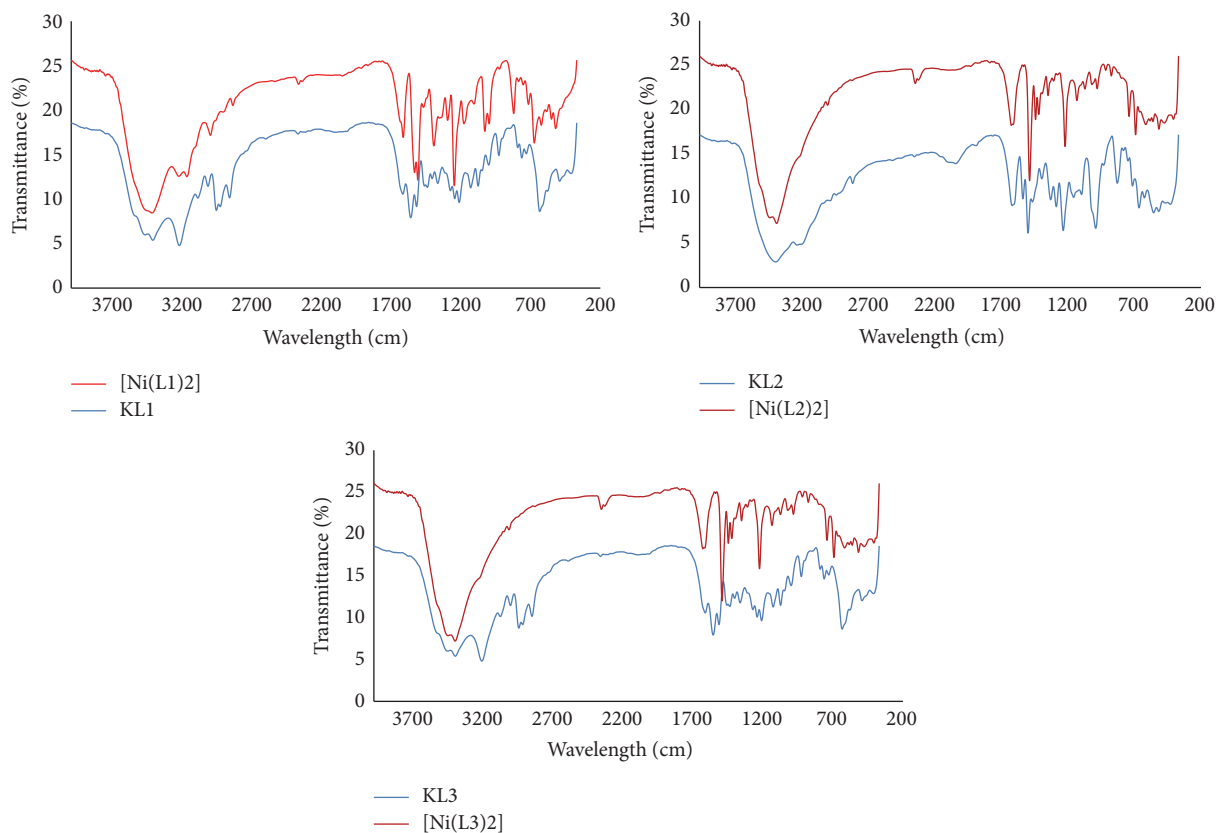


FIGURE 1: FTIR spectra of the dithiocarbamate ligands and corresponding Ni(II) complexes.

to the ligands could be ascribed to the decrease in the double bond character of $\nu\text{C}=\text{S}$ in the formation of the complex [23]. The $\nu\text{Ni}-\text{S}$ stretching vibration for $[\text{Ni}(\text{L}^1)_2]$ was observed at 470 cm^{-1} , for $[\text{Ni}(\text{L}^2)_2]$ at 457 cm^{-1} , and for $[\text{Ni}(\text{L}^3)_2]$ at 382 cm^{-1} and this confirms that the Ni ion is bonded to the dithiocarbamate through the sulfur atoms [24].

3.2. Electronic Spectra Studies of the Ligands and Complexes.

The electronic spectra of the dithiocarbamate ligands and Ni(II) complexes are shown in Figure 2. Anisidine dithiocarbamate (KL^1) has an absorption band at 297 nm assigned to the $\pi \rightarrow \pi^*$ transitions of N-C-S; the second band at 303 nm is assigned to $\pi \rightarrow \pi^*$ transitions of S-C-S of dithiocarbamate moiety, and the band that appears at 305 nm is due to $n \rightarrow \pi^*$ bonding which is located on the sulfur atom. In the Ni(II) anisidine dithiocarbamate, the intraligand charge transfer transitions occurred at 324 nm assigned to $\pi \rightarrow \pi^*$ bonding of the dithiocarbamate moiety. $[\text{Ni}(\text{L}^1)_2]$ has an absorption band at 418 nm which is assigned to the d-d transition due to the excitation of the metal ion [25]. Dibenzyl dithiocarbamate (KL^2) has intraligand charge transfer transitions at 295 nm and the corresponding complex $[\text{Ni}(\text{L}^2)_2]$ also has intraligand charge transfer transitions but it shifted to higher wavelength at 332 nm and another band at 391 which is due to metal to ligand charge transfer (MLCT) transitions. At 430 nm, there is a small band for d-d transition observed in the Ni(II) complex. KL^3 has a band at 291 nm, which is assigned to the

$\pi \rightarrow \pi^*$ transition of the dithiocarbamate moiety. But this band appeared as a broad absorption band at 397 nm in the complex, $[\text{Ni}(\text{L}^3)_2]$, which could be ascribed to the MLCT. At 418 nm, there is a small band assigned to the d-d transition [25–27]. This indicates that the complex is a four-coordinate species [25, 26].

3.3. Optical Studies of NiS Nanoparticles

3.3.1. Absorption Spectra Studies of HDA-Capped NiS Nanoparticle. The absorption spectra of the NiS nanoparticles (Figure 3) showed maximum peaks at 306, 314, and 271 nm for NiS1, NiS2, and NiS3, respectively. The absorption band edges were used to determine the band gaps energy (E_g) of the NiS nanoparticles [26]. The band gaps of all synthesized NiS nanoparticles were calculated and found to be 4.05, 3.95, and 4.58 for NiS1, NiS2, and NiS3, respectively. These were found to be blue-shifted compared to the bulk NiS [27]. This shows that the as-prepared NiS nanoparticles have very small sizes and the increase in band gaps with the decrease in particle sizes is due to quantum confinement effects in the nanoparticles [28].

3.3.2. Photoluminescence Spectra Studies of NiS Nanoparticles. The photoluminescence spectra of HDA-capped NiS nanoparticles (Figure 4) were recorded at excitation wavelength of 711 nm. The spectra showed emission maxima at 725, 720, and 622 nm for NiS1, NiS2, and NiS3, respectively.

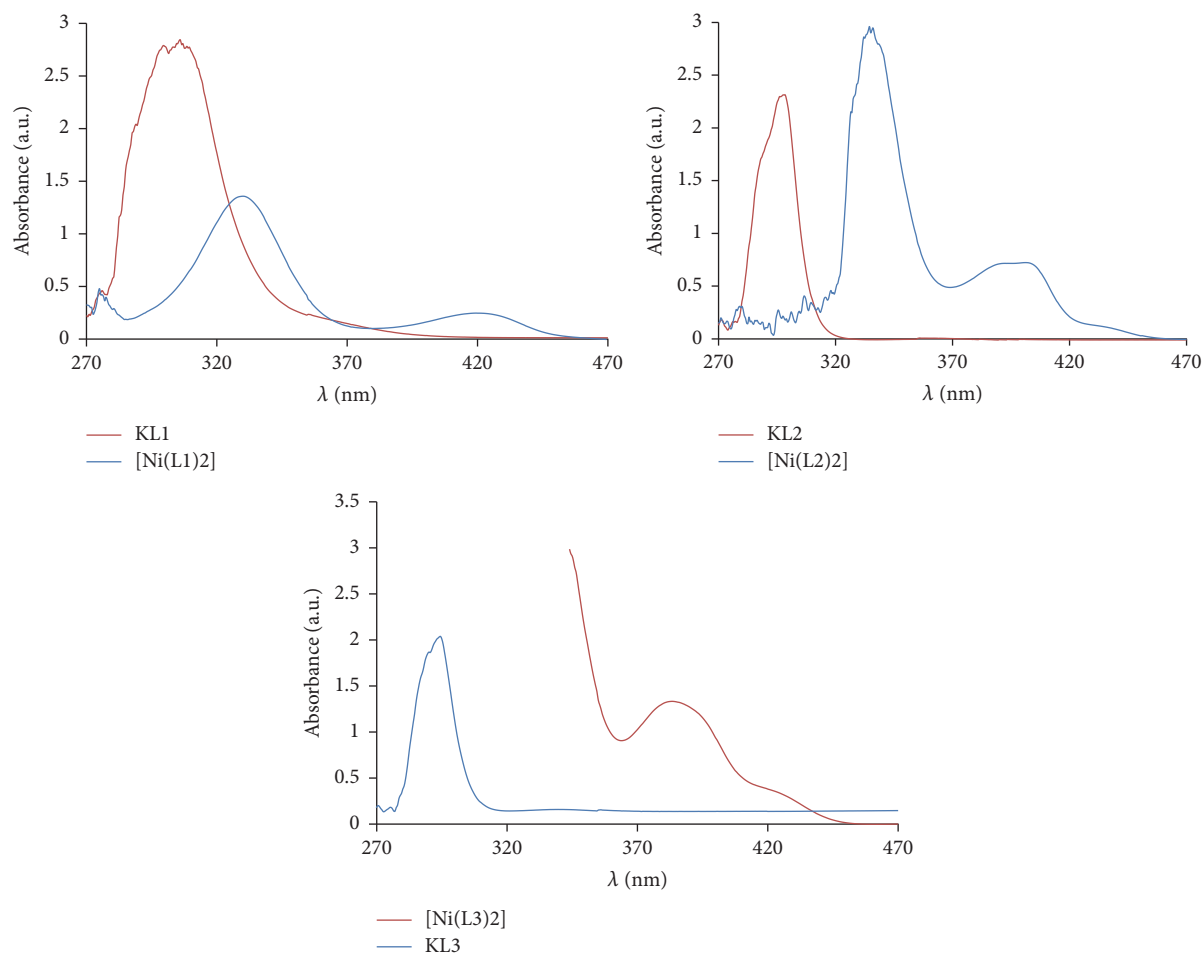


FIGURE 2: Electronic spectra of the ligands and the corresponding Ni(II) dithiocarbamate complexes.

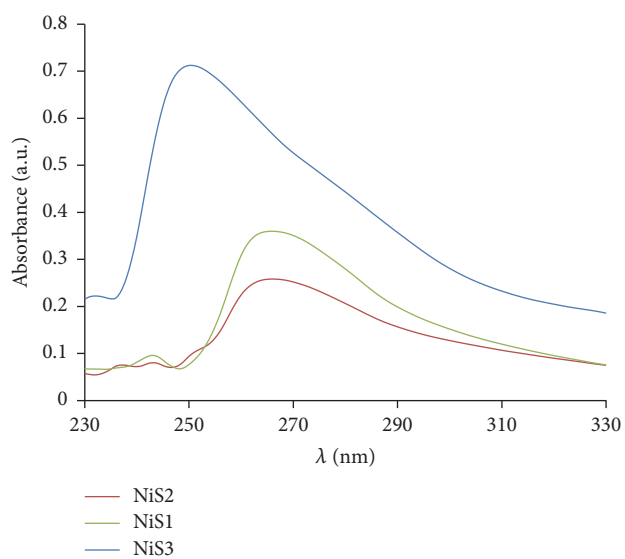


FIGURE 3: Absorption spectra of HDA-capped NiS nanoparticles.

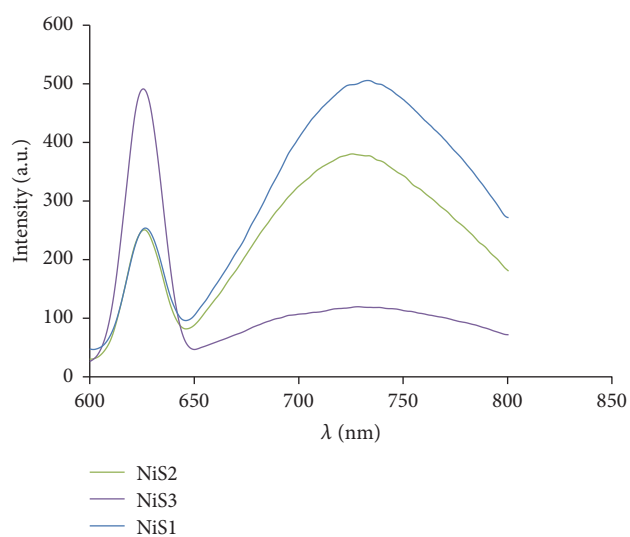


FIGURE 4: PL spectra of HDA-capped NiS nanoparticles.

The emission maxima were found to be red-shifted in comparison to the optical absorption band edges. The broadening

of the emission peaks as shown in Figure 4 could be ascribed to size distributions [29].

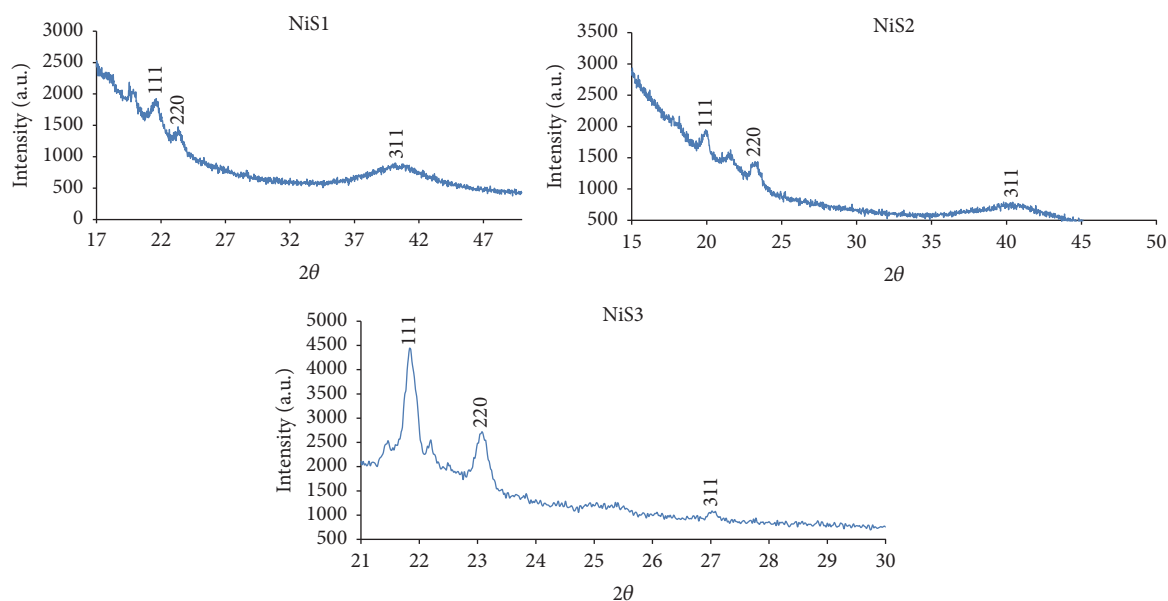


FIGURE 5: Powder XRD patterns of HDA-capped NiS nanoparticles.

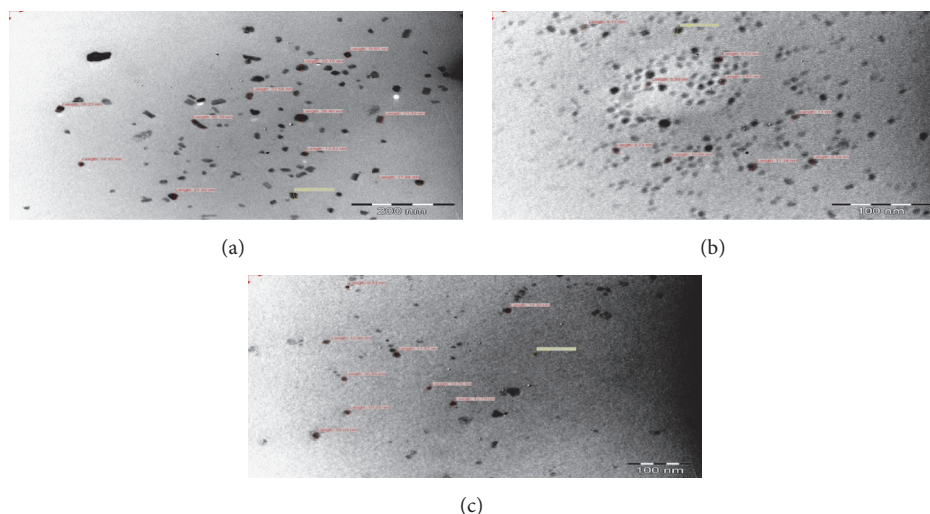


FIGURE 6: TEM image of NiS1 (a), NiS2 (b), and NiS3 (c).

3.4. Structural Studies of NiS Nanoparticles

3.4.1. XRD Pattern Studies of NiS Nanoparticles. The powder X-ray diffraction patterns of the NiS nanoparticles are shown in Figure 5. The XRD patterns of NiS1 showed three peaks at 2θ values of 21.2° , 22.5° , and 40.7° which correspond to (111), (220), and (311) Miller indices for cubic NiS nanoparticles. Three peaks at 2θ values = 19.5° , 22.7° , and 40.85° corresponding to (111), (220), and (311) planes of cubic phase of NiS2 were observed [29]. The XRD pattern of NiS3 showed three peaks at 2θ values = 21.8° , 22.9° , and 39.8° which correspond to (111), (220), and (311) Miller indices of cubic NiS nanoparticles [29, 30]. All the diffraction patterns correspond to the NiS cubic crystalline phase (ICDD card number 043-1469).

3.4.2. TEM Studies of the NiS Nanoparticles. TEM images of HDA-capped NiS nanoparticles are shown in Figure 6. TEM image of NiS1 nanoparticles showed mixture of rod-like structures and close-to-spherical shapes with uniform size distributions [29–31]. Some particles appeared elongated with some agglomeration while others are nearly spherical and triangular in shape and the particle size ranges from 12 to 38 nm [30]. The TEM image of NiS2 showed spherically shaped nanoparticles with little agglomeration and particle size ranging from 8 to 11 nm. The small size is ascribed to the quantum confinement effect. The TEM image of HDA-capped NiS3 nanoparticles showed that the particles are spherical in shape with some agglomeration in between particles and the particle size ranges from 9 to 16 nm.

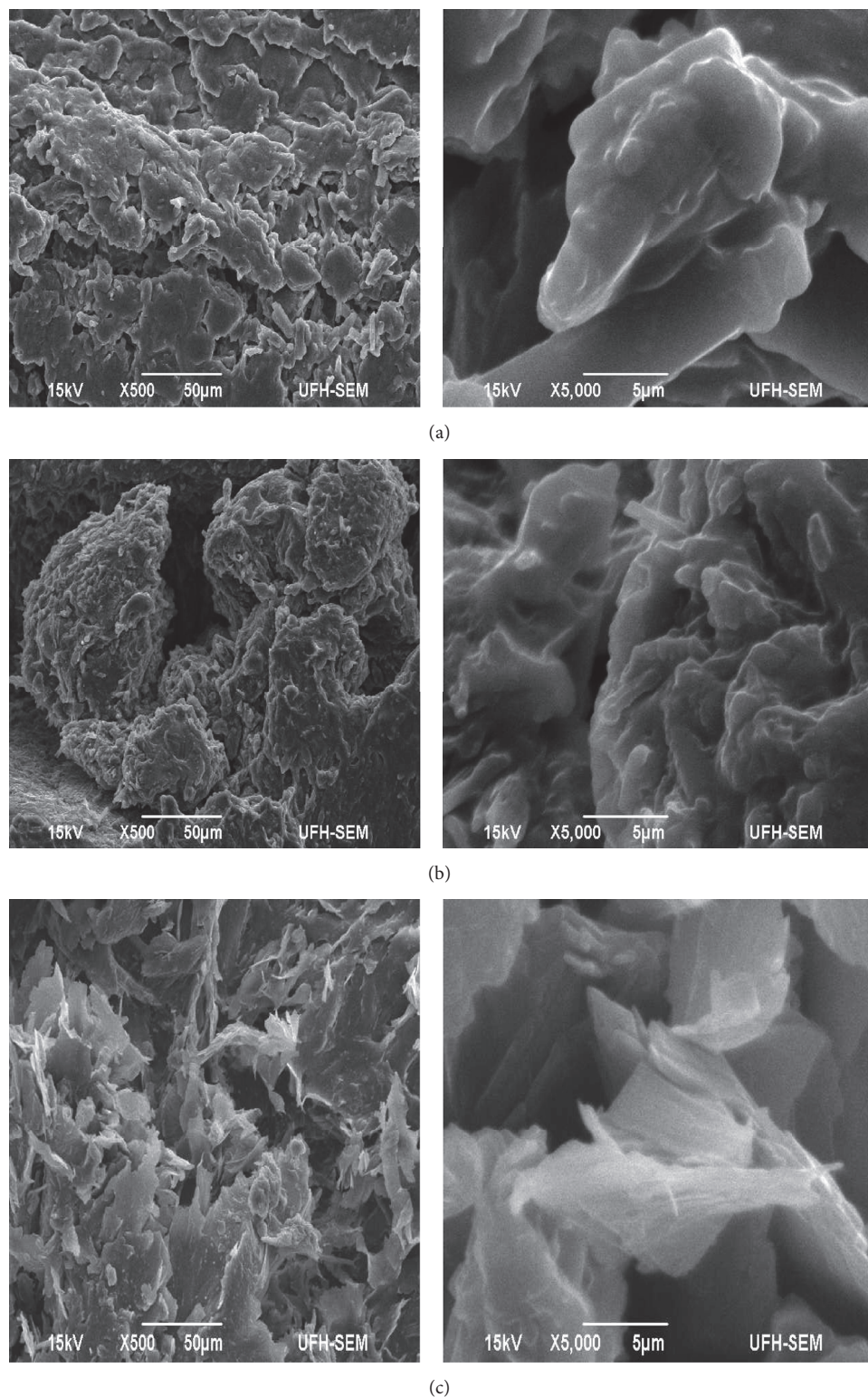


FIGURE 7: SEM images of NiS1 (a), NiS2 (b), and NiS (c).

3.4.3. SEM/EDS Studies of HDA-Capped NiS Nanoparticles. The scanning electron microscopy (SEM) images and energy dispersive spectroscopy (EDS) of NiS nanoparticles are shown in Figure 7. Figure 7(a) shows SEM images of NiS1

nanoparticles with particles with little or no agglomeration because the nanoparticles are lumps-free [31]. Figure 7(b) shows SEM images of NiS2 nanoparticles with uniform surface morphology at both low and high magnification

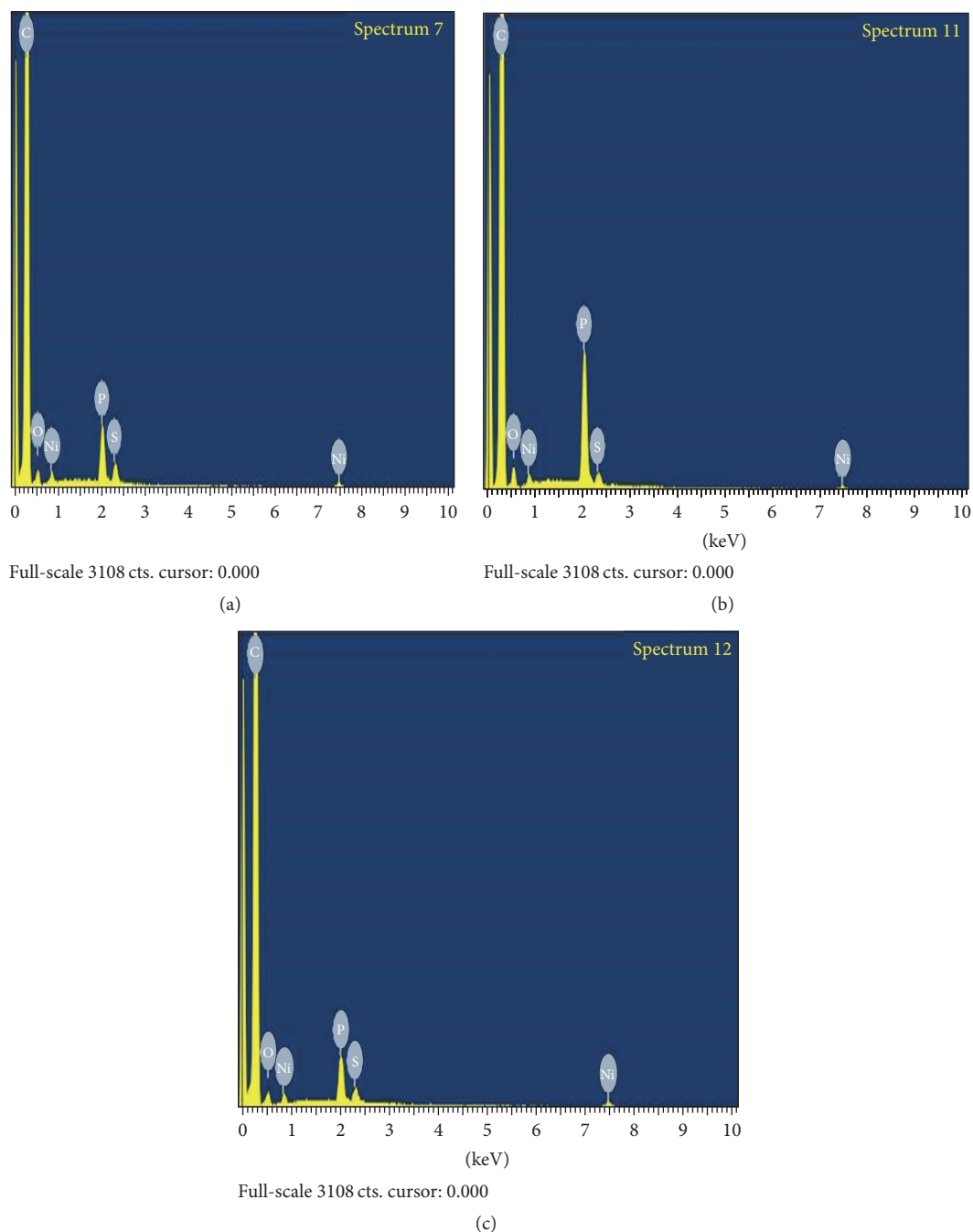


FIGURE 8: EDS spectra of NiS1 (a), NiS2 (b), and NiS3 (c) of the nanoparticles.

while the SEM images of HDA-capped NiS3 nanoparticles as shown in Figure 7(c) at both low and high magnifications have homogeneous surface morphology with cauliflower-like structures [32]. The EDS spectra of the NiS nanoparticles as presented in Figure 8 confirmed the elemental composition of the NiS nanocrystals. Other traces of elements such as O, P, and C are also observed. The observed P and O are due to trioctylphosphine (TOP), and C is attributed to hexadecylamine (HDA) [32].

4. Conclusion

Ni(II) dithiocarbamate complexes have been synthesized and characterized by UV-Vis, FTIR, and NMR spectroscopy. The complexes were formulated as four-coordinate species in which dithiocarbamate acts as bidentate chelating ligand through the sulfur atoms. The complexes were used as single source precursors and thermolysed at 220°C to prepare HDA-capped NiS nanoparticles. The absorption spectra studies

of the as-prepared nanoparticles showed that absorption band edges are blue-shifted compared to the bulk (~2.1 eV) with emission maxima that are red-shifted confirming the small crystallite sizes of the as-prepared nanoparticles. The XRD patterns revealed cubic crystalline phases for the NiS nanocrystals and TEM images showed spherical and close-to-spherical particles with size in the range 12–38 nm for NiS₁, 8–11 nm for NiS₂, and 9–16 nm for NiS₃.

Competing Interests

The authors declare that there are no competing interests regarding the publication of this paper.

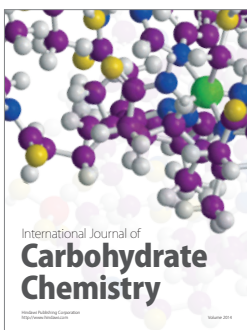
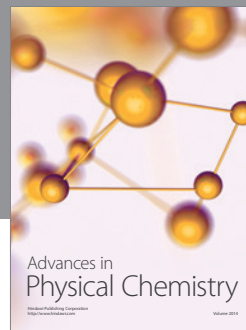
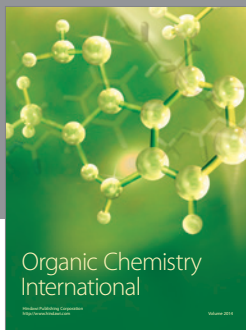
Acknowledgments

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