EAST EUROPEAN JOURNAL OF PHYSICS. 1. 146-153 (2023)
DOI:10.26565/2312-4334-2023-1-18 ISSN 2312-4334

STRUCTURAL, ELECTRICAL AND OPTICAL STUDIES OF $Ni_xCd_{1-x}S$ (x = 0.8, 0.6, 0.4 and 0.2) NANOPARTICLE SYSTEM†

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Received January 23, 2023; revised January 28, 2023; accepted February 3, 2023

This paper demonstrates the synthesis of $Ni_xCd_{1-x}S$ (x=0.8,0.6,0.4,0.2) nanoparticles by microwave-assisted chemical precipitation method. The prepared samples were characterized by XRD, EDAX, SEM, UV-VIS, and PL spectroscopy. The energy-dispersive x-ray analysis confirms the existence of Nickel, Cadmium and Sulphur in proper ratios. The DC electrical resistances were measured in the temperature range of 300 K-500 K. The temperature resistance curves of all the samples show phase transitions above a particular temperature. The UV and PL spectra of all the samples were compared and studied.
Keywords: *nanoparticles; chemical precipitation; phase transition; electrical; optical; bandgap*
PACS: 81.07.-b, 05.70.-a, 81.20.Fw, 61.05.C-, 78.20.-e, 68.37.-d, 81.07.-b, 88.40.H-, 87.64.Ee

1. INTRODUCTION

Nickel Sulphide Nanoparticles are important members of the transition metal sulphide nanomaterials, which also include Ni_3S_2, Ni_4S_3, Ni_6S_5, Ni_7S_6, Ni_9S_8, Ni_3S_4, and NiS [1]. Nickel Sulphide nanoparticles have attracted much interest because of their novel physical, optical, and electrical properties [2]. Nickel Sulphide nanoparticles have potential applications in rechargeable lithium batteries [3, 4], solar cells [5, 6], catalyst devices [7, 8], and image processing devices [9, 10]. Due to the variety of applications, different synthesis methods, such as the solvothermal method [7], the sonochemical method [8], the hydrothermal method [9], and microwave irradiation [10], were used to prepare the NiS nanoparticles.

Cadmium Sulphide (CdS) is an important II-VI semiconductor with a direct band gap of 2.4 eV at room temperature and many excellent physical and chemical properties [11]. Due to its wide band gap, it has been used in many applications such as field effect transistors [12], solar cells [13], light-emitting diodes [14], photocatalysis [15], photographic developers [16], and biological sensors [17]. Various methods have been used to synthesize CdS nanoparticles, such as sol-gel [18], aqueous precipitation [19], hydrothermal [20], sonochemical [21], and microwave heating [22].

Phase transitions in Nickel Sulphide nanoparticles were already studied by varying the thermal annealing temperature [23]. Phase transitions in Cadmium Sulphide nanoparticles have already been reported by varying pressure, modifying the bath temperature, and varying annealing temperatures [24–30]. Ni_xCd_{1-x} S (x = 0.8,0.6,0.4 and 0.2) nanoparticles have both the applications of Nickel Sulphide nanoparticles and Cadmium Sulphide nanoparticles. M. Elango and D. Nataraj studied synthesis and characterization of Nickel doped Cadmium Sulphide ($CdS:Ni^{2+}$) nanoparticles[31]. D. Wu and F. Wang synthesized NiS/CdS nanocomposite by hydrothermal process and studied their photocatalytic performance [32]. Photocatalytic Activity of Pure and Nickel Doped Cadmium Sulphide Nanoparticles were synthesized by co-Precipitation Method [33].

In the present study, we synthesized $Ni_xCd_{1-x}S$ (x= 0.8,0.6,0.4 and 0.2) nanoparticles by microwave assisted chemical precipitation and studied their phase transition through DC electrical resistance measurements at various temperatures. When compared to conventional heating, microwave irradiation can shorten the reaction time. The optical properties of the samples were compared using the UV and PL spectra of the samples.

2. EXPERIMENTAL DETAILS

2.1. Synthesis of $Ni_xCd_{1-x}S$ (x= 0.8,0.6,0.4 and 0.2) nanoparticles by microwave assisted chemical precipitation method

$Ni_xCd_{1-x}S$ (x = 0.8, 0.6, 0.4, and 0.2) nanoparticles were created using Nickel acetate, Cadmium acetate, and Sodium Sulphide. Separately, Nickel acetate and Cadmium acetate were dissolved in 20 ml of distilled water and then mixed together. The Sodium Sulphide solution obtained by dissolving 6.14 g of Sodium Sulphide in 40 ml of distilled water was added in drops to the above solution after effective stirring for 3 hours and kept undisturbed for one day. After complete precipitation, the precipitates were filtered out separately, washed thoroughly with deionized water several times, and kept in a microwaveoven. The solution was then subjected to microwave irradiation of 800 W for 20 minutes for efficient heating. The nanoparticles thus obtained were then cooled to room temperature. Finally, the nanoparticles were annealed at 100^0C for one hour to get the pure $Ni_xCd_{1-x}S$ (x = 0.8, 0.6, 0.4, and 0.2) nanoparticles. The collected $Ni_xCd_{1-x}S$

† ***Cite as:*** M.M. Rose, R.S. Christy, T.A. Benitta, and J.T.T. Kumaran, East Eur. J. Phys. **1**, 146 (2023), https://doi.org/10.26565/2312-4334-2023-1-18

(x = 0.8, 0.6, 0.4, and 0.2) nanoparticles were used for various characterizations. Table 1 shows the amount of precursor materials used to dissolve in 40 ml of distilled water.

Table 1. The amount of precursor materials taken to dissolve in 40 ml distilled water.

Sl no	Composition	Nickel acetate	Cadmium acetate
1	$Ni_{0.8}Cd_{0.2}S$	3.91gm	1.06gm
2	$Ni_{0.6}Cd_{0.4}S$	2.93gm	2.13gm
3	$Ni_{0.4}Cd_{0.6}S$	1.95gm	3.19gm
4	$Ni_{0.2}Cd_{0.8}S$	0.9gm	4.26gm

2.2. Instrumentation

X-ray diffraction (XRD) patterns of the synthesized sample were recorded on a powder X-ray diffractometer with Cu Kα radiation (λ = 1.54 Å) with 2θ ranging from angles 10°- 80°. The surface morphology of the samples has been studied using a TESCAN VEGA3 SBH scanning electron microscope. The elemental compositions were identified using an energy dispersed X-ray analysis setup attached to the scanning electron microscope. The optical absorption spectra of the synthesized nanoparticles were recorded on a UV-visible spectrometer in the wave length range of 200–900 nm. Photoluminescence measurements were performed on a Varian Cary Eclipse photoluminescence spectrophotometer in the range 300-650 nm. A compressed collection of nanoparticles (pellets) was obtained by applying a high pressure of 10 tons/cm^2**.** The resistance of the pellet form of the samples was measured using a four-probe technique.

3. RESULT AND DISCUSSION

Figure 1 shows the XRD patterns of $Ni_xCd_{1-x}S$ (x = 0.8, 0.6, 0.4, and 0.2) nanoparticles synthesized by microwave-assisted chemical precipitation method. The respective EDAX images shown in Figure 2 confirm their compositions.

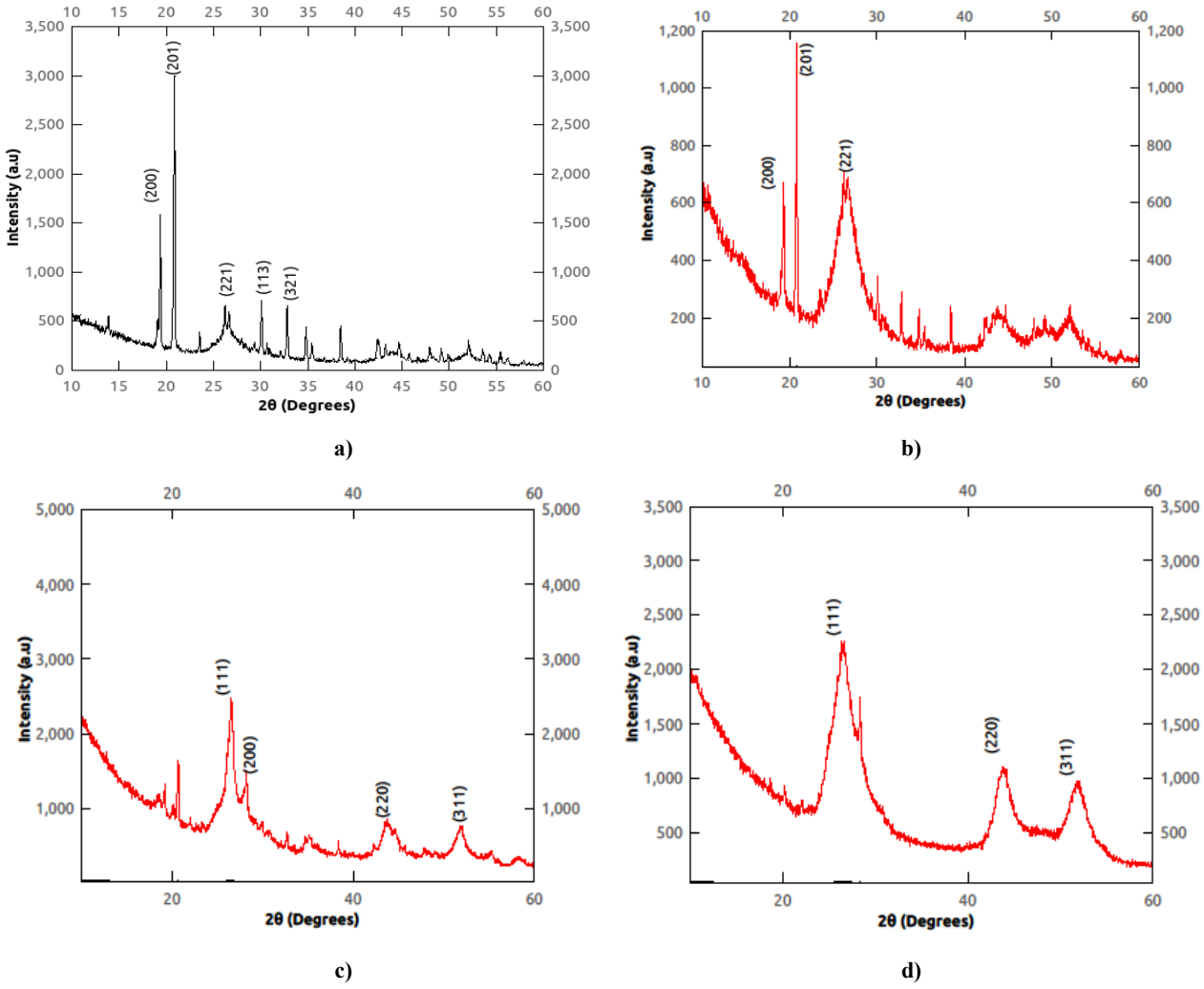

Figure 1. Indexed XRD patterns of $Ni_xCd_{1-x}S$ nanoparticles
a) $Ni_{0.8}Cd_{0.2}S$ nanoparticles, b) $Ni_{0.6}Cd_{0.4}S$ nanoparticles, c) $Ni_{0.4}Cd_{0.6}S$ nanoparticles, d) $Ni_{0.2}Cd_{0.8}S$ nanoparticles

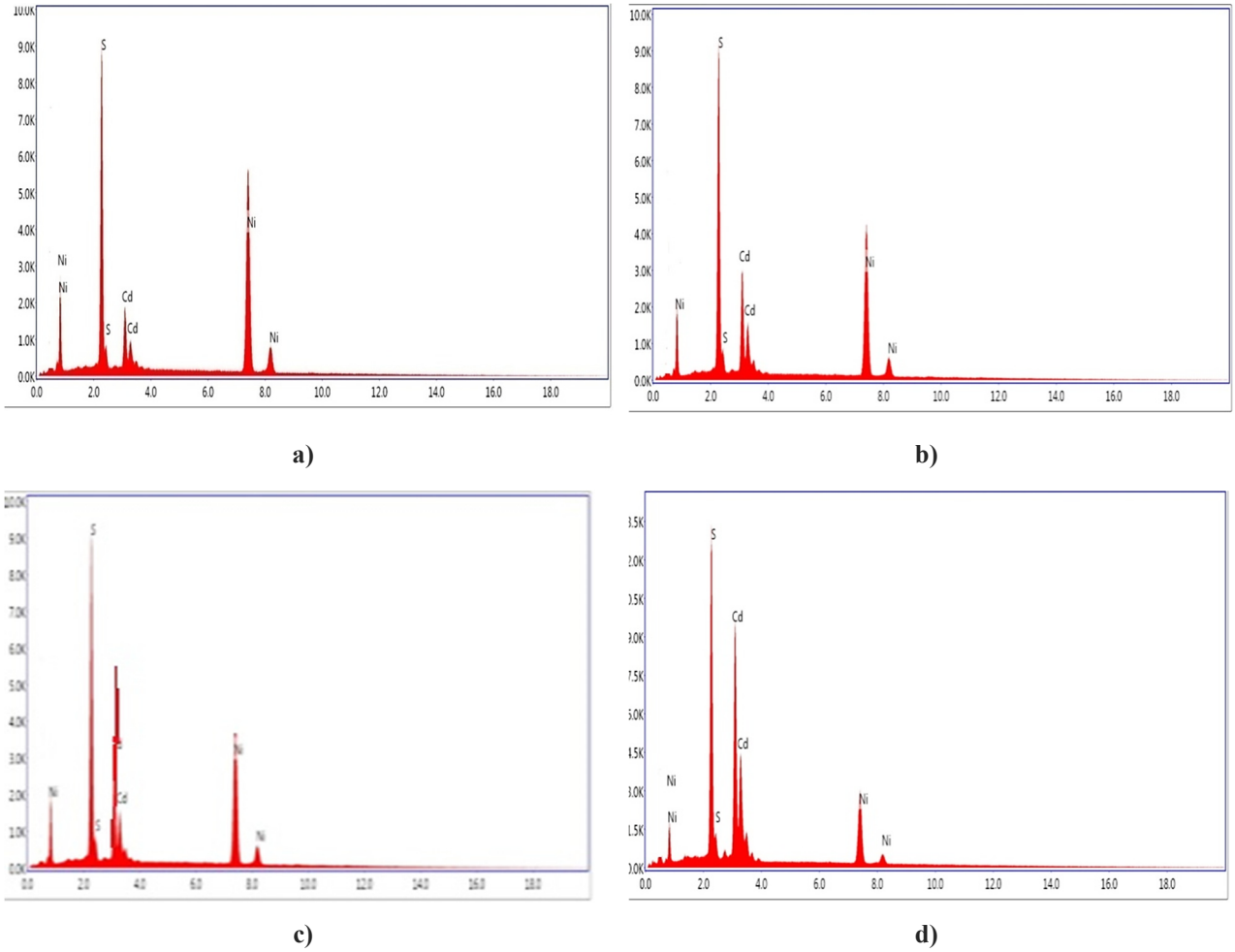

Figure 2. EDAX images of $Ni_xCd_{1-x}S$ nanoparticles
a) $Ni_{0.8}Cd_{0.2}S$ nanoparticles, b) $Ni_{0.6}Cd_{0.4}S$ nanoparticles, c) $Ni_{0.4}Cd_{0.6}S$ nanoparticles, d) $Ni_{0.2}Cd_{0.8}S$ nanoparticles

Figure 3 shows the SEM images of synthesized $Ni_xCd_{1-x}S$ (x = 0.8, 0.6, 0.4, and 0.2). SEM images show the uniform distribution of nanoparticles. Table 2 gives the composition, structure at room temperature, lattice parameters, and particle size of the synthesized samples. As the composition of Cd increases, the particle size decreases. The mixture at room temperature reveals the structure of any one of its components [34,35]. Structural studies clearly indicates that by suitably adjusting the composition, the nanoparticles can be synthesized with either orthorhombic phase or cubic phases and with desired particle size.

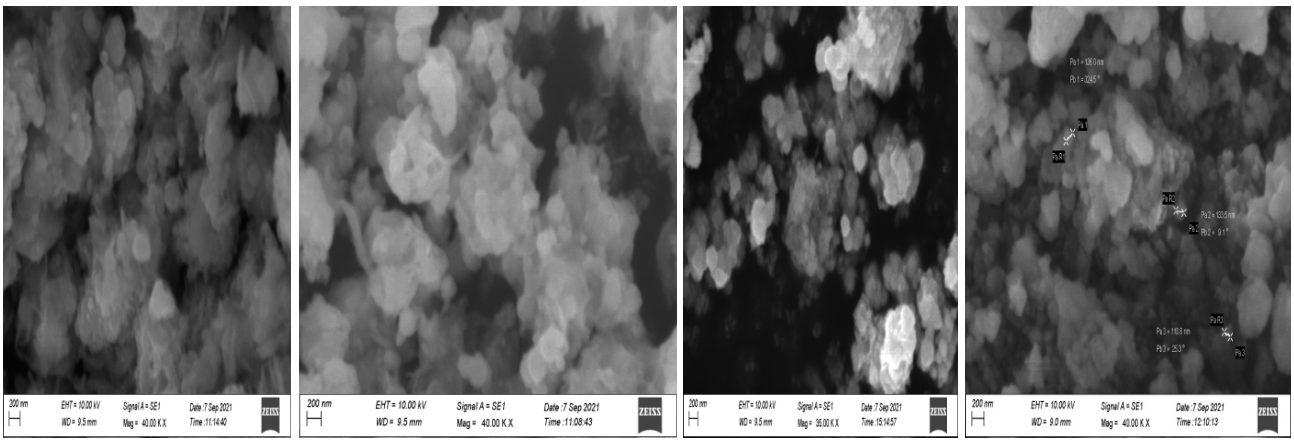

Figure 3. SEM images of synthesized $Ni_xCd_{1-x}S$ nanoparticles
a) $Ni_{0.8}Cd_{0.2}S$ nanoparticles, b) $Ni_{0.6}Cd_{0.4}S$ nanoparticles c) $Ni_{0.4}Cd_{0.6}S$ nanoparticles d) $Ni_{0.2}Cd_{0.8}S$ nanoparticles

Table2. The samples, structure at room temperature, particle size and lattice constants of $Ni_xCd_{1-x}S$ (X= 0.8,0.6,0.4,0.2) nanoparticles

The samples	Structure at room temperature	Particle size	Lattice constants
$Ni_{0.8}Cd_{0.2}S$ nanoparticles	Orthorhombic structure	44nm	a=9.29Å, b=11.12Å, c=9.39Å
$Ni_{0.6}Cd_{0.4}S$ nanoparticles	Orthorhombic structure	37nm	a=9.20, b=11.10Å, c=9.29Å
$Ni_{0.4}Cd_{0.6}S$ nanoparticles	Cubic structure	14nm	a=b=c=5.8Å
$Ni_{0.2}Cd_{0.8}S$ nanoparticles	Cubic structure	5nm	a=b=c=5.68Å

3.1. Electrical studies of $Ni_xCd_{1-x}S$ (x = 0.8, 0.6, 0.4, 0.2) nanoparticles synthesized by microwave-assisted chemical precipitation method

Resistance variations with temperature of $Ni_xCd_{1-x}S$ (x = 0.8, 0.6, 0.4, and 0.2) nanoparticles (300K-500K) are shown in Figure 4 respectively. Table.3 lists the compositions, their behaviour at room temperature, order of resistance, possible transition temperatures, and their behaviour after phase transition. By varying the composition of Nickel and Cadmium, it is possible to synthesize nanoparticles with the desired resistance and electrical behavior. The electrical properties of the sample can also be changed according to the table by increasing the temperature. This change in electrical property is due to phase transition [36].

Table 3. Composition, behavior of the sample at room temperature, order of resistance, possible transition temperatures, behavior of the sample after phase transition of $Ni_xCd_{1-x}S$(x = 0.8, 0.6, 0.4, 0.2) nanoparticles

Sl.NO	Compositions	Behaviour of the sample at room temperature	Order of resistance	Possible transition temperatures	Behaviour of the sample after phase transition
1	**$Ni_{0.8}Cd_{0.2}S$**	Semiconductor	10^8 Ohm	>353K	Conductor
2	**$Ni_{0.6}Cd_{0.4}S$**	insulator	10^9 Ohm	>353K	Semiconductor
3	**$Ni_{0.4}Cd_{0.6}S$**	Semiconductor	10^6 Ohm	>373K	Insulator
4	**$Ni_{0.2}Cd_{0.8}S$**	Semiconductor	10^6 Ohm	>363K	Insulator

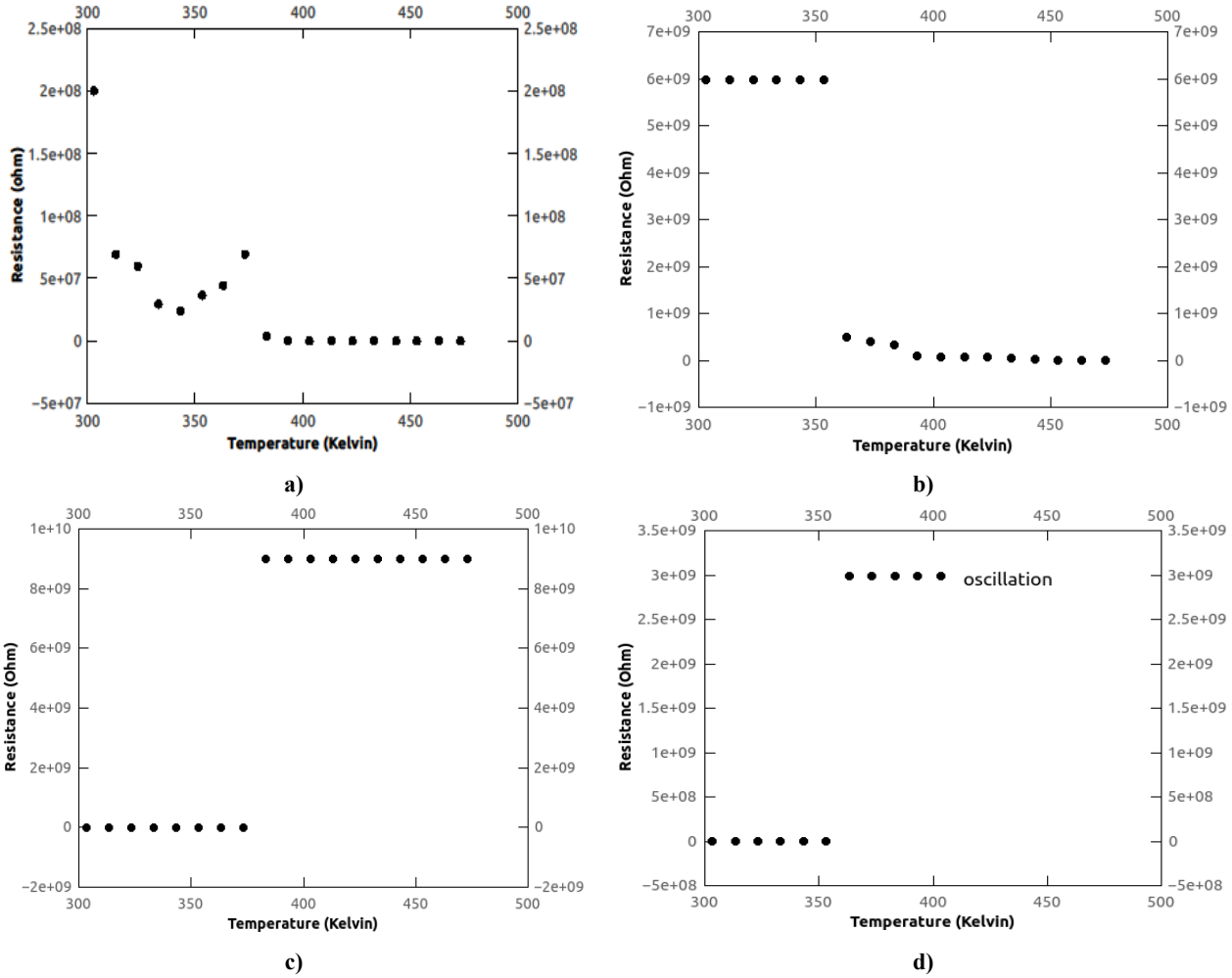

Figure 4. Variation of resistance with temperature of $Ni_xCd_{1-x}S$ nanoparticles
a) $Ni_{0.8}Cd_{0.2}S$, b) $Ni_{0.6}Cd_{0.4}S$, c) $Ni_{0.4}Cd_{0.6}S$, d) $Ni_{0.2}Cd_{0.8}S$

3.2. Optical studies of $Ni_xCd_{1-x}S$ (x = 0.8, 0.6, 0.4, 0.2) nanoparticles synthesized by microwave-assisted chemical precipitation method

UV studies. The optical absorption spectra of $Ni_xCd_{1-x}S$ (x = 0.8, 0.6, 0.4, and 0.2) nanoparticles are shown in Figure 5. The optical absorption spectrum of $Ni_{0.8}Cd_{0.2}S$ nanoparticles shows one more absorption edge near 900nm (Fig. 5a). As more and more Cd is incorporated, this absorption edge goes on decreasing and disappears in the $Ni_{0.2}Cd_{0.8}S$ nanoparticles mixture.

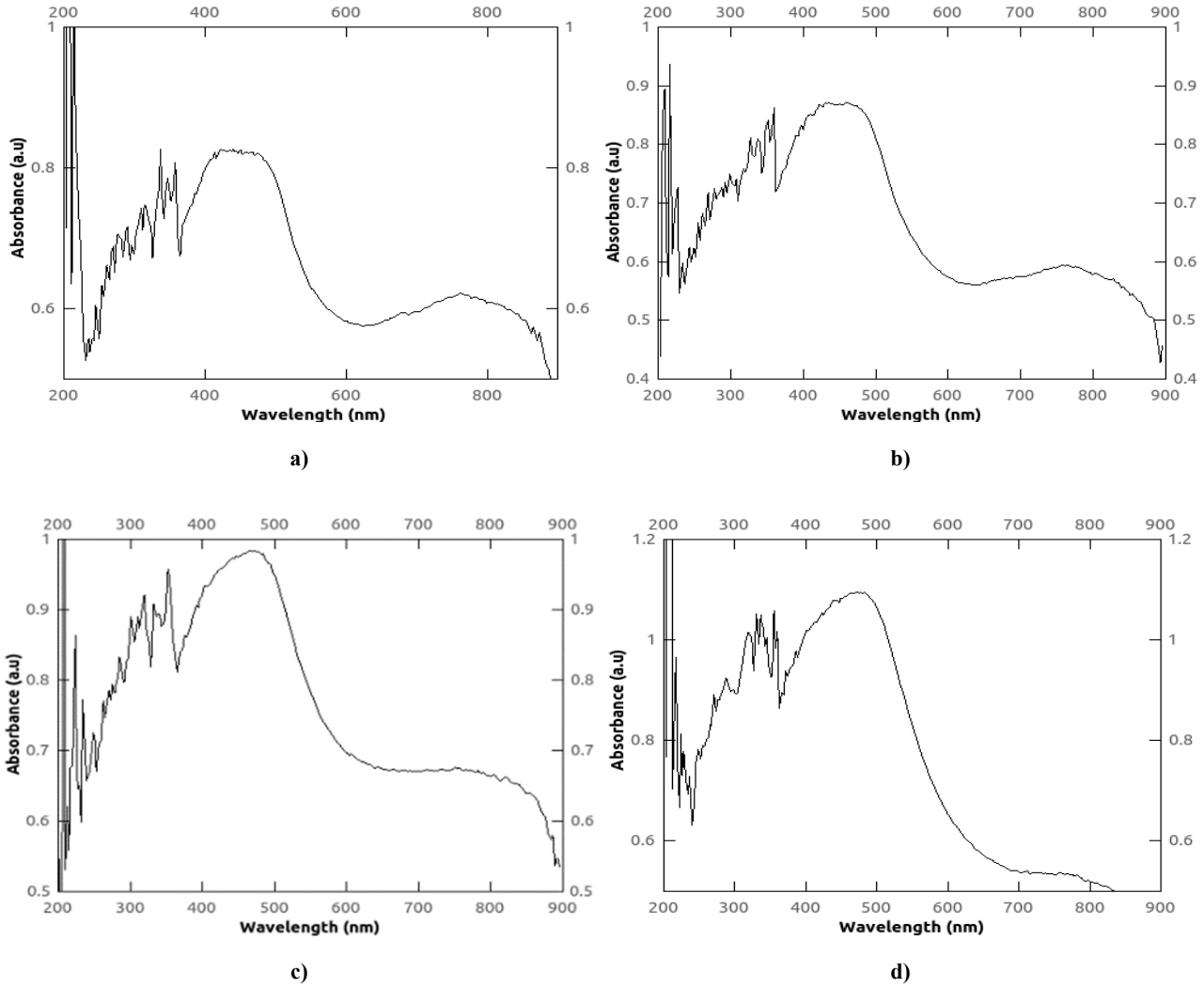

Figure 5. Optical absorption Spectrum of $Ni_xCd_{1-x}S$ nanoparticles
a) $Ni_{0.8}Cd_{0.2}S$ nanoparticles, b) of $Ni_{0.6}Cd_{0.4}S$ nanoparticles, c) $Ni_{0.4}Cd_{0.6}S$ nanoparticles, d) $Ni_{0.2}Cd_{0.8}S$ nanoparticles

The tauc's plots of $Ni_xCd_{1-x}S$ (x = 0.8, 0.6, 0.4, and 0.2) nanoparticles are shown in Figure 6. By replacing 20% of Ni by Cd in the NiS nanoparticles the bandgap energy shifts from 3.5 eV to 2.2 eV, ie the bandgap energy of CdS [37]. Hence by incorporating Cd in NiS nanoparticles the bandgap energy can be reduced to a large extent, and also the absorption shifts from UV to visible region. Hence it can be used as light-sensitive materials. The compositions and bandgap energy of $Ni_xCd_{1-x}S$ (x = 0.8, 0.6, 0.4, and 0.2) nanoparticles are tabulated in Table 4. When more Cd is incorporated, the band gap decreases.

Table4. The sample, bandgap of $Ni_xCd_{1-x}S$ (x = 0.8, 0.6, 0.4,0.2) nanoparticles

Samples	Bandgap
$Ni_{0.8}Cd_{0.2}$ S	2 eV
$Ni_{0.6}Cd_{0.4c}S$	1.9 eV
$Ni_{0.4}Cd_{0.6c}S$	1.85 eV
$Ni_{0.2}Cd_{0.8}$ S	1.8 eV

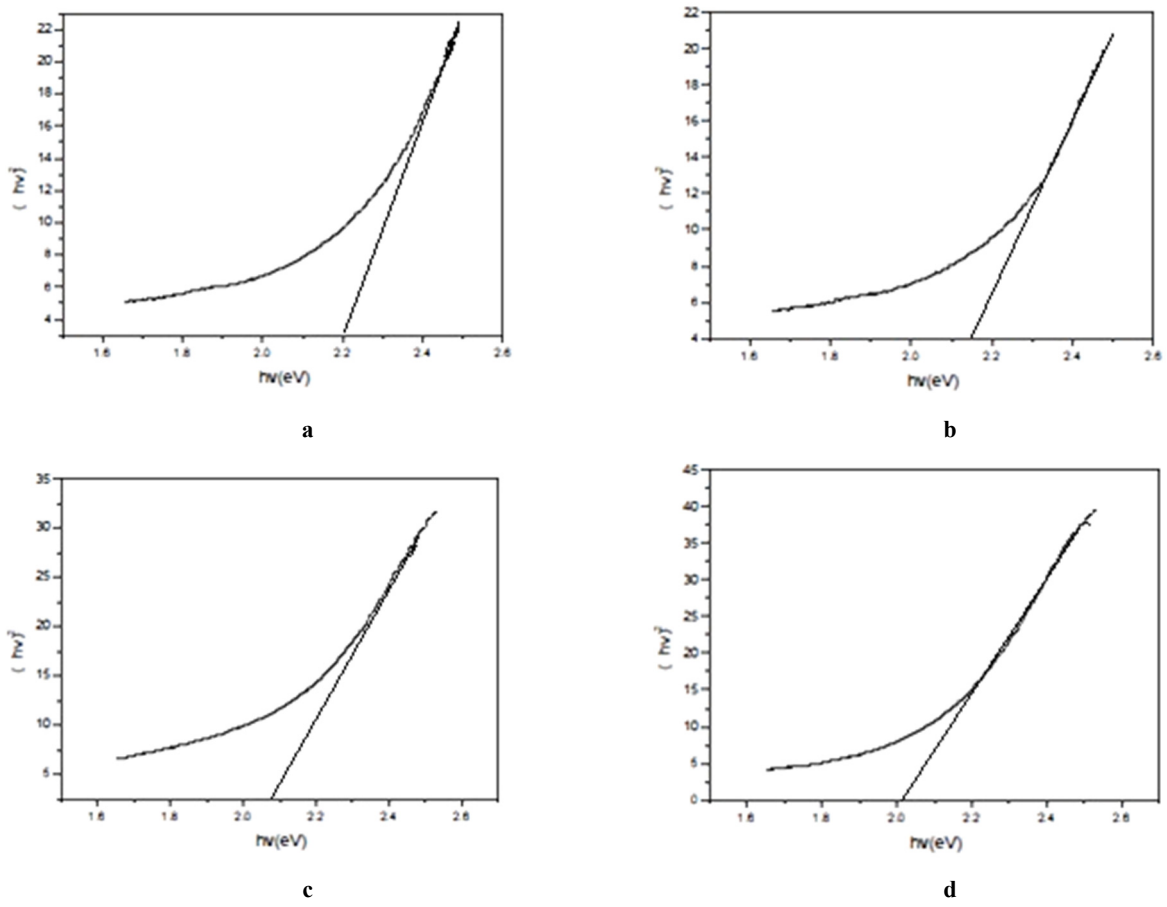

Figure 6. Tauc's plot of $Ni_xCd_{1-x}S$ nanoparticles
a) $Ni_{0.8}Cd_{0.2}S$ nanoparticles, b) of $Ni_{0.6}Cd_{0.4}S$ nanoparticles, c) $Ni_{0.4}Cd_{0.6}S$ nanoparticles, d) $Ni_{0.2}Cd_{0.8}S$ nanoparticles

PL Studies. The PL emission spectra of $Ni_xCd_{1-x}S$ nanoparticles (x = 0.8, 0.6, 0.4, and 0.2) are shown in Figure 7. As the composition of Cd increases, the emission spectrum shifts towards the higher wavelength, i.e., towards the emission wavelength of pure CdS [38]. From the luminous spectroscopy (Figure 7), it has been found that the emission peak shifts from 440 nm to 470 nm (close to the emission peak of CdS) [39]. when more Cd is incorporated. Figure 8 depicts the variation of peak positions with composition of $Ni_xCd_{1-x}S$ nanoparticles (x = 0.8, 0.6, 0.4, and 0.2). Because this variation is linear, $Ni_xCd_{1-x}S$(x = 0.8, 0.6, 0.4, and 0.2) nanoparticles can be tuned to emit different wavelengths in the range 440 nm – 470 nm by varying the Cd composition, and the sample composition can also be identified from the graph by observing the emission peak.

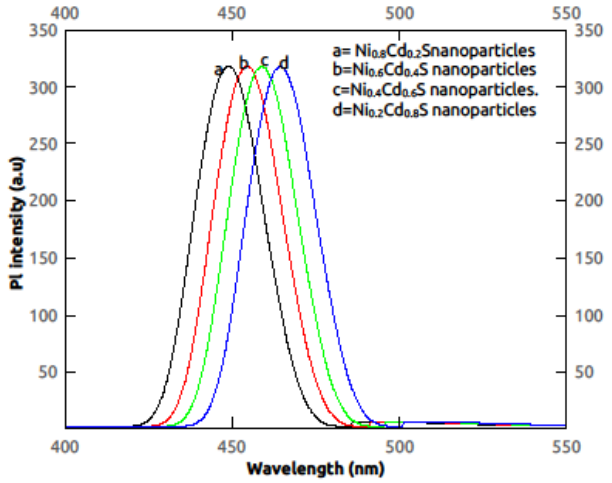

Figure 7. Pl emission spectra of $Ni_xCd_{1-x}S$ (x = 0.8,0.6,0.4,0.2) nanoparticles

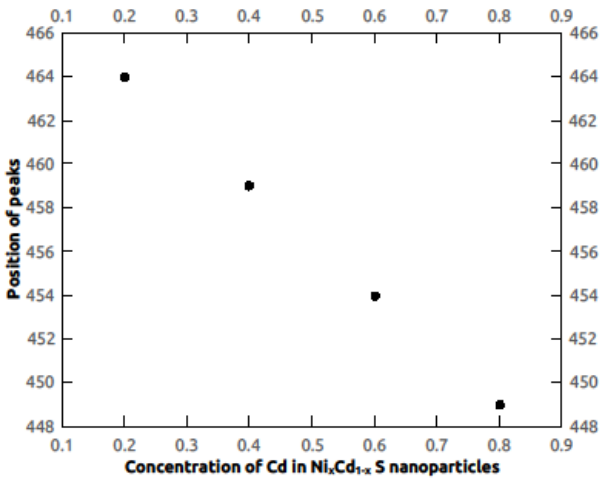

Figure 8. The variation of position of peaks with composition of $Ni_xCd_{1-x}S$ (x = 0.8,0.6,0.4,0.2) nanoparticles

4. CONCLUSION

We successfully synthesized $Ni_xCd_{1-x}S$ (x = 0.8, 0.6, 0.4, and 0.2) nanoparticles using microwave-assisted chemical precipitation, and the nanoparticles were characterized using XRD, SEM, and EDAX analyses. The DC electrical resistances were measured in the temperature range 300 K-500 K. All the samples undergo phase transition above a particular temperature. The behaviour of the samples at room temperature is entirely different from the behaviour of the samples after phase transition. From the absorption spectroscopy, it is clear that when the concentration of Cd is increased, the bandgap energy decreases. $Ni_xCd_{1-x}S$ (x = 0.8, 0.6, 0.4, and 0.2) nanoparticles can be tuned to emit different wavelengths in the range 440 nm–470 nm by varying the Cd composition, and the sample composition can also be identified from the graph by observing the emission peak.

Acknowledgements

The authors gratefully acknowledge Nesamony Memorial Christian College, Marthandam, for providing the necessary laboratory facilities to carry out this work.

Funding. This study was done towards the Ph.D. work of Mrs. Moly M. Rose and was not funded by any external agencies.
Competing Interests. We have not received any funding from any company or university, and hence, there is no conflict of interest.
Author Contributions. All authors discussed the results and contributed to the final manuscript.
Data availability statement. The data that support the findings of this study are available within the article

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СТРУКТУРНІ, ЕЛЕКТРИЧНІ ТА ОПТИЧНІ ДОСЛІДЖЕННЯ СИСТЕМИ НАНОЧАСТИНОК $Ni_xCd_{1-x}S$ (x = 0,8, 0,6, 0,4 і 0,2)

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У цій статті демонструється синтез наночастинок $Ni_xCd_{1-x}S$ (x = 0,8, 0,6, 0,4, 0,2) методом хімічного осадження за допомогою мікрохвиль. Підготовлені зразки охарактеризовані методами XRD, EDAX, SEM, UV-VIS та PL спектроскопії. Енергодисперсійний рентгенівський аналіз підтверджує існування нікелю, кадмію та сірки в належних співвідношеннях. Електричний опір постійному струму вимірювали в діапазоні температур 300 K-500 K. Криві температурного опору всіх зразків показують фазові переходи вище певної температури. УФ та ФЛ спектри всіх зразків були порівняні та досліджені.
Ключові слова: *наночастинки; хімічне осадження; фазовий перехід; електричні; оптичний; заборонена зона*