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

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$Zn_xCu_{1-x}S$ (x = 0.8, 0.6, 0.4 and 0.2) nanoparticles were synthesized by microwave assisted chemical precipitation method. The as-synthesized nanoparticles were characterized by X ray diffraction, SEM and TEM analysis to study the crystal structure, size and surface morphology. The energy dispersed x-ray analysis confirms the presence of Zinc, Copper and Sulphur in proper ratio. The D.C. electrical resistance was measured in the temperature range 300K-500K. All the samples show phase transition above a particular temperature. UV, PL and Raman spectra of all the samples were compared and studied.
Keywords: *chemical precipitation; structural; electrical; phase transition*
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1. Introduction

Zinc Sulphide is an important semiconductor material which has been extensively studied because of its physical and chemical properties. ZnS is a wide bandgap semiconductor with a band gap energy of 3.68eV [1,2]. Due to wide band gap, it is useful in optoelectronics [3] and sensors [4]. Zinc Sulphide nanoparticles have potential for various applications in the field of solar cells [5], displays [6], lasers [7] and light emitting diodes, [8] ZnS exist in two phases, ie's cubic phase and hexagonal phase [9]. Zinc Sulphide nanoparticles have been succesfully synthesized by different Methods such as sol-gel [10], sonochemical [11], microwave irradiation [12], microemulsion [13] solvothermal [14] and, hydrothermal [15].

CuS is typical p-type semiconductor that has a direct band gap of 2.5 eV [16-17]. CuS is a potential candidate that could be used in the areas of solar cell elements conversion, gas sensors, IR detectors, electrochemical cells, and photo catalysts [18-22]. The synthesis of copper sulphide has been achieved using different approaches such as microwave irradiation [23], hydrothermal [24], sol-gel [25], microemulsion [26], sonochemical [27], microwave assisted heating [28].

Phase transition in Zinc Sulphide nanoparticles were already studied by varying annealing temperature and pressure, from resistance measurements [29-33]. Phase transition in Copper Sulphide nanoparticles have been studied by previous works [34-35]. $Zn_xCu_{1-x}S$ (x = 0.8, 0.6, 0.4 and 0.2) nanoparticles have the application of both Zinc Sulphide nanoparticles and Copper Sulphide nanoparticles. W.Q Peng and G.W Cong investigated the room temperature photoluminescence of (ZnS: Cu) nanoparticles [36]. Jagadeep Kaur and Manoj Sharma studied the structural and optical studies of undoped and copper dopped Zinc Sulphide nanoparticles for photocatalytic application [37]. Chanchal Mondal performed Zns nanoflower promoted evolution of CuS/ZnS p-n heterojunction for exceptional visible light driven photocatalytic activity [38]. S. Harish and J. Archana investigated ultrafast visible light active ZnS/CuS nanostructured photocatalyst [39]. Vijayan et al studied High luminescence efficiency of Copper doped Zinc Sulfide (Cu: ZnS) nanoparticles towards LED applications [40].

In the present study we synthesized $Zn_xCu_{1-x}S$ (x = 0.8, 0.6, 0.4 and 0.2) nanoparticles by microwave assisted chemical precipitation method and studied their phase transition through D.C. electrical resistance measurements at various temperatures.

2. Experimental techniques

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

Zinc acetate, Copper acetate and Sodium Sulphide were used for the synthesis of $Zn_xCu_{1-x}S$ (x = 0.8, 0.6, 0.4 and 0.2) nanoparticles. Zinc acetate and Copper acetate were taken together in the required composition (1:2 molar ratio) and dissolved in 40 ml distilled water separately and mixed together. The amount of precursor materials taken to dissolve in 80 ml distilled water are given in Table 1. The sodium sulphide solution obtained by dissolving 6.14 gm in 40 ml of distilled water was added in drops to the above solution under effective stirring for 3 hours and kept undisturbed for one day. After performing precipitation, the precipitates were purified out several times, cleaned thoroughly with deionized water several times, and kept in a microwave oven. The solution was then subjected to microwave irradiation of 800 W for 20 minutes. The nanoparticles thus obtained were then brought to room temperature.

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The nanoparticles thus obtained were then cooled to room temperature. Finally, the $Zn_xCu_{1-x}S$ (x = 0.8, 0.6, 0.4 and 0.2) nanoparticles were annealed at 100^0C for 3 hours to get the phase pure $Zn_xCu_{1-x}S$ (x = 0.8, 0.6, 0.4 and 0.2) nanoparticles. The collected nanoparticles were used for different characterization.

Table 1. The amount of precursor materials taken to dissolve in 80 ml distilled water.

Sl no	**Expected composition**	**Zinc acetate**	**Copper acetate**
1	$Zn_{0.8}Cu_{0.2}S$	7.024 gm	1.59 gm
2	$Zn_{0.6}Cu_{0.4}S$	5.26 gm	3.19 gm
3	$Zn_{0.4}Cu_{0.6}S$	3.512 gm	4.79 gm
4	$Zn_{0.2}Cu_{0.8}S$	1.75 gm	6.38 gm

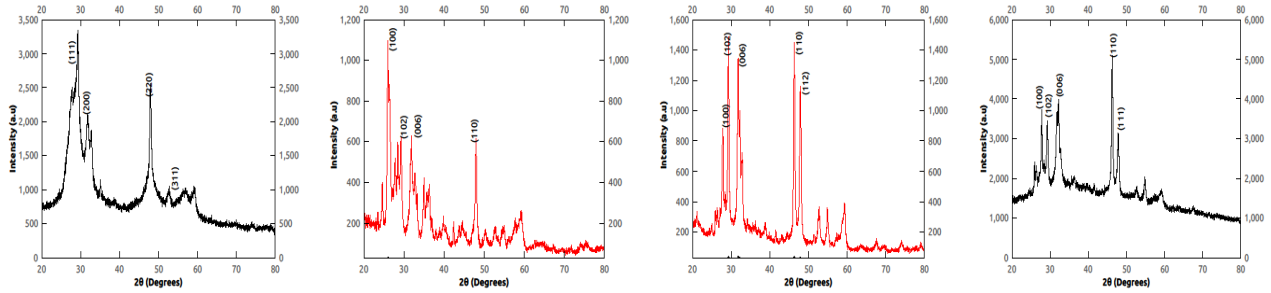
2.2. Instrumentation

X-ray diffraction (XRD) patterns of $Zn_xCu_{1-x}S$ (x = 0.8, 0.6, 0.4 and 0.2) nanoparticles were recorded on a powder X-ray diffractometer with Cu Kα radiation (λ = 1.54 Å) with 2θ ranging from angles 10° – 80°.Surface Morphology of the samples has been studied using TESCAN VEGA3 SBH Scanning Electron Microscope. The elements compositions were confirmed using an energy dispersive X-ray analysis (EDAX) set up attached with scanning electron microscope. A compressed collection of nanoparticles (pellet) was obtained by applying a high pressure of 10 tons/cm^2**.** Resistance of the pellet form of the samples were measured using four probe technique. Optical absorption spectra of the synthesized nanoparticles were recorded on UV-visible-spectrometer in the wave length range 200-900 nm. Photoluminescence measurements were performed on Varian Cary Eclipse Photoluminescence spectrophotometer in the range 300-650 nm. Raman spectrum of the $Zn_xCu_{1-x}S$ (x = 0.8, 0.6, 0.4 and 0.2) nanoparticles were recorded using peak Seeker Raman spectrometer.

3. Results and discussion

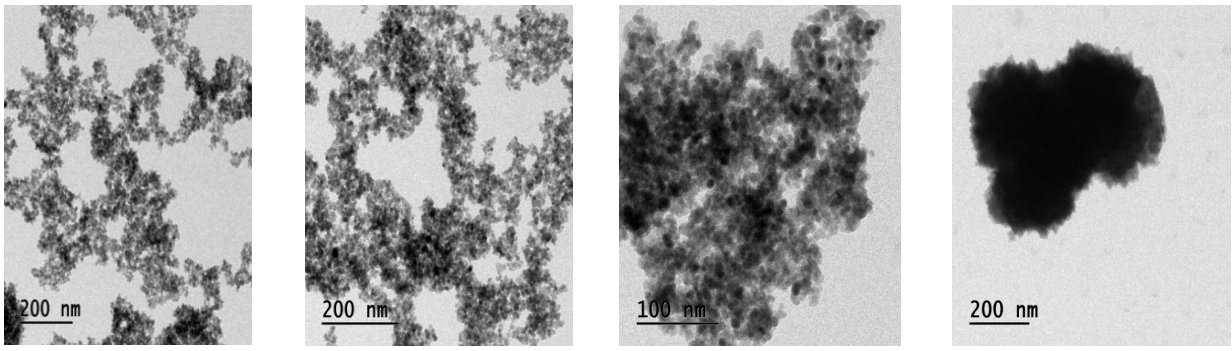
3.1. Structural studies

The indexed XRD patterns of as synthesized $Zn_xCu_{1-x}S$ (x = 0.8, 0.6, 0.4 and 0.2) nanoparticles prepared by microwave assisted chemical precipitation method are shown in Fig.1a, 1b, 1c, and 1d respectively. TEM images of all the samples are given in Fig. 2a, 2b, 2c and 2d respectively and it confirms the nanostructures. The respective EDAX images are depicted in Fig. 3a, 3b, 3c and 3d confirms the presence of Zinc, Copper and Sulphur in proper ratio. Samples, structure at room temperature, lattice parameters and particle size of the synthesized samples are given in the Table 2. As the content of Cu increases, the particle size also increases. The XRD patterns of the mixtures show that the mixtures may have the structure of any one of their components (ZnS or CuS).



a) **b)** **c)** **d)**

Figure 1. Indexed XRD patterns of nanoparticles
a) $Zn_{0.8}Cu_{0.2}S$ nanoparticles, b) $Zn_{0.6}Cu_{0.4}S$ nanoparticles, c) $Zn_{0.4}Cu_{0.6}S$ nanoparticles, d) $Zn_{0.2}Cu_{0.8}S$ nanoparticles



a) **b)** **c)** **d)**

Figure 2. TEM image of nanoparticles
a) $Zn_{0.8}Cu_{0.2}S$ nanoparticles, b) $Zn_{0.6}Cu_{0.4}S$ nanoparticles, c) $Zn_{0.4}Cu_{0.6}S$ nanoparticles, d) $Zn_{0.2}Cu_{0.8}S$ nanoparticles

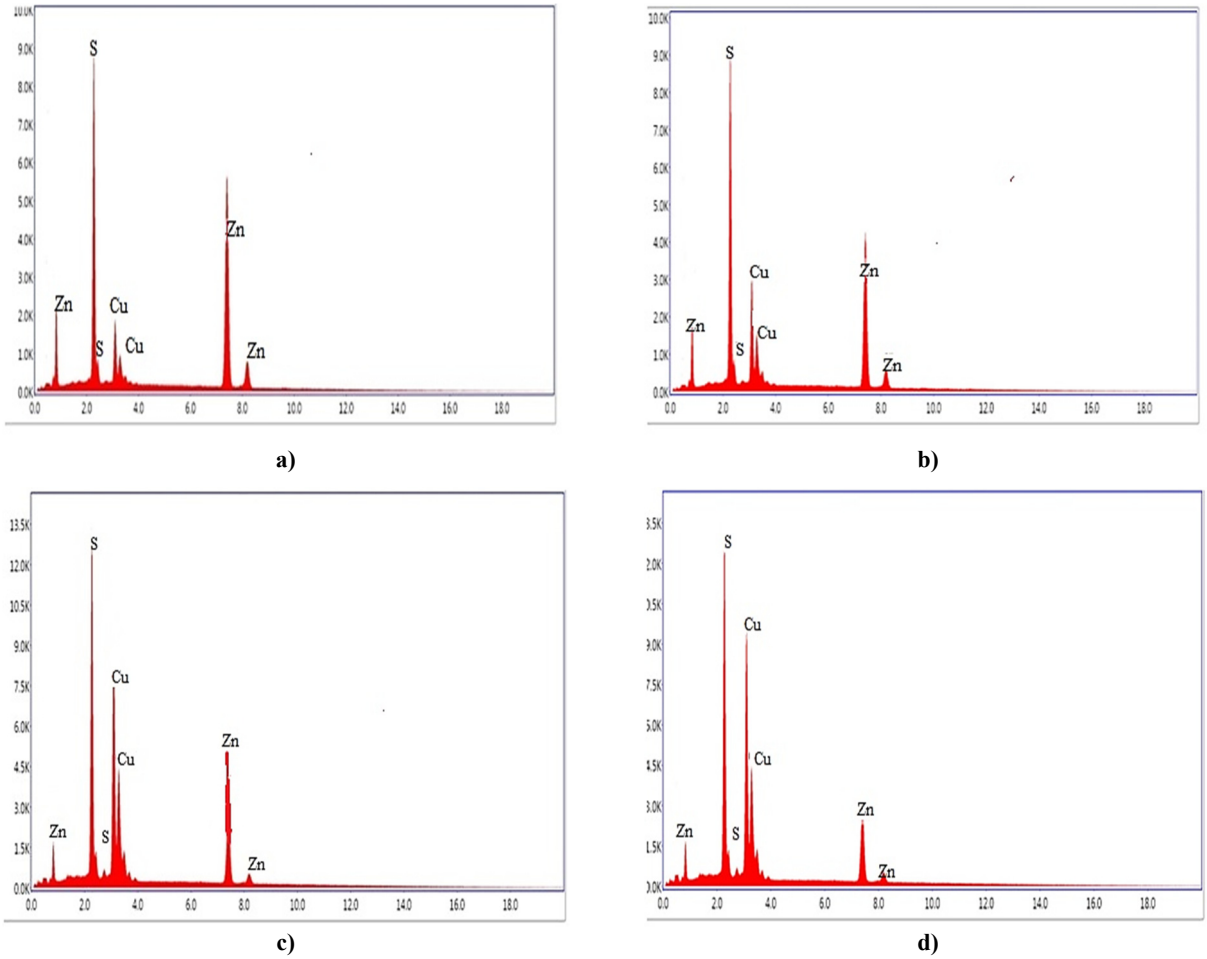


Figure 3. EDAX image of nanoparticles
a) $Zn_{0.8}Cu_{0.2}S$ nanoparticles, b) $Zn_{0.6}Cu_{0.4}S$ nanoparticles, c) $Zn_{0.4}Cu_{0.6}$ S nanoparticles, d) $Zn_{0.2}Cu_{0.8}$ S nanoparticles

Table 2. Samples, structure at room temperature, lattice parameters, particle size of synthesized Zn_xCu_{1-x} S(x = 0.8, 0.6, 0.4 and 0.2) nanoparticles

Sl no	Samples	Structure at room temperature	Lattice parameters	Particle size
1	ZnS(reported)	Face centered cubic	a=b=c=5.4 Å	
2	$Zn_{0.8}Cu_{0.2}S$	Face centered cubic	a=b=c=5.2 Å	31nm
3	$Zn_{0.6}Cu_{0.4}S$	Hexagonal	a=3.8 Å and c=17 Å	36nm
4	$Zn_{0.4}Cu_{0.6}S$	Hexagonal	a=3.9 Å and c=16.8 Å	39nm
5	$Zn_{0.2}Cu_{0.8}S$	Hexagonal	a=3.9 Å and c=16.62 Å	45nm
6	CuS (reported)	Hexagonal	a=3.8 Å and c=16.4 Å	

3.2. Electrical Studies

The D.C. electrical resistance of pellet form of the Zn_xCu_{1-x} S (x=0.8, 0.6, 0.4 and 0.2) nanoparticles synthesized were measured in the temperature range 300K-500K and is shown in Fig. 4a, 4b, 4c and 4d respectively. A discontinuity is observed in all the samples at a particular temperature due to phase transition [33]. The electrical properties of all the samples also change at this particular temperature. This change in electrical property is due to phase transition [41]. Behaviour of the sample at room temperature, order of resistance at room temperature, possible transition temperatures and behaviour of the sample after phase transition of Zn_xCu_{1-x} S (x=0.8, 0.6, 0.4, 0.2) nanoparticles are tabulated in Table 3.

The temperature resistance curve of $Zn_{0.8}Cu_{0.2}S$ nanoparticles (Fig. 4a) remains constant up to 450 K. In this region, the resistance of the sample is of the order of 10^9 Ohms, and the sample behaves as an insulator. Hence it can be utilized for the purpose of withstanding high resistance up to 450 K. As the resistance decreases rapidly with temperature above 450 K it can be used as a temperature sensor.

Fig. 5 shows the order of resistance with variations in the composition of Cu in $Zn_xCu_{1-x}S$ (x = 0.8, 0.6, 0.4 and 0.2) nanoparticles. As the composition of Cu in Zn_xCu_{1-x} S (x = 0.8, 0.6, 0.4 and 0.2) nanoparticles varies the order of resistance changes from 10^9 to 10^2 Ohms. R. Sheela Christy et al have already reported the rapid decrease in the resistance due to the incorporation of more Cu in CuS-Ag_2S nanoparticle system [42]. From the graph (Fig. 5), as the curve is linear, by varying the composition, the sample can be synthesised with the desired order of resistance for a particular purpose.

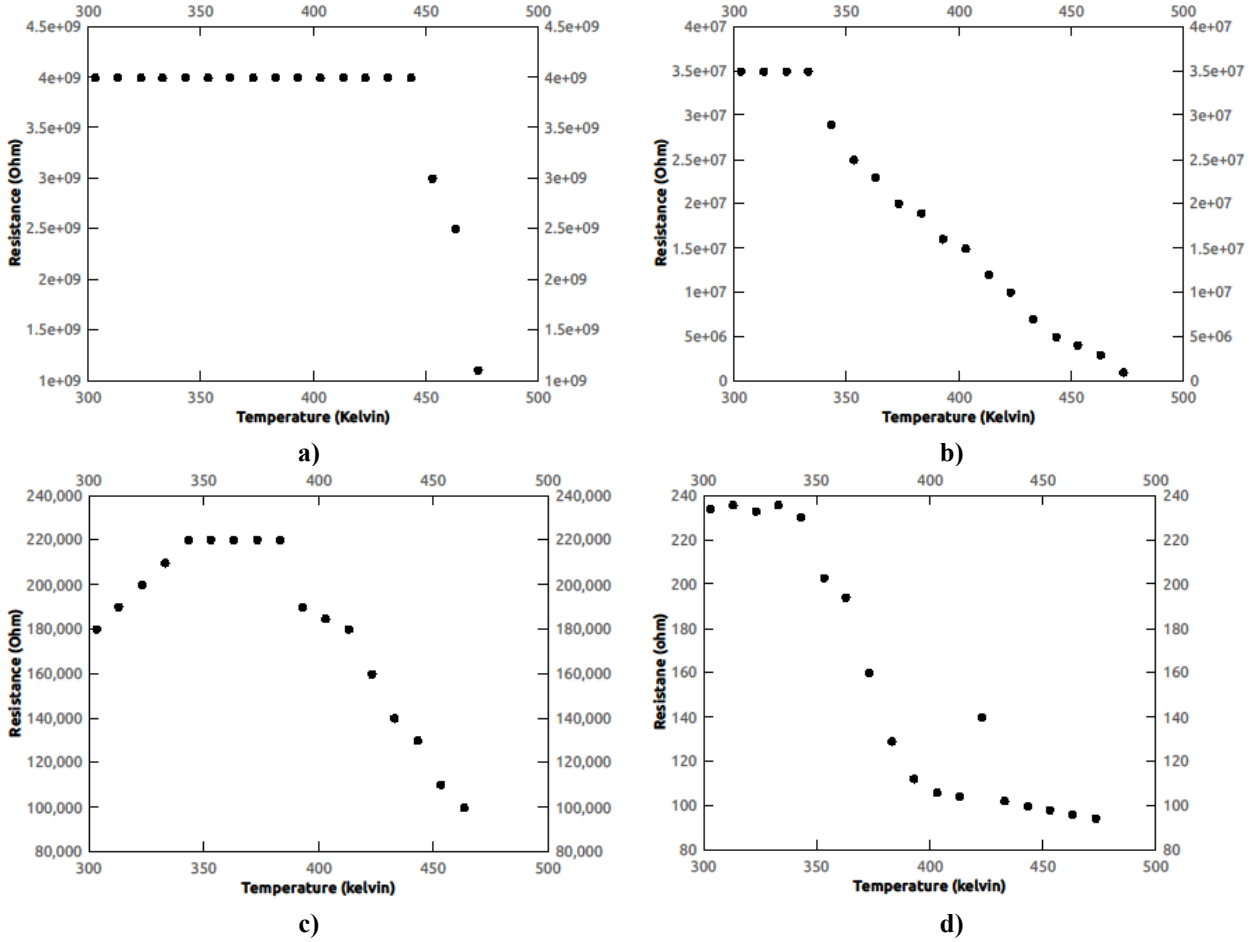


Figure 4. Temperature resistance curve of nanoparticles
a) $Zn_{0.8}Cu_{0.2}S$ nanoparticles, b) $Zn_{0.6}Cu_{0.4}$ S nanoparticles, c) $Zn_{0.4}Cu_{0.6}$ S nanoparticles, d) $Zn_{0.2}Cu_{0.8}$ S nanoparticles

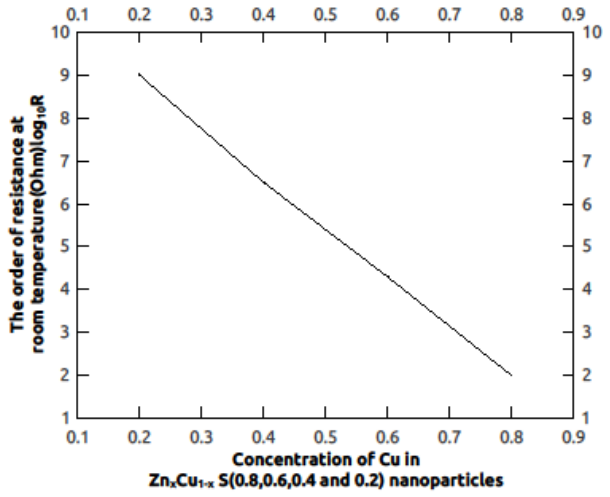


Figure 5. The variation of order of resistance with the composition of Cu in Zn_xCu_{1-x} S (x=0.8, 0.6, 0.4 and 0.2) nanoparticles

Table 3. Behaviour of the sample at room temperature, order of resistance at room temperature, possible transition temperatures and behaviour of the sample after phase transition of Zn_xCu_{1-x} S (x = 0.8, 0.6, 0.4 and 0.2) nanoparticles

Samples	Behaviour of the sample at room temperature	Order of resistance at room temperature	Possible transition temperatures	Behaviour of the sample after phase transition
$Zn_{0.8}Cu_{0.2}S$	Insulator	10^9 Ω	440K	Semiconductor
$Zn_{0.6}Cu_{0.4}S$	Insulator	10^7 Ω	340K	Semiconductor
$Zn_{0.4}Cu_{0.6}S$	Conductor	10^5 Ω	340K	Semiconductor
$Zn_{0.2}Cu_{0.8}S$	Oscillation	10^2 Ω	340K	Semiconductor

3.3. Optical studies

Fig. 6 shows the optical absorption spectra of $Zn_xCu_{1-x}S$ (x = 0.8, 0.6, 0.4 and 0.2) nanoparticles synthesised by microwave-assisted chemical precipitation method. From the absorption spectroscopy, it's clear that when more and more Cu is incorporated, the absorption edge gets shifted towards the lower wavelength region and the percentage of absorption also decreases. The samples can be synthesised with the desired absorption edge and percentage of absorption by varying the composition of Cu in the mixture for different applications.

Fig. 7 depicts the PL emission spectra of $Zn_xCu_{1-x}S$ (x = 0.8, 0.6, 0.4 and 0.2) nanoparticles synthesised by microwave-assisted chemical precipitation method. When more Cu is incorporated, the emission peaks shift from 390 nm to 370 nm. Fig. 8 depicts the variation of emission peak with composition of Cu in $Zn_xCu_{1-x}S$ nanoparticles (x = 0.8, 0.6, 0.4 and 0.2). Because this variation is linear, $Zn_xCu_{1-x}S$ (x = 0.8, 0.6, 0.4 and 0.2) nanoparticles can be tuned to emit different wavelengths in the range 390 nm – 370 nm by varying the composition of Cu, and the sample composition can also be identified from the graph by observing the emission peak.

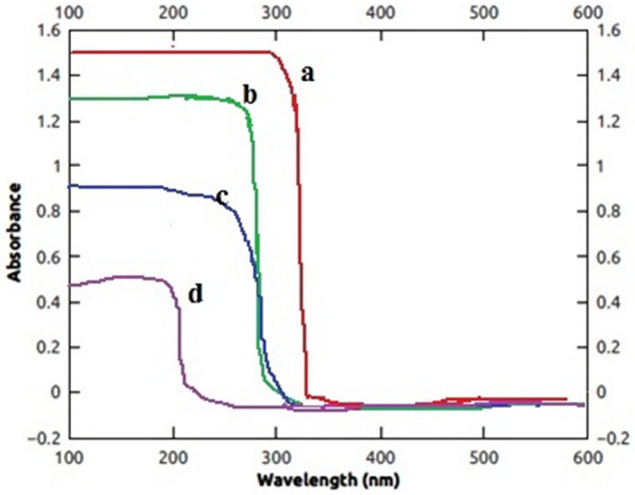


Figure 6. Optical absorption spectrum of nanoparticles a) $Zn_{0.8}Cu_{0.2}S$ nanoparticles, b) $Zn_{0.6}Cu_{0.4}S$ nanoparticles, c) $Zn_{0.4}Cu_{0.6}$ S nanoparticles, d) $Zn_{0.2}Cu_{0.8}S$ nanoparticles

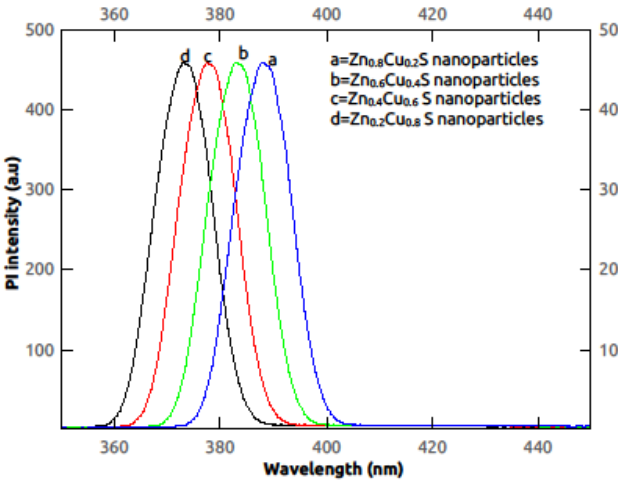


Figure 7. Emission spectra of $Zn_xCu_{1-x}S$ nanoparticles (x = 0.8, 0.6, 0.4 and 0.2)

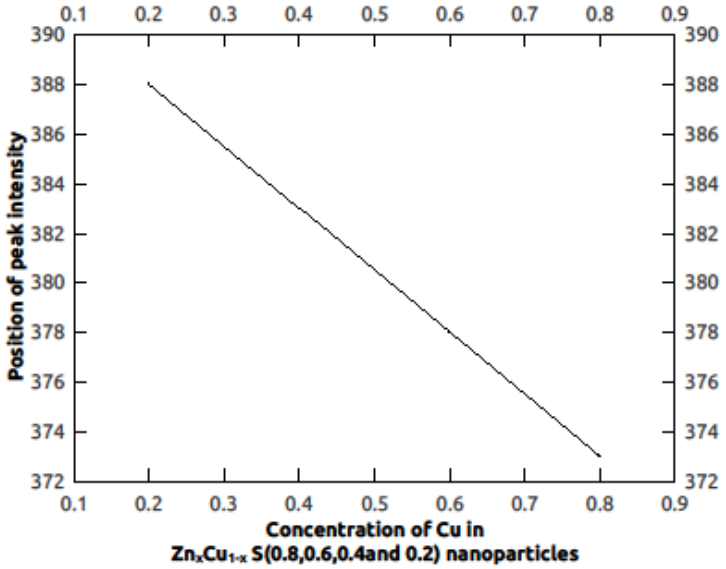


Figure 8. The plot for the variation of emission peak with the composition of Cu in Zn_xCu_{1-x} S (x = 0.8, 0.6, 0.4 and 0.2) nanoparticles synthesized by microwave assisted chemical precipitation method

3.4. Raman studies

Figures 9 a-d show the Raman spectra of $Zn_xCu_{1-x}S$ (x = 0.8, 0.6, 0.4 and 0.2) nanoparticles synthesised via microwave-assisted chemical precipitation method. The peaks are centered around 340 cm^{-1} and 400 cm^{-1}. When more Cu is incorporated, the intensity of the peaks keeps decreasing. The peak around 290 cm^{-1} was attributed to Cu-S bond vibration [43], and the peak around 400 cm^{-1} was attributed to Zn-S bond vibration [44].

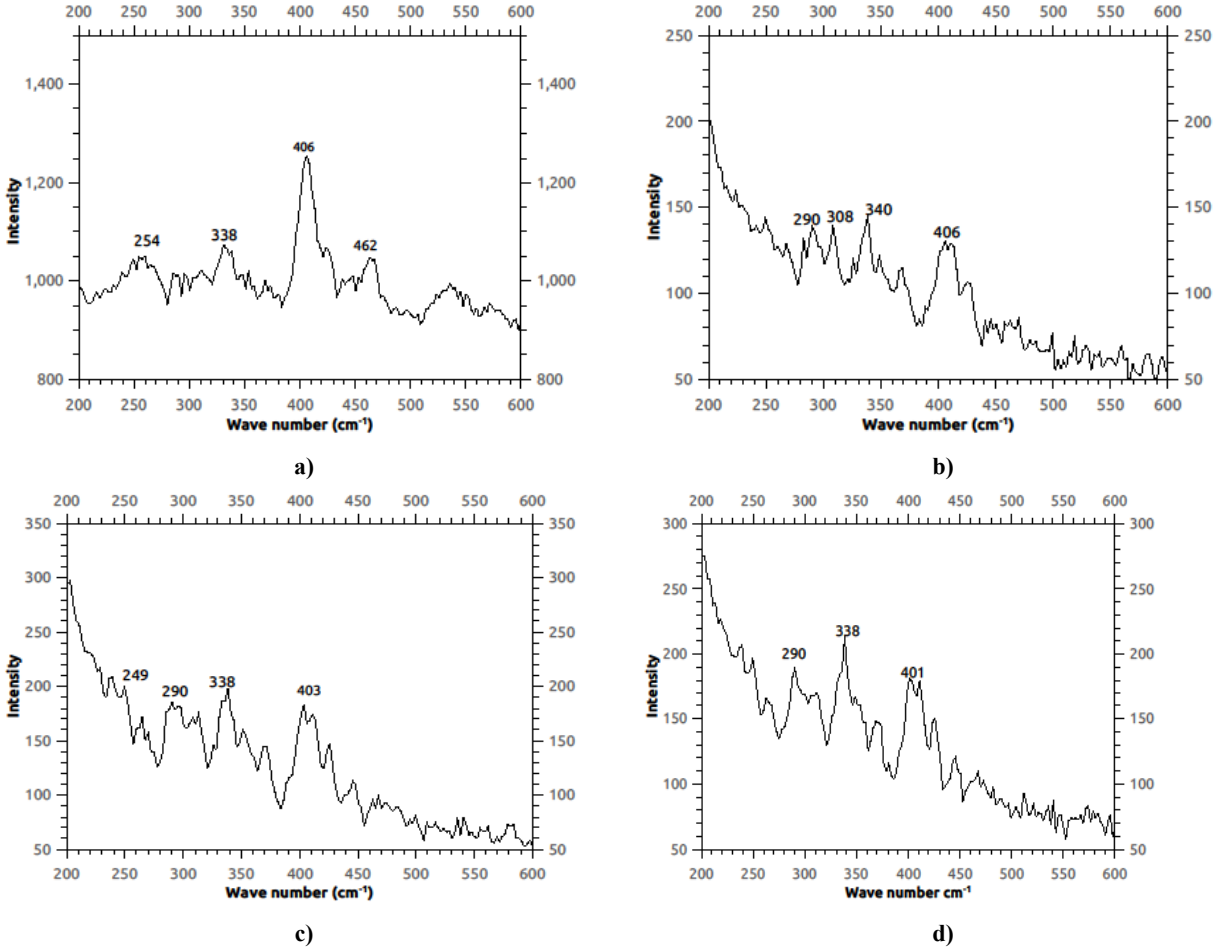


Figure 9. Raman spectrum of nanoparticles
a) $Zn_{0.8}Cu_{0.2}S$ nanoparticles, b) $Zn_{0.6}Cu_{0.4}S$ nanoparticles, c) $Zn_{0.4}Cu_{0.6}S$ nanoparticles, d) of $Zn_{0.2}Cu_{0.8}S$ nanoparticles

4. CONCLUSION

Zn_xCu_{1-x} S (x = 0.8, 0.6, 0.4 and 0.2) nanoparticles were synthesised by the microwave-assisted chemical precipitation method. The XRD patterns of the mixtures show that the mixtures may have the structure of any one of their components (ZnS or CuS). The electrical studies show that all the samples undergo phase transition above a particular temperature. Starting with $Zn_{0.8}Cu_{0.2}S$ nanoparticles, the electrical resistance of the samples is rapidly reduced as more and more Cu is incorporated. From the absorption spectroscopy, it's clear that as more and more Cu is incorporated, the absorption edge gets blue-shifted. By varying the composition of Cu, the mixture can be tuned to emit different wavelengths in the range of 390 nm to 370 nm. The peaks of both ZnS and CuS are present in the Raman spectra of the Zn_xCu_{1-x} S (x = 0.8, 0.6, 0.4 and 0.2) nanoparticles.

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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 НАНОЧАСТИНОК $Zn_xCu_{1-x}S$ (x = 0,8, 0,6, 0,4 та 0,2)

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Наночастинки $Zn_xCu_{1-x}S$ (x = 0,8, 0,6, 0,4 і 0,2) були синтезовані методом хімічного осадження за допомогою мікрохвиль. Синтезовані наночастинки були охарактеризовані за допомогою дифракції рентгенівських променів, SEM та TEM аналізу для вивчення кристалічної структури, розміру та морфології поверхні. Енергодисперсний рентгенівський аналіз підтверджує наявність цинку, міді та сірки в правильному співвідношенні. Електричний опір постійному струму вимірювали в діапазоні температур 300-500 К. Усі зразки демонструють фазовий перехід вище певної температури. УФ, ФЛ та спектри комбінаційного розсіювання всіх зразків були порівняні та досліджені.

Ключові слова: *хімічне осадження; структурний; електричні; фазовий перехід*