International Journal of
Scientific Research in Science and Technology (IJSRST)
Print ISSN : 2395-6011, Online ISSN : 2395-602X
International Conference on Advanced Materials
Held on 14, 15 December 2017, Organized by Department of Physics,
St. Joseph's College, Trichy, Tamilnadu, India

Spectroscopic Investigation of Laser Treated Nano Material Cadmium Sulphide (CdS)

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Abstract

The study of nano powder, particularly laser heat treatment of nano particles plays a major role in laser materials processing studies. The synthesis and charactersation techniques of nano particles attracted greater attention in material science due to their various applications. Cadmium sulphide is the important material used in solar cells as well as in electronics and photo optics. Now in this study we report the influence of laser heat treatment on nano powder CdS. The laser treated CdS samples are characterized and analysed by Xray diffraction, UVvisible absorption, FTIR spectra, Dielectric studies, DLS spectra and SEM. From the Xray analysis we observed that the laser treated CdS in with the structure of hexagonal and the crystalline size is in the range of 1127 nm. The UVvisible spectrum also confirms the presence of CdS nano particles with the average band gap energy of 5.1ev. The optical band gap values of CdS calculated are about 5.15.3ev. FTIR confirms the presence of CdS particles with various groups. Dielectric studies of laser treated CdS samples linear increase of values when the frequency increase (except for dipole laser heat treatment of samples for 30 minutes). Dynamic light scattering studies reveals that the particle size matches with Xray diffraction values that slightly differ with SEM studies.

1. Introduction

Cadmium sulphide nano particle belongs to the group of chalcogenides is a II–IV group semiconductor nano particles show size dependent, which finds number of applications particularly use in solar cells because of its high photo sensitivity. In recent years, greater attention has been given for synthesis and characterization of CdS because of its application in optical, electronic as well as thermo dynamic properties of particles.Several studies of CdS have been reported based on their size, shape and crystalline forms of particles. In general, the band gap energy is mainly based on particle size of the semiconducting particle[1]. With the use of laser heat treatment on semi conducting materials gives necessary enhancement either in conductivity or in band gap energy with the use of proper laser parameters [2]. This paper investigation the effect of laser irradiation on CdS nano particle with different time exposures. The structural analysis of the samples are analysed using Xray diffractometers. Similarly the spectroscopic investigations such as UV and FTIR are also investigated for with and without laser irradiated samples. Dielectric properties, DLS spectra and SEM studies show interesting information pertaining to band gap energy and particle size [3].

2. Experimental Procedure

Commercially available CdS powder (SigmaAldrich, 99.995% purity) is used for investigation (composition is given in Table1). Laser treatments are carried out by using 5.0mw low power HeNe gas laser with red light of (wavelength 633nm) and diode laser with green light of 5mw power (wave length 532nm). The structural analysis of with and without laser irradiated samples are analysed using PANalytical's Xray diffractometers (copper Kα radiation of wave length λ as 1.54060 A° and 1.54443A°). This system recorded the intensity as a function of Bragg's angle. While the average crystalline size of the particles can be estimated using full width at half maximum (FWHM) value of the xray diffraction peaks. The optical and spectroscopic investigation of the samples such as UV and FTIR

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studies are performed using Perkin Elmer UV/VIS spectrophotometer (λ 365) and Perkin Elmer FTIR spectrometer. The Dielectric studies are performed using Digital LCRZ Meter TH2816A (50Hz 200KHz) with various frequencies. The DLS spectra (Measuring the Particle Size Distribution) recorded by Particulate system Nano plus Zeta/nano particle analyzer instrument. SnO_2 nano particles are analysed using SEM (ZEISS SEM instrument) micrographs. The laser processing parameters used for laser irradiation of samples are given in Table2 and the properties of sample used is given in Table3.

Table 1. Chemical composition of CdS

Element	Content (in %)
Cadmium	77.81
Sulphide	22.19

Table 2. Laser processing parameters of samples

Sample Number	HeNe laser irradiation	Diode green laser irradiation
1	Without laser	Without laser
2	15min	
3	30min	
4		15min
5		30min

Table 3. Properties of CdS

Property	Characteristics
Solubility	soluble in acid very slightly soluble in ammonium
Magnetic susceptibility	-50.0×10^{-6} cm^3/mol
Density	4.826 g/cm^3,solid
Molar mass	144.47 g/mol

3. Results and Discussion

3.1. UVVisible studies

The absorption of UV radiations on the nano powder CdS sample with and without laser irradiation shows appreciable results. From the data the band gap energy for the laser treated sample varies from 5.1eV to 5.2eV and the sample without laser irradiation is 5.3eV. The result proves that the laser influences the nano particle CdS towards band gap energy, particularly for large laser irradiation time for the both HeNe and Diode lasers.Table4 shows the band gap energy values for different laser irradiation with two different time exposures. Figure 1(a) Figure 5(b) shows the UVVisible absorption spectrum of with and without laser irradiated samples [1].

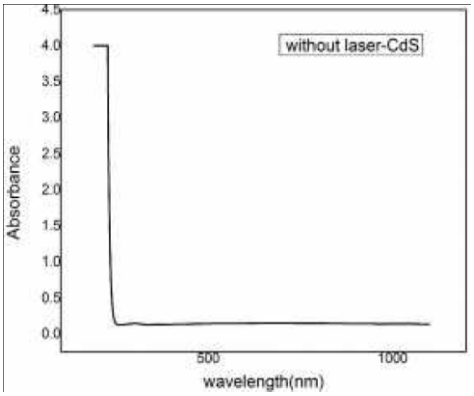

Figure 1(a). UVVis Absorption spectra cutoff wavelength (sample 1)

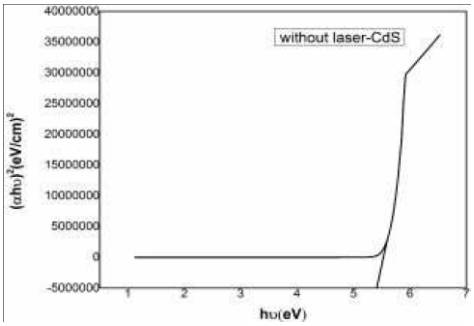

Figure 1(b). UVVis Absorption spectra band gap energy (sample 1)

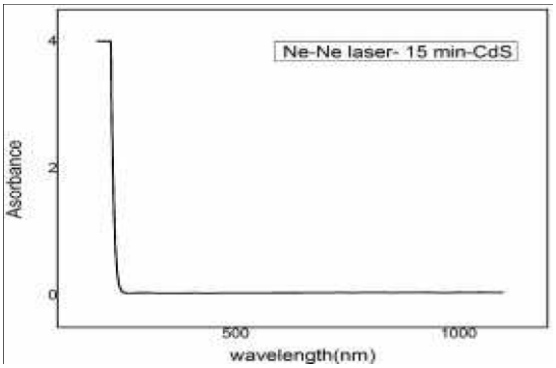

Figure 2(a). UVVis Absorption spectra cutoff wavelength (sample 2)

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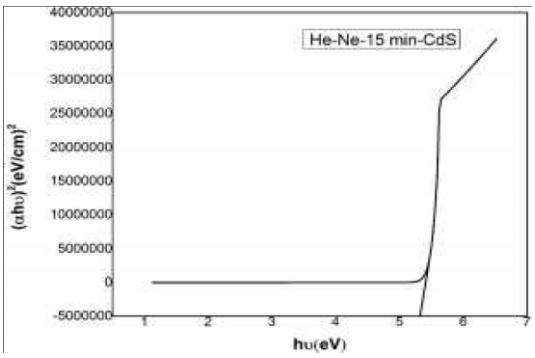

Figure 2(b): UVVis Absorption spectra band gap energy (sample 2)

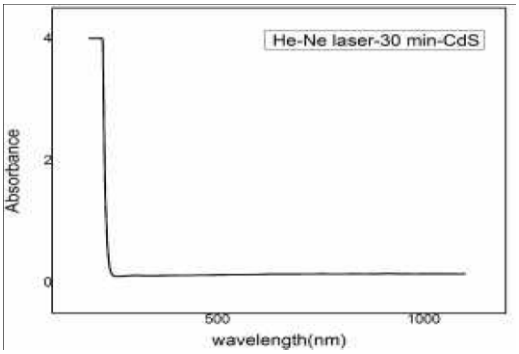

Figure 3(a). UVVis Absorption spectra cutoff wavelength (sample 3)

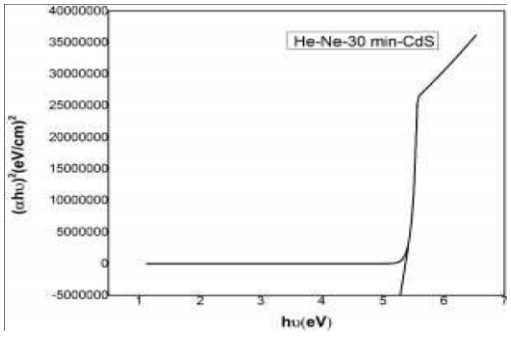

Figure 3(b). UVVis Absorption spectra band gap energy (sample 3)

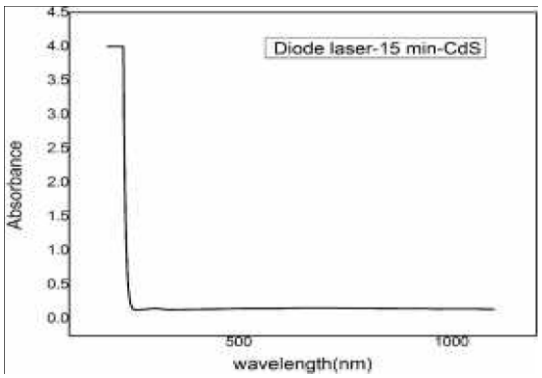

Figure 4(a). UVVis Absorption spectra cutoff wavelength (sample 4)

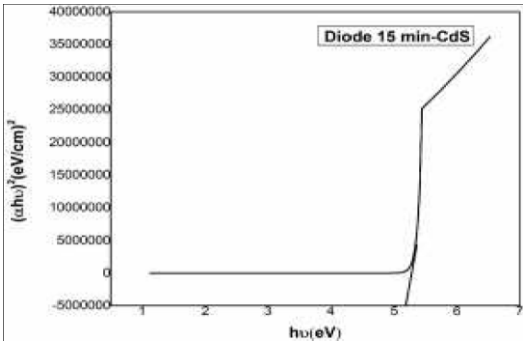

Figure 4(b). UVVis Absorption spectra band gap energy (sample 4)

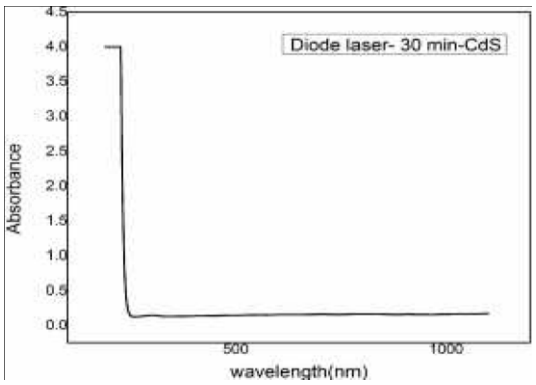

Figure 5(a). UVVis Absorption spectra cutoff wavelength (sample 5)

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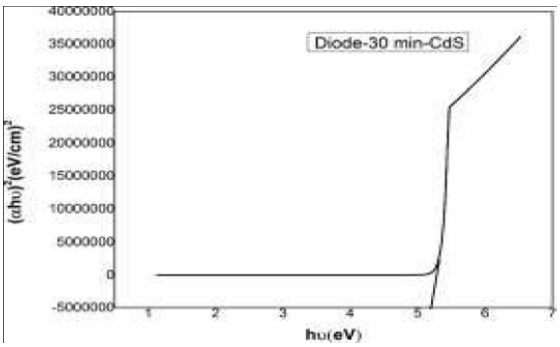

Figure 5(b). UVVis Absorption Spectra band gap energy (sample 5)

Table 4. Band Gap Energy Values For CdS Nanoparticles

Sample number	Time duration of laser irradiation (sec)	Band gap energy (eV)
1	Without laser irradiation	5.3
2	15 minutes (with HeNe laser irradiation)	5.2
3	30 minutes (with HeNe laser irradiation)	5.2
4	15 minutes (with Green Diode laser irradiation)	5.1
5	30 minutes (with Green Diode laser irradiation)	5.1

3.2. FTIR spectral studies

The FTIR spectrum of with and without laser treated CdS particles are given in Figure 6Figure 10. [2]

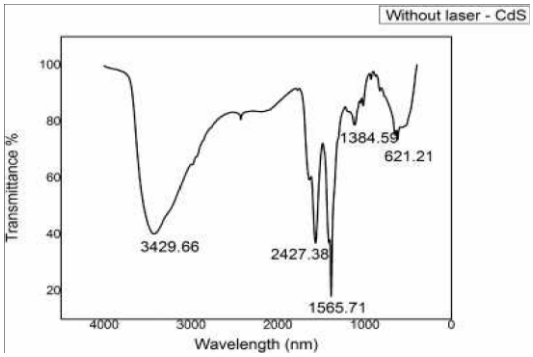

Figure 6: FTIR spectra (sample 1)

Table 5. FTIR functional analysis (Sample 1)

Group	Frequency	Vibrations	Intensity
OH (Hbonded)	3429.66	Broad Stretching	Strong
CH Alkanes	2427.38	Stretching	Strong
NH_2 Amines	1565.71	Scissoring bending	Mediumstrong
CH_2 Alkanes	1384.59	Deformation bending	Medium
SS Disulphide	621.21	Symmetry stretching	Weak

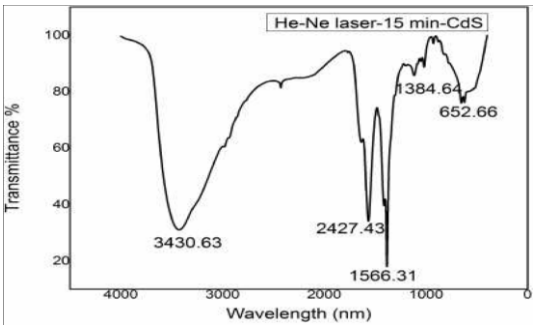

Figure 7: FTIR spectra (sample 2)

Table. 6. FTIR functional analysis (Sample 2)

Group	Frequency	Vibrations	Intensity
NH	3430.63	Stretching	Strong
NH Amines	2427.43	Stretching	Strong
CH	1566.31	Symmetry stretching	Strong
CX (iodoalkanes)	1384.64	Asymmetric stretching	Medium strong
CH_2 rocking	652.66	Bending	Weak

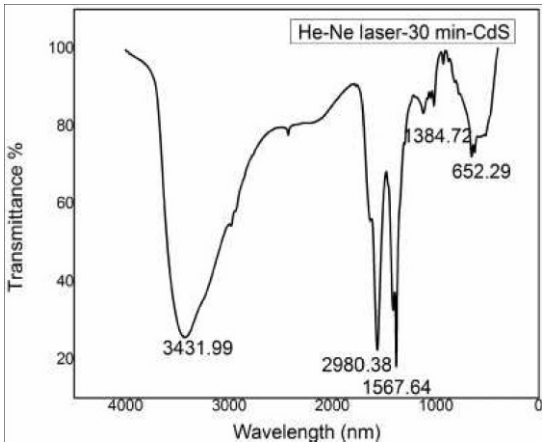

Figure 8: FTIR spectra (sample 3)

Table 7. FTIR functional analysis (Sample 3)

Group	Frequency	Vibrations	Intensity
NH	3431.99	Stretching	Strong
NH Amines	2980.38	Stretching	Strong
CH	1567.64	Symmetry	Strong

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		stretching	
CX (iodo alkanes)	1384.72	Asymmetric stretching	Medium strong
CH_2 rocking	652.29	Bending	Weak

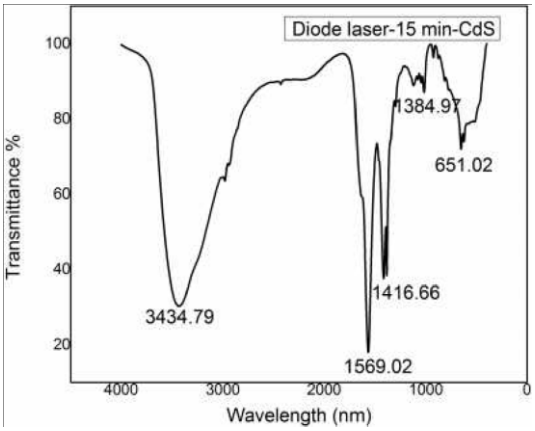

Figure 9. FTIR spectra (sample 4)

Table 8. FTIR functional analysis (Sample 4)

Group	Frequency	Vibrations	Intensity
NH (2 bonds)	3434.79	Stretching	Strong
NH_2 Amines	1569.02	Scissoring bending	Mediumstrong
α CH_2 (ketones)	1416.66	Bending	Strong
CX (bromo alkanes)	1384.97	Stretching	Mediumstrong
CH_2	651.02	Bending	Weak

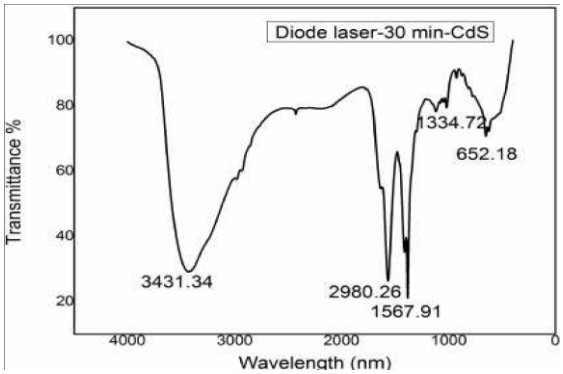

Figure 10. FTIR spectra (sample 5)

Table 9. FTIR functional analysis (Sample 5)

Group	Frequency	Vibrations	Intensity
NH (2 bonds)	3431.34	Stretching	Strong
NH_2 Amines	2980.26	Scissoring bending	Mediumstrong
S=0 (sulfoxide)	1567.91	Stretching	Strong
CX(bromo alkanes)	1334.72	Stretching	Mediumstrong
CH_2	652.18	Bending	Weak

3.3. XRay Diffraction Analysis

The Xray diffraction pattern of with and without laser treated samples are shown in Figure 11 – Figure 13.

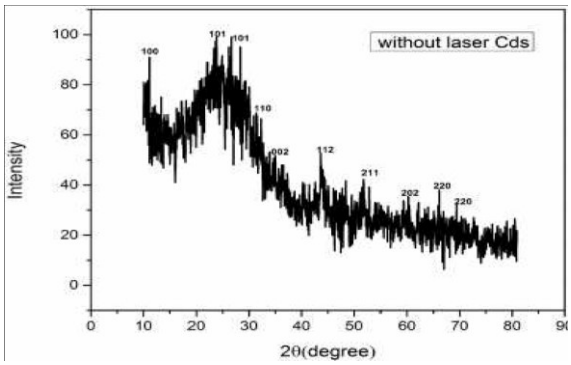

Figure 11. XRD spectrum (sample 1)

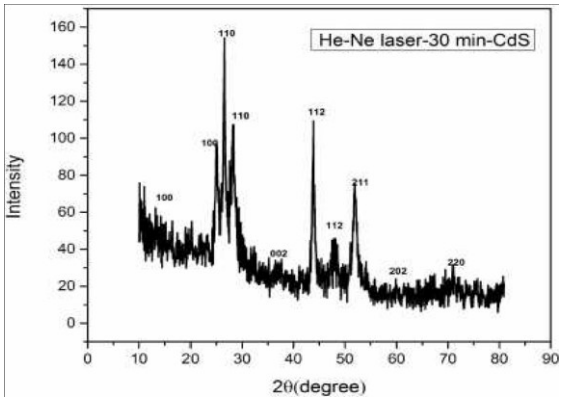

Figure 12. XRD spectrum (sample 3)

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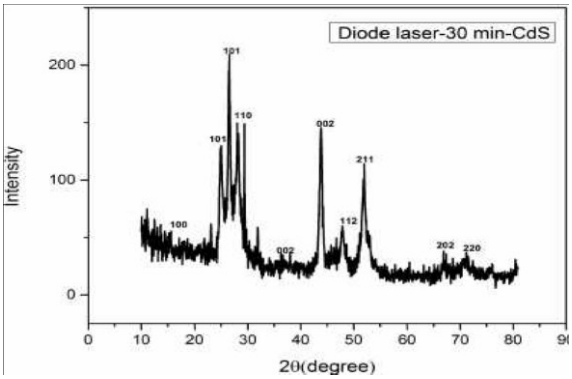

Figure 13. XRD spectrum (sample 5)

From the Xray diffraction studies, the structure of the CdS nano particle observed is hexagonal structure.The average particle size (D) was determined using the Scherer's equation [3]

$$D = K\lambda / \beta \cos\theta,$$

where, D is the crystallite size, K is the shape factor, being equal to 0.9, λ is the Xray wavelength, β is the full width at half maximum of the diffraction peak, and θ is the Bragg diffraction angle in degree. The average crystallite grain size calculated using Scherrer formula of CdS[3] of samples is given in Table 6.

Table 10. crystallite grain size of CdS samples

Sample No	Sample	Grain size (nm)
1	Without laser CdS	11.3
2	HeNe laser15minCdS	15.4
3	HeNe laser30minCdS	15.9
4	Diode laser15minCdS	26.8
5	Diode laser30minCdS	27.3

From Table10 we observed that calculated grain size is 27.3nm in sample5(Diode laser irteraction with time exposures of 30mins) which is slightly higher than the other samples.

3.4. Dielectric studies

The dielectric studies of laser treated nano particles CdS are measured using LCRZ instrument for various frequencies at room temperature (starting from 50Hz to 200 KHz).the corresponding effective capacitance (Cp) and effective resistance (Rp) are measured. Finally the dielectric constants, dielectric loss and ac electrical conductivity of the samples were calculated using the expression [4].

$$\sigma_{ac} = (f \varepsilon \tan(\delta)) / (1.8 \times 10^{10}), \quad (1)$$

where, f is the frequency applied field in Hz, (ε) is the dielectric constant or relative permittivity and tan(δ) is the dielectric loss tangent or loss factor.

$$\text{Dielectric constant } (\varepsilon) = (Cp\, L) / (\Sigma_o\, A), \quad (2)$$

where, Cp is the measured capacitance, L is the thickness of the sample, A is the electrode area and (Σ_o) is the permittivity of free space (8.854 X 10^{12} F/m).

The dielectric loss factor i.e. tan (δ) can be expressed by relation,

$$\tan(\delta) = \omega \cdot Cp.Rp, \quad (3)$$

where, Cp is the measured capacitance, Rp is the measured resistance and ω is the angular frequency. From the Fig.14(a)Fig.18(c) shows the graphical representation of dielectric constant, dielectric loss and a.c electrical conductivity.

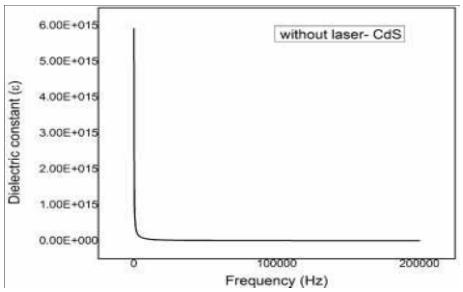

Figure 14(a). Dieletric curve of Dielectric constant (Sample 1)

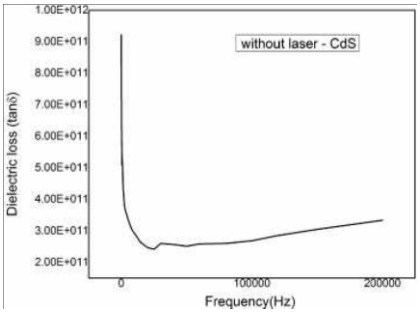

Figure 14(b): Dieletric curve of Dielectric loss (Sample 1)

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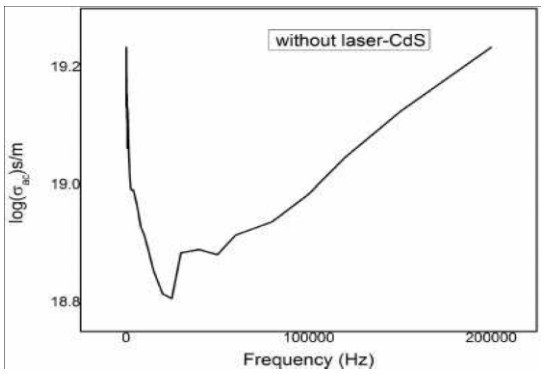

Figure 14(c). Dieletric curve of a.c electrical conductivity (Sample 1)

Table 11. Dielectric studies (Sample 1)

Frequency (KHz)	Dielectric constant (ε)	Dielectric loss (tanδ)	a.c conductivity (σ_{ac}) 10^5 s/m
10	5.0353 x10^{13}	2.9304x10^{11}	18.91368
50	1.0887x10^{13}	2.51429x10^{11}	18.88103
100	6.4643x10^{12}	2.68714x10^{11}	18.98454
150	5.2394x10^{12}	3.0492x10^{11}	19.12429
200	4.6270x10^{12}	3.33394x10^{11}	19.23402

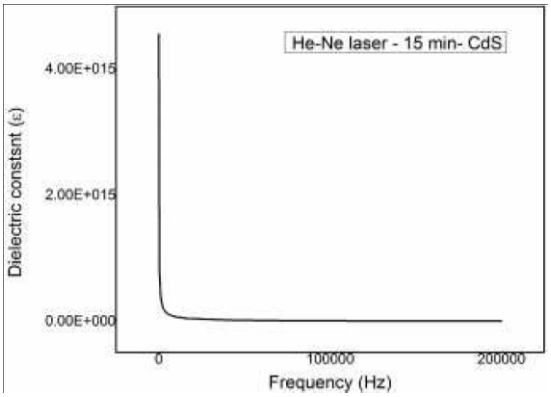

Figure 15(a). Dieletric curve of Dielectric constant (Sample 2)

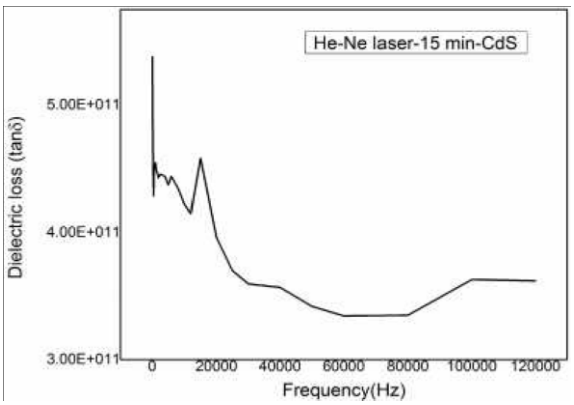

Figure 15(b). Dieletric curve of Dielectric loss (Sample 2)

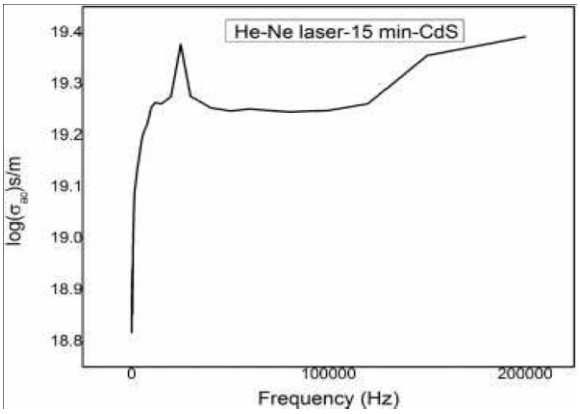

Figure 15(c). Dieletric curve of a.c electrical conductivity (Sample 2)

Table 12. Dielectric studies (Sample 2)

Frequency (KHz)	Dielectric constant (ε)	Dielectric loss (tanδ)	a. c conductivity (σ_{ac}) 10^5 s/m
10	7.2808x10^{13}	4.43897x10^{11}	19.25419
50	1.7691x10^{13}	4.22777x10^{11}	19.24722
100	9.5263x10^{12}	3.344x10^{11}	19.24792
150	7.4849x10^{12}	3.63x10^{11}	19.35492
200	6.1240x10^{12}	3.62057x10^{11}	19.39158

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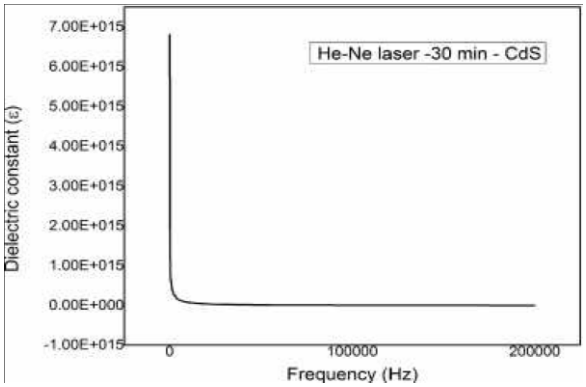

Figure 16(a). Dieletric curve of Dielectric constant (Sample 3)

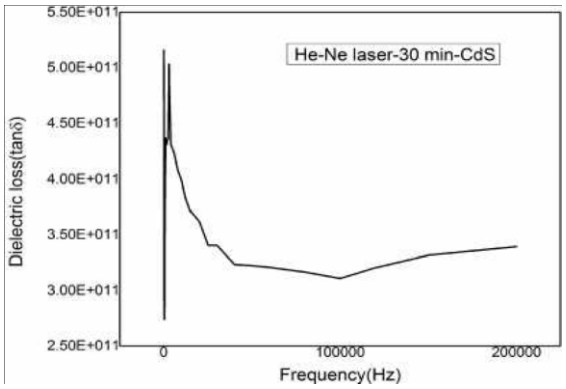

Figure 16(b). Dieletric curve of Dielectric loss (Sample 3)

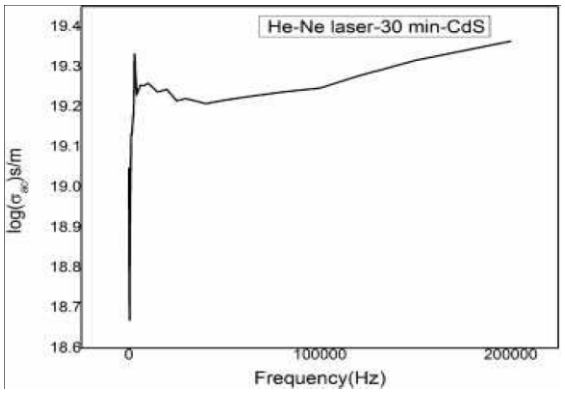

Figure 16(c). Dieletric curve of a.c electrical conductivity (Sample 3)

Table13: Dielectric studies (Sample 3)

Frequency (KHz)	Dielectric constant (ε)	Dielectric loss (tanδ)	a. c conductivity (σ_{ac})10^5 s/m
10	$8.1654x10^{13}$	$3.99771x10^{11}$	19.25852
50	$1.8372x10^{13}$	$3.22457x10^{11}$	19.21633
100	$1.0206x10^{13}$	$3.1143x10^{11}$	19.24658
150	$7.4849x10^{13}$	$3.31886x10^{11}$	19.316
200	$6.1240x10^{12}$	$3.39429x10^{11}$	19.36355

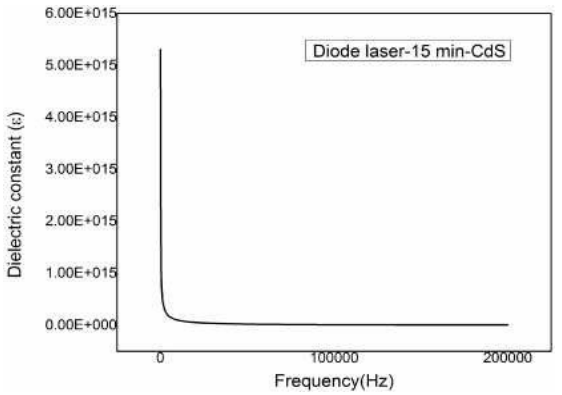

Figure 17(a). Dieletric curve of Dielectic constant (Sample 4)

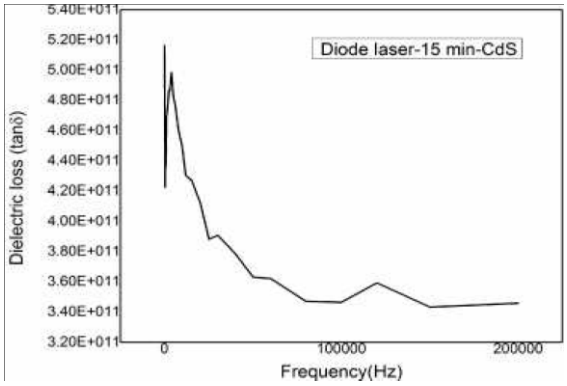

Figure 17(b). Dieletric curve of Dielectric loss (Sample 4)

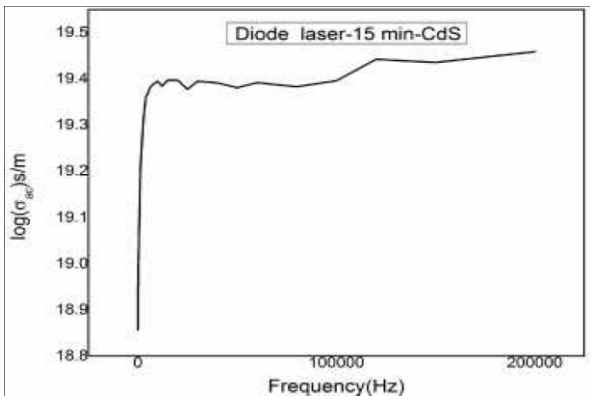

Figure 17(c). Dieletric curve of a.c electrical conductivity (Sample 4)

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Scientific Research in Science and Technology (IJSRST)
Print ISSN : 2395-6011, Online ISSN : 2395-602X
International Conference on Advanced Materials
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Table 14. Dielectric studies (Sample 4)

Frequency (KHz)	Dielectric constant (ε)	Dielectric loss (tanδ)	a.c conductivity (σ_{ac}) 10^5 s/m
10	$9.9346x10^{13}$	$4.4968x10^{11}$	19.39478
50	$2.3815x10^{13}$	$3.63x10^{11}$	19.38047
100	$1.3928x10^{13}$	$3.4633x10^{11}$	19.39579
150	$9.5263x10^{12}$	$3.432x10^{11}$	19.43529
200	$7.4849x10^{12}$	$3.4571x10^{11}$	19.45867

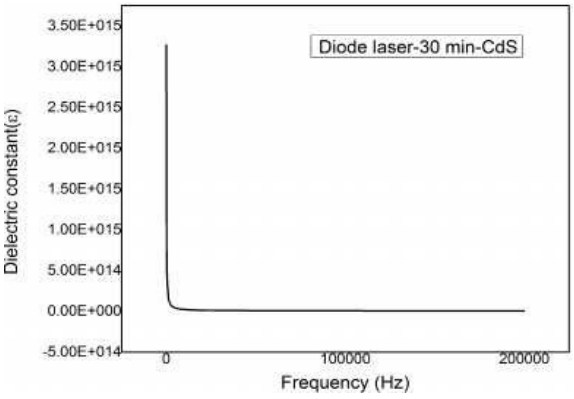

Figure 18(a). Dieletric curve of Dielectric constant (Sample 5)

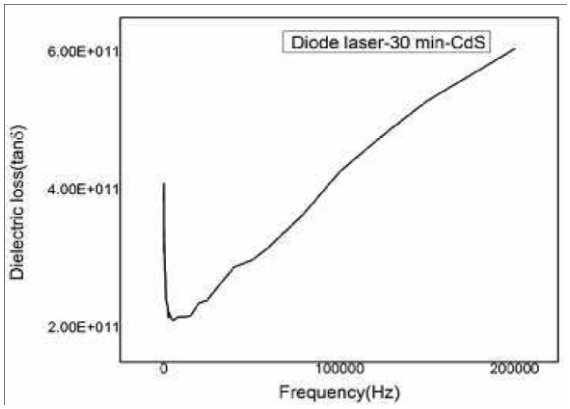

Fig 18(b): Dieletric curve of Dielectric loss (Sample 5)

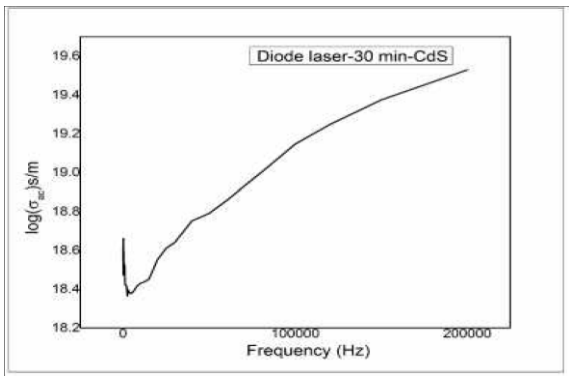

Figure 18(c). Dieletric curve of a.c electrical conductivity (Sample 5)

Table 15. Dielectric studies (Sample 5)

Frequency (KHz)	Dielectric constant (Σ)	Dielectric loss (tanδ)	a. c conductivity (σ_{ac}) 10^5 s/m
10	$2.2455x10^{13}$	$2.1572x10^{11}$	18.291934
50	$7.48498x10^{12}$	$2.9731x10^{11}$	18.7911
100	$5.91994x10^{12}$	$4.2654x10^{11}$	19.14701
150	$5.37558x10^{12}$	$5.2884x10^{11}$	19.37458
200	$5.03535x10^{12}$	$6.0468x10^{11}$	19.52932

The result shows liner decrease in dielectric constant values for increasing frequency and linear increase in dielectric loss for laser treated CdS nano particles(reverse action for without laser irradiated samples) and for a.c electrical conductivity linear increase for with and without laser irradiated samples when the frequency increases.

3.5. DLS Spectra (Measuring the Particle Size Distribution)

The DLS spectra of with and without laser treated samples are shown in Figure 19 – Figure 21.[5]

International Journal of
Scientific Research in Science and Technology (IJSRST)
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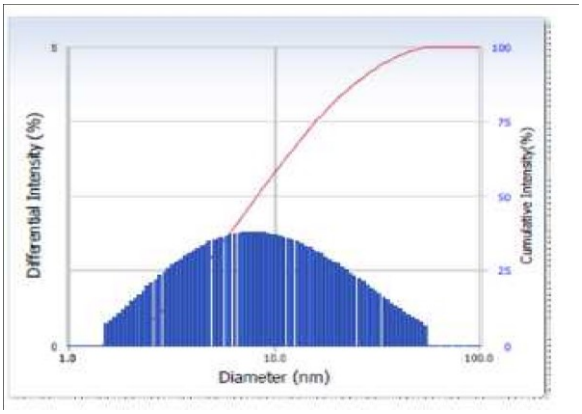

Figure 19. DLS spectra (sample 1)

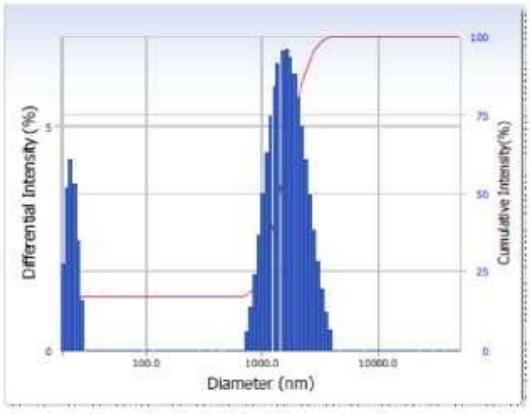

Figure 20. DLS spectra (sample 3)

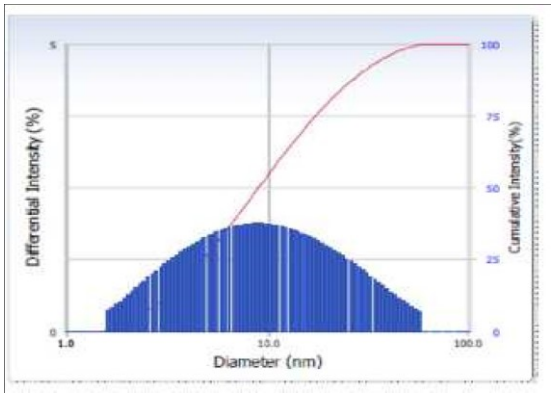

Figure 21. DLS spectra (sample 5)

Dynamic light scattering (DLS) studies revealed that the particle size distribution of Cadmium sulphide(CdS) nanoparticles are 2.1nm(sample1), 16.52nm(sample3), and 1.8 nm (sample 5).

3.6. SEM Analysis

The SEM Analysis of with and without laser treated samples are shown in Figure 22 – Figure 24. [6]

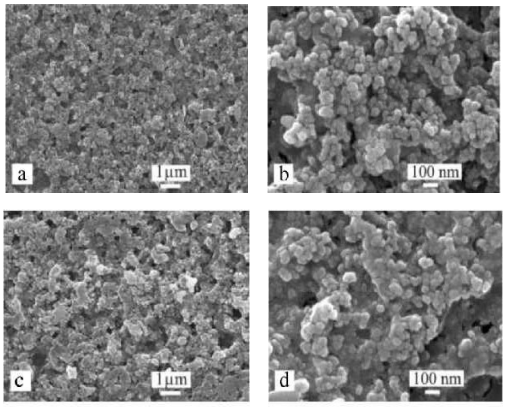

Fig 22: SEM image (sample1)

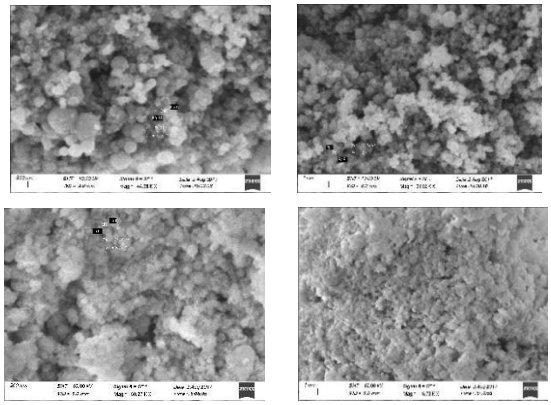

Figure 23. SEM image (sample3)

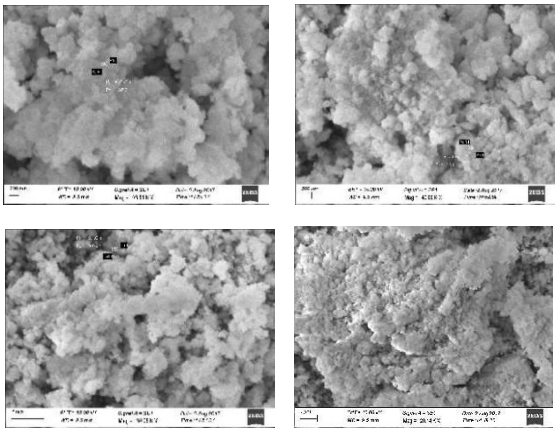

Figure 24. SEM image (sample5)

SEM analysis of Cadmium sulphide (CdS) Nano Particle, it is observed that the particles are in the

International Journal of
Scientific Research in Science and Technology (IJSRST)
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hexagonal shape within the particle size are in the range about 20 127nm (sample1), 34.9680.31nm (sample3), and 50.4867.02 nm (sample5). The average particle size observed in both SEM and XRD measurements are slightly different values.

Table 16. comparative study of particle size for Cadmium sulphide (CdS) Nanoparticle (sample 1, 3 and 5)

Sample	XRD Crystallite (nm)	DLS Particle (nm)	SEM Particle (nm)
1	11.3	2.1	20 – 127
3	15.88	16.52	34.96 –80.31
5	27.27	1.8	50.48 –67.02

4. Conclusion

In this, we investigated and reported the effect of laser heat treatment (HeNe laser and Diode laser) on nano particles CdS. The results observed are given below;

i. From the UVvisible spectrum studies, we observed thatthe band gap energy (for various laser exposures) from 5.1 eV – 5.2 eV (5.3 for sample1 without laser irradiation).
ii. The functional groups for different peaks in FTIR are analyzed and the type of vibration as well as intensity are studied.
iii. From the Xray diffraction studies, the structure of CdS nano particle (with and without laser irradiation) is hexagonal structure.
iv. The crystallite grain size of the samples calculated are 11.3 (sample1), 15.42 (sample2), 15.88 (sample3), 26.78 (sample4) and 27.27 nm (sample5) were observed from the XRD studies.
v. The dielectric properties of laser treated CdS nano particles and their relation with frequency, dielectric constant, dielectric loss and a.c electrical conductivity are investigated.
vi. Dynamic light scattering (DLS) studies revealed that the particle size distribution of Cadmium Sulphide CdS nanoparticles are 2.1nm (without laser), 16.52nm (HeNe laser treated with 30min), and 1.8 nm (Diode laser treated with 30min).
vii. From SEM analysis of Cadmium sulphide (CdS) Nano Particle, it is observed that the particles are in the hexagonal shape within the particle size are in the range about 20127nm (without laser),34.9680.31nm (HeNe laser treated with30min), and 50.4867.02 nm (Diode laser treated with30min)

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