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Amino acids (L-arginine and L-alanine) passivated CdS nanoparticles: Synthesis of spherical hierarchical structure and nonlinear optical properties



S.S. Talwatkar ^a, Y.S. Tamgadge ^b, A.L. Sunatkari ^c, A.B. Gambhire ^d, G.G. Muley ^{e,*}

- ^a Department of Physics, D K Marathe and N G Acharya College, Chembur, Mumbai, MS 440071, India
- ^b Department of Physics, Shri Shivaji College of Arts, Commerce & Science, Akola, MS 444003, India
- ^c Department of Physics, Siddharth College of ACS, Fort, Mumbai, MS 440001, India
- ^d Department of Chemistry, Shri Anand College of Science, Pathardi, Ahemadnagar, MS 414102, India
- ^e Department of Physics, Sant Gadge Baba Amravati University, Amravati, MS 444602, India

ARTICLE INFO

Article history: Received 31 May 2014 Received in revised form 24 September 2014 Accepted 30 September 2014 Available online 2 October 2014

Keywords: Amino acid CdS nanoparticles Powder XRD Photoluminescence 7-scan

ABSTRACT

CdS nanoparticles (NPs) passivated with amino acids (L-alanine and L-arginine) having spherical hierarchical morphology were synthesized by room temperature wet chemical method. Synthesized NPs were characterized by ultraviolet—visible (UV—vis) spectroscopy to study the variation of band gaps with concentration of surface modifying agents. Increase in band gap has been observed with the increase in concentration of surface modifying agents and was found more prominent for CdS NPs passivated with L-alanine. Powder X-ray diffraction (XRD) and transmission electron microscopy (TEM) analysis were carried out for the study of crystal structure and morphology of CdS NPs. The average particle size of CdS NPs calculated from Debye-Scherer formula was found to less than 5 nm and agrees well with those determined from UV—vis spectra and TEM data. Fourier transform infrared (FT-IR) spectroscopy was performed to know the functional groups of the grown NPs. Peaks in FT-IR spectra indicate the formation of CdS NPs and capping with L-alanine and L-arginine. Photoluminescence spectra of these NPs were also studied. Finally, colloidal solution of CdS-PVAc was subjected to Z-scan experiment under low power cw laser illumination to characterize them for third order nonlinear optical properties. CdS-PVAc colloidal solution shows enhanced nonlinear absorption due to RSA and weak FCA on account of two photon absorption processes triggered by thermal effect.

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1. Introduction

Nanoparticles (NPs) show unique chemical, physical, optical, electrical and transport properties, which are very different from those of bulk materials and single atoms [1,2]. The NPs have high surface to volume ratio and on account of it have high surface energy. Formation of stable NPs, avoiding agglomeration can be achieved only by minimizing this energy. This can be accomplished by capping NPs with some suitable capping agents [3,4]. Cadmium sulphide (CdS) is wide band gap II—VI semiconductor having bulk band gap energy of 2.42 eV [3] at room temperature that is why it

E-mail addresses: swarna_81@rediffmail.com (S.S. Talwatkar), ystamgadge@gmail.com (Y.S. Tamgadge), ashok.sunatkari@rediffmail.com (A.L. Sunatkari), abg_chem@ymail.com (A.B. Gambhire), gajananggm@yahoo.co.in, gajananmuley@sgbau.ac.in (G.G. Muley).

offers huge potential applications in optical devices. It exhibits size dependent properties like variation of band gap and melting point with size. As it is wide band gap semiconductor, it is used as visible light detector, window material for hetero junction solar cells and in other applications like in light emitting diodes, photo detectors, sensors and electrically driven lasers, etc [4–9]. Semiconducting NPs also show enhanced nonlinear optical (NLO) properties which include self-focussing and de-focussing phenomenon, two photon and multiphoton absorption (TPA and MPA), free carrier absorption (FCA), etc. which make them efficient candidate for optical limiting devices [10,11].

Water-soluble organic surface modifying agents, which include amino acids such as L-histidine and L-cysteine, have already been developed as capping agents for semiconducting nanocrystals [12,13]. In the synthesis of narrow range size NPs, amino acids play an important role as a capping agent. Some workers have demonstrated the synthesis and optical characterization of NPs capped

^{*} Corresponding author.

using L-valine, which showed that an amino acid ligand could be a promising biocompatible capping agent for the semiconductor nanocrystals [14,15]. L-alanine and L-arginine belong to α -amino acid with the chemical formula CH₃CH(NH₂)COOH and H₂NC(NH) C(NH)(CH)₃(NH₂)COOH, respectively. They are commercially available, inexpensive and non-biohazard materials. The α -carbon atom of L-alanine is bound with a methyl group (-CH₃), making it one of the simplest α -amino acids with respect to molecular structure. The methyl group of alanine is non-reactive. The amino group has a loan pair of electrons and capable of forming hydrogen bonds [16,17]. L-arginine has been used efficiently as capping agent in our recently reported work [18,19].

In this article, we present synthesis and characterization of CdS NPs capped with L-alanine and L-arginine. The NPs were synthesized by chemical precipitation method and characterized by ultraviolet—visible (UV—vis) spectroscopy, which gives evidence of blue shift in the absorption wavelength. The shift in the position of peaks in Fourier transform infrared (FT-IR) spectra of capped CdS NPs reveals the formation of hydrogen bonding, which prevents an aggregation of NPs and bulk crystal growth. Powder X-ray diffraction (XRD) study confirms the formation of pure phase CdS NPs having cubic crystal structure. Transmission electron microscopy (TEM) and photoluminescence (PL) studies were performed on the synthesized NPs. Nonlinear optical (NLO) properties of colloidal solution of these NPs in polyvinyl acetate (PVAc) matrix have also been studied using open aperture Z-scan technique using low power linearly polarized continuous wave (cw) He—Ne laser.

2. Experimental

2.1. Materials and synthesis

Cadmium chloride (CdCl₂, 99.9%), sodium sulphide (Na₂S, 99%), L-alanine (99.9%) and L-arginine (99.9%) of AR grade were purchase from Sigma Aldrich, USA. 1 M solutions of CdCl2 and Na2S were prepared separately by dissolving appropriate amount of solute in the 59.6 mL double distilled water. The solutions were stirred for 3 h using magnetic stirrer and filtered using membrane filter papers. Same process was adopted to prepare 0.1 M 15 mL solution of L-alanine and L-arginine in double distilled water. 14.9 mL solution from prepared stock solution of CdCl₂ was taken in four beakers to prepare four samples; first sample (CdS-pure) was prepared without any capping agent and second sample (AlCdS1) was prepared by pouring 1.49 mL of 0.1 M L-alanine solution in the second beaker to obtain CdS NPs capped with 1 mol% L-alanine. Similarly the third (AlCdS2) and fourth (AlCdS3) samples were prepared by pouring 2.98 mL and 5.96 mL of 0.1 M L-alanine solution to prepare CdS NPs capped with 2 mol% and 4 mol% L-alanine, respectively. Double distilled water was added in all four beakers to make total volume 100 mL. Finally, uncapped and capped CdS NPs were synthesized by adding drop-by-drop 14.9 mL solution of Na₂S in each beaker under continuous stirring for 3 h. The transparent solution starts to become yellow with addition of Na₂S solution. The CdS NPs without capping agent appear reddish orange in colour and the colour becomes dull with increasing concentration of L-alanine. The synthesized NPs were then separated by centrifuging for 5 min at 4000 rpm. The NPs were washed with water and ethanol followed by separation using centrifugation and dried optically to get fine powder. The synthesized NPs were used for further study. By same procedure, three more samples (ArCdS1, ArCdS2 and ArCdS3) were also prepared to get CdS NPs capped with 1, 2 and 4 mol% L-arginine. Concentrations of different reactants used in the synthesis process are summarized in Table 1. The following chemical reaction takes place during synthesis of NPs:

$$(CdCl_2)_n + (L-alanine)_m + (Na_2S)_n \rightarrow (CdS)_n - (L-alanine)_m + 2n(NaCl)$$

$$(CdCl_2)_n + (\iota\text{-arginine})_m + (Na_2S)_n \rightarrow (CdS)_n - (\iota\text{-arginine})_m + 2n(NaCl)$$

Mixing and ultrasonicating equal volumes of 50 mM CdS NPs (capped with 4 mol% L-alanine and L-arginine taken separately) and 10 wt% PVAc solution in double distilled water, colloidal solutions of CdS-PVAc were obtained in order to study their NLO properties by Z-scan.

2.2. Characterizations

UV—vis spectroscopy on the grown NPs in 10 mm quartz cuvette was carried out using UV-1700 spectrophotometer, Shimadzu-Japan. For this, powder samples of NPs were dissolved in double distilled water and were ultrasonicated to obtain colloidal suspension which was used for UV—vis study. FT-IR spectroscopy was performed on the instrument FT-IR spectrophotometer, 8400S-Shimadzu, Japan. CdS NPs were characterized for phase formation information by PANalytical (Philips) X-ray diffractometer. TEM study was performed on JEOL-JSM-6380 Transmission Electron Microscope (Japan). Photoluminescence study was carried out using Hitachi fluorescence spectrophotometer (F-7000). CdS-PVAc colloidal solutions in 1 mm path length quartz cuvette were subjected to Z-scan technique in which low power 10 mW cw laser was used to study nonlinear absorption phenomenon.

3. Results and discussion

3.1. UV-vis spectroscopy

All samples of CdS NPs were subjected to UV—vis spectroscopy to study excitonic absorption bands. Fig. 1 show absorption spectra of all synthesized CdS NPs. From figure, it is evident that absorption peak for each sample is blue shifted depending on the

Table 1Data summarizing concentration of reactants, band gaps and particle sizes of CdS NPs.

Sample name	Concentration of reactants			Experimentally determined	Particle size determined
	L-arginine/L-alanine	1 M CdCl ₂ (mL)	1 M Na ₂ S (mL)	band gap from Fig. 2 (eV)	from EMA model (nm)
CdS bulk	-	14.9	14.9	2.42 [3]	_
	0.1 M L-alanine (mL)				
AlCdS1	1.49 (1 mol%)	14.9	14.9	2.43	26
AlCdS2	2.98 (2 mol%)	14.9	14.9	2.79	5
AlCdS3	4.96 (4 mol%)	14.9	14.9	2.95	4.21
		0.	1 M L-arginine (mL)		
ArCdS1	1.49 (1 mol%)	14.9	14.9	2.423	28
ArCdS2	2.98 (2 mol%)	14.9	14.9	2.47	13
ArCdS3	4.96 (4 mol%)	14.9	14.9	2.93	4.29

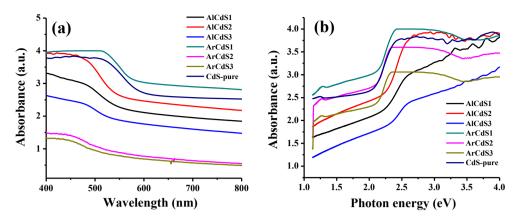


Fig. 1. Variation of absorbance of CdS NPs with (a) wavelength and (b) photon energy.

concentration of capping agent. Shift of absorption peak is more for the sample containing largest (4 mol%) concentration of L-alanine or L-arginine. Thus, NPs capped with largest concentration of Lalanine or L-arginine has smaller particle size and increased forbidden energy gap. Role of L-alanine and L-arginine as an effective surface modifying agents is thus obvious. Bulk CdS is known to have direct band gap of 2.42 eV which is reduced by the factor of exciton binding energy (67 meV) due to presence of excitons in CdS and absorption peak may be red shifted. In present case; sample CdS-pure shows absorption peak at a wavelength 517 nm. Broad absorption due to the excitons in various quantum states is clearly seen from Fig. 1. In a typical case, absorption peaks in samples AlCdS3 and ArCdS3 are due to excitons in states n = 1 and n = 2. From Fig. 1, it is also evident that L-alanine acts as a good capping agent as we get very narrow particle size and increased bandwidth for samples capped with L-alanine than that of L-arginine for equal concentrations. Fig. 2 shows plots of variation of $(\alpha h \nu)^2$ vs. photon energy ($h\nu$). The optical band gap (E_g) for direct type of semiconductor can be calculated using the equation.

$$\alpha = A(E_g - h\nu)^n / h\nu \tag{1}$$

Where, α is an absorption coefficient, A is constant and n is equal to 1/2 for direct transition [20]. The absorption coefficient α has been calculated in the wavelength range 400–800 nm using the relation = 2.303log[1/(1-A)]/cd, where 'A' is absorbance, 'c' is concentration and 'd' is thickness of the sample. The band gap energy E_g is obtained by extrapolating the linear portion of the plot to the energy axis. As obtained particle size for CdS NPs is greater

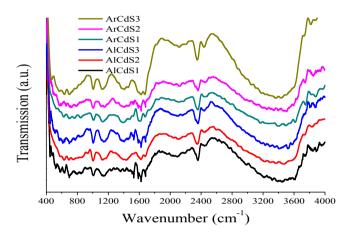


Fig. 3. FT-IR spectra of as synthesized CdS NPs using different capping agents.

than Bohr exciton radius which is 1.8 nm for present case, this regime is treated as weak confinement regime. In this regime the Coulomb's energy is stronger than the confinement effect [21] and e-h pair is confined as a whole due to which quantification of the movement of the center of mass is possible. The energies of the possible optical transitions can be obtained through the relation $E_n = E_g - E_{\rm exc} + (\hbar^2 \pi^2 n^2 / 2MR^2)$; where, $(M = m_e^* + m_h^*)$ the total mass of the e-h pair, R is radius of nanoparticle, E_g is bulk band gap energy, $E_{\rm exc}$ is the exciton binding energy, and n is the quantum number [22]. The particle size for all samples is calculated from

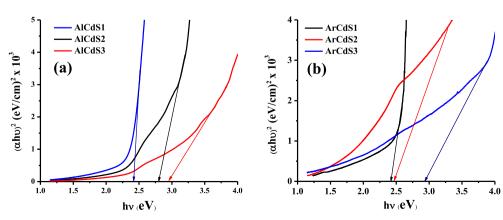


Fig. 2. Plots of variation of $(\alpha h v)^2$ with photon energy (hv) for CdS NPs capped with (a) ι -alanine and (b) ι -arginine.

UV—vis spectra by using effective mass approximation (EMA) [23]. The EMA formula can be stated as:

$$E_g = E_{bulk} + h^2 \Big/ 8r^2 \Big[\Big(1/m_e^* \Big) + \Big(1/m_h^* \Big) \Big] - e^2 \Big/ 4\pi \epsilon_0 \epsilon_r \gamma_e \eqno(2)$$

Where, E_g is band gap energy of NPs, E_{bulk} is energy gap for bulk semiconductor, m_e^* and m_h^* are effective electron mass $(0.19m_e)$ and effective hole mass $(0.8m_e)$. γ_e is Bohr exciton radius (1.8 nm), ε_r is relative dielectric constant (5.7), ε_0 is dielectric constant of vacuum and r is the radius of NPs. Particle sizes calculated and band gaps determined from Fig. 2 are listed in Table 1.

3.2. FT-IR spectroscopy

The FT-IR spectra of the samples were recorded and shown in Fig. 3 with prominent peaks. The band of peaks around 710 cm⁻¹ is assigned to the stretching vibrations of the CdS. The absorption around 1144 and 1395 cm⁻¹ are due to the bonding between S of CdS and O of H₂O and absorbed ethanol [24–26]. The absorption around 1630 cm⁻¹ is being assigned to the bending vibrations of H_2O and the peaks around 2353 and 3423 cm⁻¹ are due to the O-H stretching [26]. Peaks around 710 cm⁻¹ of CdS NPs are found to be shifted toward lower wave numbers as concentration of capping agent increases. This may be due to the formation of hydrogen bonding between CdS and L-alanine or L-arginine. However, peaks around 1144, 1386 and 2353 cm⁻¹ are found to shifted toward higher wave numbers which is due to the weakening of the bonding between oxygen atom of absorbed H₂O molecules and sulphur atom of CdS because of formation of additional hydrogen bonding with capping agent.

3.3. Structural and morphological analysis using XRD and TEM

The powder XRD patterns of L-alanine and L-arginine capped CdS NPs are presented in Fig. 4. The XRD data was analysed by using PowderX software. The peaks at diffraction angles $2\theta = 27.28$, 30.81, 45.06 and 52.19° have been assigned to (111), (200), (220) and (311) crystallographic planes of the cubic structure of CdS, respectively. Broadening of diffraction peaks with increasing the concentration of capping agent is clearly seen which confirms the decrease in particle size with increasing concentration of capping agent. Assuming spherical structure, particle size (D) of the synthesized NPs was determined by using Debye-Scherer's equation [27]:

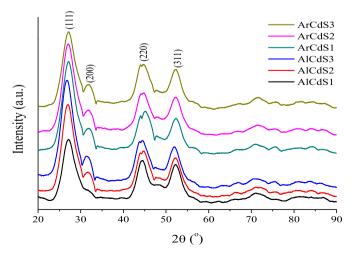


Fig. 4. Powder XRD pattern of CdS NPs.

$$D = k\lambda/(\beta \cos \theta) \tag{3}$$

Where, λ is the X-ray wavelength (1.54 Å), θ is the Bragg angle in degree, k is a constant (k=0.94) and β is width of the diffraction peak at half maxima in radians. The diffraction peaks associated with (111), (220) and (311) crystallographic planes were chosen to calculate the size of the NPs. The calculated particle sizes are summarized in Table 2. It can be seen that the size of the NPs is less than 5 nm and decreases with increasing concentration of capping agents. It is also observed that decrease in particle size is more prominent in case of L-alanine. Fig. 5 shows TEM images of CdS NPs capped with 4 mol% (a) L-alanine and (b) L-arginine. TEM analysis confirms the formation of NPs and observed size of NPs are in good agreement with XRD data and UV—vis data. All NPs show spherical morphology having uniform shape and size.

3.4. Photoluminescence study

In PL process, an electron is excited from valence to conduction band by monochromatic light having photon energy equal to or greater than band gap. The radiative recombination of an excited electron either takes place at the edge of valence band (band edge emission) or through traps within the band gap. In CdS NPs, the emission peaks are generally due to excitonic emission (at ~2.9 eV) and a broad surface states related emissions (at ~2.2 eV) [28]. Dopants can also introduce energy states in forbidden gap that give rise to different emission peaks [28,29]. In the present case, CdS NPs have been characterized for photoluminescence study by recording excitation (Fig. 6) and emission (Fig. 7) spectra of all the samples. Excitation spectra were recorded by monitoring emission at a wavelength 530 nm and show absorptions peaks at around 208, 223, 240, and 256 nm. The emission of all the samples were monitored over wavelength range 500-700 nm with an over band excitation using wavelength 400 nm (3.1 eV). The emission spectra show emission peaks centred at around 520 (2.37 eV), 554 (2.24 eV) and 588 nm (2.11 eV). The well-known excitonic green peak at ~2.9 eV is not visible in emission spectra as it is out of measuring range. The green emissions at around 520 and 554 nm are attributed to the electronic transition from the conduction band to an acceptor levels due to interstitial sulphur [30] and cadmium vacancy [28]. The yellow emission around 588 nm is attributed to the recombination via surface localized states [31] and a transition from interstitial cadmium states to the valence band [32]. Again, it can be observed that the PL intensity of band at 554 nm is more as compare to other bands. It may be due to presence of fewer traps introduced by interstitial cadmium and surface localised states. In case of CdS NPs capped with L-arginine, PL intensity increases with increases in the concentration of L-arginine, however intensity decreases with increase in the concentration of L-alanine. This may be due to presence of less trap states for the samples capped with Larginine [33]. The peak positions are not found to change that confirms the traps are dip traps and not affected by capping agent.

Table 2Size of CdS NPs calculated from XRD data.

Sample	Particle size (nm)	
AlCdS1	4.32	
AlCdS2	4.02	
AlCdS3	3.60	
ArCdS1	6.06	
ArCdS2	5.10	
ArCdS3	4.20	

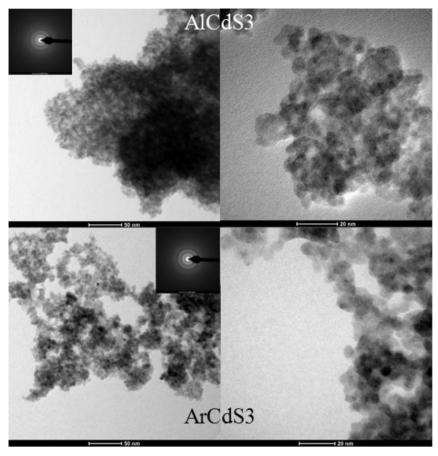


Fig. 5. TEM images of CdS NPs capped with 4 mol% (a) L-alanine and (b) L-arginine.

3.5. Z-scan study

Nonlinear absorption process was studied using open aperture Z-scan technique as proposed by Bahae et al. [34,35]. Z-Scan is a single-beam technique based on spatial beam distortion principle. It offers simplicity as well as high sensitivity for measuring the sign and magnitude of nonlinear refraction and nonlinear absorption coefficient. In this technique, the sample is translated along the direction of focused polarized Gaussian laser beam (z-direction) and its transmittance is measured with aperture in case of closed

aperture and without aperture in case of open aperture Z-scan. NLO properties of semiconducting nanomaterials are of prime importance due their active role in photonics technology. Semiconducting nanoclusters are known to show significant NLO properties because of quantum size effects and excitonic oscillator strength [36–39]. In our present case, since particle size is more than Bohr exciton radius of CdS NPs, weak confinement approach is applicable. In weak confinement regime, optical nonlinearity will be a combination of Coulomb interaction between the e-h pair and the deviation of excitonic oscillations from the simple harmonic model. There are several mechanisms reported to occur in NPs that

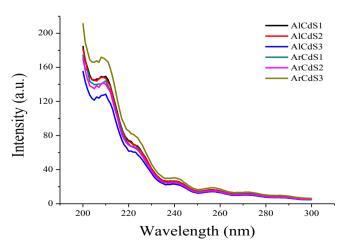


Fig. 6. Excitation spectra of CdS NPs.

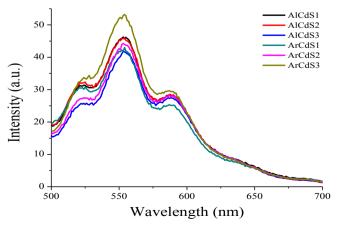
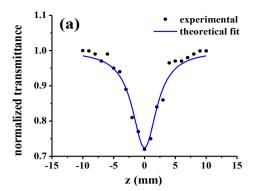


Fig. 7. Emission spectra of CdS NPs.



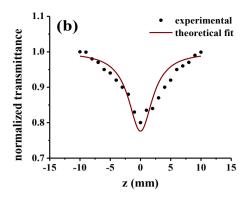


Fig. 8. Open aperture Z-scan curves for CdS-PVAc colloidal solution capped with (a) L-alanine and (b) L-arginine.

give rise to nonlinear absorption including two-photon absorption (TPA), reverse saturable absorption (RSA), transient absorption, free carrier absorption (FCA) and nonlinear scattering [40]. In case of CdS, when continuous wave (cw) laser is used as excitation source, the observed optical nonlinearity is due to intensity dependent refractive index and multiphoton absorption (MPA) processes [41].

Low power linearly polarized cw He-Ne laser (10 mW) having wavelength 632.8 nm was used to excite the sample. The photon energy ($E_p = 1.89 \text{ eV}$) of the laser beam is less than the band gap energy ($E_g \sim 3$ eV) of CdS nanocrystals and at the same time $E_p > E_g/$ 2. Hence nonlinearities from two photon absorption (TPA) are expected [40]. In the present experiment, laser beam was focused using 20 cm focal length lens. The measured laser beam waist ω_0 and the Rayleigh length z_0 were 22 μ m and 2.4 mm, respectively. Open aperture Z-scan experiment was carried out in order to estimate the intensity dependence of nonlinear absorption process for samples of CdS NPs dispersed in PVAc. Quartz cuvette having path length of 1 mm containing CdS-PVAc colloidal solution was translated through the focus ($\pm z$ direction) and transmittance with respect to z position of the sample was recorded. Plot of open aperture Z-scan for CdS NPs capped with 4 mol% L-alanine and Larginine are shown in Fig. 8. When the sample is at focus, the transmittance decreases showing well defined dip at the focus, indicating the occurrence of reverse saturable absorption (RSA) process [42]. The decrease in transmittance at the focus is more for the sample capped with L-alanine. This may be due to the enhancement of anharmonicity as CdS NPs capped with L-alanine are smaller in size. In addition to RSA, very weak contribution from FCA is reported to occur to give enhanced nonlinear absorption. As our colloidal solution is multiphase system containing CdS NPs, surface modifying agents and PVAc matrix, role of surface states on the borders of polymer-CdS NPs becomes important [43]. These surface states do not affect much on the optical properties of CdS NPs due to surface passivation by capping agents and polymer matrix. However, some unshielded surface states may contribute towards the enhancement of nonlinear absorption in the present case [33,43]. Scattering at the CdS-PVAc boundary may be neglected due to negligible difference between refractive index of CdS and PVAc [44].

Nonlinear absorption coefficient β is calculated for both the samples using following equation [34,35].

$$T(z,s=1) = \sum_{m=0}^{\infty} \frac{[-q_0(z)]^m}{(1+m)^{3/2}}$$
 (4)

Where, $q_0(z) = \beta I_0 L_{\rm eff}/(1+z^2/z_0^2)$, $I_0(13.16 \text{ MW/m}^2)$ is on-axis irradiance of laser beam at the focus (z=0), $L_{\rm eff}$ is effective thickness of sample $(L_{\rm eff}=(1-exp(-\alpha L))/\alpha)$, α is linear absorption coefficient and L is actual thickness of sample), $z_0=k\omega_0^2/2$ is

Rayleigh's range and ω_0 is beam waist. The value of linear absorption coefficient α for the evaluation of nonlinear absorption coefficient has been obtained at wavelength 632 nm using the same procedure adopted in section 3.1.

Experimental data curves are fitted with the equation (4) for two photon absorption processes and the value of β for which theoretical fits agree with experimental data is noted. With the experimental data, we have obtained $\beta = 5.93 \times 10^{-6}$ m/W for CdS-PVAc sample capped with L-alanine and $\beta = 4.81 \times 10^{-6}$ m/W for CdS-PVAc sample capped with L-arginine. This large value may be attributed to the thermally induced nonlinearity as cw laser is used. The solid line in Fig. 8 is theoretical fit to experimental data by using equation (4).

4. Conclusions

The CdS NPs passivated with L-alanine and L-arginine were successfully synthesized by chemical precipitation method and characterized by various techniques. UV-vis spectroscopy confirms the blue shift in the absorption band of CdS NPs. The increase in the band gap is more for CdS NPs capped with L-alanine than that of Larginine for the same concentration. The FT-IR spectra of capped CdS NPs reveal the capping of CdS with L-alanine and L-arginine which prevents an aggregation of particles and bulk crystal growth. XRD study shows that the NPs have cubic structure and particles have size <5 nm for 4 mol% of capping agents. TEM analysis shows spherical morphology of CdS NPs. From the photoluminescence study, it is found that the NPs show emission peaks at around 554 and 588 nm. The intensity of peak at 588 nm decreases with concentration of L-alanine. Finally, these CdS NPs dispersed in PVAc in colloidal form were subjected to study nonlinear absorption properties using open aperture Z-scan technique. CdS-PVAc colloidal solution shows enhanced nonlinear absorption due to RSA and weak FCA on account of two photon absorption processes triggered by thermal effect. Enhanced nonlinear absorption is observed for CdS NPs capped with L-alanine. The obtained value of nonlinear absorption coefficient β is as large as 10^{-6} m/W, which makes them good candidates for potential applications in optical limiters and other photonic devices.

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