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Effect of capping agents on structural and photoluminescence Properties of ZnS nanoparticles

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ABSTRACT:

In the present work, uncapped and capped ZnS nanoparticles were synthesized by chemical co-precipitation method using Thiophenol and CTAB as capping agents at room temperature. The structural properties of ZnS nanoparticles were analysed using x-ray diffractometer (XRD), transmission electron microscope (TEM), & scanning electron microscope (SEM) and their optical properties were studied using UV-VISIBLE absorption, Photoluminescence and FTIR analysis. XRD analysis showed that samples prepared were having cubic zinc blende structure with particle size in the range 2.3 nm to 3.6 nm. The sizes of samples were calculated using scherrer formula and Williamson-Hall(w-H) plots. TEM analysis revealed the formation of ZnS nanoparticles with almost uniform spherical shape. UV-VISIBLE absorption spectra showed a large blue shift . Photoluminescence analysis showed that the emission peak shifts towards shorter wavelength side as the size of the particle is reduced. In capped samples, enhanced luminescence intensities were observed. Among capped samples, Thiophenol capped ZnS nanoparticles showed maximum luminescence intensity compared to that of CTAB capped nanoparticles.

Key Words: Co-precipitation method, II-VI group Semiconductor particles, Structural, optical and Photoluminescence properties.

INTRODUCTION:

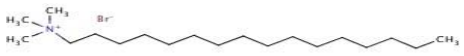
The Synthesis of nanomaterials has become an important research area in recent years due to strong dependency of the size of the particle. Among various nanomaterials, II-VI class inorganic semiconductor materials like ZnS, CdS, CdSe, ZnSe are proved to be versatile materials because of their applications in optoelectronic devices due to large variation of bandgap. ZnS has attracted much attention owing to its applications in efficient phosphors, flat panel displays, cathode ray tubes, solar cell etc. Semiconductor nanocrystals are described as a state of matter, that is intermediate between individual molecule and bulk [1-8]. Transition from bulk to nanoparticles lead to the display of quantum mechanical properties and an increased dominance of surface atoms which increases the chemical reactivity of a material. The advantage of the chemical synthesis is to produce size-controlled, un-agglomerated nanoparticles. The tunability of the properties of nanoparticles by controlling their size may provide an advantage in formulating new composite materials with optimized properties for various applications. But applications of these materials are restricted due to different non–radiative relaxation pathways. One important non radiative relaxation is surface related

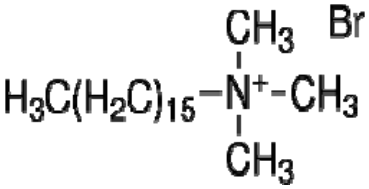
defects. Most of the physical or chemical properties exhibited by these nanoparticles are due to their crystallites. Further growth in their size is due to agglomeration of these crystallites to form primary particles. If this growth of particles is not controlled, then due to Ostwald ripening and Vanderwaals interactions between particles, they may agglomerate and settled. To control the growth of nanoparticles, organic stabilizers (polymers) NNN Cetyl Trimethylammonium Bromide(CTAB), mercaptoethanol, thiophenol, thiourea , SHMP, sodium polyphosphate, etc. can be added during the chemical synthesis for capping. Such materials have applications in luminescent devices, light emitters, phosphors ,optical sensors etc.[9-11]. Recently due to advancement of luminescent nanoparticles by successful capping, this results in fluorescent labelling by semiconductor nanoparticles in biological detection and applications in many fields.

Addition of organic compounds to inorganic semiconductors will show different morphology, size confinement, and luminescence behaviour. Many scientific methods have been employed to prepare semiconducting nanoparticles including chemical co-precipitation, chemical vapour deposition, hydrothermal and sol-gel process etc. In the present work, an attempt has been made to synthesize and characterize Thiophenol and Cetyl trimethyl ammonium bromide(CTAB) capped ZnS nanoparticles by chemical co precipitation method in order to explore various interesting properties.

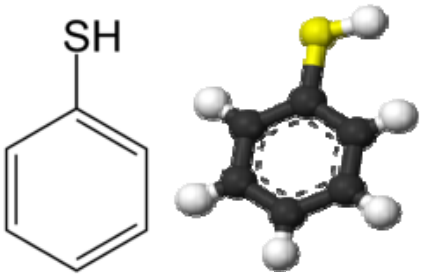
Structure of CTAB and THIOPHENOL are as shown below.

(a) CTAB





(b)Thiophenol



2. Experimental procedure

In the present study, ZnS quantum dots without and with capping agents were synthesized through chemical co-precipitation method. All the chemicals were of AR grade and were used without further purification for the preparation of nanoparticles.

The chemicals used were Zinc acetate ($Zn(CH_3COO)_2.6H_2O$), Sodium sulphide ($Na_2S.XH_2O$) and capping agents Thiophenol (C_6H_5SH) and Cetyl Trimethylammoniumbromide (CTAB) ($C_{19}H_{42}NBr$).

ZnS WITHOUT CAPPING AGENT: 0.2M Zinc acetate solution is prepared by dissolving 6.5847grams in 150ml(75ml of distilled water+75ml of methanol). 0.2M Na_2S solution is

prepared by dissolving 3.1216 grams in 200ml(100ml distilled water+100ml methanol). 0.2M sodium sulphide solution is added dropwise in to the solution of 0.2M zinc acetate, under continuous stirring using magnetic stirrer for 3-4 hours. The white precipitate is collected after centrifugation and is washed 3 to 4 times using distilled water and ethanol to remove impurities.

ZnS WITH CAPPING AGENTS: 0.1M solution of Thiophenol and 0.1M solution of CTAB were mixed separately with the 0.2M Zinc acetate solution in two different beakers for 30 minutes using magnetic stirrer. Then 0.2M Na_2S solution is added dropwise to each beaker under continuous stirring for 3 to 4 hours until the precipitates are formed. After centrifugation, precipitates were collected and washed 3 to 4 times using distilled water and ethanol to remove impurities.

The precipitates were dried using 60 watt bulb for 3 hours and crushed into fine powder using mortar and pestle.

3. INSTRUMENTATION:

The sizes of all the samples are determined using PXRD Panalytical X'PERT Pro MPD diffractometer with CuKα radiation(0.154nm). XRD data is collected over the range 20°-80° at room temperature. The particle size is calculated using Scherrer formula. Structural properties of samples are studied by SEM and TEM(Philips CM 200, Operating voltage 20-200 kV, Resolution-0.24nm) analysis. UV-VISIBLE absorption spectrum of the samples is studied from 200 nm to 600 nm. Photoluminescence properties are studied using WITEC α-300 Raman PL Setup from 250 nm to 800 nm. FTIR studies are carried out from 400 to 4000 cm^{-1}.

4. RESULTS AND DISSCUSSION:

4.1: XRD Studies:

In the XRD pattern, there were three diffraction peaks corresponds to (111), (221) and (311) planes of the cubic ZnS. The peaks were broadened due to size effect and it implies the formation of nanoparticles. The average particle size is calculated using Debye-Scherrer formula.

$$D=K\lambda/\beta\cos\theta \qquad (1)$$

where K is scherrer constant=0.9, λ is wavelength of Cu-Kα radiation=0.154nm, β is full width at half maximum(FWHM) in radians and θ is the diffraction angle, D is the size of a particle[9].

The average crystalline size is also calculated using W-H plot. William and Hall plots suggested a method of combing the particle size and lattice micro strain effects on line broadening when both were operative. $\beta=\beta_D+\beta_{\varepsilon,}$ where β is the sum of the peak width due to microstrain and due to particle size. W-H plots may be expressed in the form [10].

$$\beta\cos\theta=\varepsilon(4\sin\theta)+K\lambda/D \qquad (2)$$

The above equation represents a straight line between 4sinθ (x-axis) and βcosθ (y-axis). The slope of the line gives the strain(ε) and the intercept (Kλ/D) of this line on y-axis gives the grain size. The structural properties of the nanoparticles are as shown in Table 1.

The average strain is also calculated using Stokes-Wilson relation [11].

$$\varepsilon_{str}=\beta/4\tan\theta. \qquad (3)$$

The interplanar spacing d is calculated using the relation $d=\lambda/2\sin\theta$.

where λ is the wavelength of x-ray radiation, θ is the diffraction angle.

Lattice constant corresponding to different planes is calculated using equation(4) [11].

$$a=d(h^2+k^2+l^2)^{1/2} \qquad (4)$$

Table 1: Structural properties of ZnS (uncapped and capped) nano particles

Sample	2θ in Deg	Plane	Interplanar Spacing d (nm)	Lattice constant a (nm)	Avg crystalline size(nm)from		Strain
					XRD	W-H plot	
1. ZnS	28.87 47.92 56.95	(1 1 1) (2 2 0) (3 1 1)	0.3088 0.1896 0.1618	0.5360	3.606	4.03	0.038
2. ZnS+ Thiophenol	28.57 47.92 56.23	(1 1 1) (2 2 0) (3 1 1)	0.3111 0.1896 0.1634	0.5380	2.340	2.37	0.062
3.ZnS+CTAB	28.87 47.71 56.42	(1 1 1) (2 2 0) (3 1 1)	0.3088 0.1900 0.1623	0.5364	3.008	3.35	0.042

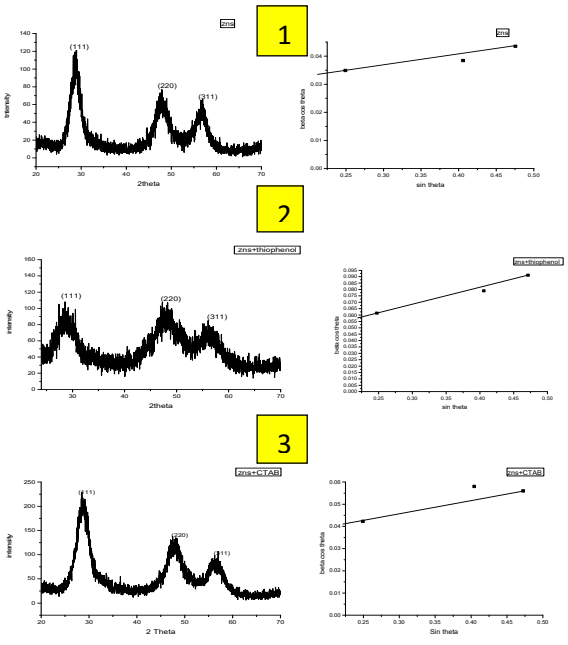


Fig 1: X-Ray diffraction and W-H Plots of (1)ZnS (2)Thiophenol+ZnS &(3)CTAB+ZnS nanoparticles.

Three broad peaks in XRD pattern corresponds to (1 1 1), (2 2 0) and (3 1 1) planes of the cubic zinc-blende phase of ZnS respectively. The crystal size obtained using W-H plot is slightly higher than the size calculated using Scherrer equation, because the Scherrer equation does not take in to consideration the effect of lattice strain in the line broadening [11].

4.2: Optical absorption studies:

The absorption spectra of ZnS nanoparticles synthesized with different capping agents are shown in fig 2.The absorption peaks were observed in the range 204.5nm to 206.5 nm.

Band gap energy is calculated using the relation

$E_g = h\vartheta = hc/\lambda$ (5)

where h, c, λ, and E_g are the plank's constant, speed of light in vacuum, wavelength and band gap energy of nanoparticles respectively.

The blue shift of the absorption edges for different nanocrystals is related to the decrease of size of the

particles and is attributed to the quantum confinement effect of nanoparticles [12].

Brus has proposed a theoretical model (Effective mass approximation EMA) relating the effective band gap of material with the particle size. Using the average crystallite size obtained from X-ray diffraction spectrum, the band gap for each particle is calculated, results are reported in Table 2. Also by applying the confinement effects the optical size of nanocrystallites is calculated using Brus equation [13].

$$E_{g(nano)}=E_{g(bulk)}+h^2/8r^2(1/m_e+1/m_h)-1.8e^2/4\pi\varepsilon_o\varepsilon_r r \quad (6)$$

where $E_{g(nano)}$ in ev is the band gap energy of bulk ZnS, m_e and m_h are electron and hole effective masses and r is the radius of the particle in nm, ε_o is the permittivity of free space and ε_r is the permittivity of the sample. For bulk ZnS E_g=3.68 eV, m_e=0.34m_o and m_h=0.23m_o ; m_o=free electron rest mass. The last term in equation (6) is the coulombic term and is generally neglected. By substituting the band gap energy values of synthesized samples in equation (6), size of nanoparticles were calculated and are reported in Table2.

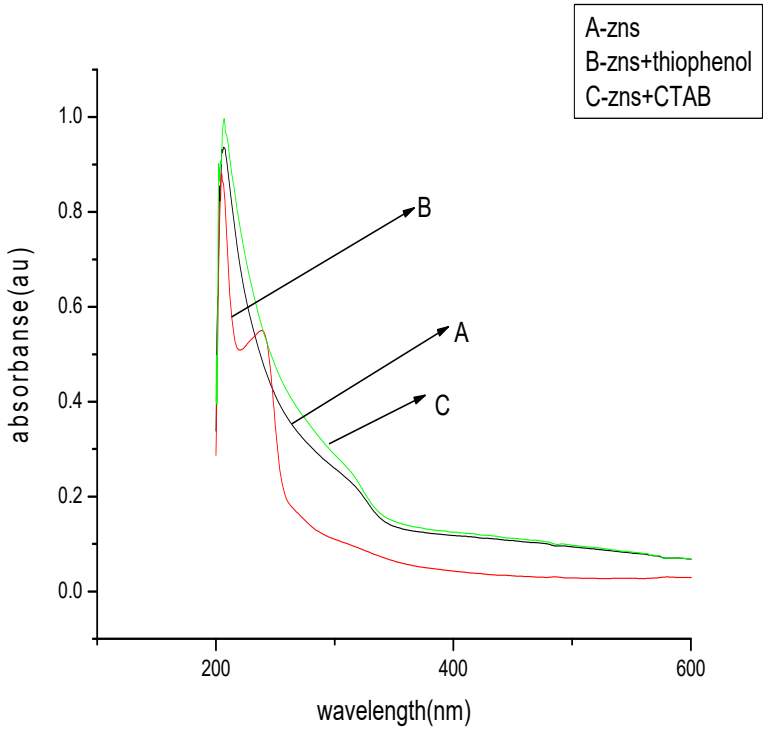


Fig 2: UV-Visible absorption spectrum of ZnS uncapped and capped nanoparticles

Table 2 : Band gap and size of ZnS (uncapped and capped) nanoparticles.

Sample	Absorption peak λ in nm	Band gap in(eV) from absorption spectra	Band gap(eV) from Brus eq.	Crystal size in nm From		
				EMA	XRD	TEM
1.ZnS	206.5	6.015	4.53	2.170	3.606	3.678
2.ZnS+Thiophenol	204.5	6.074	5.76	2.140	2.340	2.360
3.ZnS+CTAB	206.0	6.030	4.90	2.162	3.008	3.058

From these results we can conclude that as the particles size is reduced, there is a clear blue shift of band gap for all the samples with respect to bulk ZnS (band gap of bulk ZnS is 3.68 eV) for cubic structure). This is due to the modification of valence band and conduction bands by quantum confinement effects [12]. Strong confinement is observed when the particle size is smaller than the Bohr radius (approximately 2.5 nm), as a result of this strong confinement absorption peaks which appear in the absorption spectra are due to sub band transitions, so that in this case band gap can be found from the maximum of this absorption peak. The results obtained for ZnS (with and without CTAB) were nearly matched with ref (Yesu Thangam et al.)[14]. In absorption spectrum of Thiophenol capped ZnS , two exciton peaks were observed at 204.5 nm and 238.19 nm indicating the formation of different morphology due to aromatic ring and S-H group of Thiophenol. From the results it is seen that band gap calculated from Brus equation was less than the band gap calculated from absorption peak for each particle; this shows that the Brus equation cannot be expected to be quantitatively correct for very small particles.

4.3: Transmission electron microscopic (TEM) studies

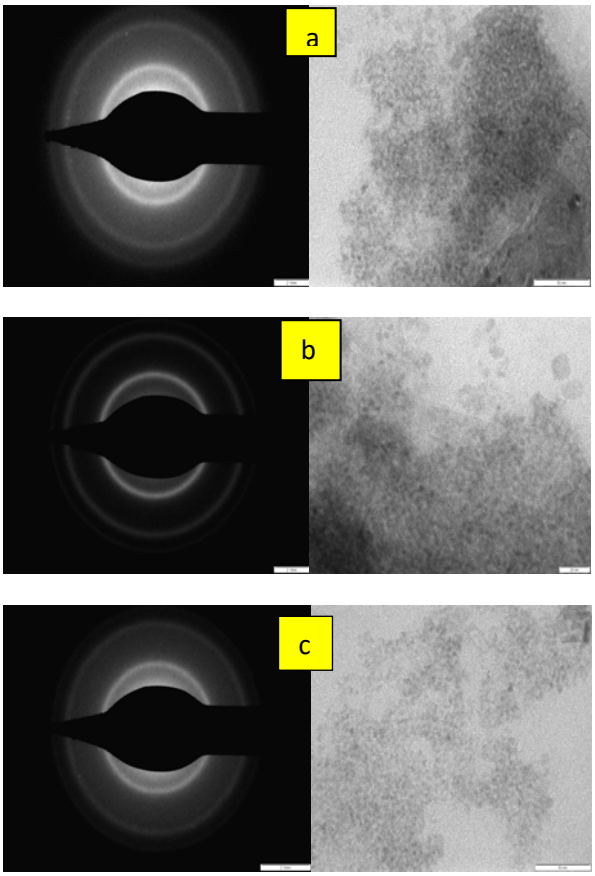


Fig 3: TEM and SAED Pattern of (a)ZnS (b)ZnS+Thiophenol and(c)ZnS+CTAB Capped nanoparticles.

The TEM and SAED images of ZnS (uncapped), Thiophenol capped ZnS and CTAB capped ZnS nanoparticles are shown in Fig 3. The size and morphology of nanoparticles were determined by TEM images. From each image, it is seen that

particles are monodispersed, almost spherical in shape, and the sizes calculated were well matched with XRD results. The selected area electron diffraction(SAED) patterns exhibit concentric circles, revealing that, the ZnS samples are nano crystalline being made up of small particles. The rings correspond to (111), (220), and (311) planes of the cubic zinc blende phase. [15]

4.4: Scanning electron microscopic (SEM) studies :

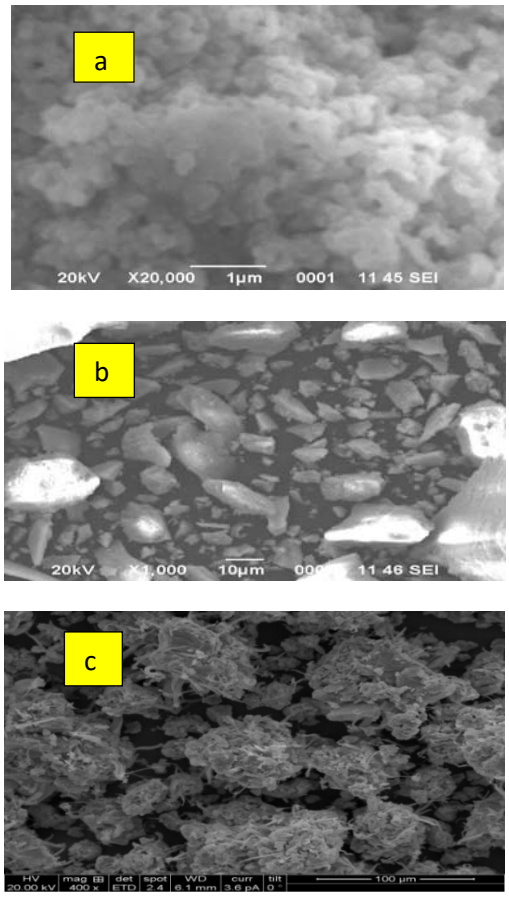


Fig 4: SEM images of (a) ZnS
(b) ZnS+Thiophenol (c) ZnS+CTAB nanoparticles.

From the above SEM images of samples with different magnifications, the formation of nano clusters was confirmed [9,16].

4.5: Photoluminescence Studies:

Room temperature Photoluminescence spectra of ZnS (uncapped and capped) nanoparticles recorded at an excitation wavelength of 355 nm are shown in Fig 5. From photoluminescence spectra, it was observed that, uncapped ZnS nanoparticles showed broad emission peak at 439 nm originated from the host ZnS. This blue emission can be ascribed to a self activated centre presumably formed between a Zn vacancy and a shallow donor associated with a Sulphur vacancy [17]. Wheareas Thiophenol and CTAB capped particles showed the peaks at 426 nm and at 431nm respectively. The emission intensity of capped ZnS nanoparticles are comparatively higher than the uncapped ZnS. This is expected because in the absence of capping agent uncontrolled nucleation and growth of particles occurred, resulting in the formation of defect states. But in capped nanoparticles, enhanced luminescence was observed due to surface modification by capping agents with the effect of minimizing surface defects and also due to transfer of energy from chemisorbed capped molecules to interstitial sites and vacancy centres. The enhancement of luminescence intensity increases with decrease of particle size, was ascribed to quantum size effect [18,19]. When the particle size decreases, the valence band edge shifts downwards. Therefore the emitted photon has comparatively higher energy, giving photoluminescence peak at shorter wavelength [20]. Among CTAB and Thiophenol capped ZnS nanoparticles, Thiophenol capped ZnS nanoparticles showed maximum luminescence intensity. This may be due to presence of aromatic ring and S-H group of Thiophenol. [21-25]

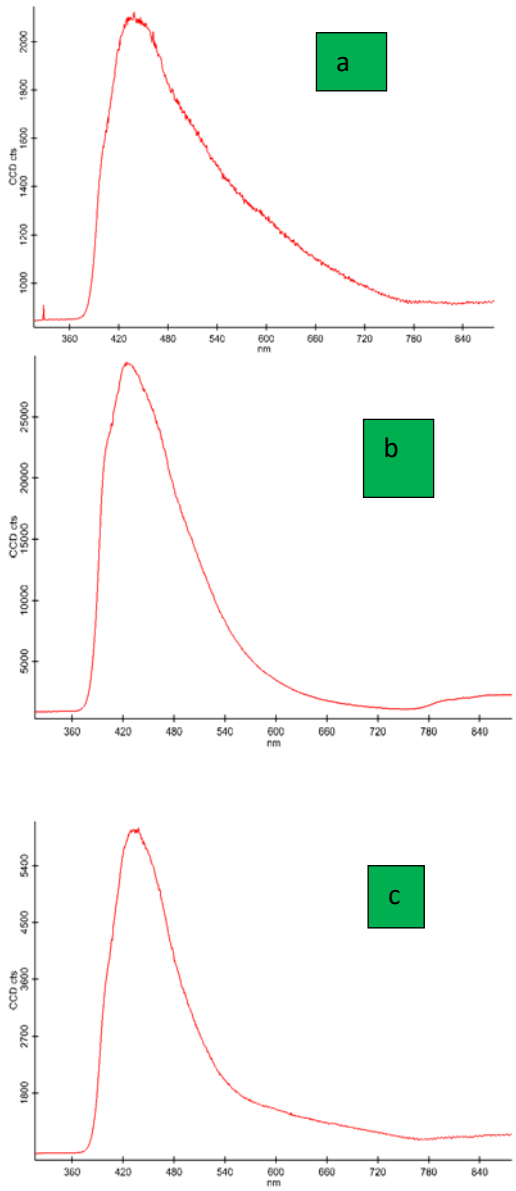


Fig 5: Photoluminescencs spectra (a) ZnS(b)Thiophenol capped ZnS, and (c) CTAB capped ZnS nanoparticles.

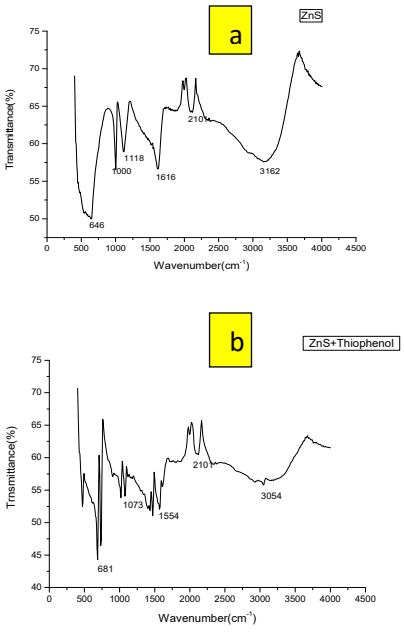
4.6: FTIR studies:

FTIR spectrum of uncapped ZnS nanoparticles is as shown in fig 6a. The broad absorption peak at 3116 cm^{-1} corresponds to O-H stretching vibration of water absorbed in the sample, peak at 1616 cm^{-1} corresponds to bending vibration of water, peak at 1118cm^{-1} corresponds to C-O stretching vibration of absorbed methanol and peak at 646cm^{-1} corresponds to ZnS vibrations.[26,27]

FTIR spectrum of Thiophenol capped ZnS nanoparticles is as shown in fig 6b. A small peak at 3054 cm^{-1} corresponds to ring C-H vibrations of thiophenol, peak at 1554 cm^{-1} corresponds to C=C skeletol vibration, peak at 1073 cm^{-1} corresponds to C-O stretching vibration of absorbed methanol, peak at 681 cm^{-1}corresponds to ZnS vibrations in the presence of thiophenol.[28,29,30]

FTIR spectrum of CTAB capped ZnS nanoparticles is as shown in fig 6c.The broad absorption peak at 3290 cm^{-1} corresponds to O-H stretching vibration of water absorbed in the sample, peak at 2926 cm^{-1} corresponds to symmetric stretch of CH_2 vibrations, peaks at 1554 cm^{-1} and 1408 cm^{-1} are attributed to C-H scissoring vibrations of $–N-CH_3$ moeity, peaks around 1000 cm^{-1} and 636 cm^{-1} corresponds to ZnS vibrations [31].

Peak around 2101 cm^{-1} in all the samples corresponds to microstructure formation of samples.



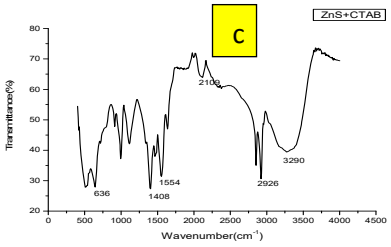


Fig 6: FTIR spectra of (a) ZnS (uncapped) (b)Thiophenol capped ZnS (c) CTAB capped ZnS nanoparticles.

5.Conclusions:

Uncapped and capped ZnS nanoparticles were successfully synthesized by co-precipitation method using Thiophenol and CTAB as capping agents. The synthesized samples were studied by different characterisation techniques, XRD, EM, TEM, UV-Visible absorption, photoluminescence and FTIR studies. XRD and SAED patterns confirm the cubic crystalline structure of ZnS. Sizes of nanoparticles calculated from Scherrer formula and W-H plot are well matched with sizes calculated from TEM images. The morphology of the particles has been identified from the SEM and TEM analysis and revealed a spherical shape. The UV-visible absorption spectrum peak for all the three samples exhibits blue shift from the bulk. Photoluminescence spectrum peak also shifted towards shorter wavelength side as the size of the particle is reduced. Capped particles showed maximum PL intensity compared to uncapped particles. Thiophenol capped ZnS nanoparticles showed maximum intensity than CTAB capped ZnS nanoparticles, this may be due to the presence of aromatic ring and S-H group in Thiophenol.

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