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Surfactant-Mediated Synthesis and Stabilization of Colloidal Nanoparticles in Aqueous Media: A Preliminary Study on Controlling Parameters

Sanjay Kumar [1], Samjeet Singh Thakur [2,*]

Abstract

Surfactants dissolved in water possess a distinctive tendency to accumulate at surfaces or interfaces, a process that involves the migration of surfactant molecules from the bulk liquid phase to the interfacial region. This adsorption tendency of surfactants is being exploited in stabilization of the Nanoparticles (NPs) synthesized through colloidal chemistry routes. In present research, preliminary studies on controlling parameters involving various surfactants has been carried out to standardized the chemical precipitation method for the synthesis of colloidal ZnS, CdS and HgS NPs in aqueous medium. Five cationic surfactants of varying hydrophobic chain length, counter ions and head group have been compared along with three anionic surfactants having different head group moieties. The aggregation tendencies of all these surfactants have been compared by determining the critical micelle concentrations (CMC) values to shortlist the most appropriate ones that may produce well dispersed spherical shaped NPs with minimum amount surfactant required. The growth rate of the NPs has been significantly controlled by the nature of surfactant. The self-aggregation of NPs has been prevented by surfactant coating at the NP surface due to changed inter-particle potential. The optimum concentrations of NPs at a particular surfactant concentration have been established through interpretation of UV-vis spectra.

Graphical Abstract
Surfactant-mediated synthesis and stabilization of colloidal nanoparticles in aqueous media:
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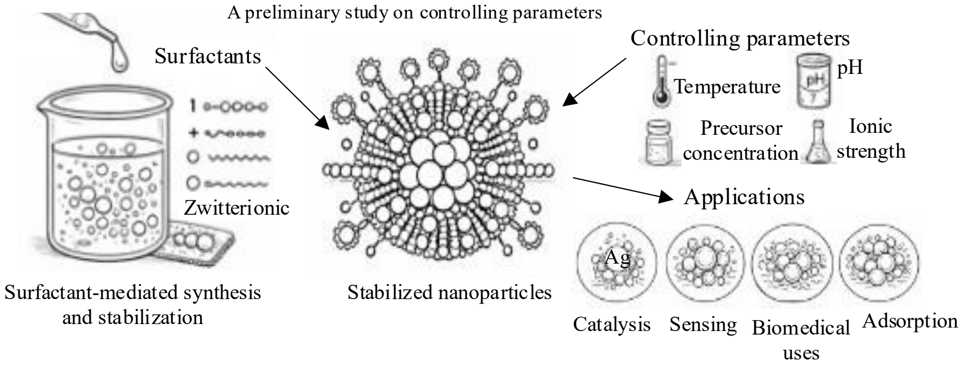

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Keywords: Critical micelle concentration, nanoparticles, surfactant, UV-vis spectroscopy

INTRODUCTION

The surfactants are the amphiphilic substances that, when present at low concentration, have property of adsorbing on surfaces or interfaces of the system and thereby altering the surface or interface free energy. Because of this property, the surface coating of NPs with surfactants is being extensively explored as a simple, effective, and economical method in aqueous as well as non-aqueous media (Singla et al., 2009) [1]. In contrast to their bare counterparts, these NPs remain well dispersed in relatively dilute solutions. At higher concentrations, they exhibit a strong propensity to self-assemble into ordered arrays when deposited on a smooth solid surface. The dynamic binding and unbinding processes (Ji et al., 2008; Studart et al., 2007; Clarke et al., 2006) [2, 3, 7] yield to the important consequence that the surfactant molecules can get off, e.g. by excessive washing or mass action by another species, which might compromise the stability of the NPs that ultimately aggregate and precipitate. In aqueous solution, strongly charged surfactant molecules, containing e.g. carboxylic or sulfonic acid groups, can stabilize the particles for longer time. In addition to impart stabilization to the NPs, the surfactant surface shell also influences the fluorescence properties of the semiconductor NPs, (Wuister et al., 2004; Bullen & Mulvaney, 2006; Kalyuzhny & Murray, 2005) [8–10] in particular the quantum yield, especially in aqueous phase. The kinetics of self-aggregation and interfacial adsorption of the surfactants over wide variety of surfaces has been extensively studied under different conditions and in presence of various other organic and inorganic moieties (Studart et al., 2007) [3].

By regulating particle dispersion, morphology, and interfacial interactions, surfactants are essential to the synthesis and functional performance of hybrid and nanostructured materials. Surfactants are frequently used as stabilizers and structure-directing agents in the creation of gelatin-based mesoporous nano-hybrids to enable the uniform distribution of inorganic phases like silica, titania, or zirconia inside the polymer matrix (Thakur, 2025) [4]. Their amphiphilic nature contributes to increased surface area and porosity by controlling the nucleation and growth of inorganic domains. Surfactant-assisted synthesis increases the adsorption effectiveness of nanohybrids for the removal of pollutants in environmental applications. Research on titania-gelatin and gelatin-silica-based materials shows that the removal of organic dyes and hazardous metal ions from aqueous solutions is greatly improved by the controlled addition of inorganic components with the help of surfactant-mediated dispersion (Thakur & Chauhan, 2014; Thakur & Chauhan, 2018) [5, 6]. Surfactants are therefore crucial instruments for enhancing functional performance in nanohybrid systems and customizing material structure. However, there exists a limited understanding about the role played by the surfactants in the stabilization and modification various properties of the semiconductor NPs.

In the view of the above discussion, the paper focus on understanding the controlling parameters during stabilization of semiconductor metal sulfide, i.e., MS (M= Zn, Cd, Hg) NPs synthesized using chemical precipitation in aqueous surfactant medium. Broadly, a series of cationic and anionic surfactants have been investigated to describe and discuss the effect of chain length, head group functionalities, counter ions and polarity of surfactants on stability metal sulfide NPs. Additionally, three anionic surfactants of identical chain length having different head groups has also been explored. The aggregation, adsorption, and other properties of these commercial surfactants have already been well established in the wide literature and being used in many important application areas (Tadros, 2005) [11]. Therefore, the general aim of work is to develop a database on the additional role of these surfactants in relation to NPs synthesis. The formation of nanocrystals by chemical precipitation method is a dynamic process, where after nucleation; the further particle growth occurs mainly via Ostwald ripening phenomenon (Wageh et al., 2003; De Smet et al., 1999) [12–13] i.e. the dissolution of small clusters in favor of the growth of larger particles. The mobility of the surface atoms and the rates at which matter enters and exits the surface must determine the surface structure of NPs at any given moment in time. These cluster mobility and growth processes are impacted by the presence of specific stabilizers or capping agents in the reaction media. The stabilizing shell that is always present on the surface of colloidal NPs controls both their growth and their characteristics. Several surfactant adsorption mechanisms on the surface of NPs during the development and stabilization processes are shown in Figure 1. Furthermore, the characteristics of NPs in colloidal solution can vary from those of a single NP and are always averaged throughout the entire ensemble (Empedocles et al., 1999) [14].

EXPERIMENTAL DETAILS

Chemicals Used

All the surfactants used in the study were of AR grade (purity > 99.5%) from Sigma Aldrich and were vacuum dried prior to use. Zinc acetate ($Zn(OAc)_2 . 2H_2O$); Cadmium acetate ($Cd(OAc)_2 . 2H_2O$);

Mercuric acetate ($Hg(OAc)_2$. $2H_2O$) were obtained from Himedia (purity > 99.0%) and Sodium sulfide (Na_2S. xH_2O) obtained from CDH (purity > 58%). These chemicals were used as received. All samples were prepared using double-distilled water (specific conductance, 2–4 μScm-1 at 303.15 K).

Preparation of Samples

For the synthesis of semiconducting (ZnS, CdS, and HgS) NPs, micellar formulations of the surfactants were prepared with surfactant concentrations above and below CMC. Different molar ratios for metal acetate: Na_2S were tested. Typically, 10 ml of a surfactant solution containing desired amount of metal acetate was slowly added into another containing Na_2S with same surfactant concentration. The solutions were formed by slow stirring to result homogeneous dispersions of the NPs. The NPs were then separated by slow evaporation of the solvent at 50–60 °C, washing with water and then with ethanol repeatedly to obtain fine powder of NPs containing surfactant. The synthesized NPs were characterized in dispersion form. A material's capacity to carry electric current is measured by its electrical conductivity, also known as specific conductance. A Pico digital conductivity meter from Lab India instruments was used to test the specific conductivity, with a precision of 0.1% and an absolute accuracy of 3%. It consists of a stone bridge circuit, a conductivity cell and a digital display. The conductivity cell was initially calibrated with KCl solution of different concentrations and specific conductivity: [KCl] = 0.01 M, κ = 1.413 mScm-1 and [KCl] = 0.10 M, κ = 12.9 mScm-1 at 298.15 K. The obtained cell constant was 1.02 cm-1. A Pt-electrode and temperature sensor were inserted into a double-walled, jacketed vessel holding the solution in which the thermostated water was circulated to measure the conductivity. An automatic thermostated bath from Julabo was used for maintaining the temperature with ± 0.01 °C accuracy. Conductivity measurements were carried out by titration of concentrated aqueous surfactant solution into water to determine the CMC values of the surfactants used for the stabilization of NPs. The optical spectra have been acquired using dual beam Jasco V-530 UV-vis spectrophotometer having range from 1100–200 nm.

RESULTS AND DISCUSSION

Preliminary Investigations on Surfactants Aggregation

The molecular structures of the cationic and anionic surfactants to be used as stabilizers for the synthesis of NPs have been depicted in Figure 2. The series of these surfactants have been selected to derive better comparative information in context of their role in tuning the sizes of the NPs. The effect of changing counter ion would be discussed by comparing various traits NPs formed using CTAB and CTAC; whereas comparison of the NPs formed in CTAC and CPyC would deduce the effect of head group variation. The series of three surfactants viz CTAB, TTAB, and DTAB have the same positively charged head group; therefore, the differences in their binding tendencies to the NP surface have been governed by length of hydrocarbon chain. The other three anionic surfactants, namely SL, SDS, and SDBS possess the same hydrocarbon chain length but different head groups. The sizes, charge, electronic polarizability and geometric features of head group of these surfactants are also different.

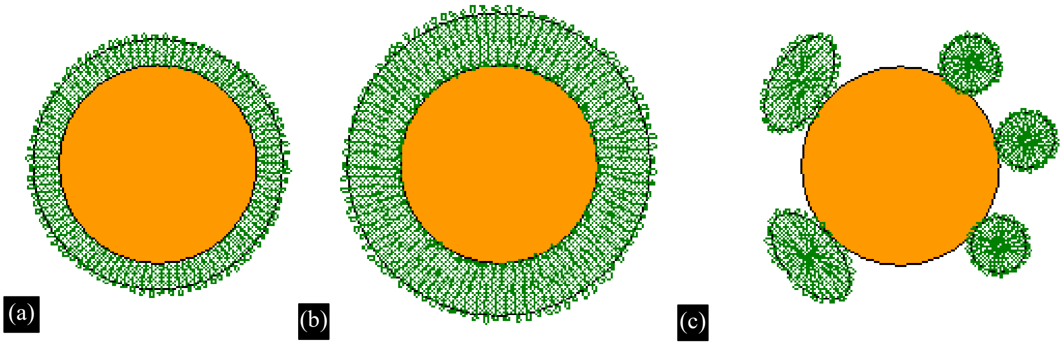

Figure 1. Different modes of surface coverage of a nanoparticle by surfactant: a) monolayer; b) bilayer; c) surfactant micelles.

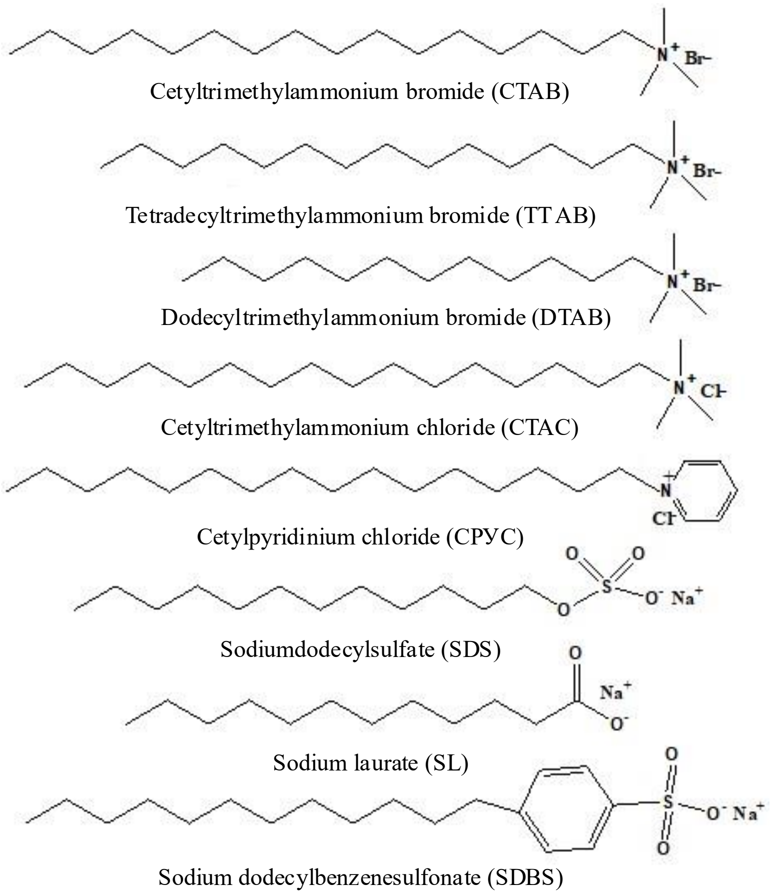

Figure 2. Molecular structures of the cationic and anionic surfactants used in the study.

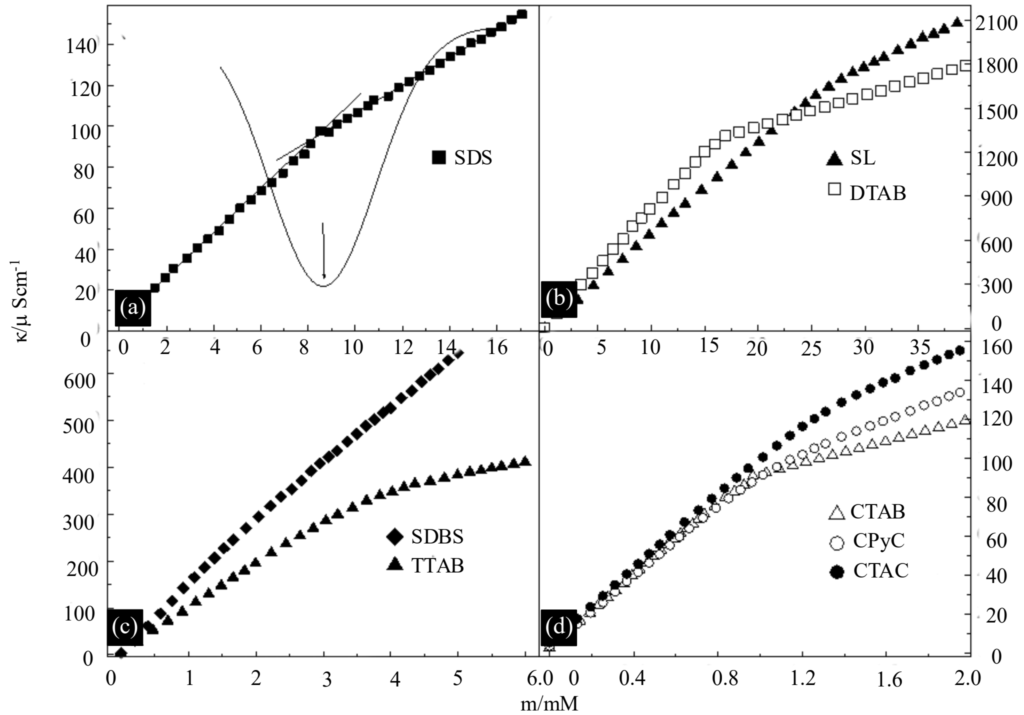

Figure 3. Variation of conductivity with m of different surfactants at 298.15 k along with (a) representative gaussian fit to the data for the determination of CMC.

Table 1. CMC values of the different surfactants.

Surfactant	CMC / mM	Surfactant	CMC / mM
CTAB	0.92±0.03	CPyC	0.92±0.03
TTAB	3.40±0.20	SDS	8.30±0.20
DTAB	15.8±0.20	SDBS	1.90±0.20
CTAC	1.30±0.10	SL	25.1±0.20

In aqueous solution, the surfactants tend to form self-aggregates when their concentration reaches a critical concentration, i.e., CMC. The CMC value of a surfactant is an important characteristic parameter to compare its various properties. In certain cases, the sudden rise in surfactant adsorption has also been assigned to the formation of surface aggregate of the surfactant molecules on the solid-liquid interface. The term "hemimicelles" (Gaudin & Fuerstenau, 1955) [15] refers to these surface aggregates that form above a critical concentration below the CMC, also referred to as the critical hemi-micellar concentration (HMC). Therefore, before investigating the various effects of surfactants on synthesis and stabilization of semiconductor NPs, it is of prime importance to compare their self-aggregation behavior in aqueous solution. The CMC values of these surfactants have been evaluated by performing the conductivity measurements in their aqueous solutions. The representative plots of conductivity, κ, as a function of surfactant concentration, m, for different surfactants at 293.15K are depicted in Figure 3.

The Williams method (Rodríguez et al., 1998) [16] has been applied to locate the CMC as intersection point of fitting lines in conductivity concentration plots above and below the break point. Additionally, the Phillips method (Phillips, 1955) [17] in classical form have also been used to confirm the CMC values by applying Gaussian fitting on second derivative of conductivity-concentration data. The obtained CMC values of the surfactants have been listed in Table 1 and found to be in close agreement with the literature values (Akhtar, 1997; Paria et al., 2003; Mata et al., 2005) [18–20].

Optimization of NPs Synthesis Conditions

Through the ionization or dissociation of surface groups or the adsorption of ions from solution onto a previously uncharged surface, the solid surfaces acquire either a positive or negative charge in the aqueous medium. Consequently, the adsorption of ionic surfactants is typically caused by the electrical double layer at the solid-liquid interface (Paria & Khilar, 2004) [21].

Similar adsorption process has been thought to be possible on the surface of freshly nucleated solid phase in chemical precipitation process, and the adsorbed surfactant can restrict the size of the synthesized material within nano regime. Surfactant molecules can adsorb onto solid substrates from aqueous solution through a variety of methods (Rosen, 1978) [22]. Surfactant adsorption often uses single ions rather than micelles (Griffith & Alexander, 1967) [23]. In the ion exchange mechanism, similarly, charged surfactant ions replace counter ions that have been adsorbed onto the substrate from the solution. Surfactant ions can also be adsorbed from solution onto oppositely charged sites that aren't occupied by counterions. The attraction between the electron-rich aromatic nuclei of the adsorbates and the positive sites on the solid adsorbent can also lead to adsorption when the surfactant has electron-rich aromatic nuclei and the solid adsorbent has strongly positive sites.

Thus, the final size and characteristics of the developing NPs would be impacted by this structure-dependent adsorption. Compared to anionic and non-ionic surfactants, cationic surfactants have a faster rate of adsorption kinetics and a larger ultimate adsorbed amount. Also, it has been established through SANS studies that shape and size of the micelles depends up on concentration of the surfactant used (Das eta al., 2012) [24]. The factors discussed above infer that during surfactant aggregation, the high surfactant concentration may itself give rise to varying shapes and sizes of micelles thereby adsorption process may get further complicated. For effectiveness and reliable results, the surfactant-NPs system should be kept as simplest possible. Therefore, to begin with, CTAB surfactant has been chosen for the preliminary study as it has lowest CMC value and simplest head group. Furthermore, its long hydrophobic chain length makes it strongly adsorbing surfactant thereby most suitable candidate for stabilization of NPs (Rosen, 1978; Griffith & Alexander, 1967) [22–23].

The ionic reaction involved in the surfactant stabilized chemical precipitation of metal sulfide (MS) NPs from metal acetates and sodium sulfide can be expressed as

$$M^{2+}(aq) + S^{2-}(aq) \xrightarrow{Surfactant} \mathrm{MS}\,(s) \qquad (M = Zn, Cd, Hg) \qquad \textbf{(1)}$$

The formation of NPs has been identified primarily through visual inspection and UV-vis spectroscopy of the dispersions. Appearance of light turbid, light yellow and brown homogeneous dispersions (Figure 4) indicated the formation of ZnS, CdS, and HgS NPs, respectively. The stability of these dispersions for months together in contrast to immediate precipitation of similar samples without surfactant further affirms the claim about the presence of NPs.

The UV-vis spectra of the samples have also been recorded to confirm the formation of the NPs and the results have been presented in Figure 5. Since the aqueous surfactant solution has been taken as reference, therefore it does not show any peak in UV-vis spectra. Also, none of the three aqueous surfactant solutions containing metal acetates showed any distinctive absorption in 200–600 nm range, whereas that containing Na2S showed a prominent peak centered at 229 nm. When the metal acetate solution was added separately into the aqueous surfactant solution containing S 2-, characteristic absorption shoulders appeared in 292–297 nm, 450–460 nm and 495–500 nm regions for ZnS, CdS, and HgS NPs, respectively with complete disappearance of peak at 229 nm. These shoulders have been regarded as exciton absorptions of respective NPs (Choi et al., 2007; Shankar et al., 2003; Qin et al., 2008) [25–27] and prove the existence of ZnS, CdS, and HgS NPs in the dispersions. Additionally, it has been observed that the absorption shoulder did not alter for a number of months, suggesting that the NPs were stable in the aqueous surfactant solution.

Since all the reaction medium's constituents – metal acetates, Na2S, and surfactants – are ionic, the concentration of each individual ion in an aqueous solution can be interpreted as the concentration of the added salts. The optimal theoretical stoichiometric ratio of $[M(OAc)_2]$:$[Na_2S]$ is anticipated to be 1:1 during the creation of MS according to reaction (1). However, because the aqueous solution of Na2S contains both aqueous H_2S and HS − as well as other sulfur oxyions like thiosulfate and sulfite, which either originate as impurities in solid Na2S or from the quick oxidation of HS − by O2, the actual [S2−] is always less than the added $[Na_2S]$ (Zhang et al., 2004) [28].

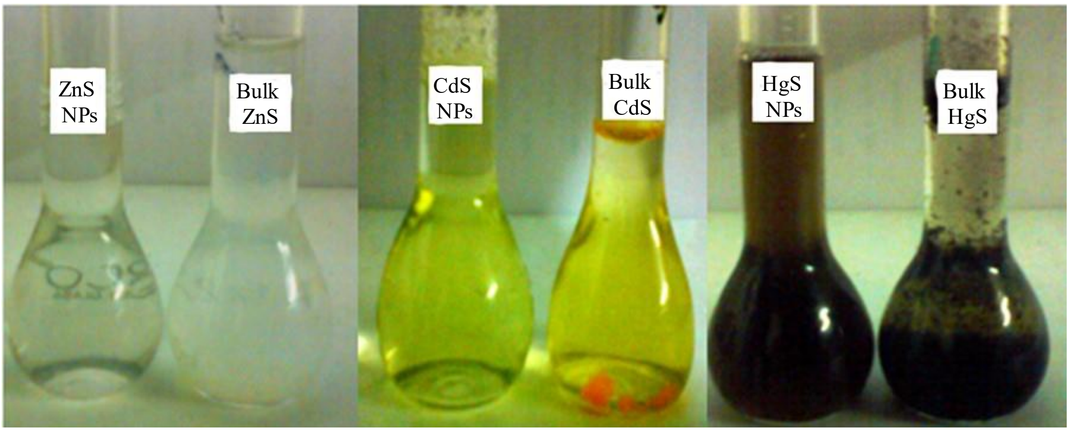

Figure 4. Pictures of the flasks containing aqueous dispersions of three ms in presence (NPs) and Absence (Bulk) of Surfactant (CTAB). Pictures taken after 1 h of Their Formation.

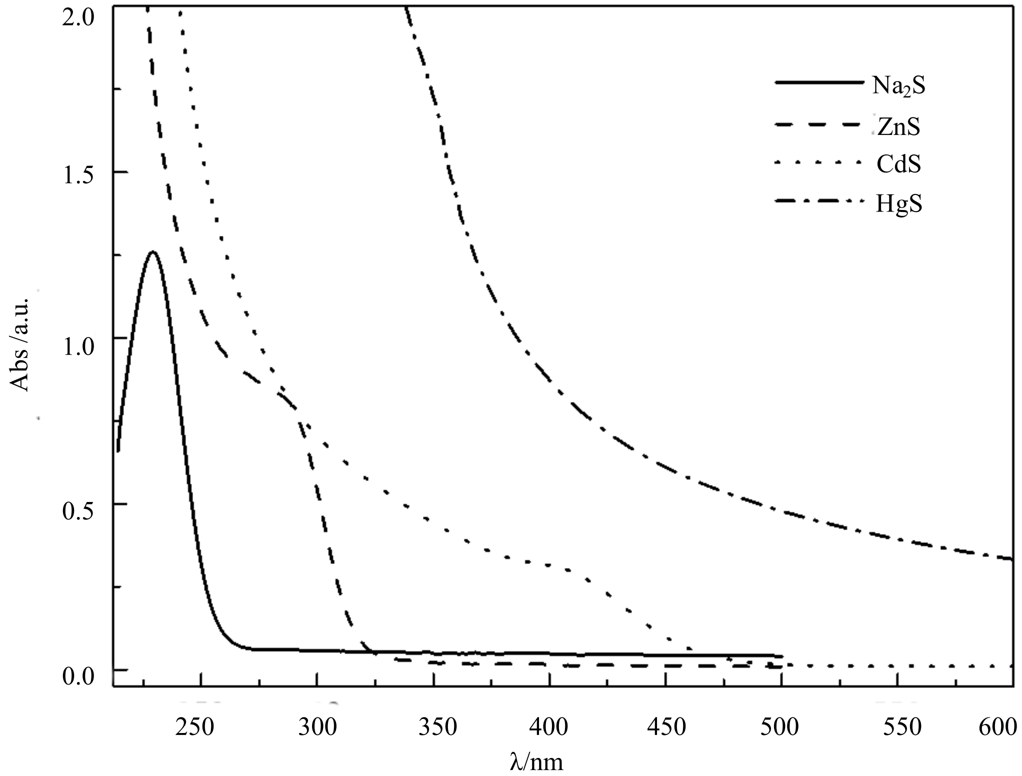

Figure 5. UV-vis spectra of aqueous CTAB solution of Na2S and different NPs.

Thus, some preliminary experiments on NPs formation in aqueous CTAB solution have been undertaken to develop an understanding about the idealized [M(OAc)$_2$]:[Na$_2$S] ratio, that would lead to optimum formation MS NPs. Figure 6 depicts the UV-vis spectra of aqueous dispersions of MS NPs (M =Zn, Cd, Hg) formed at different [M(OAc)$_2$]:[Na$_2$S] ratios keeping [M(OAc)$_2$] constant and varying the [Na$_2$S].

The spectra make it clear that the absorbance rises as [Na$_2$S] grows and reaches its maximum at [M(OAc)$_2$]:[Na$_2$S] = 1:2, indicating that NP production is at its peak at this ratio. Although the shoulder absorbance at [M (OAc)$_2$]:[Na$_2$S] = 1:3 ratio remains the same, but the shoulder have been shifted towards longer wavelength due to increased NPs size. Therefore, the ratio [Zn(OAc)$_2$]:[Na$_2$S] = 1:2 have been found to be optimum and same ratios has been used for the synthesis of all the three metal sulfide NPs.

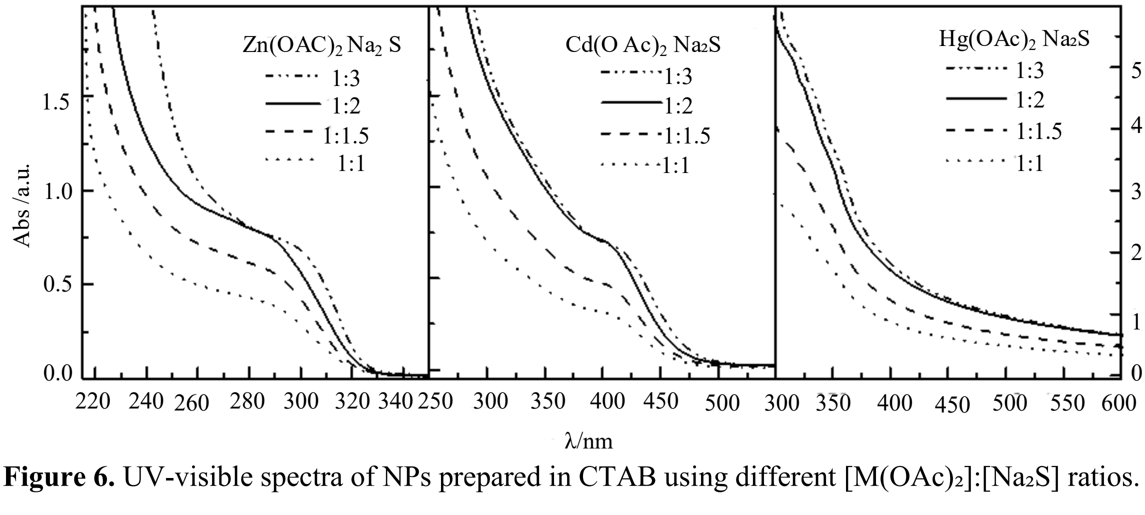

Figure 6. UV-visible spectra of NPs prepared in CTAB using different [M(OAc)$_2$]:[Na$_2$S] ratios.

CONCLUSIONS

The preliminary investigations establish a clear and systematic basis for understanding the role of surfactants in the synthesis and stabilization of metal sulfide nanoparticles. Conductivity measurements provided reliable determination of the critical micelle concentration (CMC) values using both Williams and Phillips methods, and the close agreement of the experimentally obtained CMC values with reported literature confirms the validity of the experimental approach. The aggregation characteristics of surfactants, including the possibility of hemimicelle formation at concentrations below CMC, underline their potential to strongly influence nucleation, growth, and stabilization of nanoparticles at the solid–liquid interface. The successful formation of ZnS, CdS, and HgS nanoparticles was unambiguously confirmed through visual observation and UV–visible spectroscopy. The appearance of characteristic excitonic absorption shoulders, along with the disappearance of sulfide ion absorption, provides strong evidence for nanoparticle formation. Moreover, the long-term stability of these dispersions, in contrast to rapid precipitation in the absence of surfactants, highlights the crucial stabilizing role of surfactant aggregates. The dependence of absorbance and spectral shifts on the metal acetate to sodium sulfide ratio further revealed that a 1:2 stoichiometric ratio is optimal for maximum nanoparticle formation with minimal size growth. Overall, these findings demonstrate that understanding surfactant self-aggregation behavior is essential for rational design and optimization of surfactant-assisted synthesis of stable semiconductor nanoparticles.

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