Ultrasound assisted synthesis of Cadmium Sulfide Nanoparticles and Characterization

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***Abstract*—Nanomaterials are the leading edge of nanoscience and nanotechnology. To found efficient method is a big challenge in nanotechnology. Herein we develop a green method to synthesize CdS nanoparticles. Cadmium sulfide (CdS) nanoparticles were synthesized by ultrasonication method using sodium sulfide and various salts of Cadmium. Current synthesis saves considerable energy which essential for formal synthesis. The synthesized nanoparticles were characterized by UV-Visible, XRD and STA analysis.**

***Index Terms*—Nanomaterial; Cadmium sulfide; Bath Ultrasonicator**

I. INTRODUCTION

Nanotechnology is developing and emerging multidisciplinary field attracts global attention in pharmacology[1]. Due to the proven biomedical properties, Cadmium sulfide nanoparticles (CdS NPs) have been employed in various fields of nanobiotechnology[2]. Nanoparticles (NPs) have major interest from nanostructures now a days[3]. The NPs should include 1 to 100 nm dimension in size. The great significance of such materials have physicochemical characteristics and significance[4]. The high surface-to-volume ratio gives rise to reactivity at the molecular scale[6,7]. NPs have wide range of applications in the biological and non-biological fields, diagnostics, cosmetics, agriculture, and other areas of science[8,9,10,11].Cadmium (Cd) has unique properties, such as high electrical conductivity, corrosion resistance and high malleability[12,13]. Cd has no biological function and its toxicity to humans has been confirmed[14]. Reactive oxygen species (ROS) produced as a result of oxidative stress brought on by Cd bioaccumulation in human tissues can interfere with the antioxidant defense mechanism can lead to various health problems[15]. Cadmium sulfide (CdS) has fluorescent properties which used in medicine. CdS has excellent optical and electrical properties, photocatalytic activity[16,17,18]. Different types of NPs are formed by using chemical, physical, and biological methods. The application of CdS NPs is dependent on the characteristics of the synthesized NPs, such as size, shape, and surface charge[15]. CdS NPs have drugs and diagnostic role because of non-toxicity, antioxidant, antimicrobial, anticancer and drug delivery properties[19,20,21]. In the present study, CdS NPs are synthesized by chemical precipitation techniques as this technique is simple, less time-consuming, and inexpensive. The structural and morphological characteristics of the synthesized NPs were extensively examined using X-ray diffraction (XRD) and STA analysis.

Experimental set-up

Ultrasound Set-upUltrasound for sonochemical synthesis is generated with the help of ultrasonic instrument set- up. The specification and details of the set- up, processing parameters used during experiments are as follows-

Make: Roop Ultrasonics, Gujarat

Operating frequency: 40 kHz (Inbuilt)

Rated output power: 230 W

Fig-1 Bath Ultrasonicator

II. MATERIALS AND METHODS

Analytical grade chemicals of cadmium acetate dihydrate [$CdCO_3$] (99.0%) as cadmium precursor, sodium sulfide (Na_2S, 95%) as reducing agents, and starch as capping agent used for the synthesis of CdS NPs. All chemicals and reagents used in the research were parched from Thermo Fisher Scientific Pvt. Ltd., Mumbai, India, and distilled water was used as the solvent throughout the experiment. The reagents were used directly without further purification. At first, 50 mL of 0.05 M precursor solution of cadmium acetate was mixed with 4 mL of 1% starch solution, and then 50 mL of 0.05 M sodium sulfate solution was run dropwise into the mixture solution. The mixture was stirred for 4 hr at room temperature and left for aging and sedimentation. Finally, the mixture was filtered, and the residues were washed with distilled water a couple of times and dried in a hot oven at 90°C, and then the sample was subjected to characterization.

III. PREPARATION OF CDS NANOPARTICLES

Solution-A: 2.66 gm of pure $CdCO_3$ dissolves in deionized water which makes 0.1 M solution. Solution-B: Take 0.78 gm of $Na_2S.9H_2O$ to make 0.1 M solution. Fill the burette with the solution B. Solution A placed on magnetic stirrer. Slowly add solution B in solution A under vigorous stirring. The resulting solution should turn yellow, indication the formation of CdS nanoparticles. Now take NaOH and add it to the solution to maintain the basic pH. After complete titration, the obtained solution is placed into the ultrasonic bath sonicator for 1 hour. Take out the solution from the ultrasonic bath sonicator and filter it with the whatmann filter paper no. 41. Wash the nanoparticles several times with deionized water and ethanol, dry them under oven for at least 15 to 20 minutes only, then borrow the compound from oven and crushed it.

Doping of CdS nanoparticles with Mg (1%) :
Doping needs 2.66 gm of $CdCO_3$, 0.780 gm of Na_2S and 0.1 M Mg solution.
For mg doping, 1.07 gm of mg (magnesium) is taken. Rest of the procedure is same as preparation of CdS nanoparticles.

Varying the pH of the synthesis:
Repeat the synthesis of CdS and Mg-doped CdS nanoparticles at different pH valueS (e.g., Acidic i.e. 5 and Basic i.e. 10).
Characterize the nanoparticles synthesized at different pH values using the same characterization techniques. It is seen that yield of the product is somewhat different in different salts taken.
Compare the properties of the nanoparticles synthesized at different pH values to understand the effect of pH on the synthesis.

Characterization:
X-Ray Diffraction
The XRD patterns confirmed the formation of CdS nanoparticles in different methods. The XRD pattern exhibited diffraction peaks at 26.4^o, 30.7^o and 51.6^o corresponds to (111), (220), (311) planes of cubic phase CdS nanoparticles. The synthesis and crystalline nature was significantly observed. It was observed that the diffraction peak in presence of dopping agent is broader than the pure CdS. This result confirms that the crystallite size of the CdS nanoparticle decreases to some extent.

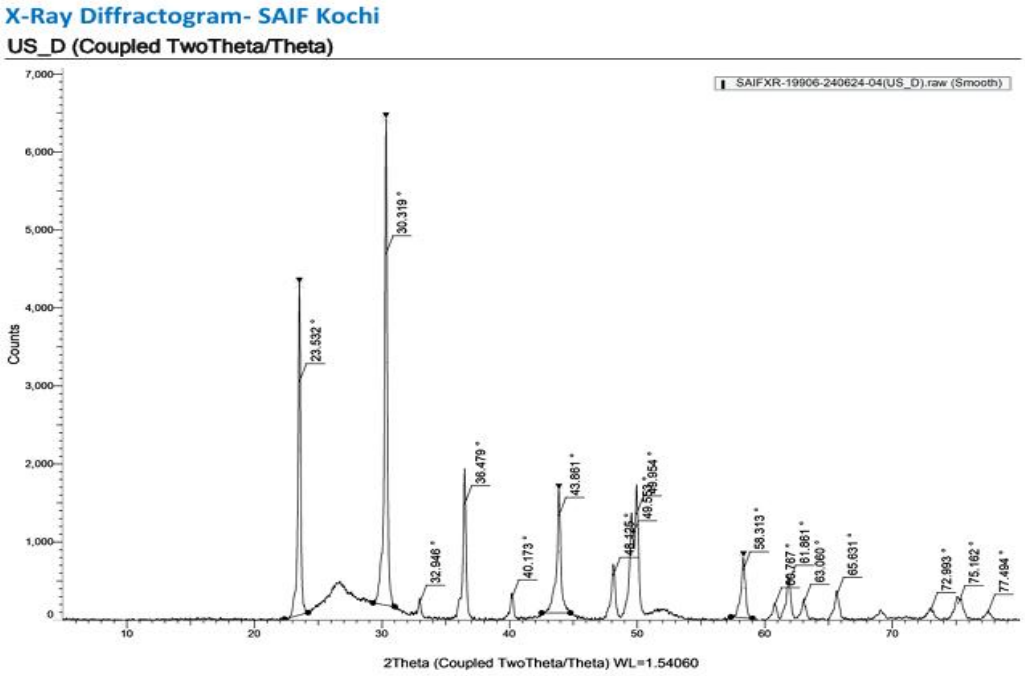

Fig.2 (a) XRD pattern of CdS nanoparticles synthesized CdS nanoparticle

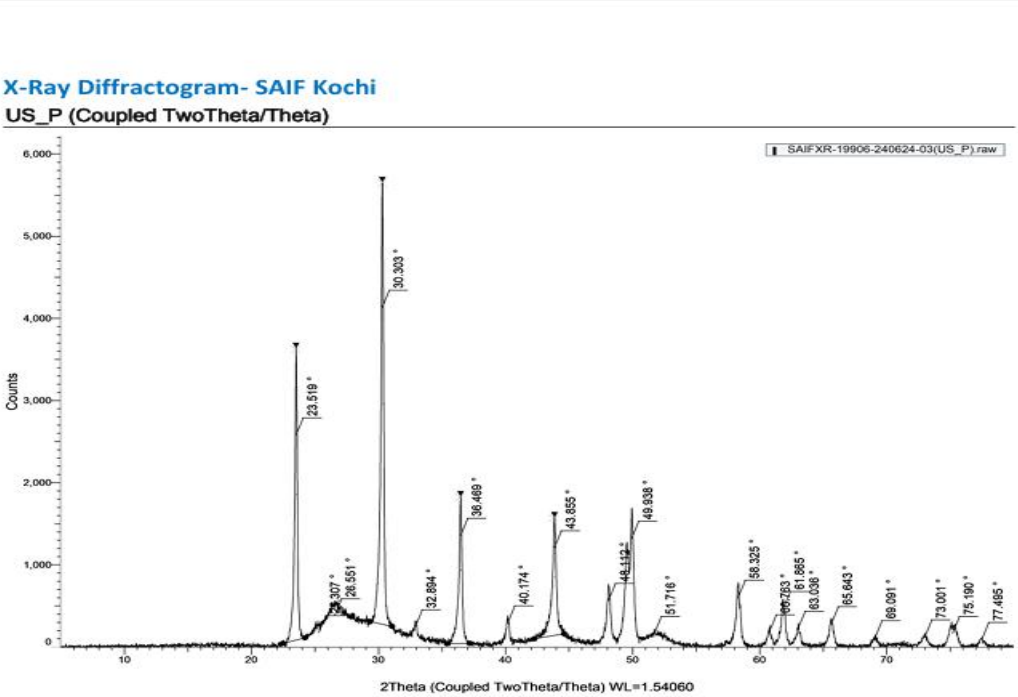

Fig-2 b) XRD pattern of CdS nanoparticles synthesized 1 % doping of Mg

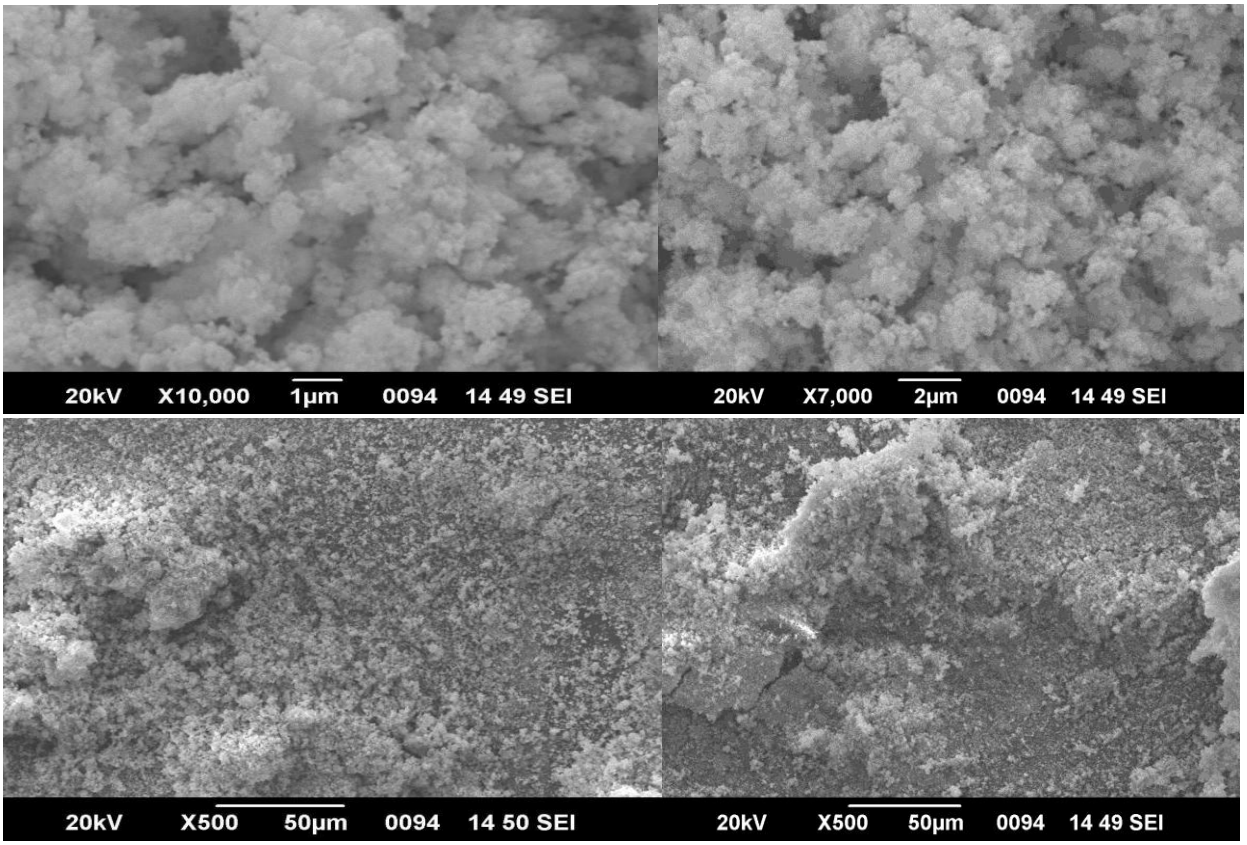

Fig-3a) SEM images of CdS nanoparticles

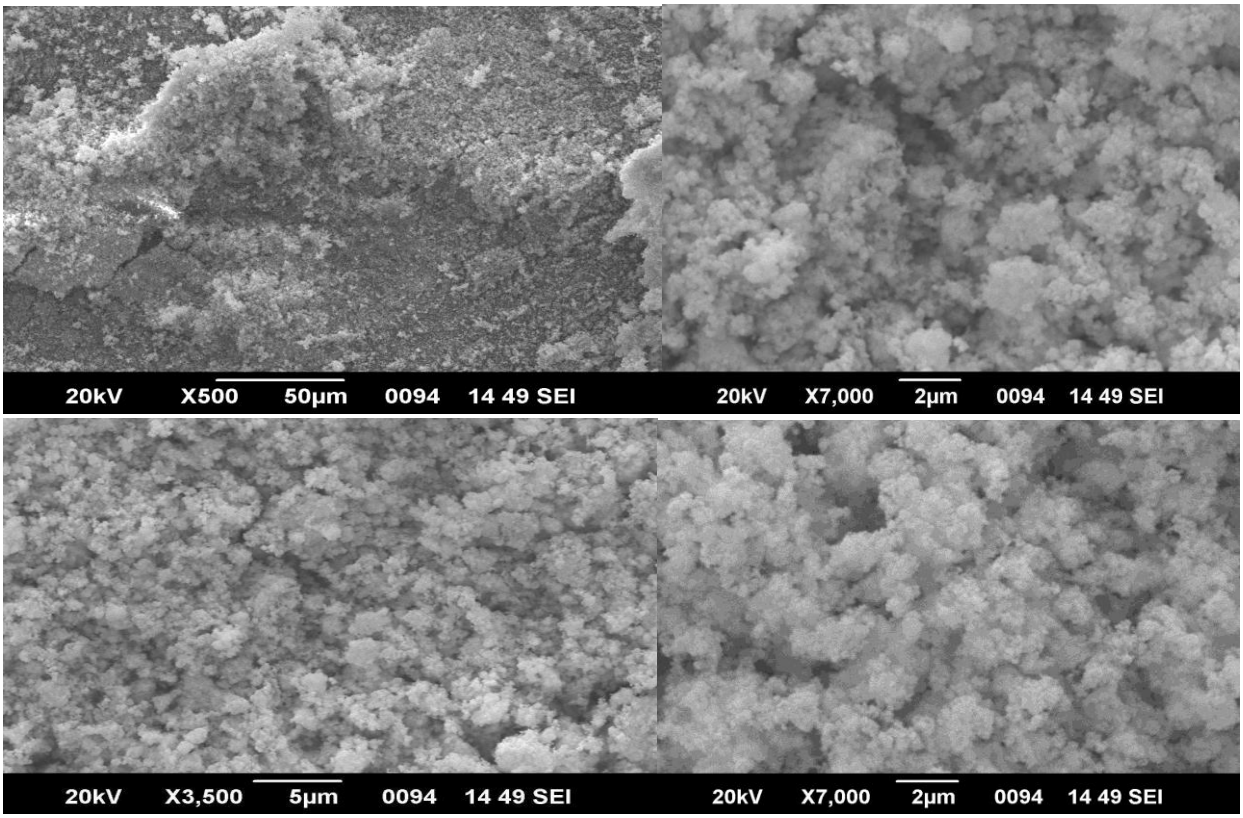

Fig-3b) SEM images of CdS nanoparticles with 1 % doping of Mg

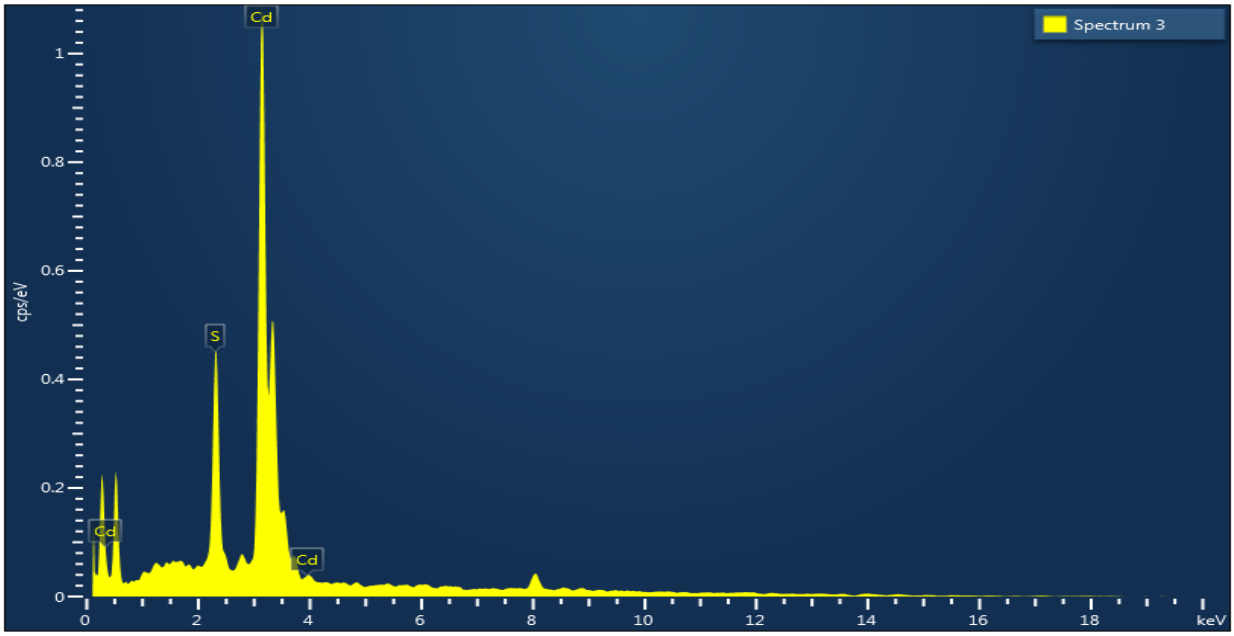

Fig-4a) EDX graph of CdS nanoparticles

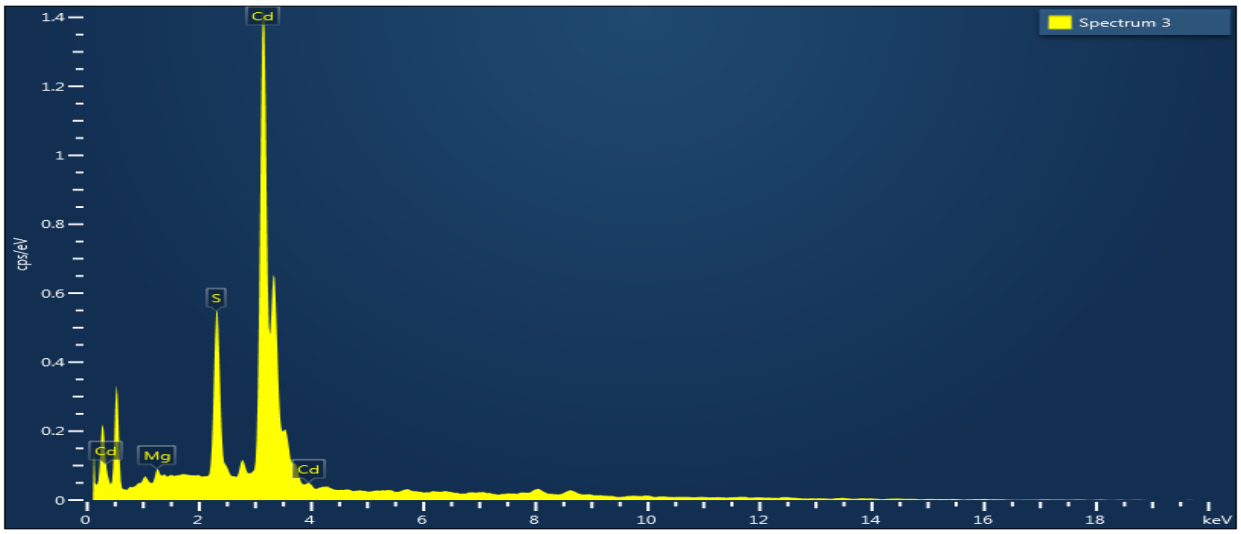

Fig-4b) EDX graph of CdS nanoparticles with 1 % doping of Mg

UV- Visible Spectra

UV- Visible Spectra Further characterization of CdS nanoparticle was confirmed by studying the optical properties. The yellow colour powder synthesized and the UV-Vis spectra were recorded by scanning in the range 200-700 nm. The UV-Vis spectra of the dispersed solution of the yellow colour powder synthesized showed absorption maxima between the wavelength 400-500 nm due to resonance band of the CdS nanoparticle. This confirms a blue shift from the bulk CdS which has absorption maxima of about 415 nm.

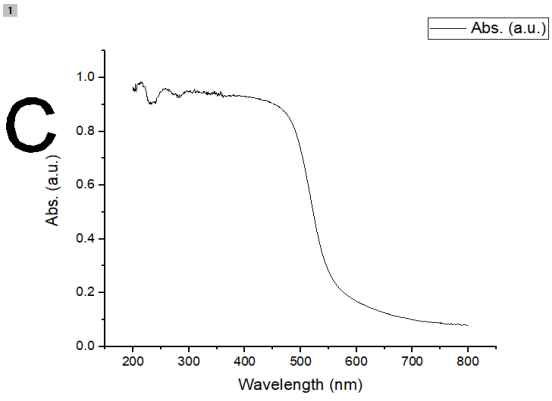

Fig-5 (a)

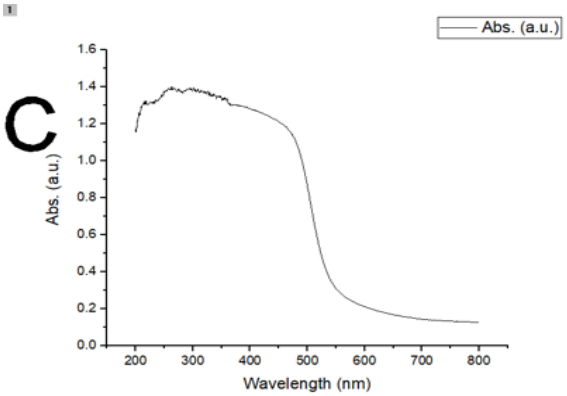

Fig-5 (b)

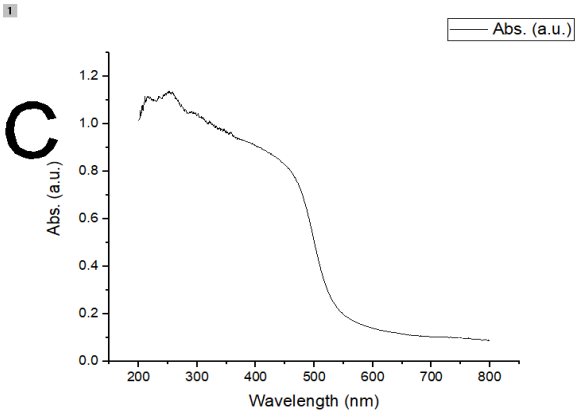

Fig-5 (c)

Fig-5 UV-Vis spectra **a)** CdS nanoparticle **b)** CdS Nanoparticles with 1 % doping of Mg **c)** CdS Nanoparticles with 5 % doping of Mg

STA Analysis-

Simultaneous Thermal Gravimetric analysis (TGA) was conducted to study the quantitative composition of CdS nanoparticles (Fig. 3a & 3b). For pure CdS nano- particles, the curves exhibit two distinct slopes. There is no initial weight loss between 30 and 300 °C means no physically adsorbed water is present in pure CdS and Mg doped CdS. This also confirms that pure and crystalline nanoparticles are obtained and no need to be calcinate under high temperature. Only loss of molecules with low molecular weight and weight loss between 400 to 500 °C indicates the complete stability towards heat. The whole weight loss of pure CdS and Mg doped CdS over the temperature range of 30–800 °C was about 22.45 % and 20.47 % respectively which reveals the good thermal stability of the prepared NPs.

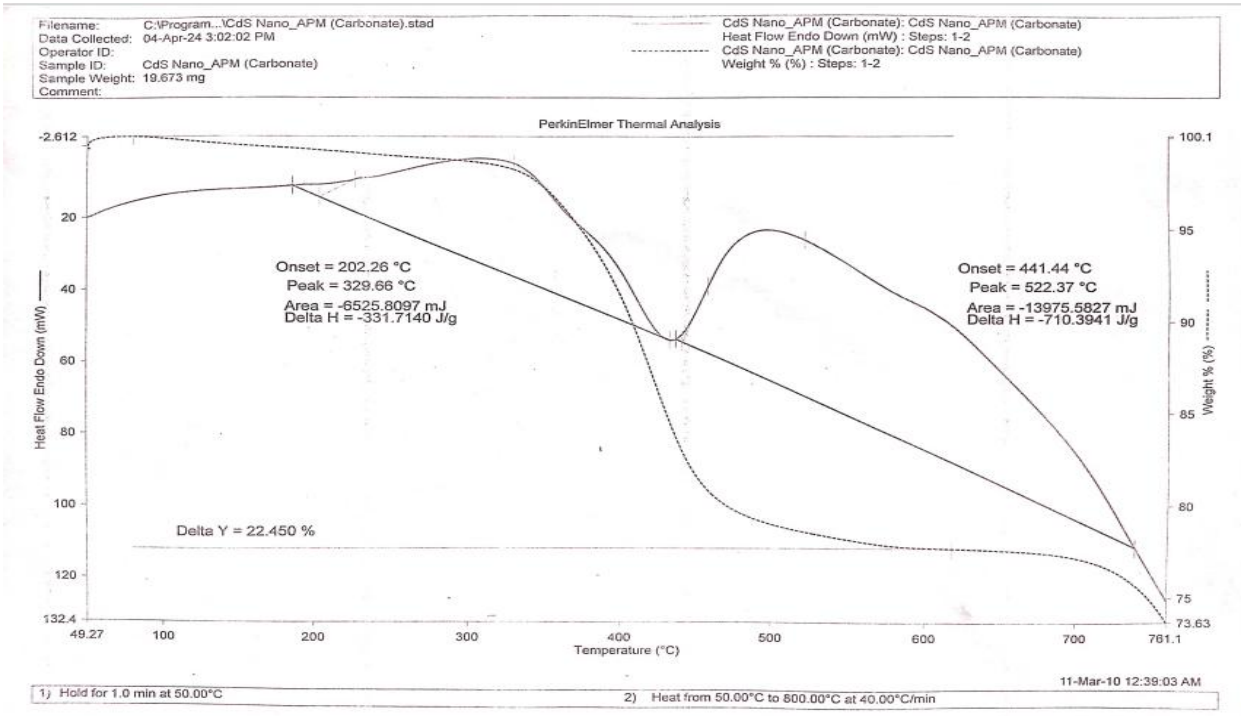

Fig-6a) STA of CdS

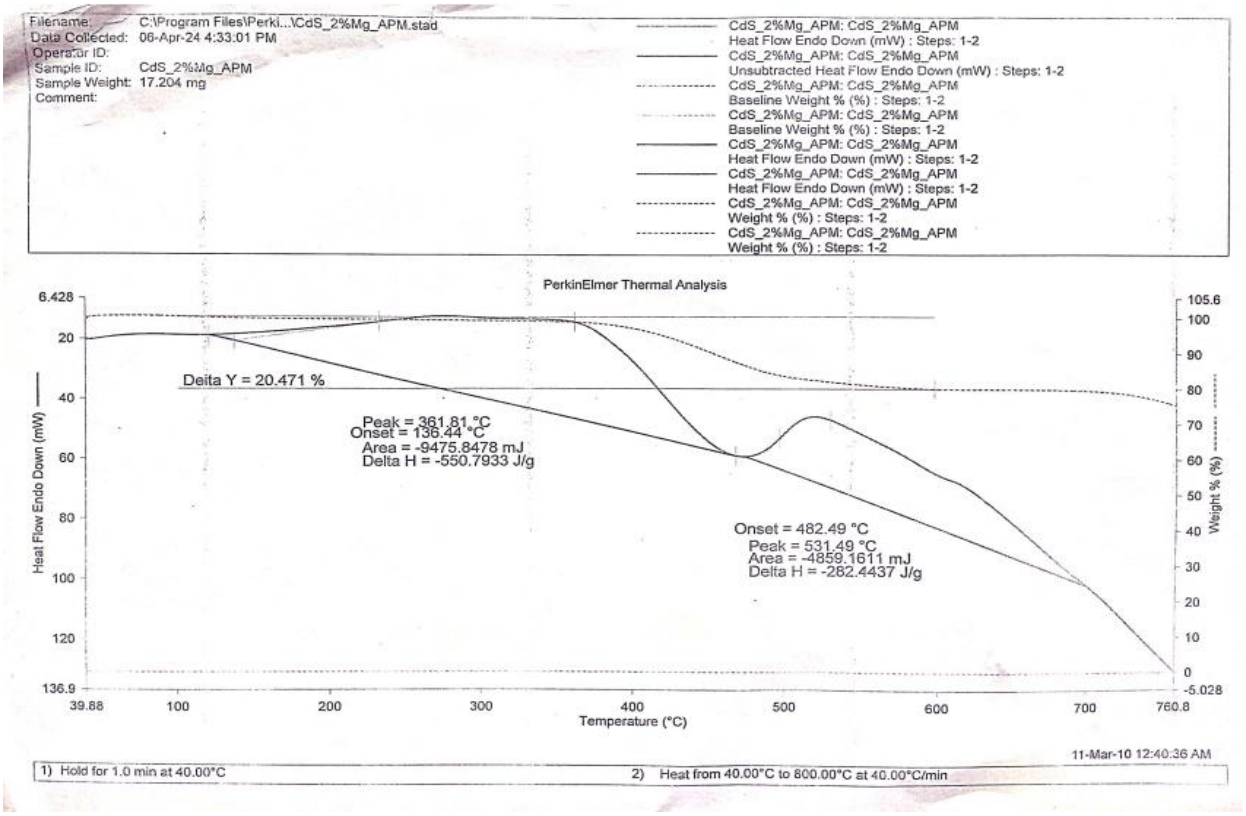

Fig-6b) STA of 1 % Mg doped CdS

IV. RESULTS

Table No- 1: CdS Nanoparticles

Sr. No.	Salt taken	MW (gm)	pH	Wt of CdS Nanoparticles (gm)	Wt of 1% Mg doped CdS Nanoparticles (gm)	Wt of 5% Mg doped CdS Nanoparticles (gm)
1	$CdSO_4$	770	5.0	0.547	0.855	0.862
2	$CdSO_4$	770	8.5	1.46	1.66	1.71

V. CONCLUSION

The stable and pure CdS NPs were successfully synthesized using various Cadmium salts by chemical precipitation method. The remarkable change has been observed changed while synthesized using ultrasonicator. The synthesis carried out at different pH values i.e. in basic and in acidic media. When we use $CdSO_4$, $CdCO_3$, $CdNO_3$ salts for synthesis, then weight of product increases and decreases for CdI_2, $CdCl_2$ at lower pH (pH=5). For doping Magnesium metal is used. The doping also varies with concentration (1%, 2% and 5%). The STA study shows that, the product is stable to heat and does not contain any other impurities. The morphology and structure of the synthesized nanoparticles were confirmed using the XRD.

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