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# Investigation of factors affecting the synthesis of nano-cadmium sulfide by pulsed laser ablation in liquid environment

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ABSTRACT

Pulsed laser ablation in a liquid medium is a promising technique as compared to the other synthetic methods to synthesize different materials in nanoscale form. The laser parameters (e.g., wavelength, pulse width, fluence, and repetition frequency) and liquid medium (e.g., aqueous/nonaqueous liquid or solution with surfactant) were tightly controlled during and after the ablation process. By optimizing these parameters, the particle size and distribution of materials can be adjusted. The UV–vis absorption spectra and weight changes of targets were used for the characterization and comparison of products.

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## 1. Introduction

In the last few decades, research on semiconductor materials in nanoscale had been increased enormously and has been extremely attractive and of interest to several fields which include optoelectronic devices, solid-state lasers and solar cells [1–3]. Among semiconductor materials, cadmium sulfide (CdS) nanostructures in the form of quantum dots, nanowires, and thin films are widely investigated by many researchers [4]. For these applications it is important to synthesize nanoparticles with the adequate size distribution, morphology and crystallinity. There are different techniques for producing materials in the nanoscale such as pulsed laser deposition, flame metal combustion, chemical reduction, photo-reduction, electrochemical reduction, solvothermal, electrolysis, microwave-induced, sono-electrochemical, aerosol flow reactor, photochemical reduction, chemical fluid deposition, spray pyrolysis, and spark discharge [5–20]. Many of these techniques use precursors and solvents, or imply chemical reactions which can contaminate the obtained nanoparticles. Pulsed laser ablation of solids in liquid environment (PLAL), one of these techniques, enables obtaining nanoparticles with no need of chemical precursors. Its simplicity together with the advantage of producing nanoparticles with small size, narrow distribution and weak agglomeration make it suitable for metal nanoparticle fabrication. This alternative physical nanofabrication method opened new routes for material processing based on the pulsed laser ablation of solids in various liquids. It could be used to produce a wide range of novel materials, such as nano-diamond and related nanocrystals, metallic nanocrystals, nanocrystal alloys, and metal oxides [21–24].

In the PLAL method, three main steps contribute to form nanoparticles from a target immersed in liquid. Only in a short period of time, typically about a few microseconds, all these steps take place and nanoparticles are synthesized. Firstly, laser pulse heats up the target surface to the boiling point, and thus, plasma plume containing vapor atoms of target is generated. Then, plasma expands adiabatically; and finally, nanoparticles are generated when condensation occurs [24]. During the condensation step, nucleation takes place, and the fine nuclei either collide and stick to each other or precipitate new materials on them which result in growth.

The efficient size control approaches employ variations of physical parameters of laser radiation, such as laser pulse energy, repetition rates, laser wavelength, focal spot size and focusing conditions [25–27]. Therefore, there are number of factors that should be taken into account during the laser–matter interaction in liquid environment. In this paper, CdS nanostructures were investigated under different promising factors, focusing conditions and ablation mechanisms, that help us to optimize laser parameters and focus on conditions to produce and control the shape/size of the desired nanostructures.

## 2. Experimental and materials

### 2.1. Materials

The materials used are Cadmium chloride hemipentahydrate ($CdCl_2 \cdot xH_2O$) of M.W. 228.35, specification assay 95%, from S.d.

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Fine-Chem. Ltd.; and Sulfur (S) of A.W. 32.97 from S.d. Fine-Chem. Ltd., Cadmium Sulfide (CdS) of M.W. 144.48 from S.d. Fine-Chem. Ltd. All chemicals were of analytical grade and used without further purification.

### 2.2. Experiment

CdS nanostructures were synthesized by a Nd:YAG laser which is a Q-switched solid state laser, which emits its fundamental line (λ) at 1064 nm producing a pulsed 7 nanosecond laser (ns), 10 Hz repetition rate and 5 mm effective beam diameter (Continuum Laser, Electro-optics, Inc. Model: PRII 8000).

The experimental setup for the generation of nanostructures by PLAL is shown in Fig. 1. The sulfur target samples used was in the form of a disc of about 0.85 cm diameter and 0.16 cm thickness. It was positioned inside a cuvette (inner dimensions 10 × 10 × 35 mm). The cuvette was filled by 1.5 ml of $CdCl_2$ solution. It was then placed onto a moving stage which could be moved in the x, y and z directions with accuracy of ±5 μm. The laser beam was focused onto the target material surface using a lens of focal length 7 cm. Laser ablation was carried out by scanning the sample. The ablation was carried out for 15 min and formation of nanostructures in the solution could be confirmed by the slight change of the color of the solvent during ablation. No bubbles were observed to adhere onto the sample surface during ablation which would have otherwise shielded or scattered the laser radiation.

The UV–visible spectra were measured in the range of 1000–200 nm by using a JASCO 570 UV–vis–NIR spectrophotometer. The shape and particle size distribution were studied using JEOL 2010 transmission electron microscopy (TEM) imaging. The samples were prepared by making a suspension from the powder in distilled water using an ultrasonic water bath. Then a drop of suspension was put on a carbon grid and left to dry. X-ray diffraction technique was used to determine the diffraction patterns of the crystal structure. An X-ray diffractometer (Shimadzu 7000, Japan), operating with Cu $K_\alpha$ radiation (λ = 0.154060 nm) generated at 30 kV and 30 mA with a scanning rate of 4°/min for 2θ values between 20 and 80°.

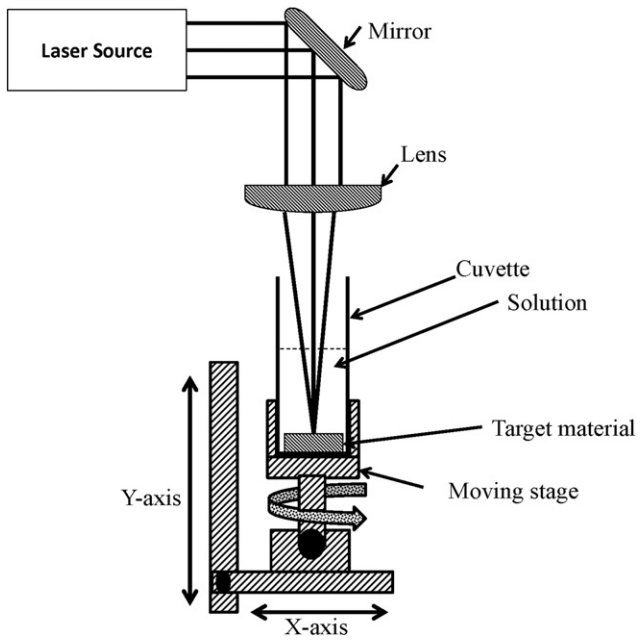


**Fig. 1.** Schematic representation of pulsed laser ablation in liquid environment technique.

## 3. Results and discussions

### 3.1. Optimization of PLAL process

The parameters of the PLAL-experiment were tightly controlled during and after the ablation process. These parameters were found to play a crucial role in controlling the shape and size of the produced nanostructures. Hence, it is worthwhile to optimize the PLAL process which can be divided into:

i) Adjustment of the PLAL system
ii) Adjustment of the ablated materials and its medium

#### 3.1.1. Adjustment of the PLAL system

Adjustment of PLAL-system depends on the study of the maximum emission and the highest sound of the plasma with respect to focal plane (Z = 0) (see Fig. 2). This study helps to achieve the best conditions to produce nanostructures. We do that by studying the sound intensity of the laser–matter interaction, UV–vis absorption spectra of the colloidal solutions, the particle size distribution of the nanostructures by using a ns-laser, 600 mW, and 1064 nm as a function of the target position approaching the focal plane (Z = 0) and fixing of all the other parameters of laser as shown in Figs. 3–5. The sound generated was recorded using a microphone connected to a computer and placed behind the laser ablation experiment. The intensity at a different focal plane with respect to the target surface was reported. Fig. 3 showed that the intensity of the sound increased as the target approached in front of the focal plane, at 450 μm, and then decreased again. Fig. 4 shows the variation of the maximum absorption of CdS nanostructures as the target approached in front of the focal plane (Z = 0) under a fixed laser energy. The absorption spectrum of the CdS nanostructure solution was measured at each position of target relative to the focal plane after 15 min of ablation by using the UV–visible spectrophotometer. After that the maximum absorption of each measurement was calculated and plotted as a function of the target position with respect to the focal plane (Z = 0). Therefore, by using the value of the absorption peak, we can estimate the concentration in the solution prepared at various target positions. So, when the production of the CdS nanostructures with high homogeneity increases at $Z_{max}$ = 450 μm, the absorption edge increases. Fig. 5 shows the particle size distribution calculated from UV–visible for ablation process as a function of the target position with respect to the focal plane (Z = 0) at fixed all the other parameters of the laser.The absorption coefficient (α), at the

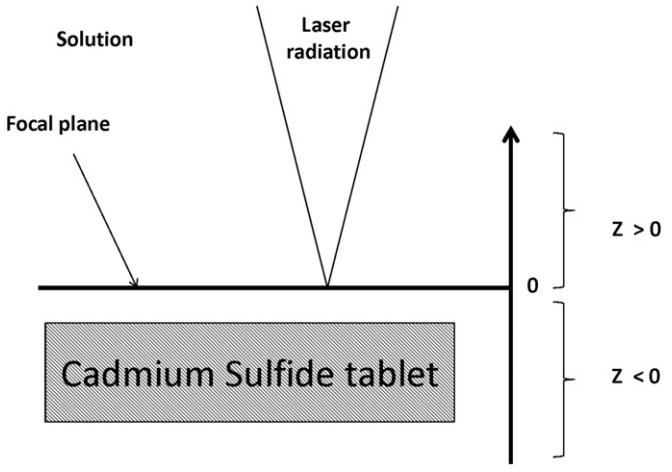


**Fig. 2.** Schematic of experimental setup of pulsed laser ablation in liquid environment.

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