SYNTHESIZE OF CDS NANOPARTICLES USING LIQUID-GAS METHOD

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Abstract. In this paper, Cadmium Sulfide (CdS) nanoparticles (NPs) was synthesized using Liquid-Gas method. After $CdCl_2$ mixed with surfactant in liquid phase, the mixture was exposed to S_2 gas from brimstone. The solution changes color to yellow which means that the CdS NPs semiconductor has been formed. It was approved by the blue-shifted of the absorption spectra of CdS colloid solution then the size (radius) of CdS NPs would be determined by using the Efros equation. The radius yielded was about 2 nm. FTIR result also confirmed the CdS bond at 509 cm^{-1}.

1. Introduction

During the past decade, semiconductors NPs have generated continuous interest because of their unique electrical and optical properties. II-VI group semiconductor has chemical stability at room temperature, wide band gap energies that are encompassing the entire visible spectrum, and direct band gap. One of the most studied systems among the II-VI group semiconductor is Cadmium Sulphide (CdS) [1, 2]. Size dependent effect of these NPs' optical and electronic properties have been studied experimentally and theoretically for possible application in solar cells, light-emitting diodes LEDs, electronic, optoelectronic, and quantum size effect semiconductors devices [3, 4].

To prepare semiconductor NPs as dispersions in organic or inorganic media or as aggregates, Cadmium Sulphide NPs has been synthesized using different methods such as Langmuir-Blodgett films [5, 6], colloid synthesis [3, 7, 8], reverse micelle [9], embedded in the polymer [10, 11] and electrostatic self-assembly [12], and green synthesizes [13, 14, 15]. One of the most important goals of nanomaterials synthesis is to control the size and stability of NPs.

In the previous research, CdS and ZnS colloid NPs were coated with organic shell containing either SO_3^- or NH_2^+ groups using thiolate ligand as a capping agent. The colloid NPs is stable. When NPs were transferred to the substrate by using the electrostatic self assembly (ESA) method, the aggregations occurred. In order to overcome this problem, a different procedure like the combination of aqueous phase and gas phase would be used. The reason is gas-phase processes are generally purer than liquid-based processes. Even the most ultra-pure water contains traces of minerals [16]. Therefore, to minimize the colloidal impurities of the NPs, after the preparation of the Cadmium salt, the solution is exposed to S_2 gas to form CdS NPs.

In this study, CdS NPs were prepared using the liquid-gas method where the sulfur (S_2) gas was from brimstone. The colloid NPs would be analyzed using a spectrophotometer for the spectra absorption. The size of the particles would be calculated theoretically using the Efros equation. The FTIR spectroscopy was used to investigate the functional groups of CdS molecule.

2. Method

2.1 Preparation of Sample

High purity chemicals purchased from Sigma-Aldrich were used to synthesize CdS colloid nanoparticles. Firstly, an aqueous solution of 0.02 M mercaptoethane sulfonate was mixed with 0.04 M solution of $CdCl_2$. All solutions were prepared using deionized water, at room temperature. Then, the solution was put in a closed chamber and exposed by S_2 gas produced from brimstone. The brimstone was taken from Ijen, East Java. The stone was crushed into powder (150 mesh) and weight for 0.0136, 0.0156 and 0.0176 g. The powder was burned in closed chamber with $CdCl_2$ solution. The result was a yellow solution of CdS colloid nanoparticles.

2.2 Experimental Methods

The solution's spectra were recorded using BEL - LGS 53 Series of UV-vis spectrophotometer at Material laboratory, Lambung Mangkurat University. It was measured by putting the NPs solution into 2 mL quartz cuvette and mounted into a sample holder in the spectrophotometer. Size of CdS nanoparticles can be evaluated from the blue shift of the absorption bands with respect to the band gap values of bulk CdS as a consequence of quantum confinement effect. In this work, the radius of semiconductor clusters is calculated using the Efros equation for the energy spectrum in NPs of direct band gap semiconductors, having parabolic *E(k)* dispersion. Assuming that the particle radius is smaller than the Bohr's exciton and the strong confinement condition, then [17]

$$E_{(n,l)} = E_g + \frac{\hbar^2}{2\,\mu\,R^2}\phi^2_{(n,l)} \qquad (1)$$

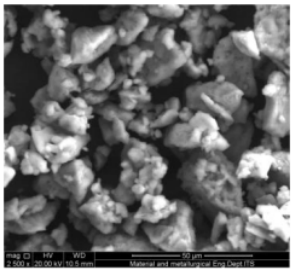
where E_g is the band gap for bulk semiconductors, μ is the reduced effective mass of exciton, $\frac{1}{\mu} = \frac{1}{m_e^*} + \frac{1}{m_h^*}$, and $\phi_{(n,l)}$ are the roots of Bessel functions (for the ground state $\phi_{(0,1)} = \pi$).

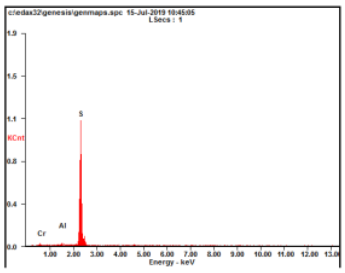
The functional groups of NPs were scanned in the infrared region of 400 to 4000 cm^{-1} using Fourier Transform Infrared Spectroscopy (FTIR) and then analyzed.

3. Result and Discussion

3.1 Sulfur Analyses of Brimstone

Firstly, brimstone was investigated by using SEM-EDX to know the sulfur contained in the stone. The result of SEM-EDX measurement could be seen in Figure 1 below. The results showed strong signals of S indicating that the average of sulfur contained in the stone was about 72 %. Sulfur is insoluble in water even when heated. To release sulfur, the stone was burned in closed chamber. To maximize the gas, sulfur was crushed into powder which passes 150 mesh sieve. The sulfur gas would react to $CdCl_2$ solution to form CdS NPs. In this study, the weight of burned sulfur was varied into 0.0136, 0.0156 and 0.0176 g. The amount of sulfur gas produced from the burning powder could not be known exactly. This is the weakness of this method.





Element	*Wt%*	*At%*
CrL	36.83	26.39
AlK	00.88	01.21
SK	62.29	72.40
Matrix	Correction	ZAF

Figure 1. SEM-EDX of brimstone at 20 KV and magnification of 2.500x.

3.2 The Absorption Spectra of CdS NPs Solution

The absorption spectra of CdS colloid solution is shown in Figure 2. As can be seen, the typical absorption spectra of CdS shows the gradually increase of absorbance to the absorption edge.

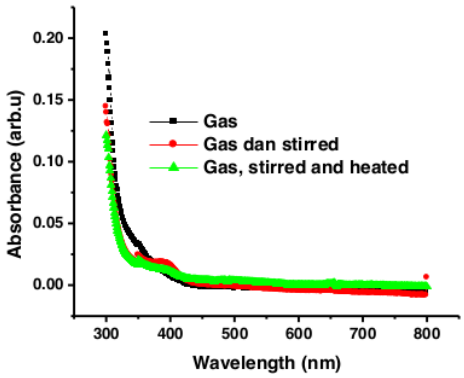
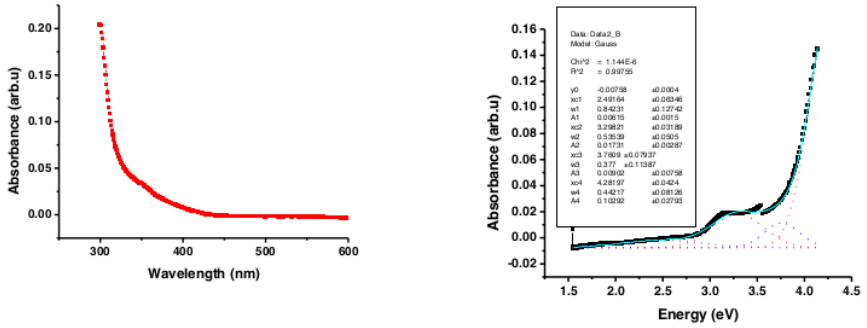


Figure 2. The UV–vis absorption spectra of CdS colloid NPs with three different treatments.

In Figure 2, it can be seen that the absorption spectra of CdS colloid NPs with three different treatments were presented. The absorption spectra of CdS NPs exposed by S_2 gas is represented by black square. The absorption spectra of CdS NPs exposed by S_2 gas while stirred is represented by red circle. And the absorption spectra of CdS NPs exposed by S_2 gas while stirred and heated are represented by green triangle. The weight sulfur powder was 0.0136 g, the results did not show a significant differences. Additional treatment, stirring and heating of the $CdCl_2$ solution showed a hump in the absorption spectrum around 400 nm which was not visible in the solution exposed to gas alone. In order to obtain the absorption peak of the CdS NPs, the UV–vis absorption spectra of CdS colloid NPs from measurement would be analyzed seperately.

The absorption spectrum curve of the CdS NP produced by exposure to S_2 gas alone is presented in Figure 3(a). In order to obtain the absorption peak, the absorption spectra of CdS colloid NPs from measurement (Figure 3(a)) was re-plot in energy coordinates. Then the Gaussian fitting of absorption spectra was performed to obtain the exact positions of absorption maxima, (see Figure 3(b)).



(a) **(b)**

Figure 3. (a) The UV–vis absorption spectra of CdS colloid NPs and (b) Gaussian fitting of the UV–vis absorption spectra of CdS

According to Nabok [6] and Yoffe [17], the blue shift value of the absorption band can be used to evaluate the size of the nanoparticles. From Figure 3 (b), the maximum absorption is generated at about 3.03 eV. In terms of wavelength, this energy corresponds to a wavelength of 409 nm. This means that there is a blue shift from bulk CdS wavelength (at about 512 nm) to CdS NPs (at about 409 nm). The observation of the blue shift of optical absorption is a typical experimental confirmation of nanoparticles' presence [17] and believed due to the effect of quantum confinement in the nanoparticles.

The observed energy dispersion may reflect the combination of the size distribution of nanoparticles and the presence of higher index energy levels of size quantization [6]. Only the first maxima (in each spectrum) corresponding to the ground state levels were chosen for further analysis. Based on Efros equation for the electron energy spectrum in nano-particles of direct band gap semiconductors, having parabolic *E(k)* dispersion [17]. Equation (1) was based on the assumption of strong confinement in the particles smaller than Bohr exciton radius, about 3 nm for CdS [17], so that electrons and holes are quantized separately the conduction and valence bands, respectively. The radius of semiconductor clusters would be calculated using Effective Mass Approximation (EMA) method above. The radius of nanoparticles of CdS NPs are presented in the table 1 below.

Table 1. The radius of CdS NPs based on results of Gaussian fitting of the absorption spectra

Method	M (g)	λ (nm)	E (eV)	ΔE (eV)	R (nm)
Gas	0.0136	409	3.03	0.61	1.93
	0.0156	409	3.03	0.61	1.93
	0.0176	410	3.03	0.61	1.93
Gas and stirred	0.0136	399	3.11	0.69	1.82
	0.0156	399	3.11	0.69	1.82
	0.0176	399	3.11	0.69	1.82
Gas, stirred and heated	0.0136	401	3.09	0.67	1.84
	0.0156	402	3.09	0.67	1.84
	0.0176	401	3.09	0.67	1.84

As can be seen in table 1, the radius of CdS NPs differ in a narrow range. Additional stirring and heating treatment provides a smaller radius. It is believed that stirring and heating would maximized the reaction between $CdCl_2$ and sulfur gas. As the result, more NPs were formed.

3.3 FTIR of CdS NPs Solution

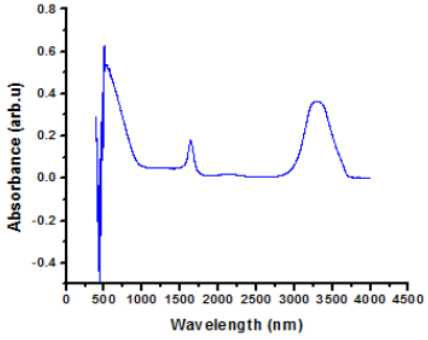


Figure 4. FTIR spectra of CdS NPs solution.

The result of Fourier Transform Infrared Spectroscopy (FTIR) measurement is presented in Figure 4. The FTIR results showed some absorption peaks in the region 4000 - 400 cm-1 which are at around 509, 1633, and 3300 cm^{-1}. The peaks occurring at 3300 cm^{-1} has corresponded to O-H stretching vibrations of the carboxyl group [15]. The peak at 1633 cm^{-1} has corresponded to C-C bending vibration. It is believed that both of these peaks were from surfactant used for capping agent which is merchapto ethane sulfonate ($C_2H_5NaO_3S_2$). It is assumed that the absorption peak at 509 cm^{-1} is corresponded to the stretching of CdS bond. According to Kumar [18] dan Rusu [19], the peak of CdS was at 500 cm^{-1} while Karthik confirmed the vibration of Cd–S bonding at 601 cm^{-1} [20]. When compared with this study, the CdS peak formed was slightly different. This is probably due to the different synthesis methods used.

4. Conclusion

The colloid of CdS NPs were successfully synthesized by using liquid-gas method. The particle core radius in the range of 1.8–1.9 nm for CdS NPs was obtained from UV–vis absorption spectra. The FTIR result showed the absorption peak of CdS at 509 cm^{-1}.

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