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Synthesis of CdS Nanocrystalline using Parkia speciosa Hassk Seeds Extract: Optic, Structure, and Morphology

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Info Artikel	**Abstrak**

Kata kunci:
Synthesis, CdS, Nanocrystalline, *Parkia speciosa Hassk*

Cadmium sulfide (CdS) nanocrystalline was successfully synthesized using *Parcia speciosa Hassk*. This study aims to utilize *Parcia speciosa hassk* seeds as a source of natural sulfur in the modification of CdS nanocrystalline. The method used is the green synthesis eco-friendly of approach. Powders of CdS nanocrystalline was characterized by X-ray diffraction (XRD), UV-Vis diffuse reflectance spectrophotometer (UV-Vis DRS), Particle size analyzer (PSA), Scanning electron microscopy-energy dispersive X-ray (SEM-EDX), Fourier transform infrared spectroscopy (FTIR). XRD shows the peak of a specific CdS nanocrystalline at 2 theta: 27.66^o, 49.92^o, and 60.13^o respectively. CdS crystal average size of 23 nm. Analysis of UV-Vis DRS showed absorption at the wavelength of 496 nm. The calculation results, obtained band gap value CdS nanocrystalline of 2.5 eV. The results of PSA showed CdS particle size distribution is ± 98 nm. SEM-EDX shows the morphological of CdS nanocrystalline has sphere shape with a component of element Cd (83%) and S (17%) respectively. Analysis of FTIR showed the presence of Cd-S vibrations at wavenumber 512 cm^{-1}.

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INTRODUCTION

Nanotechnology is an material engineering that has particle size between 1-100 nm. Many of nanomaterial applications such as in the fields of the textile industry, food products, cosmetics, medicine, and ceramics. One example of material engineering is the synthesis of cadmium sulfide (CdS) nanomaterials (Alex and John, 2001). CdS is inorganic solid classified as a type II-IV semiconductor material (Celebi *et al*, 2007). CdS has unique characteristics such as optical, electrical, and fluorescence, so it is widely used in the field of optoelectronic devices (Luccio *et al*, 2007). Materials of CdS has optical properties that work in the visible area, because it has a band gap value energy of 2.5 eV.

Materials of CdS was successfully applied as photoluminescence (Lee *et al*, 2009), photocatalytic activity (Huo *et al*, 2012), visible-light absorption (Zhou *et al*, 2014), gas sensing properties (Hasani *et al*, 2015), and methylene blue adsorption (Makama *et al*, 2016). Synthesis of CdS required a capping agent as a particle size stabilizer so that particles of CdS not agglomerate. Far there are still many used of capping agents that are not environmentally-friendly. The methods of synthesis used in growth of CdS such as arrested precipitation using organic compound: phosphine, alkylthiol, phosphine oxide, phosphate, amine or amide, nitrogen aromatic, and carboxylic acid (Suryajaya *et al*, 2012). Organic compounds used function as ligands surface passivating in the synthesis of CdS. The use of organic ligands is very dangerous and not environmentally friendly.

Besides ligands and methods, synthesis of CdS also required precursors of cadmium and sources of sulfur. The precursors that are often used in the synthesis of CdS material are cadmium chloride, cadmium sulfide, and cadmium nitrate (Lee *et al*, 2009). The source of sulfur as a modified element of cadmium from the chemical compound thiourea and the base source from the compound of NaOH, hexamine, and KOH. To reduce the use of hazardous materials, this research aims to replace the source of sulfur, and capping agents derived from natural ingredients, namely using *Parkia speciosa Hassk* seeds extract to synthesis CdS nanocrystalline.

Phytochemical test results of *Parkia speciosa Hassk* seed extract contain secondary metabolites such as alkaloids, flavonoids, and saponins. In addition, *Parkia speciosa Hassk* seeds also contain sulfur in every amino acid content. The presence of chemical compounds contained in *Parkia speciosa Hassk* seeds, the *Parkia speciosa Hassk* seeds are widely used as a prevention of kidney disease, liver, enhance immunity and antioxidants (Verawaty, 2018). In the presence of natural compounds contained in *Parkia speciosa Hassk* seeds, is expected to be a source of bases, sulfur and capping agents in the synthesis of CdS nanocrystals and can be produce optical properties, structure and morphology of CdS nanocrystals.

MATERIALS AND METHOD

Preparation of *Parkia speciosa Hassk* Seed Extract

About 10 g seed were boiled and stirring with 100 mL of distilled water for 1 hours at 60^0 C until the colour of the aqueous solution changes from watery to light yellow. After that, the yellow colored extract was filtered throught Whatman No. 1 filter paper.

Synthesis of CdS Nanocrystals using *Parkia speciosa Hassk* Seeds Extract

About 25 ml of *Parkia speciosa Hassk* seeds extract was taken from the stock solution. Then, 2 g of cadmium nitrate tetrahydrate $Cd(NO_3)\cdot 4H_2O$ was added to above extract and kept under continuous stirring at 80^0 C for 8 h. This paste was then collected in a ceramic crucible and heated in a furnace at 600^0 C for 6 h. Results of powders CdS in characterized.

Characterization of CdS Nanocrystals

UV-Vis diffuse reflectance spectrophotometer (UV-Vis DRS) were carried out with a measurements UV40404B in the wavelength range of 200-800 nm in reflectance mode. Functional group of the compound was examined by using Fourier Transform infrared spectroscopy (SHIMADZU, INDONESIA). The crystal phase information of sample was characterized from 20° to 70° in 2θ by a XRD with Cu Kα (λ=0.1546 nm) radiation (D8-Advanced, Bruker). The surface morphology of CdS nanocrystals was examined by scanning electron microscope (SEM), and the chemical composition of product were examined by energy dispersive X-ray spectroscopy (EDX, AMETEK). Particle size distribution seen using Particle Size Analyzer (PSA) Malvern ZEN 1600 with dynamic light scattering system.

RESULTS AND DISCUSSION

Solution of *Parkia speciosa Hassk* seed extract produces a yellow color and has a yield weight of 0.05% with a 5% stock solution concentration. Figure 1 shows the peak crystallinity that is typical of CdS. CdS nanocrystals has three peaks namely at the 2 theta military index: (a) 27.66°, (b) 49.92°, and (c) 60.13° respectively. This result is in accordance with JCPDS No: 10-454. The results of calculations using the Scherer"s formula, CdS nanocrystals has an average particle size of 23 nm. CdS has a cubic structure crystal shape (Makama *et al*, 2016).

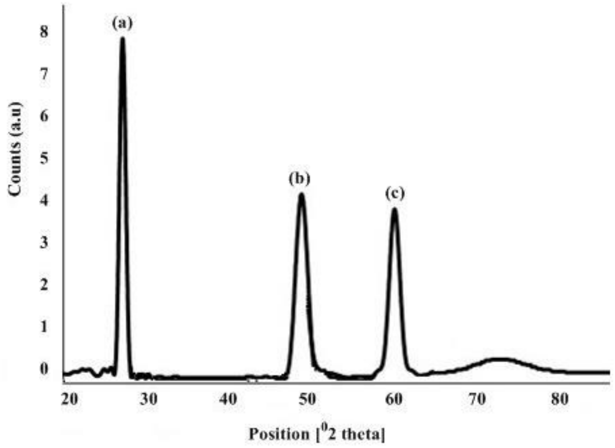

Figure 1. Powder XRD Patterns of the CdS Nanocrystals

Figure 2 shows the absorption peak of CdS nanocrystals and band gap value. The reflectance spectrum was analyzed using the Kubelka-munk equations (Thema *et al*, 2015). CdS nanocrystals has a wavelength of at λ_{max} of 496 nm (Fig.2a). The CdS nanocrystals exhibits the indirect band gap energy (Eg) of 2,5 eV (Fig.2b). Band gap values of CdS nanocrystals are characteristic and optic of CdS that works in visible light in liquid waste degradation applications (Luccio *et al*, 2007).

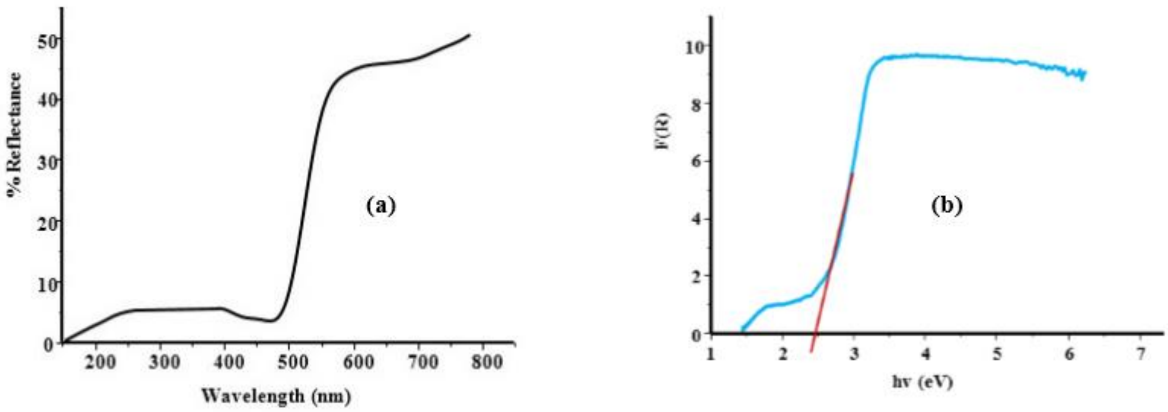

Figure 2. (a) UV-Vis DRS Spectrum and Plot of Indirect Band Gap Energy of CdS Nanocrystals

Instruments of particle size analyzer (PZA) is used for particle size distribution analysis. Figure 3 showed the distribution of CdS nanocrystals.

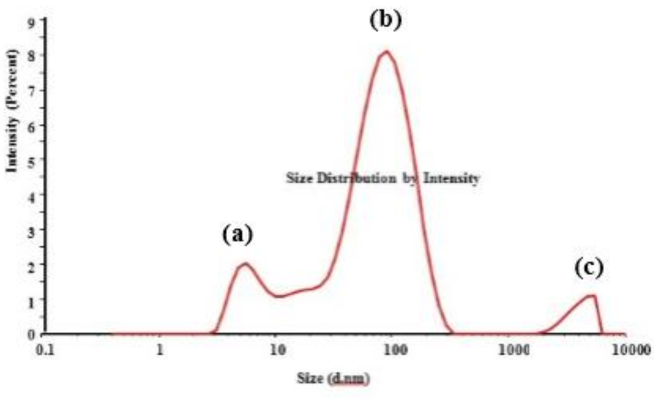

Figure 3. Distribution PSA of CdS Nanocrystals

CdS nanocrystals has 3 peaks at percent intensity of (a) 2, (b) 8.5, and (c) 1.5. Particle size distribution of CdS nanocrystals is 98 nm. The results on particle measurements using PSA are far greater than the results of the CdS crystal size on XRD. This is predicted in PSA measurements, the capping agent on the surface of CdS particles is also measured so that the particle size becomes larger. The poly disperse index (PDI) value of CdS nanocrystals was 0.6. The greater of PDI value produced in the PSA measurement, the particle size is more homogeneous.

Functional groups contained in compounds in *Parkia speciosa Hassk* seed extract were seen in characterization using FTIR. Figure 4 shows vibration of functional groups at synthesis of CdS nanocrystals. Synthesis of CdS nanocrystals using *Parkia speciosa Hassk* seed extract showed a functional group consisting of –OH at 3330 cm^{-1} and a C-H at 1420 cm^{-1}. Vibrations of Cd and S seen are strong at wavenumbers 512 cm^{-1}. This indicated that the CdS nanocrystals were successfully formed.

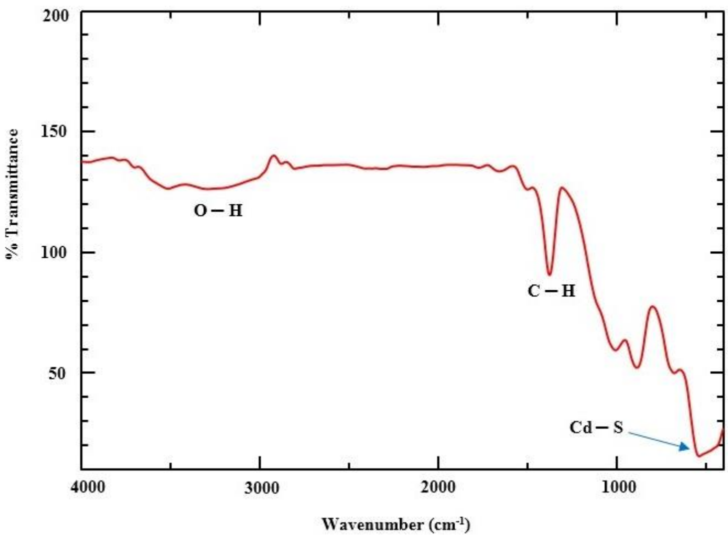

Figure 4. FTIR of CdS Nanocrystals

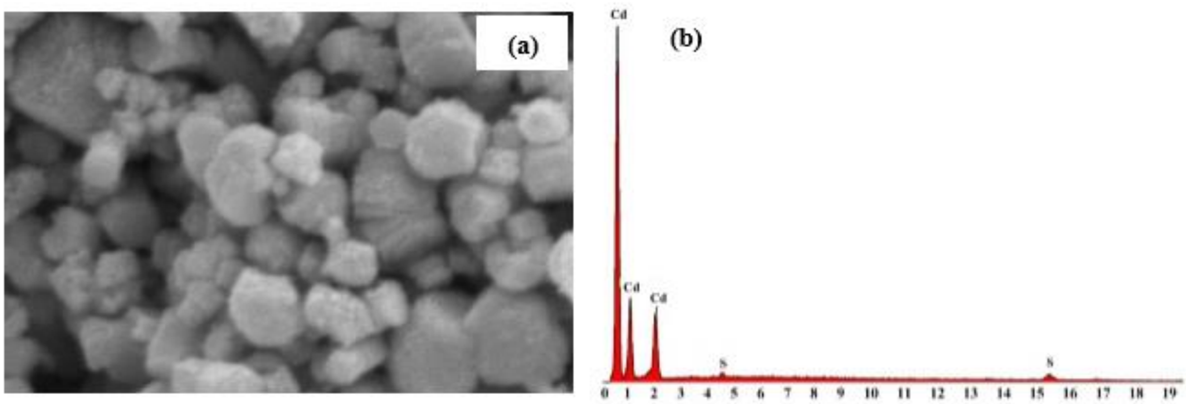

Figure 5. (a) Morphology SEM and (b) EDX of CdS Nanocrystals

Figure 5 showed the morphology shape of CdS nanocrystals with the composition of elements contained there in CdS. Morphology shape of CdS nanocrystals of sphere shaped with the composition of elements Cd and S with 83% and 17% respectively.

CONCLUSIONS

The nanocrysts of CdS was successfully synthesized using *Parkia speciosa Hassk* seeds. The optical properties of CdS nanocrystals has a band gap value of 2.5 eV with λ_{max} value of 496 nm. Crystal structure cubic with a mean CdS crystal size of 23 nm. The average CdS particle size distribution is 98 nm. There is a functional group in the *Parkia speciosa Hassk* seed extract seen in wavenumber 3330 cm^{-1} (-OH), 1420 cm^{-1} (C-H), and Cd-S vibrations at 512 cm^{-1}. Spherical shaped CdS nanocrystals with a composition of Cd and S of 83% and 17% respectively.

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