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Potency of *Parkia speciosa* Hassk seed extract for green synthesis of CdO nanoparticles and its characterization

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Abstract. CdO Nanoparticles (CdO NPs) have been green synthesized via sol-gel method using aqueous extract of *Parkia speciosa* Hassk (PSH) seed as the base source and stabilizing agent. The structural and optical properties of the formed CdO were investigated by X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy, and UV-visible diffuse reflectance spectroscopy (UV-Vis DRS). The optical band gap of CdO NPs was estimated by UV-Vis DRS spectroscopy and calculated from reflectance spectrum ~ 2.00 eV. FTIR spectrum showed the functional groups of active compound of PSH seed extract which plays a role in the CdO NPs formation.

Keywords: *Parkia speciosa* Hassk, CdO nanoparticles, green synthesis, sol-gel synthesis method

1. Introduction

Nanoparticles (NPs) show the unique chemical, physical, optical, electrical and transport properties, which are very different from those of bulk materials and single atoms [1, 2]. The NPs have high surface to volume ratio and high surface energy. The formation of stable NPs to avoid agglomeration can be achieved by minimizing the surface energy [3, 4].

Semiconductor nanoparticles have been the subject of great interest in the past years due to their optical and electrical properties. Among the group II–VI semiconductor nanoparticles, CdO is considered to be one of the promising metal oxides with n-type semiconductor and band gap of ~ 2.5 eV [5]. It has low resistivity owed to oxygen vacancies and cadmium interstitials. Due to the high electrical conductivity and optical transmittance in the visible region of solar spectrum, CdO has potential applications in flat panel display, organic light emitting diodes, gas sensors, solar cells etc. [6].

CdO NPs are known to be acutely and chronically toxic to cells and organisms among the majority components of nanomaterials [7], and the commonly used capping molecules for the synthesis of nanoparticles such as mercaptoacetic acid, ethanolamine etc. are also toxic in nature and their large scale production can induce a potential threat to the environment [8, 9]. Therefore, it is necessary to design and use methods which are based on green chemistry to synthesize nanomaterials in order to improve and to protect the environment [10].

Recently, green chemistry has attracted researchers in nanosciences field [11]. Different green sources such as bacteria [12], fungus [13], plants [14], and algae [15] are used for nanoparticles synthesis. In the present study, we have prepared the aqueous extract of *Parkia speciosa* Hassk (PSH) seeds as the base source and stabilizing agent for the synthesis of CdO NPs through a sol-gel method.



2. Materials and methods

2.1. Preparation of PSH seed extract

Parkia speciosa Hassk (PSH) plant seeds were taken from University of Indonesia area. The PSH fresh seed was dried at room temperature and 5 g of the seed powder was stirred with 100 mL double distilled water for 1 h at 70°C until the colour of colloid changes to light yellow, and filtered using a Whatman No. 1 filter paper.

2.2. Green synthesis of CdO NPs

25 mL of PSH seeds extract was used for CdO NPs synthesis through sol-gel method. 1.5 g of $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ was added into the extract and kept under continuous stirring at 85°C for 6 h. This paste was collected and heated in a furnace at 400°C for 2 h.

2.3. Characterization of CdO NPs

The UV-Vis absorption spectrum was recorded using SHIMADZU UV-Vis 2600 spectrophotometer. UV-Vis diffuse reflectance spectrum (UV-Vis DRS) was carried out using SHIMADZU UV-Vis 2450 spectrophotometer in reflectance mode in the wavelength range of 200–850 nm. The functional group of the extract compound was examined using Fourier transform infrared spectroscopy (PRESTIGE 21 SHIMADZU). The crystal phase information was characterized from $2\theta = 20\text{--}80^\circ$ by XRD with $\text{Cu K}\alpha$ ($\lambda = 0.1546$ nm) radiation (D8-Advanced, Bruker). The surface morphology of NPs was examined by JEOL JSM 6390 Scanning electron microscope (SEM) instrument operated at an accelerating voltage at 10 kV. The chemical composition of the product was examined by energy dispersive X-ray spectroscopy (EDS).

3. Results and discussion

3.1. UV-Vis absorption spectrum and DRS of CdO NPs

Figure 1 shows the UV-Vis absorption spectrum of the synthesized CdO NPs in colloidal system at maximum wavelength, λ_{max} 294 nm using UV-Vis spectrophotometry.

Figure 2a shows the DRS of CdO NPs prepared using 25 mL of PSH seeds extract. It shows an absorption inflection point at around 480 nm, which could be assigned to the excitonic absorption feature of CdO. The calculated direct band gap (E_g) from diffused reflectance spectrum was 2.0 eV as shown in

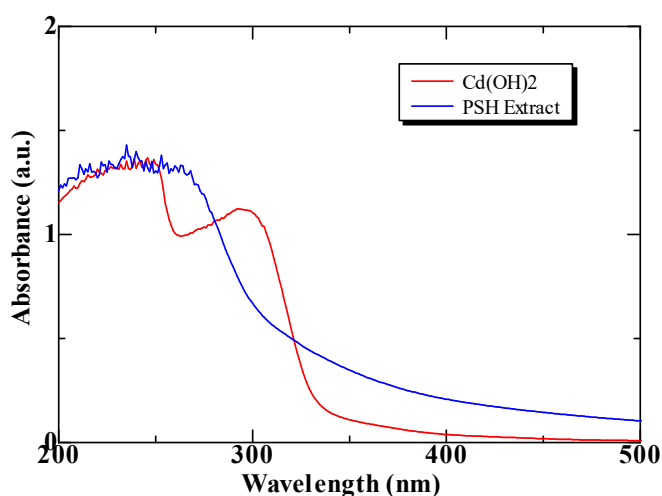


Figure 1. UV-Vis absorption spectra of the synthesized CdO NPs colloid

figure 2b, which was in good agreement with the reported values [16]. However, a small difference in E_g value was noticed due to various parameters, such as method of synthesis, size of particle, crystallinity, morphology, surface area, etc [17].

3.2. FTIR spectroscopy of CdO NPs

FTIR was used to study the purity and functional group composition of CdO NPs. The existence of strong FTIR spectrum of interactions between CdO and capping molecules were confirmed in the wavenumber region of $4000\text{--}400\text{ cm}^{-1}$ for the synthesized CdO NPs as shown in figure 3. The broad FTIR absorption band at 3457 cm^{-1} is assigned to stretching vibrations of hydroxyl group (O–H) of water molecules. The FTIR bands at 3254 and 3248 cm^{-1} are assigned to N–H asymmetric and symmetric stretching vibrations, respectively. The FTIR bands at 2970 and 2876 cm^{-1} are assigned to asymmetric and symmetric stretching vibrations of C–H group. A strong absorption band at 1366 cm^{-1} is due to wagging vibrations of C–H group [18]. The above peaks confirmed the presence of stabilizing molecules from PSH seeds extract. In the synthesized product, the metallic bonds identified near the 862 cm^{-1} due to metal-hydroxide (M–OH), and the band observed at 705 cm^{-1} is due to metal–oxygen (M–O) bond. It indicates the formation of CdO NPs.

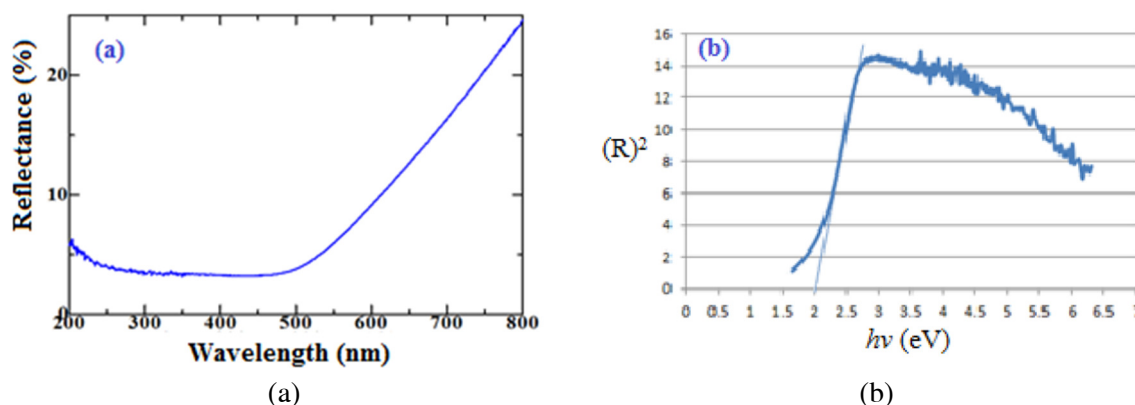


Figure 2. (a) Diffuse reflectance spectrum (b) direct band gap energy of CdO NPs.

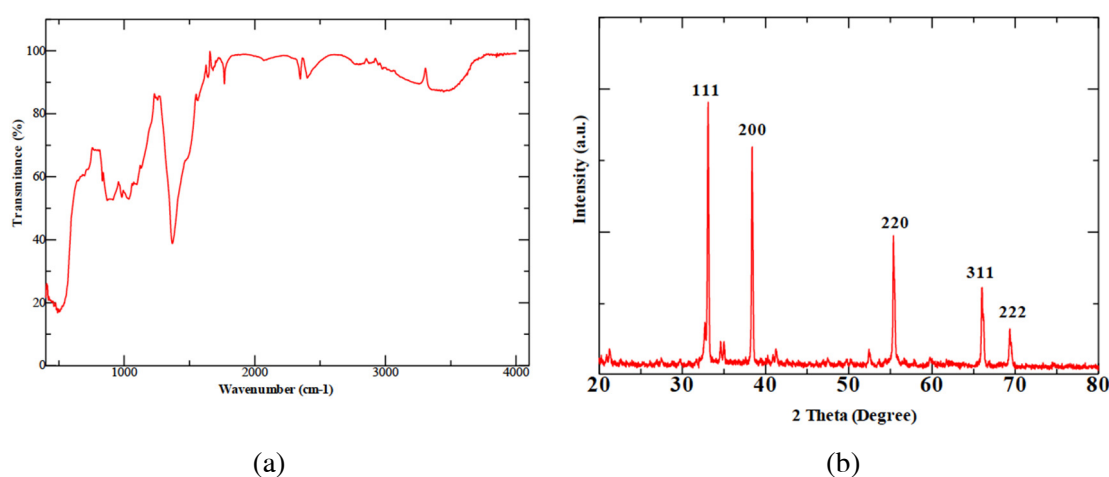


Figure 3. (a) FTIR spectrum and (b) XRD spectrum of synthesized CdO NPs.

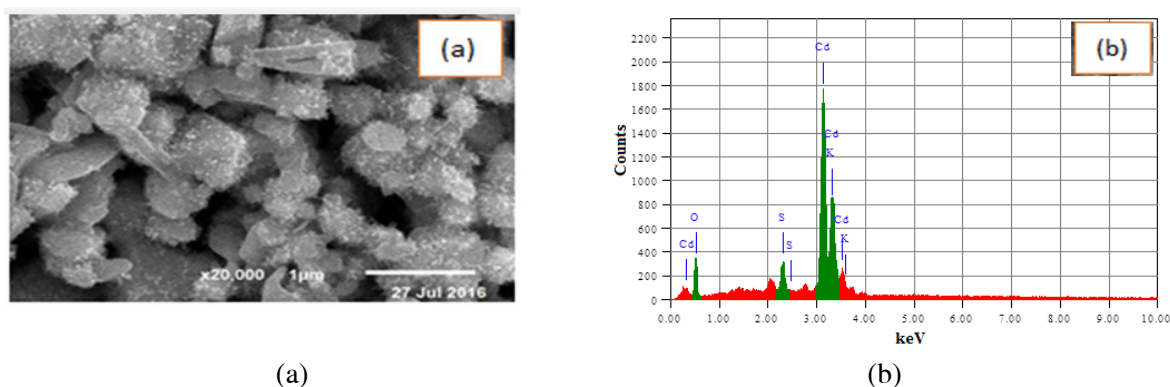


Figure 4. (a). SEM images (b). EDS spectrum of CdO NPs.

3.3. XRD of CdO NPs

Figure 3b displays a typical XRD profile of the highly crystallized sample, i.e. the powder annealed at 400°C. It exhibits five diffraction peaks at 33.07°; 38.37°; 55.37°; 66.01°; 69.35°. More accurately, the diffraction peaks can be indexed as (111), (200), (220), (311) and (222) reticular orientations of the single phase cubic Monteponite CdO structure after adjusted with JCPDS card no. 05-0640 with an average lattice parameter $\langle a_{\text{exp}} \rangle = d_{\text{hkl}} [(h^2 + k^2 + l^2)^{1/2}] = 2.23617 \text{ \AA}$. From the broadening of Bragg peaks and using the Debye-Scherrer approximation, the average size of nanocrystals was ranged from 28.92 to 44.95 nm.

3.4 SEM and EDX of CdO NPs

SEM image of calcined CdO (figure 4a) exhibits a clear alteration in morphology. The diameter of CdO NPs becomes heterogeneous with particle size between 20 and 50 nm. These CdO NPs have scales around the stick to form nanoscale stick-like morphology. In figure 4b, the observed peaks of EDS spectrum at the different energy channels can be attributed to Oxygen (O), Cadmium (Cd), Potassium (K) and Sulfur (S). Consequentially, there are the other elements observed in CdO NPs as impurities.

4. Conclusions

The CdO NPs were successfully green synthesized using *Parkia speciosa* Hassk seed extract as the base source and stabilizing agents. UV-Vis DRS data confirmed the direct band gap at 2.0 eV. FTIR spectrum revealed the CdO bond was found in the range of 400-705 cm^{-1} . The XRD data showed the well crystallized in cubic phase of CdO Monteponite structure with the average size of the nanocrystals ranges within 28.92.9 and 44.95 nm.

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