Jusami
Jurnal Sains Materi Indonesia
Homepage: http://jusami.batan.go.id
Akreditation No : 36/E/KPT/2019
Date 13 Dec 2019
ISSN 1411-1098
E-ISSN 2614-087X

SODIUM LAURETH SULFATE (SLS) DECORATED α-PBO NANOCRYSTALS: OPTICAL, STRUCTURE, AND MORPHOLOGY PROPERTIES

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Received: 4 January 2021 Revised: 14 April 2021 Accepted: 21 April 2021

ABSTRACT

SODIUM LAURETH SULFATE (SLS) DECORATED α-PbO NANOCRYSTALS: OPTICAL, STRUCTURE, AND MORPHOLOGY PROPERTIES. The α-PbO nanocrystals were successfully decorated using sodium laureth sulfate (SLS) anionic surfactant. The method used is one-pot synthesis approach. The precursor used is lead nitrate ($Pb(NO_3)_2$). The UV-Vis spectrophotometer showed the absorption peak of α-PbO nanocrystals was seen at wavelength of 237 nm and an absorbance value of 0.7. The optical properties of PbO nanocrystals can be seen at the bandgap value of 4.2 eV. FT-IR spectroscopy showed the shift of absorption peak at the wavenumber of 1358 cm^{-1}. XRD spectroscopy showed the crystals of PbO at diffraction angles (2θ) of 10-80°: 29.17, 32.54, 37.85, 39.62, 45.16, 46.21, 56.12, and 61.73 with miller indices of (111), (200), (201), (121), (220), (030), (311), and (032), respectively. The crystal size average of PbO was 56.32 nm. The results of PSA and PZC shows the particle size distribution of PbO is 71.5 nm with inter-particle charge of -25 mV. SEM-EDX data shows the PbO nanocrystals have an irregularly spherical with a compounds composition of Pb (83.12%) and O (16.88%). From the data of characterization, it can be concluded the PbO nanocrystals was successfully decorated using the surfactant anionic of sodium laureth sulfate.

Keywords: Decorated, Surfactant, PbO, Nanotechnology

ABSTRAK

NANOKRISTAL α-PbO DIDEKORASI SODIUM LAURET SULFAT (SLS) : SIFAT OPTIK, STRUKTUR DAN MORFOLOGI. Nanokristal α-PbO telah berhasil didekorasi menggunakan surfaktan anionik SLS menggunakan metode pendekatan sintesis satu pot. Prekursor yang digunakan adalah timbal nitrat ($Pb(NO_3)_2$. Puncak serapan nanokristal-PbO terlihat pada panjang gelombang 237 nm dan nilai absorpsi 0,7 pada Spektrofotometer UV-Vis. Sedangkan sifat optik dari nanokristal PbO dapat dilihat pada nilai bandgap 4.2 eV. Spektroskopi FT-IR menunjukkan pergeseran puncak serapan pada bilangan gelombang 1358 cm^{-1}. Spektroskopi XRD pada kristal PbO ditunjukkan pada sudut difraksi (2θ) dari 10°-80°: 29,17; 32,54; 37,85; 39,62; 45,16; 46,21; 56,12; dan 61,73 dengan indeks miller masing-masing (111), (200), (201), (121), (220), (030), (311), dan (032). Rerata ukuran kristal PbO adalah sekitar 56,32 nm. Hasil PSA dan PZC menunjukkan distribusi ukuran

partikel PbO yaitu sebesar 71,5 nm dengan muatan antar partikel -25 mV. Data SEM-EDX menunjukkan bahwa nanokristal PbO memiliki bentuk sferis yang tidak beraturan dengan komposisi senyawa Pb (83,12%) dan O (16,88%). Dari data karakterisasi dapat disimpulkan bahwa nanokristal PbO berhasil didekorasi menggunakan surfaktan anionik sodium lauret sulfat.

Kata kunci: Didekorasi, Surfaktan, PbO, Nanoteknologi

INTRODUCTION

Nanotechnology has a very significant development in science. Nanomaterials have a particle sizes from 1-100 nm and a unique of physical and chemical properties [1]. The interactions on the nanomaterials led researchers to modification in materials. Lead II oxide (PbO) is a metal oxide compound that is widely applied in X-ray imaging detectors and lead storage batteries [2].

Lead II oxide (PbO) is a photosensitive material so that it can be used as an optical sensor. Some other lead oxide applications are usable in supercapacitor, CO_2 photoreduction activity [3], superconductivity [4], and in-vitro biological application [5]. Due to their electronic properties and ease of production, there is great potential in its use and other materials for various applications. PbO has a two forms. The alpha (α) form is commonly referred to as litharge (red-tetragonal) [6], and a beta (β) form is widely known as massicot (yellow-orthorhombic) [7].

The synthesis of PbO can be carried out by chemical or physical routes. Thermal decomposition of lead precursors [8], spray pyrolysis [9], sonochemical [10], and electrochemical [11] methods have been applied for the synthesis of PbO. However, this process has several drawbacks. By physical means, synthesis requires high energy and high vacuum conditions, while chemical means are concerned with creating toxic and hazardous wastes that make them environmentally unfriendly [12]. To reduce energy consumption and hazardous waste, green and environmentally friendly alternative methods are proposed [13].

Apart from the method, the synthesis of PbO nanoparticles need involves base sources, reducing agents, and capping agents. Capping agent functions as a protector of PbO nanoparticles so that not agglomeration. Capping agents that have been used such as PVC polymer membranes [6], Proteins [14], and PVP matrix [15].

From the literature, α-PbO nanocrystal never been decorated using surfactant of sodium laureth sulfate (SLS). In this research, α-PbO nanocrystal will be synthesized using surfactant of sodium laureth sulfate. The synthesis process took place in one-pot by directly reacting the precursor $Pb(NO_3)_2$ with SLS surfactant at 95 °C. The α-PbO was investigated to see the optical properties, structure, and morphological.

The α-PbO nanocrystal decorated using SLS is expected to change the particles size of PbO on the nano scale. So that α-PbO which has a particle size on the nano scale.

EXPERIMENTAL METHOD

Materials and Instruments

Sodium laureth sulfate (SLS 99.9%) was purchased from Merck (Darmstad, Germany). Lead II nitrate ($Pb(NO_3)_2$ 99.99%) from Sigma-Aldrich (Missouri, United States).

The bandgap value of α-PbO nanocrystal in characterized using Shimadzu UV-Vis Diffuse Reflectance Spectrophotometer 2600 (Kyoto, Japan) with wavelength from 200-800 nm set and equiped with wolfram lamp as a light source. The vibration and functional group of α-PbO are analyzed using Shimadzu Fourier Transform Infra-Red (FT-IR) Prestige 21 Spectroscopy with wavenumbers from 4000-400 cm^{-1} set and using nernst lamps (Kyoto, Japan). Crystal of α-PbO nanocrystal in analyzed using Shimadzu X-Ray Diffractometer (XRD) 610 with Cobalt us a source of electron (Kyoto, Japan). Particle size distribution of α-PbO nanocrystal was analyzed using Particle Size Analyzer (PSA) and Potential Zeta Charge (PZC) Malvern ZEN 1600 with dynamic light scattering system (Malvern, United Kingdom). Morphological shapes of α-PbO nanocrystal were characterized using Scanning Electron Microscopy-Energy Dispersive X-ray (SEM-EDX) JEM 1400, using 350 keV of electron beam energy (Nagoya, Japan).

Method and Procedure

Synthesis of α-PbO nanocrystal using concentration of $Pb(NO_3)_2$ (0.025 M) and SLS ($9x10^{-4}$ M). About

25 mL $Pb(NO_3)_2$ solution was added 5 mL SLS. The mixture was reacted at 95 °C for 4 h. Colloid of α-PbO@SLS in furnaced at 500 °C for 2 h for the get a powders of α-PbO nanocrystal. The α-PbO nanocrystal are further investigated.

RESULTS AND DISCUSSION

The synthesis process of α-PbO nanocrystal was successfully decorated using anionic surfactant of sodium laureth sulfate (SLS).

SLS functions as a capping agent for α-PbO nanocrystal. Figure 1 is a graphical of synthesis process of α-PbO nanocrystal. Visually, the powder of α-PbO nanocrystal had a red color as shown in Figure 1(c). This indicates that a α-PbO nanocrystal was successfully formed [16].

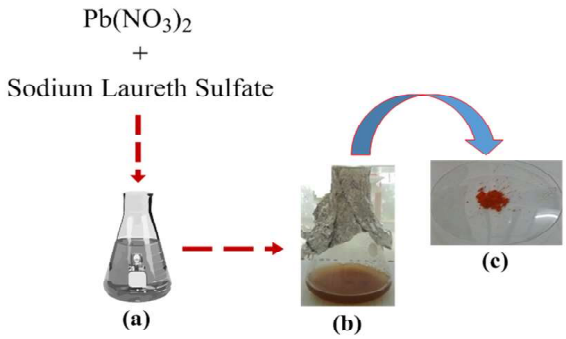

Figure 1. One-pot images of (a) precursor solution, (b) colloid of α-PbO@SLS, and (c) α-PbO powders

Analysis of UV-Vis spectrophotometer to determine the absorption peak and absorbance value of α-PbO nanocrystal. Figure 2 is the UV-Vis spectrum of α-PbO nanocrystal that shown in the wavelength range of 200-400 nm.

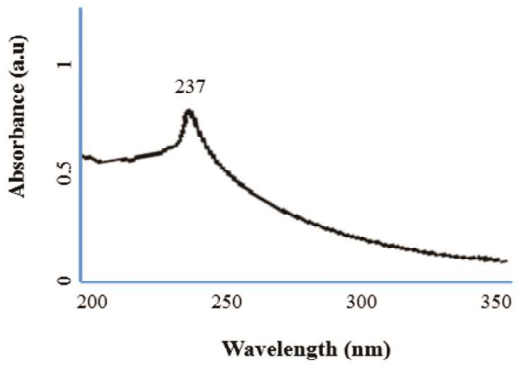

Figure 2. UV-Vis spectrum of α-PbO nanocrystal

The absorption peak of α-PbO nanocrystal can be seen at wavelength of 237 nm and absorbance value is 0.7. Previous studies, reported a absorption peak of

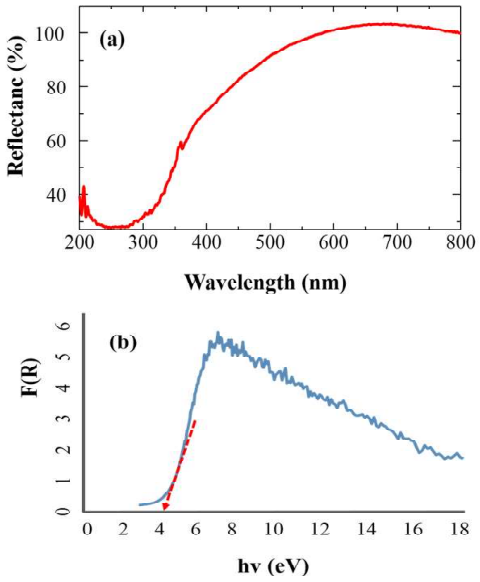

Figure 3. UV-Vis DRS spectrum of (a) α-PbO nanocrystal and (b) bandgap value

PbO nanoparticle can be seen at wavelength of 200-400 nm [17].

The UV-Vis DRS was used to determine the reflectance and bandgap value of α-PbO nanocrystal. Figure 3 shows the UV-Vis DRS spectrum of α-PbO nanocrystal.

The reflectance peak of α-PbO nanocrystal can be seen at wavelength of 330 nm. The determine of bandgap value of α-PbO nanocrystal in calculated using Kubelka-Munk equation. Tauc's Equation (1):

$$\alpha h\upsilon = A\,(h\upsilon - Eg)^n \qquad (1)$$

where α is absorption of coefficient, hv is photon energy, A is constant, Eg is bandgap energy of sample, and n is transition value of valence band to conduction band (1/2 or 2 value) [15]. After calculated, α-PbO nanocrystal has a bandgap value of 4.2 eV as shown in figure 1b. Based on the UV-Vis DRS spectrum and the bandgap value, α-PbO nanocrystal was successfully decorated using a SLS surfactant and has a optical properties in the visible light region [12].

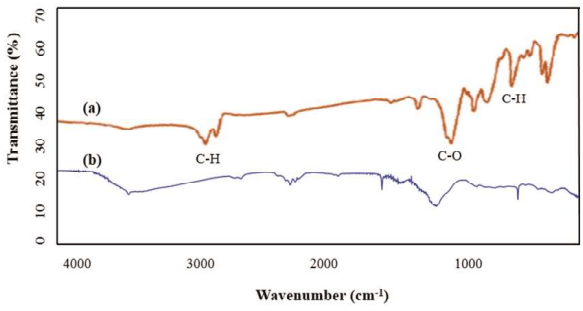

Figure 4. FT-IR spectrum of (a) SLS and (b) α-PbO nanocrystal

FT-IR spectroscopy to determine the functional groups and vibration of Pb-O nanocrystals. Figure 4 is FT-IR spectrum of SLS and α-PbO nanocrystals.

Surfactant of sodium laureth sulfate has a functional groups of C-O from ether at wavenumber of 1230 cm^{-1}, C-H from alkenes at wavenumber 875 cm^{-1}, and C-H from alkanes at wavenumber 2960 cm^{-1} as shown in Figure 4(a). Figure 4(b) shows a shift of absorption peak at the wavenumber 1358 cm^{-1} which is predicted by the strong interaction of the C-O groups from SLS which functions as a capping agent for α-PbO nanocrystals [6].

Analysis of XRD to determine the crystallinity and average size of the crystallite. Figure 5 shows the diffraction pattern of α-PbO nanocrystals at 2θ: 10-80°. α-PbO nanocrystals has a pattern at diffraction angles of(2θ) 10-80°: 29.17, 32.54, 37.85, 39.62, 45.16, 46.21, 56.12, and 61.73 as shown at miller indices of (111), (200), (201), (121), (220), (030), (311), and (032), respectively. The data corresponding with JCPDS data no. 00-005-0561. α-PbO nanocrystals synthesized using SLS has a crystal structure of tetragonal (litharge phase) [13].

The crystal size average of α-PbO nanocrystals to determine using Debye-Scherrer's equation:

$$D = \frac{0.89x\lambda}{\beta \cos\theta} \quad (2)$$

where D is particle size average, λ is wavelength used in XRD (1.54056 Å), Δθ is full widht at half maximum (FWHM), and θ is Bragg's diffraction angle. After calculating, α-PbO nanocrystals had a crystal size average of 56.32 nm.

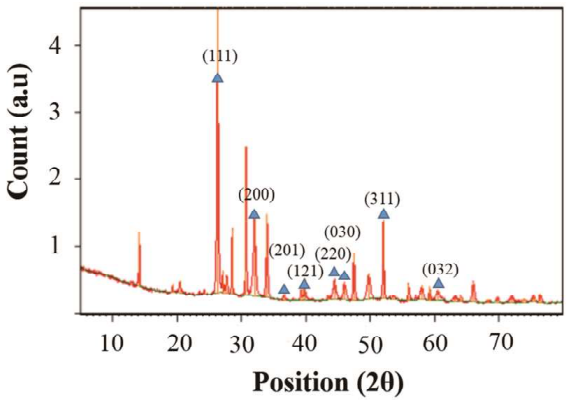

Figure 5. XRD pattern of α-PbO nanocrystal

PSA and PZC were used to determine the particle size average distribution and inter-face charge of α-PbO nanocrystals. Figure 6 shows the PSA and PZC spectra of size distribution for α-PbO nanocrystals.

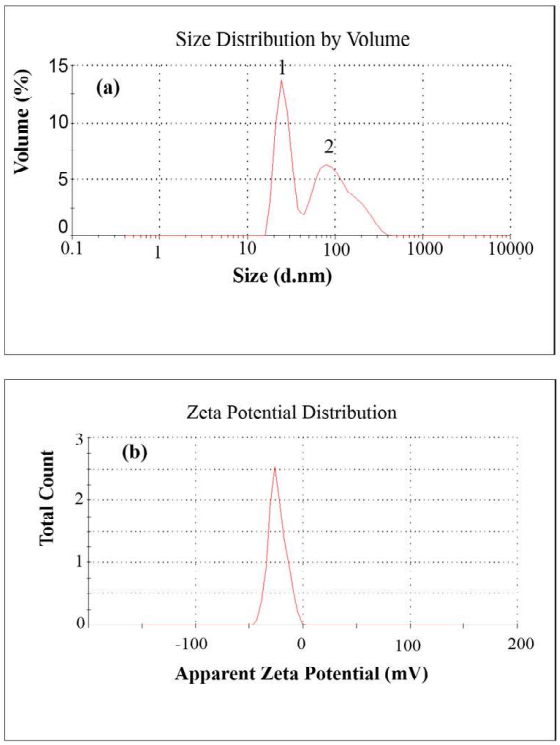

Figure 6. (a) PSA and (b) PZC spectrum of α-PbO nanocrystal

α-PbO nanocrystals has a 2 peaks with a particle size of 45 nm (peak 1) and 98 nm (peak 2). The particle size average distribution of α-PbO nanocrystals is 71.5 nm with a poly dispersity index (PDI) value of 0.3 as shown Figure 6(a) [18-20]. The results of PSA measurement showed α-PbO nanocrystals had a larger of particle size than the XRD results. It predicted, the

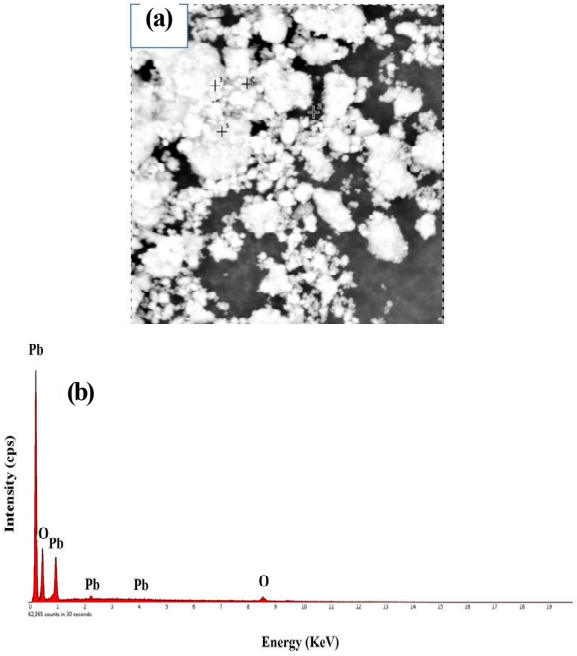

Figure 7. (a) SEM images and (b) EDX of α-PbO nanocrystals

SLS measurement which becomes the capping agent on the surface of α-PbO nanocrystals is measured. Figure 6(b). shows the α-PbO nanocrystals have an inter-particle charge value of -25 mV.

SEM-EDX was used to determine the morphological shape and atomic composition of α-PbO nanocrystals. Figure 7 is a SEM morphology image of and EDX spectrum of α-PbO nanocrystals.

α-PbO nanocrystals have an irregularly spherical morphology as shown in Figure 7(a). Figure 7(b) shows the atomic compositions of Pb and O of 83.12 and 16.88%, respectively [21-22]. From the characterization data, it can be concluded that PbO was successfully decorated using surfactant of sodium laureth sulfate. α-PbO nanocrystals has the potential as an additional material in batteries, glass materials, and catalysts.

CONCLUSION

The α-PbO nanocrystals were successfully decorated using SLS anionic surfactants. SLS functions as a capping agent on the surface of α-PbO nanocrystals. α-PbO nanocrystals have a optical properties can be seen at wavelength of 237 nm with a bandgap value of 4.2 eV. α-PbO nanocrystals have a tetragonal crystal structure (litharge phase) with an average crystallite size of 56.32 nm. The morphological shape of α-PbO nanocrystals is irregularly spherical with atomic compositions of Pb and O being 83.12 and 16.88%. α-PbO nanocrystals have a nano-scale size a potential for X-ray imaging detectors and lead storage batteries and as a contribution in the development of science and technology.

ACKNOWLEDGMENT

This Research is Supported by Institut Teknologi Sumatera under Research Grant for Hibah Publikasi GBU-45.

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