Materials Research Express

PAPER

Biofabricated SnO₂ nanoparticles using Red Spinach (Amaranthus tricolor L.) extract and the study on photocatalytic and electrochemical sensing activity

Wiyogo Prio Wicaksono, Imam Sahroni, Annisa Khafiya Saba, Ristianto Rahman and Is Fatimah

1 Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Islam Indonesia, Kampus Terpadu UII, Jl. Kaliurang Km 14.5, Sleman, Yogyakarta, 58884, Indonesia

2 Laboratory of Material for Energy and Environment, Dept. of Chemistry, UII, Yogyakarta, Indonesia

E-mail: isfatimah@uii.ac.id

Keywords: electrochemical sensor, photocatalysis, nanoparticles

Abstract

Biofabrication of SnO₂ nanoparticles (SnO₂ NPs) using Amaranthus tricolor L. extract has been conducted. Synthesis of the nanoparticles was conducted by refluxing tin chloride dihydrate as SnO₂ precursor followed by calcination at 400 °C. The formation of SnO₂ NPs was identified from UV-Visible spectrophotometric analysis and particle size analyzer. The resulting materials were characterized by x-ray diffraction (XRD), Fourier-Transform Infra-Red (FTIR), scanning electron microscope-energy dispersive x-ray (SEM-EDS), transmission electron microscope (TEM), and diffuse reflectance UV-Visible (UV-DRS). Photocatalytic evaluation of SnO₂ NPs was carried out in a photodegradation of bromophenol blue (BPB) under photocatalytic and photooxidation mechanism with the presence of H₂O₂. Physicochemical analyses confirm the formation of SnO₂ NPs providing the capability to act as photoactive material. The photocatalytic activity studied by UV-Visible spectrophotometric analysis and high performance liquid chromatography (HPLC) revealed the superior activity of the nanoparticles for decomposition of BPB. The decomposition rate of BPB under photooxidation mechanism is higher than of photocatalysis mechanism. Both mechanisms obey pseudo-second order kinetics and the decomposition was identified from the shifted spectra of treated solution. Furthermore, the electrochemical sensing activity for the detection of heavy metal Hg²⁺ showed an increasing of sensitivity and feasibility of the SnO₂/Au electrode compared to the bare Au with the limit of detection of 0.001 970 ppm. This result reveals the facile green synthesis route of SnO₂NPs with the promising activities.

1. Introduction

The formulation of metal or metal oxide in nanoparticles form is a growing research in material chemistry due to the increasing activity of the metal and metal oxide in the nanoparticles forms for many applications. SnO₂ nanoparticles (furthermore called as SnO₂ NPs) have attracted interest refer to its activity for many applications such as in catalysis, sensing, electric and optical technologies. With the band gap energy of 3.6 eV, SnO₂ is adaptive and suitable for photocatalysis application [1]. In addition, SnO₂ has also attracted many researchers as gas sensing materials [2] and electrochemical sensors [3]. As many other metal or metal oxide nanoparticles, some techniques can be utilized such as sol–gel, chemical vapour deposition, spray pyrolysis etc. Among some possible methods, green synthesis of metal or metal oxide using varied plant extracts is a popular method [4]. The utilization of active compounds from plant, or called as biosynthesis showed the sustainable, low cost, and tunable synthesis for a specific performance of metal or metal oxide. Some previous researches showed the succeed of SnO₂ NPs biosynthesis using Erwinia herbicola, Calotropis gigantean, Ficus carica, Plectranthus amboinicus [1, 5–7]. Those researches express the reliability of the synthesis and the prepared nanoparticles showed the improvement on photocatalytic activity [7].
In the present study, the green synthesis of SnO2 NPs is performed by using aqueous leaf extract of red spinach (Amaranthus tricolor L.) (figure 1). The plant is a tropical plant and growing easily in Asia countries such as in India, Sri Lanka, Philippines, and Indonesia. Phytochemical study of Amaranthus tricolor L. reported the component of Gallic acid (3,4,5-OH), myrcetin, Quercetin-3-O-rutinoside (rutin), and Apigenin-7-O-glucoside (apigetin) as main constituents [8]. The functional groups from the active compounds are predictively active for reducing Sn^{2+} in the biosynthesis, which creating the nanoparticle size. Furthermore, the SnO2 formation will be gained by the calcination process. Evaluation on photocatalytic and electrochemical activity of the nanoparticles was performed in bromophenol blue (BPB) and electrochemical sensing for detection of heavy metal Hg^{2+} in water solution.

2. Materials and methods

2.1. Materials
Amaranthus tricolor L. was obtained from traditional market in Sleman District, Yogyakarta, Indonesia. Some chemicals consist of tin (IV) chloride pentahydrate (SnCl4·5H2O), H2O2, BPB, mercury (II) chloride (HgCl2), hydrochloric acid (HCl) are obtained from Merck-Millipore (Germany).

For the preparation of leaf extract of Amaranthus tricolor L., the fresh leaves were washed with distilled water followed by boiling the mixture in water for 30 min. The chocolate solution was obtained by filtering the result. The solution was kept to attained room temperature; then the leaf extract was filtered through Whatmann filter paper No.1 and stored in the refrigerator.

2.2. Synthesis of SnO2 NPs
SnO2 NPs was synthesized by mixing 10 ml of Amaranthus tricolor L. leaf extract with SnCl2 solution followed by heating at 80 °C for 60 min. The precipitation obtained from each variation was filtered and dried in oven overnight. The dry powder was then calcined at 400 °C for 2 h to convert the nanoparticles into SnO2 NPs.

2.3. SnO2 NPs characterization
The prepared SnO2 NPs were characterized by using x-ray diffraction (XRD), gas sorption analyzer, Diffuse reflectance spectrums (UV-DRS), SEM-EDS and TEM analyses. XRD pattern was obtained Philips Benchtop XRD, while UV-DRS spectrum were obtained using UV–Vis (JASCO V-770 spectrophotometer). FT-IR spectra were obtained using a Perkin Elmer, TEM images were scanned with JEOL-JEM 2100 equipped with high-resolution Gatan CCD camera, and the SEM-EDS images were obtained using (JEOL-JSM-6360). Particle size analysis was conducted on HORIBA particle size analyzer. About 1 mg of the SnO2 NPs sample was dispersed in 10 ml water for analysis by diffusion light scattering method.

2.4. Photocatalytic activity test of SnO2 NPs
Photocatalytic activity of the synthesized SnO2 NPs was evaluated for the photocatalytic degradation of BPB in the presence and absence of H2O2 as additional oxidant. The photocatalytic experiments were conducted using closed reactor UV-light. The lamp of UVB (50 W) was used as a UV light source, a xenon lamp (50 W) was utilized as visible light source. The optimum quantity of SnO2 0.2 g was added into 500 ml of BPB solution. For photooxidation experiment, the solution was added with 1 ml H2O2 30% as oxidant. After irrigating the solution with UV-light, at periodic time intervals 0 to 60 min, 3 ml reaction mixture was withdrawn,
centrifuged, and the supernatant solution was collected and optical density (OD) measured by using UV–visible spectrophotometer. Degradation efficiency (DE) of the treatment was calculated based on following equation (1):

\[
DE = \left(\frac{[C_0] - [C_t]}{[C_0]}\right) \times 100\% \tag{1}
\]

with \(C_0\), \(C_t\) and \(k\) are initial concentration, concentration at \(t\).

In order to ensure the oxidation mechanism to the BPB solution, high performance liquid chromatography (HPLC) analysis was performed using HITACHI instrument on Kinetex C18 column, mobile phase of 0.1% formic acid and 0.1% acetonitrile.

2.5. Electrochemical sensing activity test of SnO\(_2\) NPs

Electrochemical sensing activity of the synthesized SnO\(_2\) NPs was evaluated for the detection of mercury ion (Hg\(^{2+}\)) in tap water sample. Briefly, a 0.2 ml of SnO\(_2\) NPs solution obtained by diluting 0.05 g of SnO\(_2\) NPs into 2 ml of concentrated HCl was dropped to a screen-printed gold working electrode, then the electrode was dried at 50 °C for 15 min to produce a gold modified by SnO\(_2\) NPs working electrode (SnO\(_2\)/Au). A three electrodes system consist of SnO\(_2\)/Au, platinum (Pt), and Ag/AgCl as working, counter, and reference electrode, respectively, connected to a Potentiostat (μAUTOLAB III) was employed to detect the Hg\(^{2+}\) using cyclic voltammetry technique. All of the electrochemical experiments was conducted at scan rate of 100 mV s\(^{-1}\) and range potential of 0.35 to \(-0.25\) V. The performance of SnO\(_2\)/Au working electrode was compared to the Au bare, whereas the validation parameters i.e. linearity, limit of detection (LOD), precision, and accuracy (% recovery) was also evaluated. Furthermore, the determination of Hg\(^{2+}\) in the spiked tap water sample was also conducted.

3. Results and discussion

3.1. Physicochemical characterization

The XRD pattern of synthesized SnO\(_2\) is presented in figure 2. The pattern exhibits the formation of SnO\(_2\) rutile nanoparticles with polycrystalline phase demonstrating rutile structure [JCPDS Card No: 41-1445, \(a = 4.743\) Å, \(c = 3.1859\) Å]. The crystal planes (110), (101), (200), (211), (220), (002), (112) and (310) are appeared in the pattern indicating the polycrystalline nature of powder [9, 10]. The results are similar with the pattern of SnO\(_2\) rutile reported from previous works [11].

By using Debye-Scherer equation (2):

\[
D = \frac{0.9}{\beta \cos \theta} \cos \theta \tag{2}
\]

with \(D\) is the particle size, \(\beta\) is the Full Width Half Maximum (FWHM) of the peaks, and \(\theta\) is the Bragg angle, the estimated crystallite size of SnO\(_2\) NPs is to be 18.9 nm. This value is comparable with the bio-synthesized SnO\(_2\)
NPs using *Daphne mucronate*, *Vitex altissima* (L.) and *Andrographis Paniculata* leaves with the size of 15.6, 10–20, and 27 nm, respectively [12–14].

The SEM-image of SnO$_2$ NPs (figure 3(a)) indicates that there are in tendency to be spherical form in irregular size of the particles. The EDS spectra shows that Sn is the dominant component of the materials.
By the confirmation from TEM analyses (figure 3(c)), it is seen that the irregular particles have the diameter less at the range 20–40 nm. The particle size is consistence with the particle size distribution presented in figure 3(d). The nanoparticles size is ranging at 5–100 nm with the particle size mean at 28 nm. The phenomenon of SnO₂ NPs formation within the size range was come from the reduction of Sn²⁺ by organic compounds from red spinach extract to form Sn nanoparticles before oxidized into SnO₂ during calcination process. It can be identified from the comparison on FTIR spectra of the SnO₂ NPs and Amaranthus tricolor L. leaf extract in figure 4.

The leaf extract shows the peaks associated with C–H (2970–2850 cm⁻¹ and 1400–1650 cm⁻¹), C= C (1617 cm⁻¹), and oxygen-containing groups such as carbonyl (C=O) (1720 cm⁻¹), and hydroxyl groups (O–H) observed in the region of 1000–1250 cm⁻¹. Compared to the spectrum of leaf extract, the spectrum of SnO₂ NPs shows some differences such as the disappearance of peak at 1022–1436 cm⁻¹ indicating the partial reduction of the oxygen-containing groups of the component in leaf extract. Similar pattern for the indication of reduction mechanism was also reported from previous work on SnO₂ NPs biosynthesis [15].

Schematic representation of SnO₂ NPs formation by biosynthesis is depicted in figure 5. Myrcetin, as constituent of the extract, is taken as model for reducing compound. The hydroxyls contained from the compound reduce Sn²⁺ from the precursor to form Sn nanoparticles, which composed as capping agent in creating the nanosize range of the metal nanoparticles. As the precipitate of the nanoparticles calcined, the metal nanoparticles are converted into SnO₂ NPs. The FTIR data and the interpretation is similar with the mechanism of metal oxide nanoparticles formation reported in the synthesis of SnO₂ quantum dots using Clitoria ternatea flower extract, and magnetite nanoparticles synthesis using Parkia speciosa extract [16, 17].

For photocatalyst and sensor application, optical properties including band gap energy is an important parameter [18]. The values were determined by diffuse reflectance UV-Visible spectra measurement with the result depicted in figure 6. Using Tauc plot for the diffuse reflectance spectrum, it is found that the band gap energy of SnO₂ NPs is 3.52 eV. This value is similar with SnO₂ NPs prepared using Andrographis Paniculata [19] and lays at the region of SnO₂ nanoparticle band gap energy previously reported [20].

3.2. Photocatalytic activity of SnO₂ NPs

Photocatalytic activity of SnO₂ NPs in photocatalytic degradation of BPB in the presence and absence of H₂O₂ could be seen by the kinetics curve, the spectra of initial and treated solution and also the chromatogram of initial and treated solution in figure 7.

From the kinetics curve (7a) it is seen that there is concentration reduction along time of treatment, moreover, the reduction over photodegradation in the presence of H₂O₂ is faster compared with photocatalysis mechanism.

The higher kinetics constant of photooxidation process is caused by the higher concentration of ·OH due to the presence of H₂O₂ as oxidant by following oxidation mechanism [21] (3)–(7):

\[
\text{SnO}_2 + h\nu \rightarrow \text{SnO}_2(e^- (CB) + h^+(VB)) \tag{3}
\]

\[
\text{O}_2 + e^- \rightarrow \text{O}_2^- \tag{4}
\]

\[
\text{H}_2\text{O} + h^+ \rightarrow \text{OH} + \text{H}^+\text{OH}^- + h^+ \rightarrow \text{OH} \tag{5}
\]
The fact on the presence of degradation mechanism instead of only adsorption is proven by the change of UV-Visible spectra (7b) representing that the maximum wavelength corresponding to the presence of BPB (596 nm) is disappeared after treatment. This disappearance is in line with the change of chromatogram from HPLC analysis (7c). The presence of BPB is identified from the peak at the retention time of 12.85 min, and this peak is absence after treatment. The chromatogram peaks at 12.22 min and other retention time are the indication of the degradation product.

The comparison on kinetics of BPB degradation in the presence and absence of H₂O₂ are presented in figure 8. The faster degradation is demonstrated by the addition of H₂O₂ representing the role of hydroxyl radicals accelerated by H₂O₂ dissociation [16].
The photocatalytic degradation of BPB at varied initial concentration in the presence and absence of H₂O₂ suggests that the reactions obey pseudo-first second kinetics as shown by linear plots of the kinetics (figures 8(b) and (d)) regarding to following equation (8):

\[
\frac{C_0}{C_t} = kt + 1
\]  

(8)

With \(C_0\) and \(C\) are initial BPB concentration and concentration at time of \(t\), and \(k\) is kinetics constant.

The kinetics parameters and DE values of each process are presented at table 1. The DE values are obtained refer to equation (1) from the treatment of 180 min.

The data suggest that kinetic constant and DE values of the reaction with H₂O₂ are higher than without H₂O₂. The photocatalytic degradation of kinetics was then investigated by using Langmuir-Hinshelwood (L-H)
The L-H model was widely used for heterogeneous photocatalysis and assumed that the kinetics of degradation is related with the bimolecular reaction of two surface adsorbed species. The model is mathematically given as (9):

$$\frac{1}{k} = \frac{1}{k_c k_{LH}} + \frac{C_0}{k_c}$$  \hspace{1cm} (9)

where $C_0$ is the initial concentration of BPB (in mg l$^{-1}$), $k$ is the apparent pseudo-second-order rate constant, $k_c$ is the rate constant of surface reaction (in mg/l.min) and $k_{LH}$ is the Langmuir–Hinshelwood adsorption equilibrium constant (in l mg$^{-1}$).

Table 1. Photocatalytic degradation of BPB at varied initial concentration.

| Initial concentration (mg l$^{-1}$) | R2       | Kinetics constant ($l$/mg.min) | Initial rate (mg/l.min) | DE (%)  |
|------------------------------------|----------|-------------------------------|-------------------------|---------|
| With $H_2O_2$ addition             |          |                               |                         |         |
| 10                                 | 0.9553   | 21.25                         | 3.30                    | 99.77   |
| 20                                 | 0.9458   | 6.06                          | 6.47                    | 98.02   |
| 30                                 | 0.9529   | 7.02                          | 9.43                    | 96.80   |
| 40                                 | 0.9485   | 4.65                          | 10.81                   | 90.04   |
| 50                                 | 0.9641   | 2.65                          | 13.15                   | 88.70   |
| Without $H_2O_2$ addition          |          |                               |                         |         |
| 10                                 | 0.9834   | 2.34                          | 3.10                    | 96.04   |
| 20                                 | 0.9950   | 1.39                          | 5.67                    | 89.97   |
| 30                                 | 0.9274   | 0.76                          | 7.86                    | 80.96   |
| 40                                 | 0.9666   | 0.43                          | 9.33                    | 77.87   |
| 50                                 | 0.9986   | 0.52                          | 10.21                   | 67.76   |

Figure 8. (a) Kinetics plot of BPB photocatalytic degradation at varied initial concentration without $H_2O_2$ addition (b) Pseudo-second order plot of BPB photocatalytic degradation without $H_2O_2$ addition (c) Kinetics plot of BPB photocatalytic degradation at varied initial concentration with $H_2O_2$ addition (b) Pseudo-second order plot of BPB photocatalytic degradation with $H_2O_2$ addition.
The plot and data listed in table 2 indicate that based on R² values, the kinetics fit with Langmuir-Hinshelwood model. By both with and without H₂O₂ addition KLH values are lower than kc, suggesting that the adsorption mechanism give less effect on the degradation mechanism [22, 23].

The Langmuir-Hinshelwood plot of BPB photocatalytic degradation with and without H₂O₂ is presented in figure 9.

Furthermore, the test by comparing light source was conducted. The kinetics data are presented in table 3. The data showed that SnO₂ NPs is photocatalytic active not only under UV light, but also under visible light, even though lower in either initial rate or degradation efficiency. It is linearly related with the band gap energy of SnO₂ NPs that lies on UV region.

In general, based on the particle size and photocatalytic activity in dye degradation, the synthesized SnO₂ NPs in this research is comparable with previous works (table 4).

### 3.3. Electrochemical sensor activity of SnO₂NPs

Electrochemical sensor activity of SnO₂NPs was evaluated as material sensing for heavy metal Hg²⁺. Figures 10(a) and (b) depict the cyclic voltammogram of (A) bare Au and (B) the SnO₂/Au in the presence of 0.032 ppm Hg²⁺ in HCl electrolyte and the background. A small oxidation peak at 0.04 V was observed using the

![Figure 9. Langmuir-Hinshelwood plot of BPB photocatalytic degradation with and without H₂O₂.](image-url)
bare Au electrode, whereas two bigger oxidation peaks at 0.007 and 0.06 V were observed using SnO$_2$/electrode which predicted as oxidation peak of the reduced form of SnO$_2$NPs and Pb$^{2+}$, respectively (equations (10) and (11)). In addition, a small reduction peak of Hg$^{2+}$ at −0.17 V using bare Au electrode was also observed. Meanwhile, a reduction peak at −0.13 V was predicted for both reductions of SnO$_2$ and Hg$^{2+}$. Table 5 shows the comparison of signal to background ratio of the bare Au and SnO$_2$/Au.

Table 4. Comparison on photocatalytic activity of biosynthesized SnO$_2$ NPs.

| Plant extract          | Particle size (nm) | Photocatalytic activity                                      | References     |
|------------------------|-------------------|-------------------------------------------------------------|----------------|
| Plectranthus ambotinus | 63                | SnO$_2$ NPs has 91% degradation efficiency for rhodamine B   | [24]           |
| Jujube fruit           | 18                | DE of 90% and 83% for methylene blue and eriochrome black T  | [25]           |
| Bacterium Erwinia herbicola | 15–40           | DE of 93.3, 97.8, and 94.0% for methylene blue, erichrome black T, and methyl orange | [18]           |
| Persia Americana       | 4                 | Material was very active in degradation of phenol red        | [15]           |
| Litsea cubeba          | 30                | SnO$_2$ NPs has high activity in congo red degradation       | [15]           |
| Red spinach            | 10–40             | SnO$_2$ NPs exhibits DE of 99.77% for BPB photocatalytic degradation | This work      |

bare Au electrode, whereas two bigger oxidation peaks at 0.007 and 0.06 V were observed using SnO$_2$/electrode which predicted as oxidation peak of the reduced form of SnO$_2$NPs and Pb$^{2+}$, respectively (equations (10) and (11)). In addition, a small reduction peak of Hg$^{2+}$ at −0.17 V using bare Au electrode was also observed. Meanwhile, a reduction peak at −0.13 V was predicted for both reductions of SnO$_2$ and Hg$^{2+}$. Table 5 shows the comparison of signal to background ratio of the bare Au and SnO$_2$/Au electrode. An increase of signal to background ratio from 1.60 to 2.49 (almost twice) reveals the modification of SnO$_2$NPs to the bare Au has been significantly increased the Hg$^{2+}$ signal.

\[
\text{Sn(s)} + 2\text{H}_2\text{O}(l) \rightarrow \text{SnO}_2(s) + 4\text{H}^+(aq) + 4\text{e}^{-} \tag{10}
\]

\[
\text{Hg(s)} \rightarrow \text{Hg}^{2+}(aq) + 2\text{e}^{-} \tag{11}
\]
Furthermore, the separation between oxidation and reduction peak ($\Delta E$) of Hg$^{2+}$ (figure 10) could be used to evaluate the electron transfer system of Hg$^{2+}$ on the bare Au and SnO$_2$/Au electrode using the following equation (6). Table 6 shows the smaller $\Delta E$ of SnO$_2$/Au compared to the bare Au electrode reveals that the modification of bare Au electrode by SnO$_2$NPs has increased the rate of electron transfer that improved the electrochemical sensing performance. $\Delta E$ is calculated by following equation (12):

$$\Delta E = \frac{E_{pa} - E_{pc}}{n} \tag{12}$$

with $n = 2$ is the number of transferred electrons.
The linear correlation was observed from 0 to 0.032 ppm of Hg\(^{2+}\) with an R\(^2\) of 0.9924 as shown in figure 11. The LOD was estimated from the oxidation peak current using the equation (13):

$$\chi_{LOD} = \frac{3 \cdot \text{Sa}}{\text{b}}$$

(13)

where \(\text{Sa}\) and \(\text{b}\) is the standard deviation of intercept and slope of the linear curve, respectively. An estimated LOD of 0.001 970 ppm was achieved, whereas the precision (\(n = 10\)) at 0.032 ppm of Hg\(^{2+}\) gave a relative standard deviation (RSD) of 26.73%. The high RSD value may due to weakly surface adhesion by the physical modification of SnO\(_2\) NPs to the Au electrode. In spite of the fact, an RSD Horwitz ratio (%RSD)/RSD Horwitz) of 1.27 was achieved using the equation of %RSD Horwitz = \(2^{1/2} - 0.5 \log C\), where C is Hg\(^{2+}\) standard solution. A method could be said has good repeatability if the ratio is less than 2 [26]. Thus, it can be concluded that the method has proper repeatability (precision).

The low LOD below the maximum EPA regulation (0.002 ppm of Hg in drinking water) [27] that successfully achieved reveals the possibility the use of the electrode to be used in real sample monitoring. For the real sample application, cyclic voltammetry technique using the SnO\(_2\)/Au electrode was employed to monitor a spiked tap water sample resulted in a good % recovery up to 96.4%.

4. Conclusion

Biofabrication of a spherical SnO\(_2\) NPs synthesized using *Amaranthus tricolor* L. leaf extract has been successfully conducted. The nanoparticles exhibit rutile phase in spherical forms with a diameter less than 100 nm, and the band gap energy of 3.52 eV. The nanoparticles demonstrated capability as photocatalyst for bromophenol blue photocatalytic degradation over with and without H\(_2\)O\(_2\) addition under UV and visible light which obey pseudo-second order kinetics. Furthermore, the SnO\(_2\) NPs also showed the satisfied electrochemical sensing activity for Hg\(^{2+}\) monitoring by increasing the electrode conductivity, surface area, as well as adsorption effect.

ORCID iDs

Is Fatimah © https://orcid.org/0000-0001-5551-6563

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