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Synthesis of SnO_2 Nanoparticles from Metals by Electrochemical Approach: An Innovative Solution for Functional Materials

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ABSTRACT

Purpose of the study: The aim of this study was to produce SnO2 nanoparticles by electrochemical method using tin metal and hydrochloric acid as electrolyte solution.

Methodology: Synthesis of SnO_2 nanoparticles was carried out using an electrochemical method with tin electrodes and HCl solution. Variations in potential (10–100 V) and HCl concentration (0.005–0.06 M) were observed for optimization. Characterization using UV-Vis, FTIR, XRD, and Zeta Sizer showed that this method was effective in producing SnO_2 nanoparticles with crystal structure, composition, and size distribution that could be optimized.

Main Findings: This study successfully synthesized SnO_2 nanoparticles from tin metal using an electrochemical method with HCl solution. The optimum potential for electrolysis was 60 V, with a HCl concentration of 0.06 M producing the largest nanoparticles (83.11 nm) at a wavelength of 207 nm and an absorbance of 3.068. XRD characterization showed a diffraction pattern according to the SnO_2 standard, while FTIR confirmed the Sn–O vibration at 580 cm^{-1}.

Novelty/Originality of this study: The characterization results show a correlation between HCl concentration with nanoparticle size and absorbance wavelength, which provides new insights into controlling the optical properties and structure of SnO_2 nanoparticles.

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1. INTRODUCTION

Nanotechnology is a branch of science that studies the design, manufacture, and application of nanomaterials with a focus on the relationship between the physical properties of materials and their geometric dimensions [1]-[3]. In recent decades, nanotechnology has developed rapidly, enabling innovations in nanoscale material synthesis methods [4]-[6]. Materials with optical, electronic, magnetic, and catalytic properties that can be controlled through size, shape, and composition offer great potential for various technological applications [7]-[9]. This discovery makes nanotechnology one of the main pillars in the development of modern science. This development paves the way for understanding the unique properties of materials at the nanoscale.

Nanomaterials are materials with one dimension less than 100 nm, including various forms such as nanotubes, nanofibers, and nanoparticles [10]-[12]. Changes in material properties at the nanoscale often occur due to differences in atomic or molecular structure compared to the macroscale [13]-[15]. This phenomenon makes

nanomaterials superior in terms of chemical reactivity, mechanical strength, and optical properties. One important nanomaterial is metal nanoparticles, which can be made from various elements in the periodic table [16]-[18]. This material has great potential for application in various advanced technologies.

Tin oxide is one of the n-type semiconductor materials with a large band gap, which has been used for various applications such as photocatalysts, gas sensors, batteries, and antibacterial and antioxidant materials [19]-[21]. These applications often use tin oxide in the form of nanoparticles to improve its performance. Various forms of tin oxide nanoparticles, such as nanorods, nanosheets, and nanospheres, provide unique characteristics that are not possessed by large-scale materials [22]-[24]. With its superior properties, tin oxide is one of the best candidates for further development.

Various methods for the synthesis of tin oxide nanoparticles have been carried out, such as microwave, hydrothermal, sol-gel, microemulsion, and sonochemical methods [16], [25], [26]. These methods have been proven to be able to produce nanoparticles with controllable sizes. However, obstacles arise in its application, such as long process times and difficulties in large-scale production [27], [28]. On the other hand, the electrochemical method offers a solution to these limitations. This method is not only efficient and environmentally friendly, but also allows better control of particle size through potential regulation and solution concentration.

Previous studies have shown the success of the electrochemical method in the synthesis of tin oxide nanoparticles [29], [30]. These studies prove that variations in time, solution concentration, and potential can affect the size and characteristics of the resulting nanoparticles [7], [31]. This shows the great potential of the electrochemical method to produce high-quality tin oxide nanoparticles. This method also allows the manufacture of materials with uniform structures that can be applied to various technological needs.

In addition to tin oxide, the electrochemical method has been used to synthesize other metal nanoparticles such as gold and nickel. Previous studies have shown that this method can produce metal nanoparticles with controlled sizes and properties by adjusting the synthesis parameters [32], [33]. This method also shows flexibility for use on various types of metals, making it a reliable and efficient approach. With these advantages, the electrochemical method is one of the main choices in the synthesis of nanoscale materials.

This research has high novelty and urgency in the field of functional materials and nanotechnology. Its novelty lies in the use of an electrochemical approach to synthesize SnO_2 nanoparticles directly from base metals, which offers a more environmentally friendly, energy-saving, and efficient method compared to conventional methods such as sol-gel or hydrothermal. This approach also allows more precise control of particle morphology and size, which is essential in applications such as gas sensors, optoelectronic devices, and photocatalysts [34], [35]. The urgency of this research is driven by the increasing global need for high-performance functional materials that can be produced sustainably, along with the pressure to reduce the environmental impact of material production processes. Thus, this research makes a significant contribution to the development of green technologies in the synthesis of metal oxide-based materials.

This study aims to synthesize tin oxide nanoparticles from tin metal using an electrochemical method with a hydrochloric acid electrolyte solution. Various variations in potential and solution concentration were applied to observe their effects on the size and characteristics of the resulting nanoparticles. The results of this study are expected to provide new contributions in the field of nanoparticle synthesis using electrochemical methods. With a simpler, more efficient, and environmentally friendly approach, this method is expected to be a better alternative compared to conventional synthesis methods. This research also provides a basis for the development of tin oxide-based material applications in the future..

2. RESEARCH METHOD

2.1. Tools and materials

The equipment used in this study includes a heater (hot plate), Ohaus analytical balance, power supply for nanoparticle synthesis, heat sink cable, quartz cuvette, magnetic stirrer, 100 mL bottle, glassware (50 mL, 100 mL, and 200 mL beakers, 100 mL measuring flask, 100 mL measuring cylinder, dropper, watch glass, funnel, and others). While the instruments used for the characterization of SnO2 nanoparticles are GENESYS 10S visible light (UV-Vis) spectrophotometer, Malvern Zetasizer Nano Series Instruments, Shimadzu Fourier Transform Infrared (FTIR) spectrophotometer and Philips X' Pert MPD (Multi Purpose Diffractometer) XRD using Cu Kα1 radiation (λ = 1.540598 nm), Kα2 (λ = 1.544426 nm). Some of the materials used in this study were tin foil (Merck, 99.9%), hydrochloric acid (Merck, 37%) and aquademin.

2.2. Research Procedures

2.2.1. Tin Electrode Preparation

Tin electrodes were made using the standard method that was carried out by Husna in 2011 on gold metal. Tin electrodes were made from tin foil. The tin foil was melted and formed into bars with a diameter of 4 mm, a thickness of 1 mm and a length of 10 cm. This description can be seen in Figure 1. To clarify the description in detail, a flow diagram was made in the study which can be seen in the appendix.

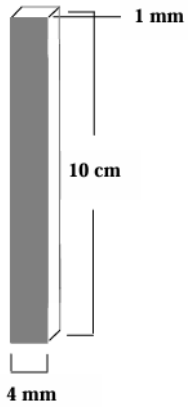

Figure 1. Tin Electrode

2.2.2. Synthesis of SnO2 Nanoparticles

In this tin nanoparticle synthesis, a set of electrolysis cells with tin as electrodes on the cathode and anode were used. The electrolyte solution used in this synthesis was hydrochloric acid solution. The electrolysis cell scheme is in Figure 2.

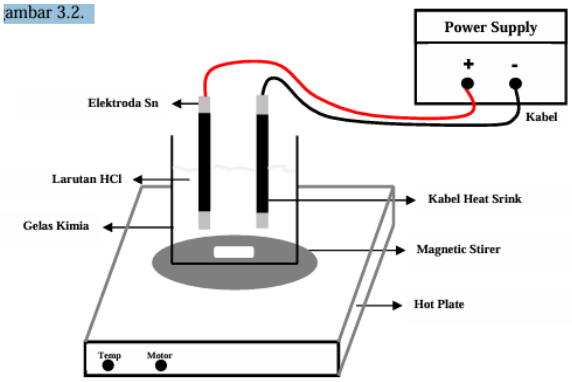

Figure 2. Schematic of Electrolysis Cell for Synthesis of SnO2 Nanoparticles

In the synthesis of SnO2 nanoparticles, observations were made on the effect of potential and the effect of HCl electrolyte solution concentration.

2.2.3. Characterization of SnO2 Nanoparticles

The SnO_2 nanoparticle colloids from each treatment were characterized using a UV-Vis spectrophotometer at a wavelength of 190–400 nm to determine the maximum absorbance. The measurement was carried out by inserting the blank solution into cuvette A and the SnO_2 nanoparticle colloid into cuvette B (size 10 x 10 x 43 mm), then inserted into the UV-Vis instrument. The maximum wavelength was determined first, followed by measuring the absorbance of the blank and SnO_2 nanoparticle colloids at that wavelength. The SnO_2 nanoparticles produced from electrolysis at a concentration of 0.06 M HCl and a potential of 60 volts initially formed a yellow colloid, which then turned into a white precipitate. This precipitate was separated from the solution and heated at 100 °C for 24 hours to produce powder. The powder was characterized using FTIR by preparing the sample in the form of pellets. Pellets were made by mixing SnO_2 nanoparticle powder with KBr powder, then ground until homogeneous, put into a holder, and pressed before being analyzed using FTIR to determine the components in the sample.

Crystal structure characterization was carried out using XRD. SnO_2 nanoparticle powder was put into a crucible, and measurements were taken at an angle of 10–100 θ. Diffraction data were analyzed to confirm the crystal structure of the nanoparticles.

To determine the size of SnO_2 nanoparticles under optimum conditions, the Zeta Sizer instrument was used. The colloidal sample was put into a cuvette, its absorbance was measured, and its refractive index was determined. The sample in the cuvette was then put into the Zeta Sizer instrument, and the program was operated with the absorbance and refractive index data of the sample. The results in the form of peaks in the size distribution of nanoparticles were displayed after the process was complete.

3. RESULTS AND DISCUSSION

SnO_2 nanoparticles were synthesized using an electrochemical method, which is a method that can control the size of the particles to be produced.

3.1. Electrolysis and Formation of SnO_2 Nanoparticles

In the synthesis of SnO_2 nanoparticles, the synthesis process using the electrochemical method begins with the ionization of the HCl solution, namely:

Ionization : $HCl_{(aq)} \longrightarrow H^+_{(aq)} + Cl^-_{(aq)}$

Next, an oxidation reaction occurs at the anode (Sn) and a reduction reaction at the cathode (Sn), namely:

Cathode (Sn) : $4H_2O_{(l)} + 4e^- \longrightarrow 4OH^-_{(aq)} + 2H_{2(g)}$
Anode (Sn) : $Sn_{(s)} \longrightarrow Sn^{4+}_{(aq)} + 4e^-$

Redox reaction : $4H_2O_{(l)} + Sn_{(s)} \longrightarrow 4OH^-_{(aq)} + Sn^{4+}_{(aq)} + 2H_{2(g)}$
Synthesis reaction of SnO2 nanoparticles:

$$Sn^{4+}_{(aq)} + 4OH^-_{(aq)} \longrightarrow SnO_{2(s)} + 2H_2O_{(l)}$$

In the synthesis of SnO_2 nanoparticles, what is observed is the effect of HCl concentration in the solution. This HCl solution functions as an electrolyte solution in the electrolysis cell. The HCl compound is ionized into H^+ and Cl^- ions. The electrode at the cathode, which is reduced is H_2O producing OH^- ions and H^2 gas. While at the anode, Chloride ions can activate the dissolved metal at the anode, namely SnO which is oxidized to $Sn4^+$.

3.2. FTIR Analysis Results

SnO2 nanoparticles produced from electrolysis at a concentration of 0.06 M HCl with a potential of 60 volts are in the form of yellow colloids that turn into white deposits. The deposits are separated from the solution and then heated at a temperature of 100°C for 24 hours. The powder obtained after heating is characterized using an FTIR instrument to determine the composition contained therein. The spectrum of SnO2 nanoparticles is shown in Figure 3.

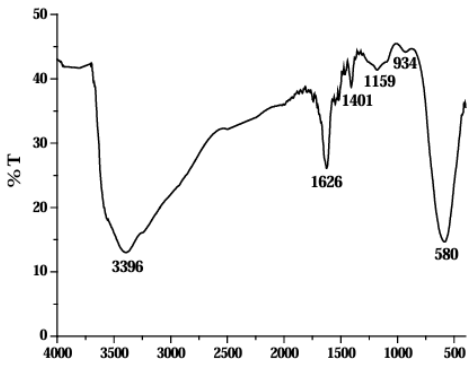

Figure 3. FTIR spectrum of SnO2 Nanoparticles

Figure 3 shows the FTIR spectrum of SnO_2 nanoparticles. The absorption peak at 3396 cm^{-1} is the stretching vibration of the OH group, while the peak at 1626 cm^{-1} indicates the bending vibration of the adsorbed water molecules. At a wave number of 934 cm^{-1}, the absorption describes the interaction of the oxygen surface with Sn metal (Sn-O). At a wave number of 1401 cm^{-1}, there are bending vibrations of H-O-H in water. In addition, the absorption at 1159 cm^{-1} shows the typical vibration of the hydroxyl group surface. The antisymmetric vibration of Sn-O was identified in the region of 400–700 cm^{-1} in this study showing a peak at a wave number of 580 cm^{-1}. Such vibrations in the synthesized samples confirm the presence of SnO_2.

3.3. XRD Analysis Results

The X-ray diffraction pattern of SnO_2 nanoparticles powder synthesized using electrochemical method showed several strong peaks, including (110), (101), (200), (211), (310), (112), (202), and (321) at 2θ angles. Three sharp peaks were identified, namely (110), (101), and (211), located at 2θ angles of 26.58°, 33.39°, and 52.04°, respectively. These peaks were consistent with the standard diffraction pattern of SnO_2, indicating that the

resulting nanoparticles had a tetragonal structure. These results confirmed that SnO_2 with a tetragonal structure was successfully synthesized using electrochemical method.

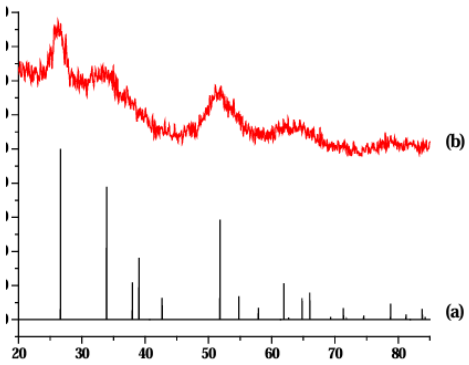

Figure 4. XRD spectra of (a) Standard SnO2 (b) SnO2 Synthesized by Electrochemical Method

3.4. Zeta Sizer Analysis Results

Colloidal SnO_2 nanoparticles at optimum conditions of potential influence with electrolysis using HCl concentration of 0.02 M and potential of 60 volts were analyzed using Zeta Sizer instrument. This analysis was conducted to determine particle size and particle size distribution. The results of the analysis using HCl concentration of 0.02 M at potential of 60 volts are presented in Table 1, while the distribution of particle size distribution is shown in Figure 5.

Table 1. Results of Zeta Sizer Instrument Analysis during Electrolysis with 0.02 M HCl Concentration at 60 Volt Potential

No.	Peak 1	Peak 2	Peak 2	% Intensity 1	% Intensity 2	% Intensity 3
1.	58.91	0	0	100	0	0
2.	59.66	0	0	100	0	0
3.	61.40	0	0	100	0	0

The table above shows stable results in the measurement of SnO2 nanoparticle colloids using the zeta sizer instrument. This can be seen from the peaks produced, namely only 1 peak each produced from 3 repetitions. In Figure 5, the particle size produced at peak 1 is 58.91 nm, peak 2 with a size of 59.66 nm and peak 3 with a size of 61.40 nm. The three peaks from these measurement results produced an average size of 59.99 nm. Thus, the size of the SnO2 nanoparticle colloid obtained during electrolysis using a concentration of 0.02 M HCl at a potential of 60 volts is 59.99 nm.

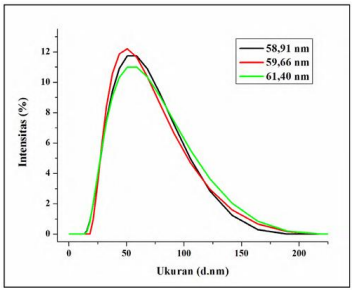

Figure 5. Distribution of Size of Analysis Results of Zeta Sizer Instrument during Electrolysis with HCl Concentration of 0.02 M at Potential of 60 Volts

Colloidal SnO_2 nanoparticles at optimum conditions of concentration influence with electrolysis using HCl concentration of 0.06 M and potential of 60 volts were analyzed using Zeta Sizer instrument. This analysis was conducted to determine particle size and particle size distribution. The results of the analysis using HCl concentration of 0.06 M at potential of 60 volts are presented in Table 2, while the distribution of particle size distribution is shown in Figure 6.

Table 2. Results of Zeta Sizer Instrument Analysis during Electrolysis with 0.06 M HCl Concentration at 60 Volt Potential

No.	Peak 1	Peak 2	Peak 2	% Intensity 1	% Intensity 2	% Intensity 3
1.	82.11	0	0	100	0	0
2.	84.11	0	0	100	0	0
3.	83.48	0	0	100	0	0

Table 2 shows stable results in the measurement of SnO_2 nanoparticle colloids using the Zeta Sizer instrument. This stability can be seen from the consistent peaks, with only one peak produced in each repetition (3 times). In Figure 4.14, the particle size produced at peak 1 is 82.11 nm, peak 2 is 84.11 nm, and peak 3 is 83.48 nm. The average particle size of these three measurements is 83.11 nm. Therefore, the size of the SnO_2 nanoparticle colloid obtained during electrolysis with a concentration of 0.06 M HCl at a potential of 60 volts is 83.11 nm.

The results of the analysis using the zeta sizer instrument from the optimum measurement of the influence of potential and concentration show that the higher the concentration of HCl used, the particle size obtained also increases, namely 59.99 nm for a concentration of 0.02 M HCl and 83.11 nm for a concentration of 0.06 M.

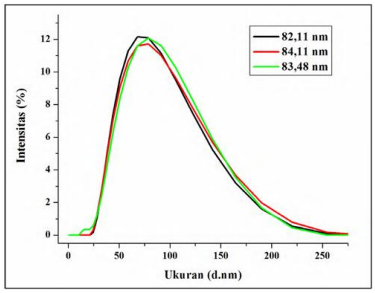

Figure 6. Distribution of Size of Analysis Results of Zeta Sizer Instrument during Electrolysis with HCl Concentration of 0.06 M at Potential of 60 Volts

The results of the analysis show that variations in HCl concentration as an electrolyte in the electrolysis process affect the size of the SnO_2 particles formed. Increasing the HCl concentration from 0.02 M to 0.06 M results in an increase in particle size, indicating that the ion concentration in the solution plays a role in the growth process of nanoparticle crystals. Possibly, at higher concentrations, the number of Cl^- ions interacting with the electrode surface increases, accelerating the rate of Sn^{4+} formation and increasing the chance of particle aggregation, so that the resulting particle size becomes larger.

The FTIR spectrum successfully confirmed the presence of typical functional groups of SnO_2, especially the antisymmetric vibration of Sn-O appearing in the range of 400–700 cm^{-1}. In addition, the presence of hydroxyl groups and adsorbed water indicated that the resulting particles still maintained interactions with the wet environment of the electrolysis process. The presence of OH^- also indicated that the synthesis took place in a wet atmosphere, which is typical in the electrochemical approach, and gave certain characteristics to the surface of the nanoparticles, which could affect the reactivity or further applications of the material [36], [37].

XRD analysis confirmed the successful synthesis of SnO_2 with a typical tetragonal structure. The obtained diffraction patterns were in accordance with the JCPDS standard for SnO_2, indicating that the electrochemical method used was not only effective in producing nano-sized particles, but also in maintaining the purity and regularity of its crystal structure. This is important to ensure the performance of the material in further applications, since the crystallographic properties greatly affect the conductivity, stability, and surface reaction ability [38], [39].

The particle size distribution indicated by one peak in each measurement with Zeta Sizer reflects the stability of the colloidal system formed [40], [41]. This stability is important because uniform particles not only indicate successful synthesis, but also provide advantages in applications such as sensors and catalysts, which require size consistency for optimal performance. Small and uniform sizes also expand the specific surface area, which is an important factor in improving the efficiency of material function [2], [42].

This research has a significant impact on the development of more efficient and sustainable nanoparticle synthesis methods, especially in producing SnO_2 as a functional material with broad application potential in the fields of gas sensors, photocatalysis, and electronics. The electrochemical approach used opens up opportunities for large-scale production at lower costs and with minimal environmental impact [43], [44]. However, this study also has limitations, including limited testing of the long-term stability and real application performance of the resulting nanoparticles. In addition, this study has not yet discussed in depth the effect of variations in electrochemical parameters on the crystal structure and functional properties of SnO_2, so further research is still needed to strengthen the generalization of the findings.

4. CONCLUSION

This study shows that SnO_2 nanoparticles can be synthesized directly from tin metal using an electrochemical method with hydrochloric acid electrolyte solution. The electrolysis potential affects the precipitation process, with an optimum potential of 60 volts. The concentration of HCl used affects the size and amount of SnO_2 nanoparticles produced, where a concentration of 0.06 M HCl produces a maximum absorbance value of 3.068 at a wavelength of 207 nm.

Characterization using XRD shows that the resulting SnO_2 nanoparticles have a tetragonal crystal structure, with three sharp peaks at (110), (101), and (211) at 2θ angles of 26.58°, 33.39°, and 52.04°, respectively. The FTIR spectrum confirms the presence of SnO_2 through the antisymmetric vibration of Sn–O at 580 cm^{-1}.

The results of the Zeta Sizer analysis show the relationship between wavelength and nanoparticle size. At 0.02 M HCl concentration, a wavelength of 203 nm was obtained with a nanoparticle size of 59.99 nm, while at 0.06 M HCl concentration, the wavelength increased to 207 nm with a nanoparticle size of 83.11 nm. These results confirm that the electrochemical method is an efficient approach for the synthesis of SnO_2 nanoparticles with good size control. Further research is suggested to explore the optimization of electrochemical parameters to improve the efficiency and control of the size of the produced SnO_2 particles. In addition, further application tests in the fields of sensory, photocatalysis, or energy storage need to be conducted to test the functional performance of the synthesized nanoparticles.

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