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by Dedi Rohendi

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Synthesis and Characterization of Amorphous TiO₂ Anode Prepared by Anodizing Method for Na-ion Batteries

Dwi Indrawati¹, Nirwan Syarif^{2,3*} and Dedi Rohendi^{2,3}¹Master Program, Department of Chemistry, Faculty of Mathematics and Natural Sciences, University of Sriwijaya, Jl. Padang Selasa no. 524, Bukit Lama, Ilir Barat, Palembang, Indonesia 30121²Department of Chemistry, Faculty of Mathematic and Natural Sciences, University of Sriwijaya, Jl. Palembang-Prabumulih KM-35 Indralaya, Ogan Ilir, Indonesia, 30662³Center of Research Excellent in Fuel Cell and Hydrogen, University of Sriwijaya, Jl. Srijaya Negara Bukit Besar, Palembang, Indonesia, 30139*Corresponding Author: nsyarif@unsri.ac.id

Abstract

Sodium ion battery (SIB) has engage sustainable alternative on replacing lithium ion batteries. The lower potential and larger ionic value has demanded on seeking a potential anode material. Amorphous TiO₂ prepared by electrochemical anodization technique has provide a suitability as anode SIB materials, which approved by conducting XRD, FTIR, and SEM-EDX measurements. Electrochemical performance was tested with variations in the concentration of NaOCl₄ electrolyte in propylene carbonate (PC) solvent. Diffraction pattern of TiO₂ showed the only peaks of Ti was observed at $2\theta = 40.34^\circ$. IR spectrogram shows that the absorption band at 507 cm^{-1} is the peak of the vibration characteristics of the Ti-O bond and the peak of 975 cm^{-1} correspond to Ti-O-Ti bond. SEM-EDX image analysis showed the morphology of TiO₂ was smooth without pore with a ratio of Ti: O elemental composition of 1: 2. Voltammogram showed the anodes in 0.5 M NaOCl₄ electrolyte in PC solvent has a good stability potential windows with the high current density 1.2 mA and the capacity of 0.037 F/g.

Keywords: Anodizing, Na-Ion Battery, Cyclic Voltammetry, TiO₂, Propylene Carbonate

Abstrak (Indonesian)

Baterai natrium ion dapat digunakan sebagai alternatif berkelanjutan untuk menggantikan baterai lithium ion. Nilai potensial yang rendah dan volume ionik yang besar sehingga perlu dicari material anoda yang ideal. Amorf TiO₂ yang disintesis dengan menggunakan teknik anodisasi memiliki kesesuaian sebagai bahan anoda SIB, material dikarakterisasi menggunakan XRD, FTIR, and SEM-EDX. Pengujian elektrokimia dilakukan dengan variasi konsentrasi elektrolit NaOCl₄ dalam pelarut propilen karbonat (PC). Difaktogram TiO₂ menunjukkan hanya puncak Ti yang teramati pada sudut $2\theta = 40,34^\circ$. Pola spektrum IR pada TiO₂ menunjukkan terjadinya pita penyerapan pada $507\text{-}590\text{ cm}^{-1}$ yang merupakan vibrasi *stretching* dari ikatan Ti-O dan pita penyerapan pada 975 cm^{-1} yang sesuai dengan vibrasi *stretching* dari ikatan Ti-O-Ti. Analisis citra SEM-EDX menunjukkan morfologi TiO₂ halus tanpa pori dengan perbandingan komposisi unsur Ti:O yaitu 1:2. Voltammogram menunjukkan anoda amorf TiO₂ menggunakan elektrolit NaOCl₄ 0.5 M dalam pelarut PC memiliki stabilitas yang baik dengan kepadatan arus 1,2 mA dan kapasitansi 0,037 F/g.

Kata Kunci: Anodisasi, Baterai Na-Ion, Voltametri Siklik, TiO₂, Propilen Karbonat

INTRODUCTION

Lithium-ion batteries have been widely used in large-scale electric energy storage applications for renewable energy and smart grid, but the availability of lithium metal is limited so it is necessary to look

for other alternatives such as sodium metal. Sodium metal has physical and chemical properties similar to the lithium because it is in the IA group in the periodic system of elements [1]. Sodium-based batteries need to be studied as an ideal alternative in replacing lithium ion batteries because the

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availability of sodium metal is very abundant and the charge transfer of sodium ions in electrolytes is easier than lithium ion [13] due to their lower solvation enthalpy because of the weaker Lewis acidit [13] [2]. The low solvation enthalpy of Na-ion shows lower activation barriers of Na-ion insertion, so indicated the faster charge-discharge characteristics of SIBs than LIBs [3]. Sodium batteries have a lower potential value compared to the lithium and an ionic volume is twice as large as lithium ions, so a material that is easily inserted into a sodium battery is needed [4]. The anode of lithium-ion batteries that are more often used is graphite material. Graphite cannot be used as an anode in sodium-ion batteries because the size of the ion/cation radius of sodium metal is much larger, resulting in de-intercalation of sodium ions [5].

New materials have been introduced and evaluated as electrode materials for sodium-ion batteries (SIBs) in recent years. Similar to lithium-ion (LIBs) batteries, metal oxide compounds and layered compounds have been widely investigated as anode materials in sodium-ion batteries. One of the electrodes used in these two types of batteries is titanium.

Titanium-based electrode materials have recently received wide attention because they have high thermal stability, are not toxic to humans and the environment, and are very good at degrading pollutants [6]. Guo [7] reported that titanium-based electrodes play a role in enhancing structural stability at the cathode, high battery safety and charge transfer providers. Titanium dioxide (TiO_2) is the most studied material due to its abundance and structural diversity. Titanium oxide has been developed as an anode in micro-biofuel cells [8], solar cells [9], super capacitors [10], and sodium-ion battery anodes [11].

Titanium dioxide (TiO_2) has been one of the best candidates for LIB anode materials [12]. The operating voltage of TiO_2 anode ($\sim 1.5 \text{ V vs. Li}^+/\text{Li}$) is higher than that of the anode material based on carbon ($\sim 0.1 \text{ V vs. Li}^+/\text{Li}$) [13]. It is of great interest to investigate the utilization of TiO_2 for Na-ion batteries because of its success as an anode material for Li-ion batteries. Amorphous TiO_2 electrodes can increase the diffusion of ion transport.

Electrochemical performance of TiO_2 with Na^+ ions achieved by reducing the particle size for shorten the migration length for Na^+ insertion. Titanium oxide (TiO_2) can be synthesized using sol-gel methods [11], hydrothermal [14], and anodizing methods [15]. The anodizing technique is a method that is relatively simple, inexpensive and the particle

size obtained was uniform. The particle size has a very significant effect on the sodium process intercalation into amorphous TiO_2 [1]. Amorphous TiO_2 which was synthesized by the anodizing technique is able to store sodium for up to 15 cycles with a reversible capacity of 150 mAh/g. The increased concentration of the interface area at amorphous materials can form percolation pathways to facilitate ion diffusion.

In this study, the electrochemical properties of amorphous TiO_2 with variations in the concentration of NaOCl_4 electrolyte in propylene carbonate (PC) solvent were described. NaOCl_4 in PC solvent has a high conductivity value [16][17], so that it can increase the electrochemical performance of the anode.

The synthesis of TiO_2 anode by the anodization technique will be conducted. The resulting anode will be characterized using XRD, FTIR, and SEM-EDX. The characterization of electrochemical properties of the TiO_2 anode was carried out by using the cyclic voltammetry (CV) method with various concentrations of NaOCl_4 electrolyte (0.2, 0.5, 0.8, 1.0, and 2.0).

MATERIALS AND METHODS

Materials

Chemicals used were analytical grade i.e. pure titanium thin-foil grade 2 (0.1 mm), acetone, ethanol, deionized water, hydrofluoric acid (HF), binchotan carbon, sodium perchlorate (NaClO_4), and propylene carbonate (PC).

Synthesis of Amorphous TiO_2 Anode

Amorphous TiO_2 was synthesized by electrochemical anodization. Pure titanium thin-foil with a size of 3.25 x 1.25 cm was cleaned by soap and rinsed with deionized water. It was cleaned by acetone, ethanol, and deionized water rinse and dried at room temperature. Titanium thin-foil is inserted into the chamber containing 50 mL of 1% HF electrolyte solution using a DC current with a voltage of 25 V for 20 minutes. Titanium substrate was used as anode. Binchotan rod was used as the cathode. The distance between anodic and cathodic electrodes was 20 mm. Anode and cathode were connected to power supply with copper wire. The voltage between anode and cathode was 25 volt. During the reaction process, magnetic stirring was performed to ensure the homogeneity of the electrolyte solution concentration and temperature uniformity. Samples were dried at room temperature and then heated at 110 °C for overnight.

Materials Characterization

The powder samples were characterized by X-ray powder diffraction (XRD) using a Rigaku Miniflex 600 diffractometer to determine the crystal size and lattice parameters, FTIR using Thermo Scientific Nicolet iS10 for detect functional groups, and Scanning electron microscope (SEM) images were taken using a VEGA 3 TESCAN for characterizing surface morphology.

Electrochemical Measurements

Measurements were conducted in potentiostat in voltammetry mode and set in half cell configuration. The anode was respectively placed in the chamber as the working electrode, the reference electrode was Ag / AgCl and the counter electrode was with Pt wire connected. The three electrodes were connected to the potentiostat which works at scan rate of 5, 25, 50, and 100 mV/s. The concentration of electro. NaOCl₄ in PC solvent as varied in 0.2 M, 0.5 M, 0.8 M, 1.0 M, and 2.0 M

Data Analysis

The capacity can be determined using shown in equation 1:

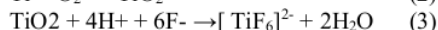
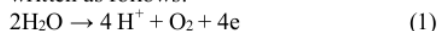
$$C = \frac{\Delta Q}{w \times \Delta V} = \frac{\int I dV}{s \times \Delta V \times w} \quad (1)$$

Where C is capacity, I is the current, V is the voltage, s is the scan rate, and w is the weight.

RESULT AND DISCUSSION

Synthesis and Characterization of Amorphous TiO₂ Anodes

The TiO₂ anode was prepared by the anodization technique. The technique was used to grow the oxide layer on a titanium plate substrate with the help of electric current [15]. The electrode used a Ti metal plate which as an anode as well as a precursor and matrix to support TiO₂/Ti, while binchotan carbon is used as a cathode. The anode will undergo an oxidation reaction and the cathode reduction reaction occurs. The Ti plate will react with the oxygen anion O²⁻ dissolved in the electrolyte solution of fluoride acid to formed a thin layer on the Ti surface. The oxygen contained in water acts as an electron carrier that diffuses towards the anode cell, while in the cathode cell the electrons react with H⁺ ions to formed hydrogen gas bubbles (H₂) on the binchotan carbon surface. The reaction that occurs is written as follows:



During the anodization process, the color of Ti sheets changes from violet to blue, green, and finally gold. The color changes may be related to the thickness increasing of the TiO₂ layer or TiO₂ nanotube formation [18]. The oxide layer formed on the Ti surface is an amorphous phase [1][19]. Figure 1 showed the diffractogram amorphous TiO₂, but only peaks from Ti were observed in the XRD patterns of the as-formed TiO₂ [20][21].

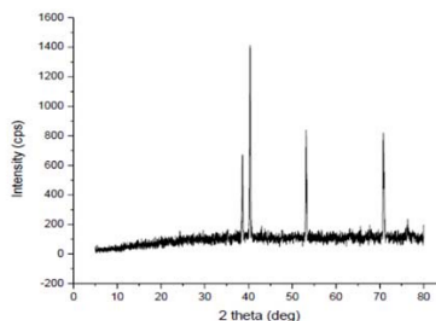


Figure 1. XRD pattern of TiO₂/Ti

Spectrophotometry IR is a method that can be used to analyzed chemical compounds with identify the functional group of materials. FT-IR spectra of the amorphous TiO₂ in Figure 2 show that peaks corresponding to stretching vibrations Ti-O and Ti-O-Ti bonds are around 500 cm⁻¹ to 1000 cm⁻¹ [22]. This broad absorption band shows the difference in vibration from the Ti-O-Ti and Ti-O bonds which reflects the formation of TiO₂. The peaks that appear at the wave number 507 - 590 cm⁻¹ are the stretching vibrations of the Ti-O bonds and the absorption in the 975 cm⁻¹ region is the Ti-O-Ti stretching vibrations [22]. Based on the results of FTIR analysis, it strengthens the assumption that the process of making TiO₂/Ti electrodes using the anodizing method approach has been successfully reached.

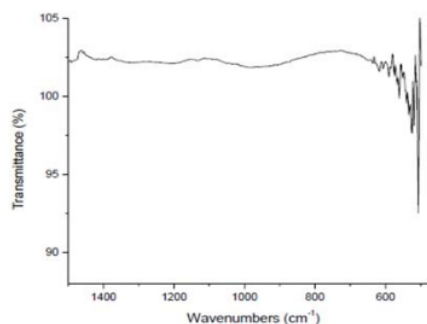


Figure 2. FT-IR analysis of TiO₂/Ti

Structural characterization of the TiO_2 was performed using a SEM-EDX with magnifications of 10 kx and 200 kx showed figure 3. Synthesis of TiO_2/Ti using the anodization method produce TiO_2/Ti that has pores morphology due to the influence of electrolytes, currents, and potential differences. These factors have been confirmed by changing the chemical composition, crystallinity, and morphology of the Ti substrate. The SEM image results showed that no pores were formed has been reported by Ng and co-workers [23]. It was inferred that due to the ineffectiveness of the HF solution in the formation of porous titanium oxide is lower than the rate of dissolving the oxide. The formation of the pores were provided by the solvation of fluoride complex ion $[\text{TiF}_6]^{2-}$. The dissolving of Ti^{4+} ion caused the pore merging and ultimately loss the pore structures [24].

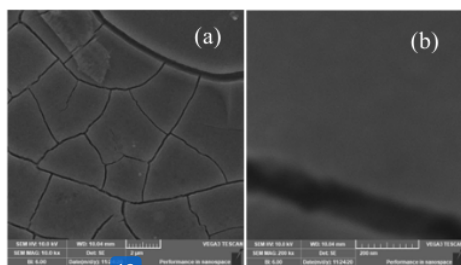


Figure 3. SEM images of the synthesized TiO_2/Ti at different magnifications (a) 10 kx (b) 200 kx

Elemental composition of TiO_2/Ti and determining whether foreign elements are introduced to the lattice of TiO_2 . The investigation was conducted by using EDX techniques. Figure 4 shows the EDX spectra that show the presence of Ti elements is 59.96%, O elements are 33.41%, C elements are 2.60% and fluoride elements are 4.02%. The EDX spectra indicate that the presence of Ti, O, F, and C in sample and that the ratio of Ti:O is approximately 1:2. This indicates the structure of the TiO_2 with the presence of carbon and fluorine from organic electrolytes attached to the TiO_2 lattice [15][25].

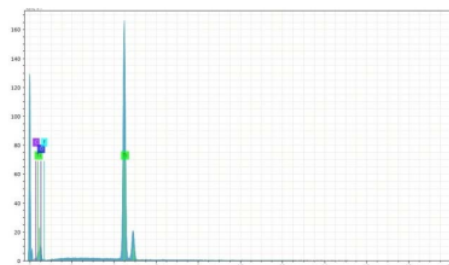


Figure 4. EDX spectrum of the synthesized TiO_2/Ti

Electrochemical and Performance Measurement

The characterization of electrochemical properties of the TiO_2 anode was provided by potentiostat. The cyclic voltammetry (CV) method employed at scan rate of 5, 25, 50, and 100 mV/s in potential windows of -1.5 to 1.5 volts. The corresponding CV profiles are as shown in Figure 5. It can be shown the clear shape of the CV profiles that changed with the concentration of electrolyte. The results show that the electrochemical properties of the anode with 0.5 M sodium perchlorate electrolyte with propylene carbonate solvent gave a good performance. At concentrations of 0.2 M and 0.5 M, the redox peaks of the anode were clearly visible, while for concentrations of 0.8, 1.0 and 2.0 M. It gave a small current response and an oval curve without redox peaks. The highest current response in the CV profile was generated by the concentration of 0.5 M NaOCl_4 i.e. 1.2 mA with an anodic peak at -0.8 V. The general results obtained were higher than those reported by [16] namely 0.2 mA.

The high concentrations of electrolytes produce low electric currents due to the ion mobility [26]. The increasing of the concentration at 0.8, 1.0, and 2.0 M of NaOCl_4 , the viscosity of the electrolyte were observed to be increased [16]. The ion transfer in this electrolyte is slightly reduced due to the low ion conductivity of the electrolyte.

Ponrouch [17] reported that PC solvent has a high conductivity value due to the influence of a large dielectric constant for the degree of dissociation of the salt. This increases the dissociation of salts which increases the mobility of ions at the electrode, increase the formation of a stable SEI and can reduce the battery operating temperature [27].

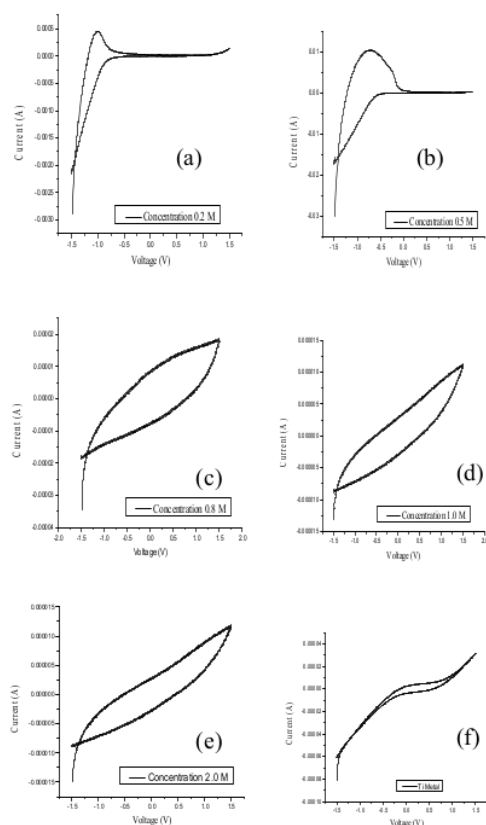


Figure 5. CV profiles of TiO_2/Ti with different NaOCl_4 electrolytes concentrations in PC solvent (a) 0.2 M (b) 0.5 M (c) 0.8 M (d) 1.0 M (e) 2.0 M and (f) Ti Metal

Besides, the high electrolyte conductivity value of NaOCl_4 will provide good electrochemical properties.

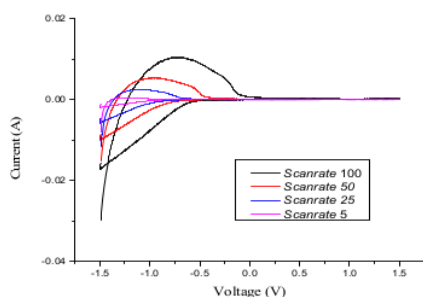


Figure 6. Voltammogram of amorphous TiO_2 anode with 0.5 M NaOCl_4 in PC at different scan rate

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Figure 6. shows the window stability of 0.5 M NaClO_4 electrolyte in PC solvent at a scan rate of 5, 25, 50, and 100 mV/s. The voltammogram shows the overlap between the scan rate and the split peaks that increase with scan rate speed used. The higher of scan rate used the blanch is getting wider and the current increases, it can be related to the ion diffusion resistance in certain micro pores. Especially the portion of the micro pores is accessible by the electrolyte. The anode has good stability in electrolytes after running several times with the resulting capacity of 0.037 F/g.

CONCLUSION

The amorphous TiO_2 anode was synthesized by electrochemical anodization technique and has been successfully confirmed its performance. The diffractogram of TiO_2 showed the only peaks from Ti were observed at $2\theta = 40.34$. The TiO_2 IR spectrogram shows that the absorption band at 507-590 cm^{-1} belong to the peak of the vibration characteristics of the Ti-O bond and the peak of 975 cm^{-1} corresponds to the Ti-O-Ti bond. SEM-EDX image analysis shows the smooth morphology of TiO_2 without pores with elemental composition a ratio of Ti: O (1: 2). CV profile shows the anode using 0.5 M NaOCl_4 electrolyte in PC solvent resulting in the high current density 0.012 A and the capacity of 0.037 F/g

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