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# Extraction of Silica ($SiO_2$) from Rare-Earth Metal Zircon ($ZrSiO_4$) as Lithium-Ion Battery Anode Material

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## Abstract

Zircon is a rare metal natural mineral composed of zirconia and silica. The silica content in zircon can be used as anode material for lithium-ion batteries because of the simple preparation and high theoretical capacity. Silica is obtained from the zircon extraction process with 5 M NaOH solution with aging time variation to determine the silica surface area. The $SiO_2$/C composite was obtained from the solid-state reaction process of a mixture of silica and graphite. The silica results obtained were tested for characteristics using X-ray diffraction (XRD), Surface area analyzer (SAA), and Scanning electron microscopy/Energy dispersive X-ray (SEM/EDX). Battery performance test with silica as an anode to determine the number of cycles, capacity, and Coulombic efficiency using Cyclic voltammetry (CV) and Charge/Discharge Cycle (CDC) tests. Based on the results of battery performance testing, the battery used silica with an aging time of 18 h at an annealing temperature of 800°C resulted in a first cycle discharge of 222 mAh/g, first cycle charge of 311 mAh/g, and Coulombic efficiency of 71.4%. Whereas used silica with an aging time of 24 hours at an annealing temperature of 800°C resulted in a first cycle discharge of 182 mAh/g, a first cycle charge of 262 mAh/g, and a Coulombic efficiency of 70%.

## 1. Introduction

Rare earth metals are metal minerals whose existence in nature is very rare. Generally, there are four minerals containing rare earth metals: monazite, bastnasite, xenotime, and zircon [1]. Rare earth metals are used as superconducting materials, rechargeable batteries, and cracking catalysts [2]. However, in its development, one of the minerals containing rare earth metals, namely zircon ($ZrSiO_4$), is still not optimally utilized in electronic and green energy products such as lithium-ion battery anodes. Utilization as anode lithium-ion batteries because lithium-ion batteries have high energy capacity, long life cycle, and low environmental impact. The availability of zircon ($ZrSiO_4$) as a mineral containing rare earth metals in nature can reach 58% [3].

Zircon ($ZrSiO_4$) is a natural mineral composed of zirconia ($ZrO_2$) and silica ($SiO_2$), which have a stable bond. Silica ($SiO_2$) content in zircon ($ZrSiO_4$) is utilized in electronic and green energy products as an anode material for lithium-ion batteries. Some studies use silica ($SiO_2$) as an anode material due to its much lower volume expansion than Si and its high theoretical capacity (1965 mAh $g^{-1}$). $SiO_2$ has a low cost and preparation process, making it a promising candidate as anode material for LIB [4]. However, $SiO_2$ has poor conductivity, so it is necessary to add agents that can increase the conductivity of $SiO_2$ by adding graphite using the solid-state reaction method to form a $SiO_2$/C composite [5]. Mixing graphite with $SiO_2$ can reduce the volume changes in the electrodes and increase electrical conductivity [6]. Research has shown that $SiO_x/C$ will experience a rapid reduction in volume expansion after 40 cycles, and the return efficiency reaches 83.5% after 100 cycles [7]. The source of silica ($SiO_2$) in zircon ($ZrSiO_4$) will be used as anode material for lithium-ion batteries can be obtained from the extraction process using a mechanochemical method followed by acid and alkali fusion [8]. In general, extraction is a process of separating solutes with solvents where the solvent will extract the desired material without dissolving other substances [9]. Several factors affect the extraction process, including temperature, concentration, solvent solution, extraction time, and stirring [10]. Zircon ($ZrSiO_4$) is decomposed into zirconia ($ZrO_2$) and silica ($SiO_2$) based on the reaction of $ZrSiO_4 \rightarrow ZrO_2 + SiO_2$.

To improve lithium-ion batteries' performance, paying attention to the surface area of the material used for lithium-ion battery anodes is necessary. The manufacture of $SiO_2/C$ aims to accelerate the transfer of electrons and produce a high capacity [11]. One of the efforts to expand the surface of $SiO_2$ is to adjust the aging time in the $SiO_2$ extraction process. The purpose of this research is to determine the extraction process of silica ($SiO_2$) in zircon ($ZrSiO_4$) through aging time variations to determine the optimal particle surface area and to determine the performance of lithium-ion batteries with silica ($SiO_2$) from zircon ($ZrSiO_4$) as the anode source. Moreover, this research will be able to contribute to research on science in the fields of electronics and renewable energy in the creation of LIB by utilizing the potential of the rare earth mineral zircon.

## 2. Experiments

### 2.1. Materials

The materials used in this research were zircon sand, 5 M NaOH (Merck), 2 M HCl, 11 M HCl, distilled water, graphite (Gelon LIB. Co. Ltd., China), NMC (Nickel, Mangan, Cobalt) cathode (Gelon LIB. Co. Ltd., China), AB (acetylene black) (Gelon LIB. Co. Ltd., China), CMC (carboxy methyl cellulose) (Gelon LIB. Co. Ltd., China), SBR (Styrene butadiene rubber) (Gelon LIB. Co. Ltd., China), copper foil, aluminum foil, Polypropylene separator (Celgard), and electrolyte solution $LiPF_6$ (Gelon LIB. Co. Ltd., China).

### 2.2. Extraction of Silica from Zircon

Zircon sand was obtained from a Yogyakarta ceramic store with mineral content including zirconia of 36.8%, silica of 21.13%, and alumina of 1.16% [12]. A 25 grams of zircon sand was extracted using 400 mL of 2 M HCl. The mixture was heated at 100°C for 1 hour under stirring at 300 rpm. Then, it was neutralized using 200 mL of distilled water until the pH was neutral. Neutralized zircon, reacted with 150 mL of 5 M NaOH (Merck) using a magnetic stirrer, was heated at 250°C for 1 hour under stirring at 300 rpm. The reaction results with NaOH was left overnight to form a zircon precipitated and a sodium silicate solution. The resulting reaction was filtered to obtain a sodium silicate solution and precipitated zircon. The sodium silicate was precipitated using 50 mL of 11 M HCl until silica gel formed. Silica gel was allowed to stand for various aging times: 18 and 24 hours. The hierarchical porous structure of the minimum $SiO_2$ begins to form when the aging time is 18 hours [11]. The resulting silica gel was filtered using filter paper and washed with distilled water until the pH was neutral. The gel was then dried using a furnace at a temperature of 500, 700, and 800°C for 3 hours to form silica powder [8].

### 2.3. $SiO_2$ Composite Manufacturing with Solid-State Reaction

Silica that has been obtained from the extraction process was composited with graphite. $SiO_2$ and graphite were mixed with a weight ratio 1:9 with 0.3 grams of silica and 2.7 grams of graphite. Using the solid-state reaction method, the $SiO_2$ and graphite mixture was ground with a mortar and pestle to obtain $SiO_2/C$ [13].

### 2.4. Silica Characterization Test

Characterization was carried out on extraction samples with aging time variations of 18 hours and 24 hours using X-ray Diffraction (XRD, EQ-MD-10 Precision Mini XRD) to see the extracted $SiO_2$ crystalline phase, using X-ray source from Cu K$\alpha$ ($\lambda$= 0.154 nm), 2$\theta$ from 17°-71°. Scanning Electron Microscope/Energy Dispersive X-Ray (SEM/EDX, JEOL Benchtop JCM 7000) with a high voltage of 15 kV was used to analyze the morphology and content of silica. A surface Area Analyzer (SAA, Quantachrome Novatouch Lx4) was utilized to determine the particle surface area.

### 2.5. Battery Performance Test

Battery assembly testing must be done before performing a battery performance test. At the battery assembly stage, it started by dissolving $SiO_2/C$: AB (Acetylene Black): CMC (Carboxymethyl Cellulose): SBR (Styrene Butadiene Rubber) material in a ratio of 93:1:1:5 with distilled water. The ingredients were mixed using a mortar and pestle to form a paste, and then anode slurry was formed. The resulting paste (anode slurry) was evenly coated with Cu-foil using a doctor blade and dried in the oven. The dry film was pressed and then cut to fit the size of the battery using a slitting machine. The nickel sheet was attached to the side of the film using a welder machine. The resulting thin anode layer with a separator-anode-separator-cathode arrangement was made using a winding machine. The rolled film was inserted into a cylinder cell using a spot welder machine and then covered with a cap welder using a grooving machine. The cylinder cell was put into a vacuum oven at 50-70°C overnight. The cathode used was NMC, where the cylindrical battery was made in a glove box by adding an electrolyte solution. Cylinder cells were packed using a sealing machine and wrapped using a hot rolling machine [13]. In testing battery performance, the test equipment used was cyclic voltammetry (CV) and charge/discharge cycle (CDC), which obtained the results of cycles, capacity, and battery voltage [14].

## 3. Results and Discussion

### 3.1. Results of $SiO_2$ Extraction from Zircon

Samples with varying heating temperatures and aging times were obtained after the extraction. The temperature variations were 500, 700, and 800°C, with aging time variations of 18 and 24 hours for the annealing temperature of 800°C. Figure 1 depicts the extraction of silica from zircon.

Based on Figure 1, it can be seen that samples $SiO_2$-500, $SiO_2$-700, and $SiO_2$-800 have different colors and textures. Sample $SiO_2$-500 has a dark color with a rough and lumpy texture because it contains organic matter ash. Sample $SiO_2$-700 is brighter than sample $SiO_2$-500 with less lumpy texture because the black color will be decomposed to produce white bone. Meanwhile, sample $SiO_2$-800 has the brightest color and finer texture according to $SiO_2$ specification. This color difference is due to temperature variations [8]. The extraction results for every 25 grams of zircon are shown in Table 1.


**Figure 1**. The final products of $SiO_2$ with an aging time of 18 hours and at different annealing temperatures, (a) $SiO_2$-500, (b) $SiO_2$-700, (c) $SiO_2$-800, and (d) $SiO_2$-800 with an aging time of 24 hours

**Table 1**. Percentage of extraction silica from zircon

| Sample | Mass of Extraction (%) |
|---|---|
| $SiO_2$-500 | 9.2 |
| $SiO_2$-700 | 17.6 |
| $SiO_2$-800 18 hours | 21.6 |
| $SiO_2$-800 24 hours | 26.8 |

### 3.2. Crystal Phase Characterization

Characterization of crystal phase using the XRD test with a voltage of 40 kV, current of 35 mA, and angle range was 10-80°. Figure 2 shows the XRD results of silica variation temperature and aging time. Figure 2(a) shows that the silica is already crystalline at a heating temperature of 500, 700, and 800°C. However, based on the diffractogram results, the highest peak proves the best crystallinity at a temperature of 800°C. High crystallinity will affect the structural stability of silica. The higher the crystallinity, the higher the stability of the silica structure, which affects the performance of silica as an anode material. Therefore, variations of aging time were carried out for 18 and 24 hours at a temperature of 800°C. In the XRD test in Figure 2(b) with a heating temperature of 800°C, the aging time of 18 and 24 hours shows that zircon contains silica with a cristobalite phase [15]. Based on the data JCPDS: 36-1451, the appearance of cristobalite-phase silica appears at the top of the graph with an angle of 21.89, 25.52, 28.36, 31.67, 36.01, 42.52, 46.78, 53.81, 56.39, 60.25, 64.89, and 72.99. If the heating temperature increases, the crystal size will decrease except at 700°C [16]. Furthermore, the crystallite size was calculated using Scherrer Equation (1).

$$D = \frac{k\lambda}{B_{hkl}Cos\theta_{hkl}} \quad (1)$$

where, k is shape factor (0.9), λ is X-ray wavelength (0.154 nm), $B_{hkl}$ is the half-width of the diffraction band (FWHM) in radians, and $\theta_{hkl}$ is Bragg diffraction angle in radians.

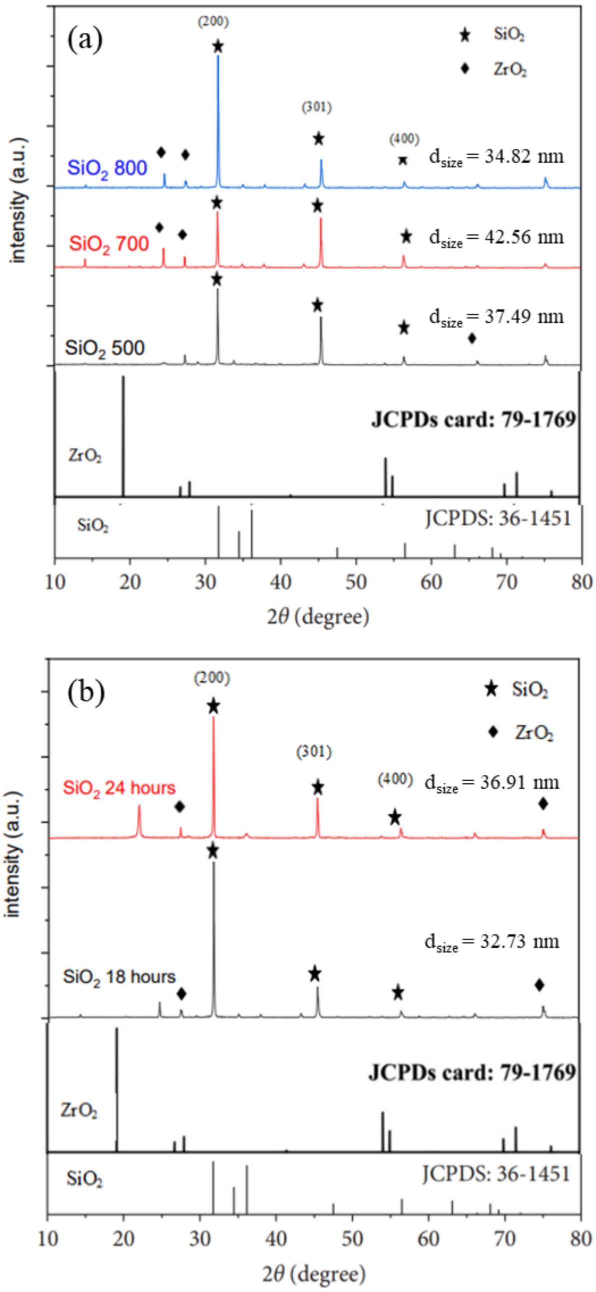


**Figure 2**. XRD patterns of silica of (a) temperature variation, (b) aging time variation

### 3.3. Morphological and Chemical Composition of Silica

Figure 3(a) shows the morphology of silica with an aging time of 18 hours ($SiO_2$-18 hours) at an annealing temperature of 800°C, which consists of entirely primary particles which aim to increase the surface area of the silica. Figure 3(b) depicts the silica morphology after

24 hours of aging ($SiO_2$-24 hours), which consists of primary and secondary particles. Small primary particles and adherent particles make up the secondary particles. The silica content is shown in Table 2 by the SEM/EDX analysis. From the results of the EDX analysis, it is possible to identify the types of chemical composition of each sample. Sample $SiO_2$-18 hours showed that element O had a percentage of 25.00%, and element Si had a percentage of 5.74%. In comparison, sample $SiO_2$-24 hours showed that element O had a percentage of 36.48%, and element Si had a percentage of 18.04%.

Furthermore, elements such as Na and Cl were still detected in the two samples, which could be attributed to the reactants used during extraction. This data confirms that $SiO_2$ was successfully extracted during the zircon mineral extraction process. The sample that aged for 24 hours has a larger silica composition than aging in 18 hours because that aging can increase the nucleation rate and reduce the induction period and crystallization time.

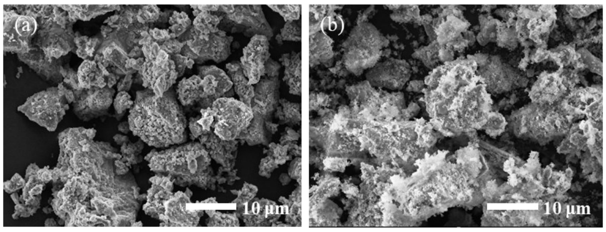


**Figure 3**. SEM images (500× magnification) of (a) $SiO_2$-18 hours, (b) $SiO_2$-24 hours

**Table 2**. Chemical composition of silica

| Sample | %Mass | | | | |
|---|---|---|---|---|---|
| | O | Na | Si | Cl | Al |
| $SiO_2$-18 hours | 25.00 | 29.90 | 5.74 | 36.70 | 5.74 |
| $SiO_2$-24 hours | 36.48 | 17.77 | 18.04 | 27.70 | |

### 3.4. Surface Area Analysis of Silica

Silica surface area was characterized using SAA (Surface Area Analyzer) at 200°C for 2 hours of degassing time. Figure 4 shows the SAA results of $SiO_2$-18 hours and $SiO_2$-24 hours.

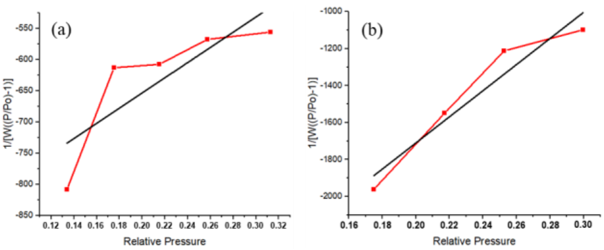


**Figure 4**. The surface area of (a) $SiO_2$-18 hours, (b) $SiO_2$-24 hours

Figure 4(a) shows the surface area analyzer (SAA) results with an aging time of 18 hours, producing a surface area of 8.7271 $m^2/g$ with a correlation coefficient of 0.845044. Figure 4(b) shows the results of the surface area analyzer (SAA) test with an aging time of 24 hours, producing a surface area of 0.887 $m^2/g$ with a correlation coefficient of 0.972199. The analysis of the results of the surface area analyzer (SAA) test reveals that the sample with an aging time of 18 hours ($SiO_2$-18 hours) has a larger surface area than the sample with an aging time of 24 hours ($SiO_2$-24 hours). The large surface area of the silica structure will result in a high discharge capacity so that it is suitable for use as an anode material [13].

### 3.5. Battery Performance Test Results

The first three cycles of the battery performance test were performed using cyclic voltammetry (CV, NuVant EZWare Potentiostat) and the charge discharging cycle (CDC, Neware Battery Analyzer 8 Channel). The theoretical capacity of the $SiO_2/C$ anode material is 300 mAh/g. Figure 5(a) shows the results of the CDC test on sample $SiO_2$-18 hours with first cycle discharge of 222 mAh/g, first cycle charge of 311 mAh/g, and 71.4% of Coulombic efficiency. Figure 5(b) shows the results of the CDC test on sample $SiO_2$-24 hours, which has sample $SiO_2$-24 hours with first cycle discharge of 182 mAh/g, first cycle charge of 262 mAh/g, and Coulombic efficiency of 70%. The CDC performance includes first cycle discharge capacity, first cycle charge capacity, and Coulombic efficiency is tabulated in Table 3. The battery efficiency was calculated using Equation 2.

$$Efficiency = \frac{Discharge\ Capacity}{Charge\ Capacity} x\ 100\% \quad (2)$$

**Table 3**. CDC performance for sample $SiO_2$-18 hours and $SiO_2$-24 hours

| Sample | 1-st Cycle Discharge Capacity (mAh/g) | 1-st Cycle Charge Capacity (mAh/g) | Coulombic Efficiency (%) |
|---|---|---|---|
| $SiO_2$-18 hours | 222 | 311 | 71.4 |
| $SiO_2$-24 hours | 182 | 262 | 70 |

$SiO_2$ with a large surface area produces a high discharge capacity, affecting battery efficiency. $SiO_2$-18 hours has a larger surface area than $SiO_2$-24 hours, so a decrease in efficiency can occur in $SiO_2$-24 hours because the surface area of $SiO_2$-24 hours is smaller than the surface area of $SiO_2$-18 hours. Figures 5a and 5b show a decrease in capacity from the first to the second cycle caused by forming a solid electrolyte interphase (SEI). A SEI is generated on the anode during the first few charging cycles of lithium-ion batteries. The SEI forms a passivation layer on the anode surface, preventing further electrolyte decomposition and allowing for the long calendar life required by many applications. The irreversible formation of SEI and the irreversible formation of $Li_2O$ and lithium silicates occur during the $SiO_2$ lithiation. The electrochemical reaction that may occur during the $SiO_2$ lithiation is listed in Equations 3-6 [4].

$$SiO_2 + 4Li^+ + 4e^- \rightarrow 2Li_2O + Si \quad (3)$$

$$2SiO_2 + 4Li^+ + 4e^- \rightarrow Li_4SiO_4 + Si \quad (4)$$

$$3SiO_2 + 4Li^+ + 4e^- \rightarrow Li_2SiO_3 + 2Si \quad (5)$$

$$5SiO_2 + 4Li^+ + 4e^- \rightarrow Li_2Si_2O_5 + Si \quad (6)$$

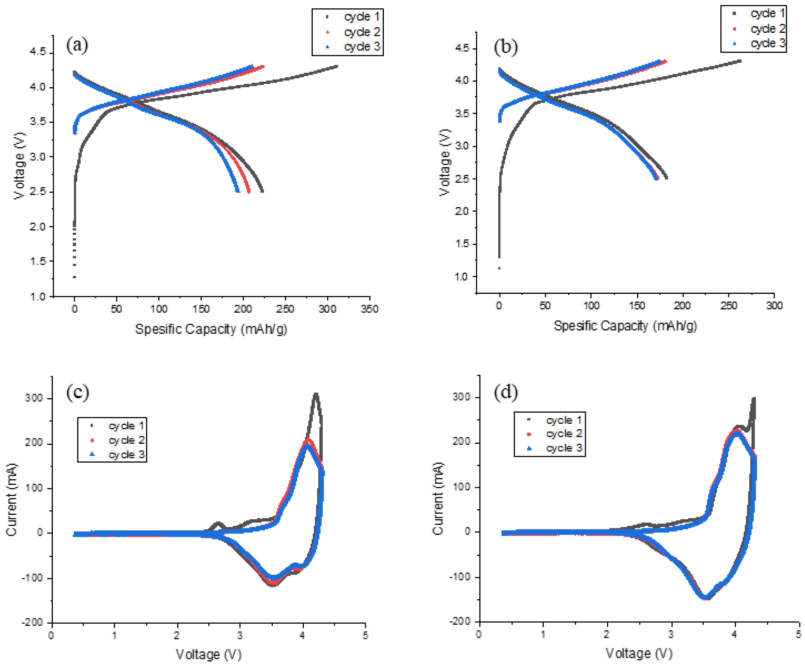


**Figure 5**. Charge discharge curve (a) $SiO_2$-18 hours, (b) $SiO_2$-24 hours and cyclic voltammetry curve of a battery using a silica anode (c) $SiO_2$-18 hours, (d) $SiO_2$-24 hours

**Table 4**. Current and voltage obtained from cycle 1 for the sample $SiO_2$-18 hours and $SiO_2$-24 hours

| Sample | Current (mA) | | Voltage (V) | | ΔV |
|---|---|---|---|---|---|
| | Oxidation | Reduction | Oxidation | Reduction | |
| $SiO_2$-18 hours | 309.03 | -116.05 | 4.20 | 3.51 | 0.69 |
| $SiO_2$-24 hours | 296.06 | -148.57 | 4.30 | 3.57 | 0.73 |

The CV test results show the relationship between the applied potential and the measured current, shown in Figures 5(c) and (d). The CV results are obtained by the oxidation and reduction processes, as shown in Table 3. Theoretically, the amount of current only affects the voltage value, while battery performance is affected by the voltage range (ΔV). Based on Table 4, it is known that of the three existing cycles, cycle 1 has a small voltage range. The smaller the voltage range, the better the battery capacity [17].

## 4. Conclusion

The three stages of the silica extraction from zircon are through immersion of zircon with HCl, reaction with NaOH, and precipitation with HCl. The highest silica and optimal surface area were obtained from the 18-hour aging time variation ($SiO_2$-18 hours) of 8.721 $m^2/g$. The percentage of elements O is 36.48%, and Si is 18.04%. The silica obtained was composited with graphite ($SiO_2/C$) as the anode material for lithium-ion batteries and obtained the highest battery performance with a capacity of 222.7 mAh/g at an aging time variation of 18 hours ($SiO_2$-18 hours) with an efficiency of 71.4%.

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