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Preliminary Study of Production Electrolytic Silicon from Silica Amorphous in Alkaline Media

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ABSTRACT

This study used direct electrowinning to examine the viability of producing silicon from amorphous silica in alkaline conditions. With low-cost electrodeposition at room temperature. A new source of SiO_2 *from geothermal sludge. Before electrolysis, the samples were analyzed using various analytical techniques (XRF and XRD) to ascertain their chemical composition and mineralogy. At low temperatures (30°C), the direct electrowinning method using amorphous silica suspended in sodium hydroxide (NaOH) was introduced. Current density adjustments were made to determine the direct electrowinning method's ideal operation. XRD and XRF analyses were performed on cathode deposits and residues in electrochemical cells to ascertain the chemical composition and current recovery. At a current density of 1250* A/m^2*, the low-temperature electrolysis current efficiency on sodium silicate solution can approach 34% recovery. Geothermal sludge was used to investigate the process, and the problematic adsorption of amorphous silica particles on the cathode caused the process's mediocre performance.*

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1. Introduction

Indonesia's high fossil energy consumption demand increases the yearly carbon dioxide (CO_2) produced. Based on the 2021 climate transparency data. Total CO_2 emissions across sectors have exceeded 500 metric tons of CO_2/year, and it is projected that in 2020 2030, it will increase to 1800 metric tons of CO_2/year. However, it is targeted that by 2030, Indonesia will emit 400 metric tons of CO_2/per year. Energy is the most dominating sector in producing carbon dioxide gas emissions. The energy composition within Indonesia's power generation framework exhibits a pronounced reliance on conventional fossil fuels, constituting approximately 75% of the total energy output. While these conventional energy sources have historically dominated the energy landscape, their utilization presents inherent challenges attributed to environmental repercussions and finite availability. In contrast, the renewable energy segment has manifested noteworthy advancement, contributing substantively with an approximate share of 20% in the overall energy matrix. Geothermal energy is prominent among renewable energy sources, accounting for approximately 11% of the aggregate energy production. This particular energy source capitalizes on the Earth's internal heat reservoirs, thus offering an environmentally sustainable and perennial energy supply. Furthermore, the biomass sector contributes approximately 7.8% to the energy mix, signifying the productive exploitation of organic matter for energy generation. This facet aligns adeptly with Indonesia's agrarian endowments, rendering biomass a promising avenue for sustainable energy sourcing. The renewable energy sector's discernible attribute of yielding a 20% reduction in carbon emissions relative to fossil fuels is of paramount significance. This reduction underscores a substantial stride towards attenuating the ecological impact and advancing the overarching agenda of environmental stewardship. The prevailing energy confluence underscores Indonesia's dual pursuit of energy diversification and the amelioration of carbon emissions. This nuanced trajectory reflects the nation's concerted endeavor to navigate the intricate interplay between energy demand, environmental imperatives, and sustainable development compared to fossil fuels. Therefore, to support the reduction of CO_2, increasing the renewable energy sector is necessary [1], [2].

Indonesia, located in the Ring of Fire, has a geothermal energy potential of about 28.91 GW, although less than 5% of these resources have been used thus far. Unfortunately, it can be shown in Fig. 1. that Indonesia's geothermal power plant electricity output has only increased gradually thus far. The Indonesian government aims to use geothermal power plants more frequently. The effectiveness of energy generation depends on the geothermal fluid's quality; such fluid is only sometimes compatible with current technologies. When geothermal fluid is acidic, scales can form because of the high concentration of dissolved minerals, as seen in Fig. 2., which happened in Dieng's geothermal power plant and frequently interrupts electricity production. A severe issue with geothermal power plants using a binary cycle system is silica scaling. The brine from the separator in the wet geothermal fluid may have large quantities of silica under oversaturated conditions [3]. The silica in the brine tends to create scales on the surfaces of downstream equipment without any scaling prevention measures [4]. Due to the high silica content and high productivity in the facility to create silicon, silica scaling from the geothermal plant in Dieng, Indonesia, can be used as an amorphous silica source [5].

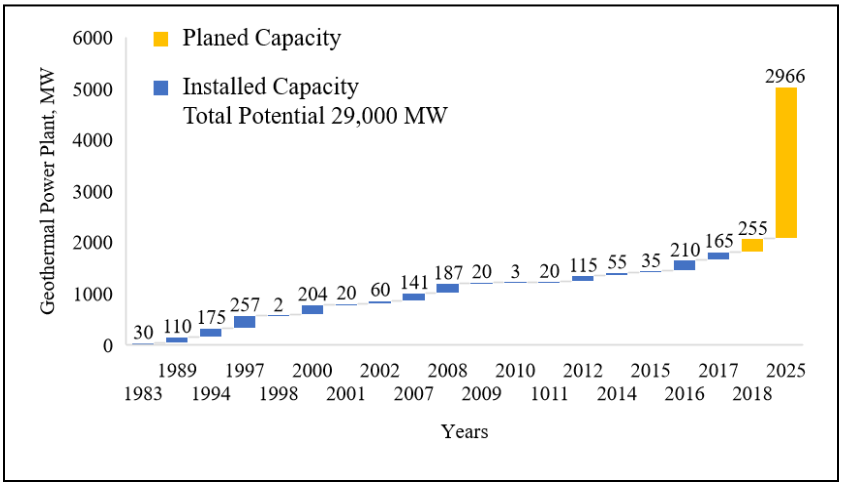

Fig. 1. Generation from Indonesian geothermal power plants: past, present, and future [6]

Fig. 2. After applying acidification to avoid silica scaling, a pipe at the Dieng Geothermal Power Plant became clogged.

A raw material with a high silica content is geothermal sludge (85-95%) [7]–[9], so the silica in geothermal sludge has great potential for further processing. Molten electrolysis-based processes for the electrodeposition of iron have emerged as an alternative route for directly converting suspended silica dioxides to metallic silicon (Si) without greenhouse gas emissions, replacing the traditional method of silicon production in electric furnaces [10]–[19]. Based on this report, a new method for obtaining silicon from geothermal sludge has been proposed in this study. The silica in the geothermal sludge is reduced to silicon by an electrochemical process. Treatment conditions, such as current density, have been investigated in this study. Hopefully, this research can improve the beneficiation of geothermal sludge originating from geothermal sludge in Indonesia with a high silica composition and apply the knowledge gained from research on silica electrolytic reduction to more intricate geothermal sludge beneficiation scenarios.

2. Research Methodology

2.1. Materials

A geothermal sludge sample was obtained from Dieng, Central Java, Indonesia. The sample as used 100 mesh was used for screening the samples. The chemical analysis of the samples used in this study is shown in Table 1. The chemical study revealed that the primary component of geothermal sludge is SiO_2. Furthermore, mineralogical characterization was conducted by X-ray diffraction (XRD), and the pattern is shown in Fig. 3. As can be seen, Amorphous silica is the classification for geothermal silica. It was present as a series of 20° to 25°-degree firm diffraction peaks in the XRD investigation. Additionally, the solid phase of the silica structure from geothermal power plants is amorphous. A ratio of SiO_2/NaOH solution ton obatain a solid liquid ratiof of 1:1 g/mL. Sodium hydroxide pellets (98% purity, Emsure Merck, Germany) were dissolved in deionized water to create the NaOH solution. The 37% hydrochloric acid (Emsure Merck, Germany) was used to create the one mol/L hydrochloric acid solution to clean the electrodes.

Table 1. XRF of Geothermal Sludge

Metal Oxides	Percentage (%)
SiO_2	96.179
P_2O_5	0.823
Fe_2O_3	1.316
SO_3	0.742
CaO	0.491
Other	0.449

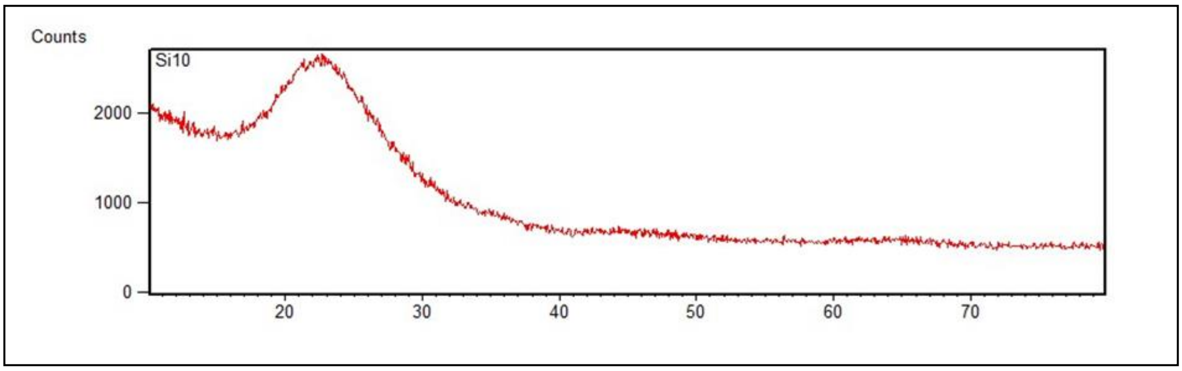

Fig. 3.X-ray diffraction of silica sludge

2.2. Cell and electrodes

The procedure is carried out in an electrolysis cell of a 300 mL borosilicate glass beaker (70 mm diameter, 80 mm height). The experiment used 250 mL of 3.3 mol/L NaOH solution and 40 g of SiO_2 added to the cell. In Fig. 4, the experimental setup is depicted.

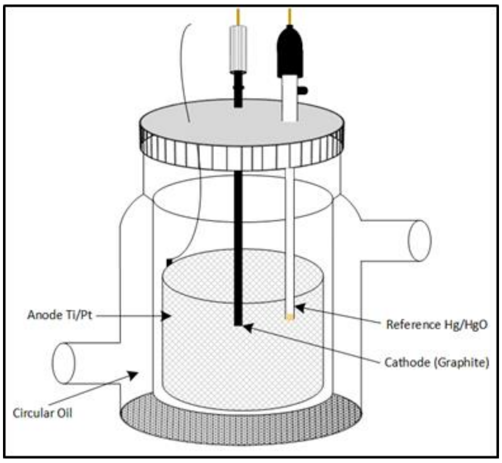

Fig. 4. Electrochemical cell

2.3. Estimating the faradaic yield

Using Faraday's law [20]–[22], it is possible to determine the theoretical mass of the metal mass Si, assuming that silicon is deposited using all of the provided electrons:

$$m_{si,theoritical} = \frac{Mr_{Si}.I.\Delta t}{n.F} \quad (1)$$

Where I is the current (A), Mr. Si is the silicon's molar weight of the metal (28.08 g mol^{-1}), Δt is the experiment's runtime, n is the number of electrons involved, and F is Faraday constant ($C.mol^{-1}$). By dividing the mass of the deposit, the faradaic yield was calculated. To determine deposit mass, the working electrode was weighed before the experiment, and when the electrolysis was complete, the cathode was dried at 100 °C. The electrode was considered a final weight after 12 hours, and the deposited mass was determined using the mass difference between the last and initial weights of the experiment.

$$Faradaic\ yield\ (\%) = \frac{Deposit\ mass}{m_{si.theoritical}} x100 \quad (2)$$

3. Results and Discussion

3.1. Cyclic Voltammetry

The dependency of the electrochemical window of electrolytes on working electrode material has already been reported [23]–[25]. It has been proven that noble metal electrodes, such as gold and platinum, exhibit low electrochemical windows compared to other electrodes, such as glassy carbon electrodes in this ionic liquid [26]. The platinum wire (1 cm^2) was used for the cyclic voltammetry measurements, which were done in a 3.3 mol/L NaOH solution at 30 °C with a ten mV/s scan rate. The result is that voltammetric curves are presented in Fig. 5. Cyclic voltammetry was used to determine the mechanism for several reactions in the sodium silicate electrolysis process.

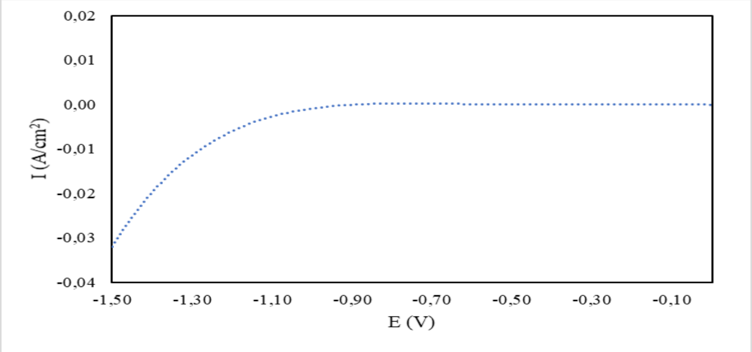

Fig. 5. Cyclic voltammetry in sodium silicate with 3.3 mol/L NaOH solution at 30 °C

Silica oxide from sodium silicate is reduced to metallic silicon in the region of cathodic potentials from –0.8 V to –1 V with a peak at –0.9 V. More Evolution Hydrogen will evolve at negative potentials lower than –0.9 V. The peak observed at cathodic potentials between –0.8 V and –1.2 V is attributed to the reduction of Si4+ to Si, which always occurs in the system under study. The peak observed at anodic scanning at about –0.4 V is attributed to the re-oxidation of silica. From several experiments, we have observed that the present porous structure of Si can be deposited using the galvanostatic method at a very low over the potential of -0.9V (vs Pt).

3.2. Effect of current density on the electrolysis of sodium silicate

Current density can be defined as the rate of charge flow across any electrode cross-section. Considering that it controls the production rate and depends on the faradaic yield, current density (cd) is crucial when evaluating electrochemical processes. Variation-applied currents were used in Galvanostatic mode experiments at 625 A/m^2, 937.5 A/m^2, and 1250 A/m^2 to determine the optimal current density. This variation was performed constantly (temperature 30 °C with a solid/ liquid ratio of 1/1 g/mL). As shown in Fig. 6., the current density level affects the faradaic yield of the sodium silicate electrolysis process. The faradaic efficiency at a very low current density (625 A/m^2) was discovered to be 8.0%. Efficiency rose to 14% when the current density was 937.5 A/m^2. On the other hand, the faradaic yield dropped to 34% at 1250 A/m^2 for a higher current density.

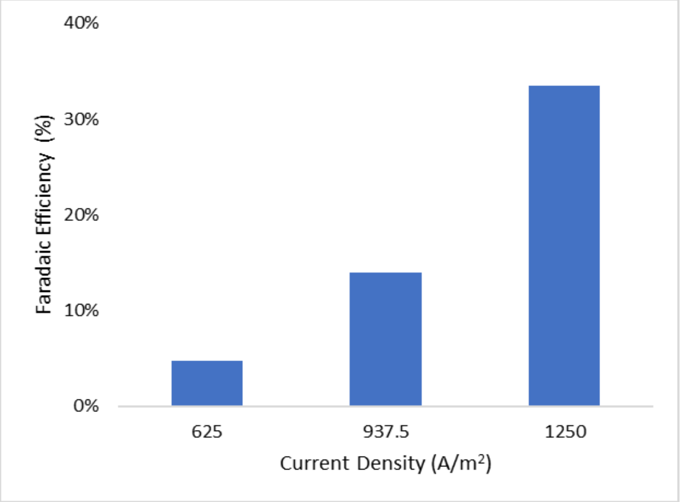

Fig. 6. Effect of the current density on the faradaic yield in the electrolysis of sodium silicate at 3.3 mol/L NaOH and 1/1 g/mL of solid/liquid for 2 hours at 30 °C

Silicon production from the electrolytic treatment of sodium silicate is poorly efficient. At a high current density, unwanted cathodic reactions, such as the hydrogen evolution reaction, occur in the system. The production of gas evolution will increase with the increased current density, causing Almost no gas bubbles to be brought to the surface. Due to a gas phase in the electrolyte solution, a three-phase foam presumably increasingly stabilized by the suspension's increased viscosity develops, resulting in decreased electrical conductivity [24]. Additionally, a process with deposited sodium silicate particles at the cathode—electroactive silicon particles coat the cathode—can explain the low Faraday efficiency.

4. Conclusion

We have demonstrated the electrochemical reduction of sodium silicate in concentrated NaOH electrolytes to produce silicon. First, various techniques (XRF and XRD) were used to evaluate samples of amorphous silica to ascertain their chemical and mineralogical contents and particle size distribution. After acid washing, the silica mainly included amorphous silica. It investigated how current density affected the maximum potential and optimal faradaic yield. The most effective faradaic outcome, using sodium silicate solution, was 34% at 1250 A/m^2. These laboratory-scale studies show that direct electrolysis of sodium silicate is viable and offers a potential method for producing iron from this naturally occurring mineral in a manner that emits no carbon dioxide.

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