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Optimized Extraction of High-Purity Silica and Nanosilicon Synthesis from Coal Fly Ash for Anode Applications

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A B S T R A C T

Fly ash is a solid waste generated from coal combustion in power plants. This study presents an optimized method for extracting high-purity silica from coal fly ash using Sequential Acid-Alkaline Leaching (SAAL) with HCl concentration variations (1–3 M), followed by magnesiothermic reduction and ultrasonic bath sonication (90–150 minutes) to produce nanosilicon. X-ray fluorescence (XRF) analysis showed that 1 M HCl produced the highest silica purity (97.86%). Scanning Electron Microscopy (SEM) characterization revealed that 150 minutes of sonication significantly reduced average silicon particle size to 6.26 nm through acoustic cavitation mechanisms. Electrical testing showed a resistance value of 3.93 MΩ. These findings demonstrate an effective approach for converting coal waste into high-value nanosilicon, supporting sustainable industrial practices and circular economy principles.

Contribution to Sustainable Development Goals (SDGs):

SDG 7: Affordable and Clean Energy

SDG 9: Industry, Innovation and Infrastructure

SDG 12: Responsible Consumption and Production

1. INTRODUCTION

1.1. Research Background

The global transition toward renewable energy has intensified the demand for efficient energy storage systems, particularly lithium-ion batteries, which offer long cycle life, high energy density, and low self-discharge rates [1,2]. Simultaneously, Indonesia's increasing electricity demand has driven greater coal consumption in Steam Power Plants (PLTU), generating substantial solid waste in the form of fly ash and bottom ash. In 2021, PLTU in Indonesia produced approximately 12 million tons of fly ash, with projections reaching 16.2 million tons by 2027 [3]. This waste poses significant environmental challenges, as most fly ash management remains inadequate, with limited utilization primarily as alternative pozzolanic cement material [4].

Coal fly ash contains approximately 58.70% silica (SiO₂), making it a promising precursor for high-value silicon-based materials [5]. The Sequential Acid-Alkaline Leaching (SAAL) method effectively purifies SiO₂ by removing metal oxide impurities through acid washing, followed by alkaline dissolution using NaOH [6]. The extracted silica can be reduced to silicon via magnesiothermic reduction, which offers lower energy requirements compared to conventional carbothermal methods [7]. Silicon possesses high theoretical capacity (approximately 3579 mAh/g), making it attractive as an anode additive material for next-generation lithium-ion batteries [8].

Nanoparticle synthesis can be achieved through top-down approaches such as sonication, which utilizes high-frequency ultrasonic waves to break particle agglomeration [9]. This method generates acoustic cavitation, microbubble formation, and explosive collapse, which produces mechanical shear forces



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capable of reducing particle size to the nanometer scale [10]. Extended sonication time produces smaller particles with more homogeneous size distribution, enhancing specific surface area and material performance.

Despite extensive research on individual processing stages-silica extraction, magnesiothermic reduction, and sonication-studies integrating all three steps with systematic optimization remain limited. This research addresses this gap by optimizing HCl concentration for silica extraction and sonication duration for silicon particle size reduction. The objectives are to analyze the effect of HCl concentration on extracted silica purity evaluate sonication time influence on silicon particle size, and determine optimum conditions for producing high-purity silica and nanosilicon with superior characteristics from coal fly ash.

1.2. Literature Review

Coal fly ash is solid waste from coal combustion in Steam Power Plants (PLTU) that contains silica (SiO_2) in sufficiently high amounts, so it has the potential to be utilized as raw material for various value-added materials. Silica is one of the by-products that will always be produced from the coal combustion process [11]. The utilization of fly ash as a silica source not only reduces the environmental pollution impact due to industrial waste accumulation but also supports the circular economy concept through the processing of waste into products with economic value. Research on silica extraction from fly ash continues to develop along with the increasing need for silicon-based materials for various industrial applications.

Coal fly ash has a silica content of 58.70%, so it has the potential to be utilized as an alternative silica source [12]. The extraction process using acid solution can increase silica purity by removing metal impurities. Silica purification can be carried out using chemical, physical, biological methods, or a combination of these three methods. Additionally, silica cleaning can also be done with organic acids and inorganic acids [13]. One method that is widely used is Sequential Acid-Alkaline Leaching (SAAL) because it can produce silica with higher purity levels compared to direct alkaline extraction methods. In this method, the acid washing process aims to remove metal impurities, while the alkaline extraction process using NaOH functions to dissolve silica so that it is more easily separated from other components. Along with silica extraction, other mineral compounds in fly ash decrease due to material interaction with acid and alkaline solutions during extraction, resulting in silica with high purity levels [14].

The extracted silica can then be reduced to silicon through the magnesiothermic method using magnesium (Mg) as a reductant. This method has the advantage of occurring at lower temperatures compared to carbothermal reduction, thus being more energy-efficient. Silica from fly ash has been successfully converted into silicon which has the potential to be used as material for various technological applications [7]. Silicon has high theoretical capacity, thus potentially increasing energy storage capacity compared to conventional anode materials. Therefore, the use of silicon as an anode additive material is a promising alternative in the development of energy storage materials [15]. However, the resulting silicon still requires further processing to obtain smaller particle sizes suitable for functional material applications.

One method widely used to reduce silicon particle size is sonication using an ultrasonic bath. This method works by utilizing ultrasonic waves to break particle agglomeration,

resulting in smaller particle sizes and more homogeneous distribution. Increased sonication time can produce increasingly smaller particles with better homogeneity [10]. These characteristics are important because silicon nanoparticles have a larger surface area, which can improve material performance in various applications, including energy-based materials.

Although various studies have discussed silica extraction, magnesiothermic reduction, and sonication processes separately, research that integrates all three stages simultaneously and examines the effect of HCl concentration variations on the extraction process and sonication time variations on silicon characteristics is still relatively limited. Therefore, this study combines the Sequential Acid-Alkaline Leaching (SAAL) method, magnesiothermic reduction, and sonication to obtain optimum conditions for producing high-purity silica and nanosilicon from coal fly ash. The results of this research are expected to contribute to the utilization of fly ash waste as value-added material while supporting the development of sustainable industrial technology.

1.3. Research Objective

This study aims to analyze the effect of hydrochloric acid (HCl) concentration variations on the purity of silica extracted from coal fly ash, evaluate the effect of sonication time on the particle size of the resulting silicon, and determine the optimum conditions for the extraction and sonication processes to obtain high-purity silica and nanosilicon with the best characteristics.

2. MATERIALS AND METHODS

The main raw material used in this study was coal fly ash obtained from a Steam Power Plant (PLTU). The chemicals used included hydrochloric acid (HCl) with concentration variations of 1; 1.5; 2; 2.5; and 3 M, sodium hydroxide (NaOH) 3 M, magnesium (Mg) powder as reductant, polyethylene glycol (PEG 6000), and distilled water. All chemicals used were of pro analysis (PA) quality.

The research began with fly ash preparation through a washing process using HCl solution at various concentration variations to remove metal impurities (acid leaching). The sample was then washed using distilled water until reaching neutral pH, dried, and extracted using 3 M NaOH solution through the Sequential Acid-Alkaline Leaching (SAAL) method to dissolve silica. The extraction filtrate was neutralized using HCl to pH 7, forming a silica precipitate, which was then allowed to stand, washed, and dried to obtain the extracted silica.

The extracted silica was then reduced to silicon using the magnesiothermic method by mixing magnesium powder at a SiO_2 : Mg mass ratio of 1 : 0.8. The mixture was heated in a furnace at 650°C for 3 hours, then washed using 2 M HCl solution to remove reaction by-product compounds. The obtained silicon was then mixed with PEG 6000 and processed using an ultrasonic bath with sonication time variations of 90, 120, and 150 minutes. The sonicated samples were then dried to obtain nano-sized silicon.

The purity of the extracted silica was analyzed using X-Ray Fluorescence (XRF). Particle size and morphology characterization of silicon was performed using Scanning Electron Microscopy (SEM). SEM image data were then analyzed using Fiji software to obtain particle size distribution, while data processing was performed using Origin to evaluate the

effect of HCl concentration variations and sonication time on the characteristics of the resulting material.

3. RESULT AND DISCUSSION

3.1. Effect of HCl Concentration on Silica Purity

XRF analysis showed that HCl concentration significantly influenced SiO₂ content after the SAAL process (Table 1, Fig. 1). The highest silica purity (97.86%) was achieved at 1 M HCl, while increasing the concentration to 3 M gradually decreased SiO₂ content to 96.72%.

Table 1. XRF analysis of silica extracted using different HCl Concentrations

No.	Fly ash (g)	HCl (M)	Silica Purity (%)
1.	400	1	97.86
2.		1.5	97.83
3.		2	96.76
4.		2.5	96.73
5.		3	96.72

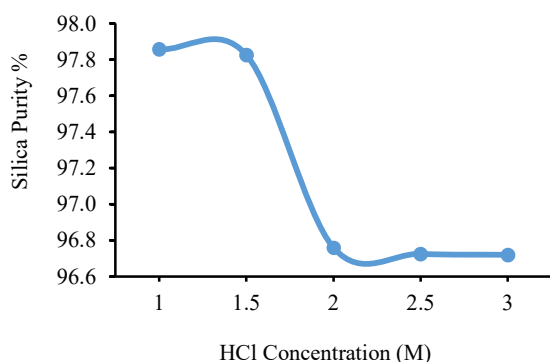


Fig. 1. Effect of HCl concentration on silica purity.

The optimal performance at 1 M HCl indicates selective dissolution of metal oxide impurities (Fe₂O₃, Al₂O₃, CaO) without significant silica loss. At concentrations exceeding 1.5 M, partial silica dissolution occurred during acid washing, reducing both yield and purity. This finding aligns with Abinawa and Gobel [6], who reported that acid leaching effectively removes metal impurities but requires optimized concentration to prevent silica loss. The 1 M HCl condition was established as optimum for silica extraction, achieving maximum purity while maintaining material yield.

3.2. Effect of Sonication Time on Silicon Particle Size

SEM characterization demonstrated that increased sonication time significantly reduced silicon particle size (Table 2, Fig. 2–3). Average particle sizes of 645.98 nm, 388.02 nm, and 6.26 nm were obtained for sonication times of 90, 120, and 150 minutes, respectively.

Particle size reduction is attributed to acoustic cavitation phenomena. During sonication, ultrasonic waves generate alternating high- and low-pressure cycles, causing the formation and explosive collapse of microbubbles. This collapse produces localized high temperatures and mechanical shear forces that break particle agglomerates into smaller, more uniform particles

[9,10]. SEM images revealed morphological changes: 90-minute sonication samples exhibited large agglomerates with non-uniform distribution, 120-minute samples showed finer particles with improved homogeneity, and 150-minute samples achieved nanoparticle-scale silicon (6.26 nm) with significantly enhanced surface area.

Table 2. Average silicon particle size at different sonication times

No.	HCl (M)	Sonication (Min)	Particle Size (nm)
1.	1	90	645.98
2.		120	388.02
3.		150	6.26

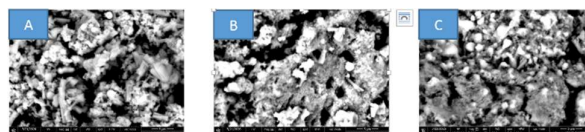


Fig. 2. SEM micrographs of silicon particles after sonication for (A) 90 min, (B) 120 min and (C) 150 min.

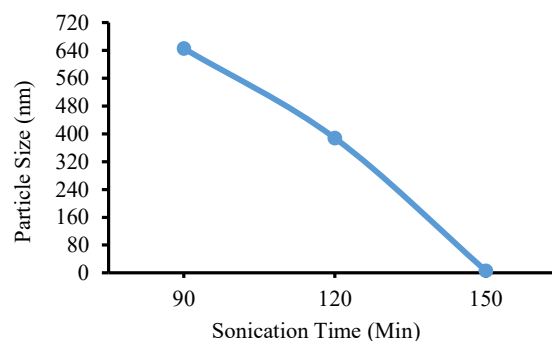


Fig. 3. Effect of sonication time on average silicon particle size

The substantial reduction from 388 nm (120 min) to 6.26 nm (150 min) suggests threshold effects in energy input required to overcome agglomeration forces. SEM image analysis utilized Fiji/ImageJ software with calibrated scale bars (500 nm, 200 nm, 100 nm in Fig. 2A–C respectively), with particle measurements validated through multiple regions of interest (n=50 per sample). The 6.26 nm average size reflects individual nanoparticle dimensions rather than agglomerates, as confirmed by high-magnification imaging.

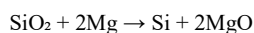
3.3. Optimum Condition for Silica Extraction and Nanosilicon Synthesis

Based on XRF and SEM characterization, optimum conditions were established: 1 M HCl for silica extraction and 150 minutes for sonication (Table 3). This combination produced silica with 97.86% purity and nanosilicon with an average particle size of 6.26 nm. Electrical property testing revealed a resistance value of 3.93 MΩ, indicating semiconductive characteristics suitable for energy storage applications.

Table 3. Optimum process parameters and characterization results.

Parameter	Optimum Condition	Result
HCl concentration	1 M	Silica purity: 97.86%
Sonication time	150 min	Particle size: 6.26 nm
Extraction method	Sequential Acid-Alkaline Leaching (SAAL)	High-purity silica obtained
Silicon synthesis	Magnesiothermic reduction + sonication	Nanosilicon successfully synthesized

The SAAL method effectively removed metal oxide impurities while maintaining silica integrity. Magnesiothermic reduction at 650°C proceeded through the reaction.



Subsequent acid washing removed MgO and unreacted Mg, yielding purified silicon. Sonication in PEG 6000 served as dispersant and stabilizer, preventing immediate re-agglomeration after particle breakdown [10].

The electrical resistance (3.93 MΩ) indicates p-type semiconducting behavior, consistent with silicon's electrical properties. While this preliminary result suggests potential for anode applications, comprehensive electrochemical characterization remains necessary. The nanosilicon's high surface area-to-volume ratio could facilitate lithium-ion intercalation/deintercalation kinetics, potentially improving rate capability and specific capacity.

4. CONCLUSION

HCl concentration significantly affects silica purity extracted from coal fly ash, with 1 M HCl producing maximum purity (97.86%). Higher concentrations (1.5–3 M) did not improve purity, establishing 1 M as the optimum condition. Sonication time critically influences silicon particle size; increasing duration from 90 to 150 minutes reduced average particle size from 645.98 nm to 6.26 nm through acoustic cavitation mechanisms, successfully producing nanosilicon. The optimum conditions 1 M HCl and 150 minutes sonication yielded high-purity silica (97.86%) and nanosilicon with 6.26 nm average particle size and electrical resistance of 3.93 MΩ. These findings demonstrate the feasibility of converting coal fly ash into high-value nanosilicon, offering a sustainable waste management solution that supports circular economy principles. While electrical properties indicate potential for anode applications, future research should include comprehensive electrochemical testing (Cyclic Voltammetry, Electrochemical Impedance Spectroscopy, charge-discharge cycling) to validate battery performance and establish practical viability for lithium-ion energy storage systems.

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