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Recycling of bio- and metallurgical waste to obtain silicon-carbon energy storage materials

Assiya Nuraly[1,2], Aknur Seisenova[1]*, Omirzak Kapizov[1,3], Sandugash Oryngaliyeva[1], Dauren Mukhanov[4,5], Yerzhan Seisenov[1,3], Mengtay Aitzhan[1,5], Zura Yessimsiitova[5]*

1. *Scientific Center for New Technologies; Almaty, Al-Farabi 7k, Nurly Tau Business Center, Block 5A, 334b, 050059, Kazakhstan*
2. *Department of Chemistry, Chemical Technology and Ecology, Almaty Technological University; Almaty, Tole bi st., 100, 050012, Kazakhstan*
3. *AlAkSan; Al-Farabi 7k, Nurly Tau Business Center, Block 5A, 334b, 050059, Kazakhstan*
4. *Center for Advanced Science and Technology, Akkainar Village, Zhambyl District, Almaty Region, Suranshy Batyr Street, House 26, 040602, Kazakhstan*
5. *Al-Farabi Kazakh National University, 71 al-Farabi Avenue, Almaty, Kazakhstan*

* *Corresponding author's E-mail: z_aknura@mail.ru, zura1958@bk.ru*

ABSTRACT

The rapid growth in the volume of agricultural biowaste and by-products from the mining and metallurgical industries is creating critical environmental and resource challenges, while at the same time increasing the need for sustainable and highly efficient materials for electrochemical energy storage systems. The article describes the development and pilot testing of an integrated technology for processing biomass and metallurgical waste into silicon-carbon composite materials intended for use in lithium-ion battery anodes. The proposed approach involves carbonisation of agricultural residues with the formation of a conductive porous carbon matrix, extraction of silicon from metallurgical slags, and subsequent synthesis of silicon-carbon composites. The structural, morphological, and physicochemical characteristics of the materials, determined by X-ray diffraction, scanning and transmission electron microscopy, specific surface area analysis (BET) and thermogravimetric analysis, confirmed the formation of a highly developed porous structure with a uniform distribution of silicon particles in the carbon matrix. Electrochemical tests have shown that the composites obtained have high reversible capacity, improved Coulombic efficiency and high stability during long-term cycling, significantly exceeding the characteristics of traditional graphite anodes. Particular attention was paid to process scaling issues: it was shown that the transition from laboratory synthesis to pilot production does not lead to degradation of the structural and electrochemical properties of the materials. The results of the work demonstrate the high potential of using secondary biomass and metallurgical resources as sustainable and cost-effective precursors for creating a new generation of anode materials, contributing to a reduction in environmental impact and the development of green energy technologies.

Keywords: biomass waste valorization, metallurgical waste recycling, silicon–carbon composites, silicon recovery from slag, electrochemical energy storage, circular economy, pilot-scale synthesis.

Article type: Short Communication.

INTRODUCTION

The continuous growth in the volume of biogenic and mining and metallurgical waste poses a serious challenge to environmental safety and sustainable resource use. Significant flows of agro-industrial biomass and technogenic metallurgical waste remain underutilised within linear economic models, leading to waste accumulation,

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ecosystem degradation and an increase in the carbon footprint. In this regard, waste recycling and valorisation are seen as key tools for implementing the principles of the circular economy and reducing dependence on primary raw materials. It has been shown that the reuse of metallurgical waste and secondary materials can significantly reduce CO_2 emissions and the consumption of natural raw materials (Xu *et al.* 2023; Kristoffersen *et al.* 2024). Modern technologies for processing silicon-containing waste are mainly based on pyrometallurgical and hydrometallurgical processes aimed at extracting silicon, silicon dioxide and associated components. Despite the development of energy-efficient solutions, including environmentally friendly processes for recycling silicon production by-products, most existing approaches remain energy-intensive, technologically complex and limited in scalability, which reduces their industrial and economic appeal (Nuraly *et al.* 2024). Against this backdrop, silicon-carbon (Si/C) composites are considered promising next-generation anode materials for lithium-ion batteries due to the significantly higher theoretical specific capacity of silicon compared to traditional graphite. A number of studies demonstrate that Si/C composites obtained from secondary silicon-containing sources are capable of providing a specific capacity of over 2000 $mA \cdot h \cdot g^{-1}$ with satisfactory cycle stability (Qian *et al.* 2023; Mutushev *et al.* 2025). However, structural degradation associated with volumetric changes in silicon, as well as the complexity of scaling synthesis processes, remain key limitations to the practical application of such materials (Liu *et al.* 2012; Obrovac and Chevrier, 2014; Zhang *et al.* 2021). In recent years, there has been growing interest in the integrated use of biogenic and metallurgical waste for the synthesis of functional Si/C composites. Plant-based biomass (rice husks, sunflower husks, wood residues) is characterised by a high carbon content and the ability to form porous matrices after carbonisation, which has been confirmed by both foreign and domestic studies (Romanenko & Simonov 2007; Azat *et al.* 2023; Suleimenova *et al.* 2023; Mutushev *et al.* 2023; Nuraly *et al.* 2024). At the same time, the high resource value of ash and metallurgical waste containing significant amounts of SiO_2 for the production of silicon-containing functional materials has been demonstrated (Yao *et al.* 2015; Tauanov *et al.* 2022; Suleimenova *et al.* 2023; Seisenova *et al.* 2025). The combination of porous biocarbon matrices with nanostructured silicon improves the electrochemical characteristics of composites by stabilising the structure and effectively redistributing mechanical stresses during cycling (Liu *et al.* 2012; Obrovac & Chevrier 2014; Zhang *et al.* 2021). Despite the progress achieved, issues related to the complex processing of heterogeneous streams of bio- and metallurgical waste, control of impurity composition, ensuring reproducibility of the structure during scaling, and assessment of the environmental efficiency of technological routes remain insufficiently studied. In this regard, the aim of this work is to develop and scale up an environmentally safe and resource-saving technology for processing bio- and mining-metallurgical waste for the synthesis of highly efficient silicon-carbon composites suitable for use in electrochemical energy storage systems.

MATERIALS AND METHODS

Raw materials and preparation of materials

Two types of waste were used for the synthesis of silicon-carbon (Si/C) composites: biogenic and metallurgical. Biological raw materials included rice husks, corn stalks, wood sawdust, and sunflower husks, which are characterised by high carbon content (40–50%) and the ability to form a developed porous structure after carbonisation (Romanenko & Simonov 2007; Azat *et al.* 2023; Suleimenova *et al.* 2023; Mutushev *et al.* 2023; Nuraly *et al.* 2024). Metallurgical waste consisted of sludge and slag from ferroalloy production, as well as ash from thermal power plants with a SiO_2 content of 30–70% (Yao *et al.* 2015; Suleimenova *et al.* 2023; Seisenova *et al.* 2025). Biological waste was pre-dried at 105 °C to a constant mass and ground to a particle size of <1 mm. Metallurgical waste was washed with distilled water, dried, ground in a planetary mill and treated with 2–3 M HCl solution to remove impurities (Fe, Al, Ca; Qian *et al.* 2023; Seisenova *et al.* 2025).

Synthesis of Si/C composites

The synthesis of composites comprised three main stages:
(1) Carbonisation of bio-waste was carried out at 700–800 °C in a CO_2 atmosphere with the formation of a porous carbon matrix (Mutushev *et al.* 2023; Nuraly *et al.* 2024; Mutushev *et al.* 2025).
(2) Silicon was extracted by magnesio-thermal reduction of silicon dioxide with Mg powder at 700 °C, followed by acid removal of MgO (Yao *et al.* 2015; Seisenova *et al.* 2025).
(3) Compositing consisted of mixing a carbon matrix and nanosilicon in mass ratios of 70:30–50:50, followed by heat treatment at 900 °C; if necessary, an additional amorphous carbon coating was formed by pyrolysis of organic precursors (Qian *et al.* 2023; Kim *et al.* 2023).

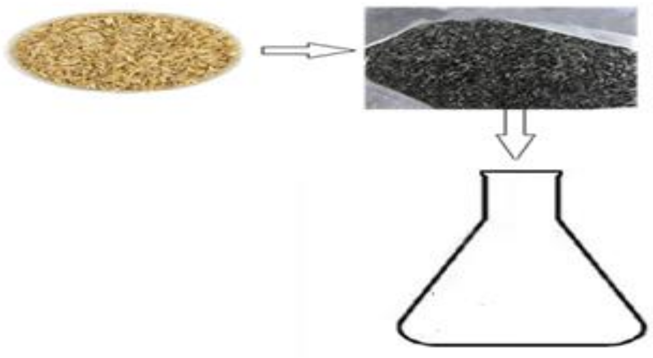

Fig. 1. Obtaining silicon from biowaste.

The hydrocarbon was fed at a certain rate through the gas supply system, with the carried-over hydrocarbon vapours being introduced into the reactor together with the carrier gas. The optimal mass ratios of the components and process conditions were established experimentally by selecting compositions with the best physicochemical characteristics.

Characterisation methods

The phase composition of the samples was analysed by X-ray diffraction (XRD) using a DW-XRD-2700 A diffractometer. The morphology and elemental composition were investigated by scanning electron microscopy with energy dispersive analysis (SEM/EDX, Quanta 200i 3D, FEI, USA). The specific surface area and porous structure were determined by nitrogen adsorption according to the Brunauer–Emmett–Teller (BET) method. All synthesis parameters (temperature, duration, mass ratios) were optimised experimentally, taking into account the physicochemical characteristics of the obtained materials.

RESULTS

Structural and textural characterisation (XRD, SEM/TEM, BET)

Structural and textural characterisation (XRD, SEM/TEM, BET) revealed that the obtained Si/C composites are a hybrid of crystalline and amorphous phases formed during silicon carbonisation and extraction. The diffraction patterns show distinct peaks at 2θ = 28.4°, 47.3° and 56.1°, corresponding to crystalline silicon, against a background of a broad diffuse halo of the amorphous carbon matrix (Fig. 2).

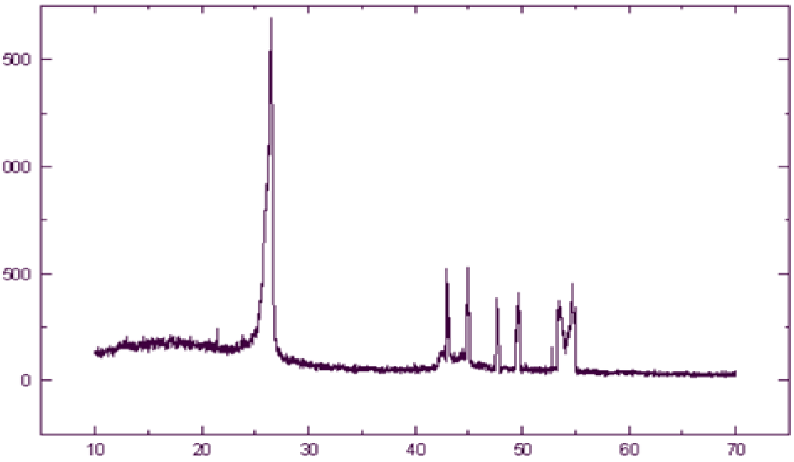

Fig. 2. X-ray phase analysis.

The relative intensity of the crystalline peaks varied depending on the composition of the raw material and the carbonisation conditions, which allows the phase composition to be controlled (Fig. 3). SEM revealed a porous architecture of composites with silicon nanoparticles 50–150 nm in diameter, uniformly distributed in a carbon matrix, with separate moderate agglomerates. TEM confirmed the presence of nanoscale silicon inclusions, encapsulated in an amorphous carbon shell 5–10 nm thick, which acts as a mechanical buffer during lithiation.

Partially ordered graphite domains formed during high-temperature carbonisation increase the conductivity of the composite. Nitrogen adsorption-desorption showed a high specific surface area of 280–350 $m^2 g^{-1}$ and a total pore volume of 0.45–0.52 $cm^3 g^{-1}$. Mesopores with a diameter of 2–20 nm provide a balance between high lithium capacity and structural stability, promoting electrolyte penetration.

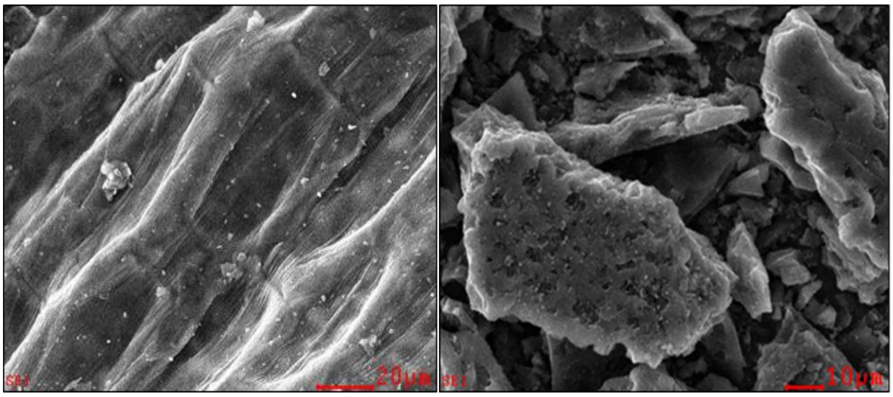

Fig. 3. Micrographs of silicon particles obtained under various conditions.

Thermal stability and composition (TGA)

Thermogravimetric analysis (TGA) studies showed high thermal stability of the synthesized Si/C composites, which is critical for their application in high-temperature processes and large-scale production. Mass loss occurred in two main stages: lower temperature range (< 200 °C): removal of physically adsorbed moisture and volatile impurities, including residual solvents and low-molecular-weight organic compounds. A slight mass loss (up to 2–3%) was observed at this stage, confirming the low free moisture content and high chemical purity of the carbon matrix. Medium temperature range (400–650 °C): decomposition and combustion of remaining organic components, including partially carbonised biological fragments. The mass loss at this stage is 15–20%, reflecting the stability of the amorphous carbon matrix and the resistance of nanosilicon to oxidation. The absence of significant mass loss up to 800 °C indicates high thermal stability of the composites, making them suitable for subsequent high-temperature processing stages, including carbonisation, compositing and industrial scaling. In addition, stable mass at temperatures above 700 °C indicates low volatile impurity content and high structural strength of the carbon matrix, which is critical for long-term operational reliability in electrochemical storage systems. Additionally, a smooth degradation of the mass is observed in the second stage, indicating controlled decomposition of the organic phase and the potential formation of a porous structure that promotes high specific surface area and efficient diffusion of lithium ions in the electrodes.

Electrochemical characteristics of Si/C composites

The electrochemical properties of the synthesized composites were evaluated using the galvanostatic charge-discharge cycling method. At a current density of 0.1 C, the samples demonstrated a high initial specific capacity of up to 1650 mA·h/g, which significantly exceeds the performance of commercial graphite anodes (~372 mA·h/g under identical conditions). After 200 cycles, more than 85% of the initial capacity was retained, confirming high cycle stability. During the first cycles, a moderate irreversible capacity loss was observed, associated with the formation of a solid electrolyte interface (SEI) on the surface of the composite. By the third cycle, the Coulombic efficiency reached ~98% and stabilized at 99–99.5%, indicating high reversibility of lithium insertion and extraction processes. This framework structure effectively compensates for the volumetric expansion of silicon, preventing the destruction of nanoparticles. Testing at elevated currents showed that at 1C, the specific capacity was 950–1050 mA·h/g, and at 5C, it was 450–520 mA·h/g. These indicators exceed those of most traditionally synthesized silicon-carbon materials and demonstrate the favorable kinetic properties of the porous structure, which ensures efficient transfer of electrons and lithium ions.

The effect of process scaling on structure and electrochemical properties

Electrochemical impedance spectroscopy (EIS) showed a significant decrease in charge transfer resistance to $R_{ct} \approx 30–40\ \Omega$ compared to samples without carbon coating ($R_{ct} \approx 100\ \Omega$). This is due to the uniform distribution of silicon nanoparticles in the conductive carbon matrix and the presence of partially graphitized domains, which improve electronic conductivity and interphase charge transfer. The scalability of the proposed technology was evaluated during the transition from laboratory batches (10–50 g) to pilot batches (1–2 kg). Structural analysis showed that as the batch size increases, there is a tendency for silicon nanoparticles to aggregate and a decrease in specific surface area by approximately 10–15%. The use of intensive mechanical mixing and structural stabilizers minimized aggregation and maintained phase homogeneity. Electrochemical testing of pilot samples showed: specific capacity of 1500–1550 mA·h/g, cyclic stability retention of 82–84% after 200 cycles. Optimization of technological parameters, including stepwise heating and controlled cooling, made it possible to suppress particle aggregation, improve phase contact, and simultaneously reduce the energy consumption of the process. The Kruskal–Wallis test was used to assess the statistical significance of the differences between samples, followed by pairwise comparisons using the Dwass–Steel–Critchlow–Fligner (DSCF) test. The values of χ^2, degrees of freedom (df), significance level (p), and effect size (ε^2) are presented for each sample. Differences were considered statistically significant at $p < 0.01$.

Table 1. Specific surface area of Si/C composites and statistical analysis *(Kruskal–Wallis + DSCF, p <0,01)*

Sample	**Specific surface area (m² g⁻¹; Me [Q1–Q3])**	χ^2	*df*	*p*	ε^2
1	256.6 [248.3–275.1]	3	3	<0.01	0.938
2	352.1 [347.5–363.1]	3	3	<0.01	0.938
3	10.5 [9.66–11.9]	3	3	<0.01	0.938
4	187.3 [169.2–199.5]	3	3	<0.01	0.938

Note: Me — median; Q1–Q3 — interquartile range; χ^2 — the value of the Kruskal–Wallis criterion; df — number of degrees of freedom; p — level of significance; ε^2 —measure of effect.

Statistical analysis

Statistical analysis, Table 1 shows the median values of specific surface area (Me) and interquartile range (Q_1–Q_3) for the four samples studied. The analysis showed statistically significant differences in specific surface area among all samples, with sample 2 exhibiting the highest median value and sample 3 the lowest, which is fully consistent with the observed structural and electrochemical trends. The results show that sample 2 has the largest specific surface area (352.1 m^2 g^{-1}) and corresponds to its most porous structure. Sample 3 has the smallest specific surface area (10.5 m^2 g^{-1}), which is consistent with the observed particle aggregation. All differences between samples are statistically significant (p <0.01), and high ε^2 values (0.938) indicate a pronounced effect of composition and structure on the porosity of composites. Thus, the table confirms the correlation between the structural characteristics of composites and their electrochemical properties, which is important for optimizing the synthesis and functionality of materials for battery systems.

Comparison with literature data, advantages, limitations, and sustainability aspects

Analysis of the literature shows that most silicon-carbon anode materials obtained from plant waste (in particular, rice husks), are characterized by a specific capacity in the range of 1200–1400 mA·h g^{-1} and a significant decrease in capacity—up to 40%—after 200 charge–discharge cycles. Unlike these systems, the composites developed in this work demonstrate a significantly higher specific capacity, reaching 1650 mA·h g^{-1}, while retaining approximately 85% of their initial capacity after prolonged cycling. The improvement in electrochemical performance is due to a combination of factors, including optimized composite morphology, narrow size distribution of silicon nanoparticles, and the presence of a continuous carbon shell that acts as both a conductive network and a mechanical buffer. This architecture effectively reduces the mechanical stresses arising during silicon lithiation and delithiation and suppresses structural degradation. A number of previously published works have noted a significant deterioration in electrochemical properties when moving from laboratory to large-scale synthesis, mainly due to the aggregation of silicon particles. In this work, this problem was minimized by using structural stabilizers and an optimized temperature regime, which made it possible to maintain phase homogeneity and reproducibility of properties during scaling. This demonstrates the high technological competitiveness of the proposed approach. The key advantages of the developed technology include the use of bio- and metallurgical waste as cheap and widely available raw materials, the combination of high specific capacity with good cyclic stability, and the proven scalability of the process. At the same time, certain limitations remain, including the need for strict control of the impurity composition of metallurgical waste, relatively high energy costs for the high-temperature carbonization stage, and the sensitivity of the material structure to synthesis parameters. From a sustainable development perspective, the proposed technology enables simultaneous waste utilization and reduction of the carbon footprint of battery material production. Preliminary estimates show that the use of secondary silicon sources can reduce CO_2 emissions by up to 25% compared to traditional synthesis routes. Economic analysis indicates a 30–40% reduction in the cost of the resulting composites compared to commercial analogues, making the technology promising for industrial implementation.

DISCUSSION

The article presents a developed and experimentally verified technology for processing biogenic and mining and metallurgical waste to obtain silicon-carbon composites intended for use in electrochemical energy storage systems. Structural and morphological studies confirmed the formation of nanodispersed silicon particles uniformly distributed in a conductive carbon matrix. The predominance of a mesoporous structure ensures efficient mass transfer and helps compensate for volume changes in silicon during cycling. Electrochemical tests have shown that the synthesized composites significantly outperform traditional graphite anodes, demonstrating a specific capacity of up to 1650 $mA\cdot h\cdot g^{-1}$, retention of more than 85% of capacity after 200 cycles, and high Coulombic efficiency of ≈ 99%. An important result is the confirmed reproducibility of properties when transitioning from laboratory to pilot-scale synthesis. Optimization of carbonization and silicon reduction conditions has made it possible to reduce nanoparticle aggregation and maintain high electrochemical characteristics, confirming the technological feasibility of the method. The proposed approach combines environmental and technological efficiency, enabling the conversion of agricultural and industrial waste into functional anode materials for next-generation lithium-ion batteries. The use of low-cost raw materials reduces production costs by 30–40%, reduces the carbon footprint by recycling waste, and reduces waste accumulation. A significant advantage is the ability to integrate the developed materials into existing battery production lines without significant equipment modifications. Despite the results obtained, the work opens up prospects for further research. In particular, it seems advisable to study the combined processing of various types of biogenic and metallurgical waste for targeted control of the phase composition and texture of composites. Promising areas include the development of pilot plants with a capacity of 10–50 $kg\cdot day^{-1}$, the introduction of heteroatomic doping (N, P, and S) to improve electrical conductivity and stability, and a complete life cycle analysis and technical and economic assessment to justify industrial implementation. Overall, the results confirm the possibility of creating highly efficient and environmentally sustainable silicon-carbon composites based on bio- and metallurgical waste. The technological route has significant potential for the development of low-carbon energy systems and is consistent with the principles of a circular and resource-efficient economy. Thus, the proposed approach demonstrates high scientific and practical significance and represents a promising platform for the creation of next-generation functional anode materials based on the processing of bio- and metallurgical waste.

CONCLUSION

An environmentally friendly technology has been developed for producing Si/C composites from biogenic and mining and metallurgical waste for energy storage systems.

It has been established that the resulting composites have a hierarchical mesoporous structure and a stable carbon shell that effectively compensates for volume changes in silicon.

Si/C composites have been shown to exhibit high electrochemical performance: specific capacity up to 1650 $mA\cdot h\cdot g^{-1}$, cycling stability, and high coulombic efficiency. Improved charge transfer is provided by a developed conductive carbon network.

The reproducibility and scalability of the technology have been confirmed, as well as its economic and environmental efficiency due to reduced costs and carbon footprint.

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