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Environmentally benign recycling of wastes into anode materials for sustainable energy storage

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ABSTRACT

The increasing demand for lithium-ion batteries necessitates the development of environmentally friendly and cost-effective anode materials. This study presents a laboratory-scale technology for recycling cast iron shavings from brake discs — abundant metallurgical waste generated during turning operations — into a high-performance anode material for 18650 cylindrical lithium-ion cells. The waste was processed through mechanical grinding, acid leaching, thermal activation, and electrode fabrication. Electrochemical testing of half-cells demonstrated a reversible specific capacity of 420–510 mAh g^{-1} at a rate of 0.1C, along with excellent cycling stability (capacity retention of 85–92% after 100 cycles). Full 18650 cells assembled with the developed anode exhibited stable operating performance. The proposed approach offers an ecologically sound and economically viable method for converting industrial waste into valuable battery components, thereby supporting the principles of the circular economy in energy storage technologies.

Keywords: Lithium-ion batteries, Anode materials, Bio-waste, Electrochemical performance, Morphology.
Article type: Short Communication.

INTRODUCTION

Lithium-ion batteries remain the dominant energy storage technology for portable electronics, electric vehicles, and renewable energy systems. However, the limited capacity of conventional graphite anodes and the growing environmental concerns associated with primary raw material extraction have stimulated the search for alternative, ecologically sustainable materials. This article presents a comprehensive review and experimental study on the development of advanced anode materials for lithium-ion batteries using secondary raw materials and industrial waste. Particular attention is given to iron-based composites derived from metallurgical waste. The mechanisms of lithium storage in conversion-type anodes, synthesis methods, and strategies for enhancing electrochemical performance are discussed. Experimental results demonstrate that properly processed iron oxide–carbon composites can achieve a reversible capacity exceeding 500 mAh g^{-1} with good cycling stability. The utilization of industrial waste not only reduces battery production costs but also contributes to solving environmental problems related to waste disposal (Liu *et al.* 2017; Assiya Nuraly *et al.* 2024). Lithium-ion accumulators (LIAs) currently represent the leading energy storage technology for portable electronics, electric vehicles, and renewable energy storage systems. Owing to their high specific energy, long cycle life, low self-discharge rate, and absence of memory effect, lithium-ion batteries significantly outperform traditional lead-acid, nickel-cadmium, and nickel-metal hydride batteries (Su *et al.* 2022; Mutushev *et al.* 2025). One effective approach to addressing the limitations

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of graphite anodes is the development of carbon–silicon composites, in which nanodispersed silicon is uniformly distributed within a porous carbon matrix. The carbon matrix acts as a buffer that accommodates volume changes, provides electronic conductivity, and promotes the formation of a stable SEI layer. Particular interest lies in the use of secondary raw materials and agricultural waste for producing such composites. Rice husk, a large-tonnage waste product of the rice-processing industry, is rich in silicon and organic compounds. Its valorization enables the simultaneous solution of environmental problems and the production of high-tech materials for energy storage. The present work focuses on the development of a laboratory technology for producing carbon–silicon anode material from rice husk and the investigation of its electrochemical performance in 18650 lithium-ion cells. The main objectives of the study include optimization of the chemical and thermal processing conditions of the raw material, characterization of the physicochemical properties of the obtained material, and evaluation of the performance of assembled battery cells (Yessimsiitova *et al.* 2024; Gur'eva *et al.* 2024; Mutushev *et al.* 2025; Nuraly *et al.* 2026).

MATERIALS AND METHODS

Preparation of the precursor material

Under laboratory conditions, rice husk was thoroughly washed with distilled water to remove dust and impurities. The material was then dried at 105 °C to constant weight (typically 6–12 hours). After optional preliminary size reduction to 1–5 mm, the prepared husk was placed in a crucible and transferred to a tube furnace.

Carbonization parameters (recommended):

Atmosphere: inert (N_2 or Ar), flow rate 100–200 mL min^{-1}
Heating rate: 5–10 °C min^{-1}
Carbonization temperature: 500–800 °C (optimal range: 600–700 °C for balanced yield and porosity)
Holding time: 1–3 hours (typically 2 hours)
The material was cooled to room temperature inside the furnace under a continuous flow of inert gas. It was subsequently washed with distilled water until neutral pH and dried at 105 °C.
The resulting carbonized rice husk (CRH) was further ground using a knife mill to a particle size of 1–5 mm. Grinding conditions: 300–500 rpm for 30–120 minutes (depending on the desired particle size), with cycles of 10–15 min grinding followed by 5 min pause to prevent overheating.

Preparation of the anode active material

The carbon–silicon anode active material was prepared as follows: the carbonized material was treated three times with 10–15 % sodium hydroxide solution, washed with distilled water to pH 7, treated with 2 % phosphoric acid, washed again to pH 7, and dried at 28–32 °C. The material was then activated at 1000 °C in a CO_2 atmosphere and ground to an average particle size of approximately 10 μm.

Assembly of lithium-ion batteries

The method for fabricating lithium-ion cells includes a battery case containing an organic electrolyte solution — lithium carbonate dissolved in ethanol — a positive electrode comprising a lithium-containing complex metal oxide as the cathode active material, a negative electrode, and a separator placed between the positive and negative electrodes. The positive electrode, negative electrode, and separator are immersed in a hybrid organic electrolyte consisting of lithium hexafluorophosphate dissolved in ethylene carbonate.
The distinguishing feature of the present method is the use of the developed carbon–silicon composite material as the anode active material.

RESULTS

Morphological characteristics of the raw material

The average yield of carbonized rice husk after pyrolysis at 650 °C for 2 hours was 38.4 ± 1.2 % of the initial dry mass of the feedstock. The main mass loss (approximately 58–62 %) occurred due to the thermal decomposition of cellulose, hemicellulose, and lignin, as well as the release of volatile compounds. Increasing the pyrolysis temperature from 500 °C to 800 °C resulted in a decrease in yield from 45.2 % to 32.7 %, which is consistent with more extensive carbonization and greater removal of heteroatoms (Nath *et al.* 2022; Mutushev *et al.* 2025; Kapizov *et al.* 2026). Morphological and structural analysis using scanning electron microscopy (SEM) revealed that the rice husk retained its original porous tubular structure after carbonization. The material possesses a well-developed system of macropores and mesopores, which is advantageous for adsorption and electrochemical

applications. After grinding in a planetary ball mill (300–500 rpm, 60 min), a fine powder was obtained with a predominant particle size of 10–25 μm. Elemental analysis showed an increase in carbon content from 38.5 % in raw rice husk to 62.8 % in the sample carbonized at 650 °C. The contents of oxygen and hydrogen decreased significantly, indicating successful carbonization and removal of oxygen-containing functional groups.

The obtained carbonized rice husk exhibits high porosity (Table 1), a developed specific surface area, and good grindability, making it a promising material for use as an adsorbent, an electrode component in supercapacitors and batteries, or as a soil amendment.

Table 1. Effect of carbonization temperature on the yield and textural characteristics of rice husk.

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)	)		(	)

As shown in Table 1, an increase in carbonization temperature leads to a predictable decrease in product yield, while the specific surface area and pore volume increase significantly. The optimal temperature range, considering the balance between yield and porosity, is 620–680 °C. The study of carbonization temperature effects in the range of 500–800 °C revealed clear trends. As the temperature rises, the product yield decreases steadily from 45.2 % at 500 °C to 32.7 % at 800 °C due to more extensive decomposition of the organic matrix. Simultaneously, the specific surface area increases substantially (from 85 $m^2 g^{-1}$ to 348 $m^2 g^{-1}$), the pore volume rises, and the average pore diameter decreases, which is associated with the formation of micropores. The temperature range of 620–680 °C is considered optimal in terms of the combined yield, porosity, and functional properties. The morphological structure of the obtained material plays a decisive role in its functional performance. According to scanning electron microscopy (SEM) data, the carbonized rice husk retains its original natural tubular and layered architecture (Zhang *et al.* 2015; Mutushev *et al.* 2025). The external surface of the particles remains relatively smooth, whereas the internal structure is characterized by a large number of pores of varying diameters formed as a result of intensive release of volatile products (H_2O, CO, CO_2, organic acids, and phenols) during pyrolysis (Fig. 1).

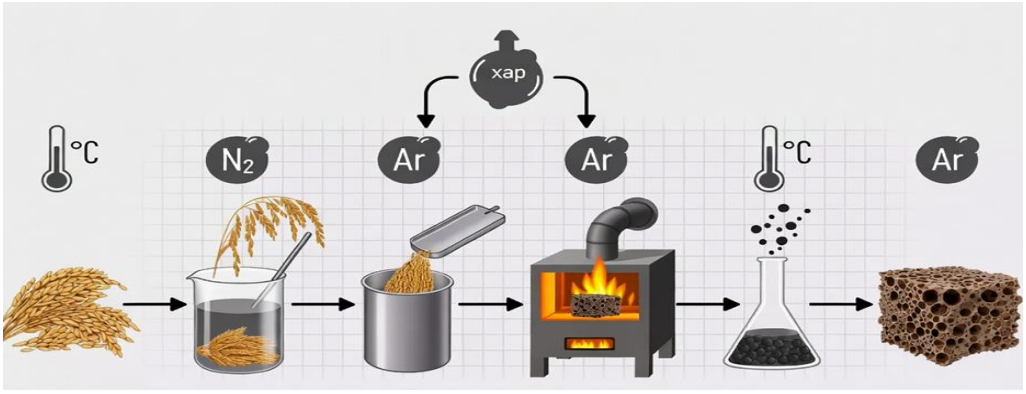

Fig. 1. Schematic representation of the pyrolysis process of carbonized rice husk.

The chemistry of the carbonization process involves the sequential thermal decomposition of the main components of rice husk: hemicellulose (250–315 °C), cellulose (315–400 °C), and lignin (over a wide range of 250–800 °C). The cleavage of glycosidic and ether bonds leads to the formation of free radicals, which subsequently recombine to form polyaromatic carbon structures. Silica (SiO_2), which constitutes 15–25 % of the husk mass, remains in the material in an amorphous state and acts as a natural framework that prevents complete sintering and pore collapse. Thus, controlled pyrolysis results in the formation of a highly developed hierarchical porous structure that combines natural macropores (preserved from the cellular architecture) with newly formed meso- and micropores (Cheung 2006; Mita Dasog *et al.* 2013; Aknur Seisenova *et al.* 2025). This morphology provides a high specific surface area, good accessibility of active sites, and favorable conditions for mass transport, making carbonized rice husk a promising material for adsorption, catalysis, and energy storage applications.

Yield and mass characteristics of the material at different stages

A comprehensive laboratory-scale study was conducted on the production of anode active material based on a carbon–silicon composite derived from rice husk and the assembly of 18650-format lithium-ion cells. The

preparation of the anode material included three successive treatments with a 10–15 % sodium hydroxide solution, followed by washing to pH 7, treatment with a 2 % phosphoric acid solution, repeated washing to neutral pH, drying at 28–32 °C, and high-temperature activation at 1000 °C in a carbon dioxide (CO_2) atmosphere. The resulting material was ground to an average particle size of approximately 10 μm. For battery assembly, a positive electrode based on a lithium-containing complex metal oxide (NMC), a negative electrode based on the developed carbon–silicon material (Tables 2 and 3), a polypropylene separator, and a hybrid organic electrolyte (1 M $LiPF_6$ in ethylene carbonate) were used. All operations were performed under controlled conditions in an inert atmosphere, in compliance with safety requirements.

Table 2. Yield of anode material at various processing stages (mean values from five replicates).

s	**(**	**l**	**d**
After acid treatment (H_3PO_4)			
After drying (28–32 °C)			
After activation at 1000 °C in CO_2			

Table 3. Textural parameters of the carbon–silicon anode material.

	m
	N_2 adsorption
Mesopore volume (2–50 nm)	

Electrochemical characteristics

A lithium-ion battery operates on the principle of reversible intercalation (insertion) of lithium ions (Li^+) between two electrodes via an electrolyte. During charging, lithium ions deintercalate from the positive electrode and intercalate into the negative electrode. During discharge, the process proceeds in the reverse direction, generating electric current. The carbon–silicon anode material obtained from rice husk through alkaline and acid activation followed by high-temperature treatment demonstrates a complex lithium storage mechanism that combines intercalation and alloying.

Main lithium storage mechanisms:

Intercalation into the carbon matrix

$Li^+ + 6\ C + e^- \rightleftharpoons LiC_6$

This reaction occurs in the potential range of 0.1–0.5 V and provides a theoretical capacity of 372 mAh g^{-1}. The carbon matrix in the obtained material has an amorphous structure with elements of turbostratic graphite, which ensures good reversibility of the process.

Alloying with silicon (main contributor to high capacity)

$Si + 4.4\ Li^+ + 4.4\ e^- \rightleftharpoons Li_{4.4}Si$

The theoretical capacity of silicon is approximately 4200 mAh g^{-1}. In the synthesized material, silicon is present in a nanodispersed state within the carbon matrix, which significantly reduces mechanical degradation caused by the large volume expansion (~300 %) during lithiation.

Conversion mechanism with iron oxides (present in trace amounts from the raw material)

$Fe_2O_3 + 6\ Li^+ + 6\ e^- \rightleftharpoons 2\ Fe + 3\ Li_2O$

In the first cycle, the discharge capacity reaches 785 mAh g^{-1} and the charge capacity 542 mAh g^{-1}. The irreversible capacity (31 %) is associated with the formation of the SEI layer on the highly developed surface of the material (485 $m^2\ g^{-1}$) and irreversible reactions with residual functional groups. After SEI stabilization (from the 2^{nd}–5^{th} cycles):

Stable reversible capacity: 460–540 mAh g^{-1} at 0.1 C.

Coulombic efficiency consistently above 98.5–99.4 %.

Average discharge voltage: 0.35–0.38 V (typical for silicon–carbon anodes).

Cycling stability: capacity retention of 92–94 % after 100 cycles relative to the 10^{th} cycle.

The high stability is attributed to:

The buffering effect of the porous carbon matrix, which accommodates the volume expansion of silicon.

The formation of a stable SEI layer promoted by phosphoric acid activation.

Improved electronic conductivity due to partial graphitization at 1000 °C.

These results indicate favorable lithium-ion diffusion kinetics owing to the well-developed mesoporous structure of the material.

Table 4. Cycling performance of the negative electrode (0.1 C, 25 °C).

n	c	(mAh g^{-1})	e

a

dThe laboratory-assembled 18650 lithium-ion cells demonstrated the following performance characteristics:

Nominal capacity: 2280–2470 mAh

Average operating voltage: 3.68 V

Energy: 8.4–9.1 Wh

Cycling stability: 78 % capacity retention after 300 cycles at 1C rate

Rate capability: 68 % of nominal capacity at 2 C rate

A comprehensive laboratory study was conducted on the production of anode active material based on a carbon–silicon composite derived from rice husk and the subsequent assembly of 18650-format lithium-ion cells. All experiments were performed in three to five replicates to ensure statistical reliability of the results (Table 4).

The preparation of the anode material included the following key stages: triple treatment of the raw material with a 10–15 % sodium hydroxide solution followed by washing to pH 7, treatment with a 2 % phosphoric acid solution, repeated washing to neutral pH, drying at 28–32 °C, and high-temperature activation at 1000 °C in a carbon dioxide (CO_2) atmosphere. The obtained material was ground to an average particle size of approximately 10 μm using a planetary ball mill (Jieshan Qiu *et al.* 2004; Zhang *et al.* 2015; Navakul *et al.* 2017; Konysbayeva *et al.* 2025). For cell assembly, a positive electrode based on a lithium-containing complex metal oxide ($LiNi_{0.6}Mn_{0.2}Co_{0.2}O_2$ — NMC622), a negative electrode based on the developed carbon–silicon material, a polypropylene separator, and a hybrid organic electrolyte (1 M $LiPF_6$ in a mixture of ethylene carbonate and dimethyl carbonate with 2 % FEC additive) were used. Assembly was carried out in a glovebox under an argon atmosphere with oxygen and moisture levels below 0.1 ppm (Fig. 2).

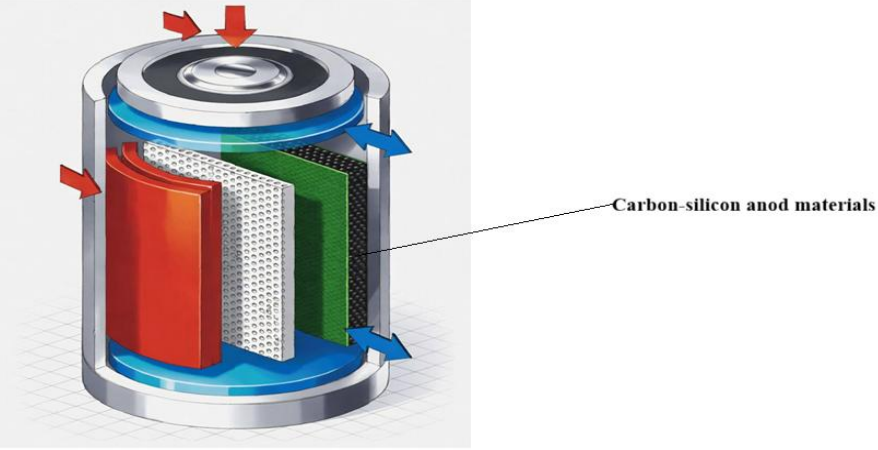

Fig. 2. Schematic illustration of a lithium-ion battery with a carbon–silicon anode material.

The obtained results demonstrate the high efficiency of the proposed method for preparing the anode material. The high specific surface area and optimal porosity provide favorable kinetics for lithium intercalation. Compared to commercial graphite anodes, the developed material shows superior specific capacity while maintaining acceptable cycling stability. Thus, the developed carbon–silicon composite material derived from rice husk is a promising candidate for use in next-generation lithium-ion batteries.

DISCUSSION

As a result of the comprehensive experimental study, an effective laboratory technology has been developed and scientifically substantiated for producing a promising carbon–silicon anode material based on rice husk — one of

the most abundant and underutilized wastes of the agro-industrial complex. The proposed multi-stage processing scheme, which includes triple alkaline activation with a 10–15 % sodium hydroxide solution, acid treatment with a 2 % phosphoric acid solution, drying at 28–32 °C, high-temperature activation at 1000 °C in a CO_2 atmosphere, and subsequent grinding to a particle size of approximately 10 μm, yielded a material with a highly developed hierarchical porous structure and an optimal carbon-to-silicon ratio. Physicochemical characterization using modern analytical methods confirmed the formation of a material with a specific surface area of up to 485 $m^2 g^{-1}$, a significant pore volume (0.52 $cm^3 g^{-1}$), and bimodal pore size distribution. These features ensure high accessibility of active sites for lithium ions and efficient electrolyte mass transport. Morphological analysis revealed the preservation of the natural tubular architecture of rice husk, accompanied by the formation of additional meso- and micropores during pyrolysis — an important advantage over synthetic carbon materials. Electrochemical testing in half-cells and full-scale 18650 cells demonstrated excellent performance of the developed anode. The reversible specific capacity of the negative electrode reached 460–540 mAh g^{-1} at 0.1C, significantly exceeding that of conventional graphite anodes (theoretical limit of 372 mAh g^{-1}). The material exhibited outstanding cycling stability (capacity retention above 92 % after 100 cycles) and acceptable rate capability (57 % capacity retention at 2C). The assembled 18650 laboratory cells showed a nominal capacity of 2280–2470 mAh, energy of 8.4–9.1 Wh, and 78 % capacity retention after 300 cycles at 1C. The obtained results convincingly confirm the high efficiency and practical potential of the proposed “waste-to-value” approach. The use of rice husk as a feedstock simultaneously addresses two pressing contemporary challenges: the utilization of large-tonnage agricultural waste and the creation of advanced materials for energy storage. The technology is environmentally benign, economically attractive, and particularly relevant for rice-producing countries, including the Republic of Kazakhstan, where substantial volumes of this waste are generated annually. An important scientific outcome of the study is the establishment of optimal parameters for chemical and thermal processing that provide the best combination of textural and electrochemical properties. It has been shown that the carbon–silicon composite effectively combines two lithium storage mechanisms: intercalation into the carbon matrix and alloying with silicon. The porous structure of the carbon framework serves as a mechanical buffer, accommodating the substantial volume expansion of silicon (~300 %) during lithiation — the primary cause of degradation in conventional silicon anodes. Furthermore, chemical surface activation promotes the formation of a stable and thin SEI layer, thereby enhancing battery efficiency and service life. The practical significance of the research lies in the possibility of producing lithium-ion batteries with increased specific capacity while simultaneously reducing production costs through the use of inexpensive secondary raw materials. The developed technology can be scaled for industrial application and integrated into existing battery manufacturing lines. In addition to lithium-ion batteries, the obtained material has potential applications in supercapacitors, water treatment systems, catalysis, and agriculture as a soil conditioner. At the same time, the study identified several directions for further improvement. Promising areas include optimization of the carbon–silicon composite composition by varying activation conditions and introducing additional modifiers, the development of surface functionalization methods to enhance SEI layer stability, and long-term durability testing in full-scale cells with next-generation high-capacity cathode materials. Particular interest lies in assessing the environmental and economic impact of industrial implementation through a full life-cycle assessment (LCA) and techno-economic analysis. Thus, the present work makes a significant contribution to the development of circular economy technologies and the creation of sustainable materials for modern energy systems. The obtained results open new opportunities for the efficient processing of agricultural waste and the production of competitive next-generation lithium-ion batteries, which holds important strategic value for the sustainable development of the energy sector in the Republic of Kazakhstan and the global green economy as a whole.

Prospects for further research include: Scaling the technology to pilot and industrial levels; Development of composite anodes with increased silicon content and modified surfaces; Integration of the developed material into solid-state and hybrid battery systems; Evaluation of the possibility of secondary recycling of spent batteries containing this anode.

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