ACTA METALLURGICA SLOVACA
2025, VOL. 31, NO. 4, 219-224

RESEARCH PAPER

Synthesis of silicon carbide from secondary silicon-containing materials

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Received: 11.09.2025
Accepted: 18.10.2025

ABSTRACT

This study presents an experimental approach to the synthesis of silicon carbide (SiC) using technogenic and biogenic wastes—microsilica and rice husk. The synthesis was carried out by carbothermal reduction in a self-designed and constructed laboratory resistance furnace. The starting materials were subjected to preliminary mechanical activation in a planetary centrifugal mill, which enhanced their reactivity. The morphology and elemental composition of the samples were examined using scanning electron microscopy (SEM) and energy-dispersive spectroscopy (EDS). The results demonstrated the formation of characteristic SiC crystals and confirmed their composition, close to stoichiometric. X-ray diffraction analysis revealed the presence of β-SiC (3C) and hexagonal polytypes (4H, 6H-SiC), as well as secondary phases of silicon dioxide (cristobalite, tridymite), reflecting the specific features of the reaction at high temperatures. It was established that mechanical activation promotes more intensive SiC formation and reduces the amount of residual oxide phases. The obtained results confirm the potential of utilising secondary silicon-containing resources for SiC synthesis, which is significant for the development of environmentally oriented technologies and for expanding the raw material base for high-value-added materials.

Keywords: silicon carbide, waste, microsilicon, rice husk, petroleum coke, pyrolysis, mechanical activation, synthesis

INTRODUCTION

In the context of the rapid growth of industrial production and the expansion of the agricultural sector worldwide, the volume of waste containing valuable components is steadily increasing. Among the most significant in terms of both quantity and environmental impact are microsilica and rice husk. According to the World Steel Association, global crude steel production amounted to 150.1 million tons in 2025 [1]. Considering the silicon content during steel alloying at 1.0 to 3.5-4.0%, the global demand for low-impurity ferrosilicon can be empirically estimated at 5.2-6.0 million tons per year [2]. Since microsilica is a byproduct of the production of high-silicon ferroalloys, on average, 50-250 kg of microsilica is generated per ton of ferrosilicon produced [3]. According to the Food and Agriculture Organization of the United Nations, annual global rice production exceeds 760 million tons, resulting in approximately 150 million tons of rice husk [4]. Rice husk is removed during the first stage of rice processing, accounting for about 20% of the weight of paddy rice [5]. The use of microsilica and rice husk possesses significant environmental potential. When these materials are applied in SiC synthesis, a dual ecological effect is achieved: the utilization of industrial and agricultural wastes and the reduction of anthropogenic environmental impact through the conservation of natural resources. Both materials contain a considerable amount of amorphous silica, making them promising raw materials for the production of functional materials. Current trends in metallurgy and materials science focus on developing new approaches to recycle technogenic and agricultural wastes and convert them into highly demanded products. Thus, materials previously regarded solely as environmental burdens can be transformed into valuable resources, particularly SiC. Due to its unique properties, SiC is widely used in various industries, including metallurgy, electronics, mechanical engineering, and aerospace [6].

SiC is a refractory compound characterized by high hardness, thermal stability, corrosion resistance, and excellent dielectric properties [7]. SiC - based materials can operate under high temperatures and high voltages and are widely used in semiconductor manufacturing [8]. The global demand for smart consumer electronics is growing exponentially, creating favorable conditions for the expansion of the SiC market. Owing to its wide band gap properties, SiC is increasingly recognized as a key technology for efficient power electronics [9]. According to Data Bridge Market Research, the SiC market was valued at USD 1,035.65 million in 2022 and is projected to rise sharply to USD 3,212.42 million by 2030, with a compound annual growth rate (CAGR) of 15.20% during the forecast period 2023-2030 [10]. This sharp market growth is closely linked to the broad adoption of SiC across industries.

SiC products are classified into black, green, and other types of SiC. According to Global Market Insights, the black SiC segment accounted for 42% of the market share in 2024. Black SiC is widely used in industries such as automotive, aerospace, and metallurgy due to its high hardness and strength. It is also applied for grinding, polishing, and cutting in these sectors. The green SiC market accounted for 38% of the market share in 2024. Green SiC is a high-purity material that makes it practical and reliable for a wide range of applications, owing to its high thermal conductivity and excellent wear resistance. Typically, green SiC is used for precision grinding of cutting tools and for lapping semiconductor materials. The remaining types of SiC held 20% of the market share, being mainly utilized as refractory materials and for related applications.

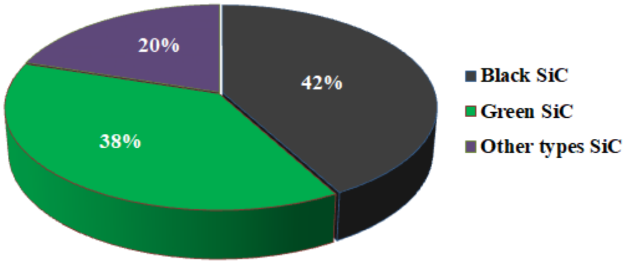


Fig. 1 Market share of different types of SiC products [11]

In the metallurgical industry, SiC is used as a deoxidizer in steel production. SiC primarily dissolves in the oxygen furnace to process large amounts of hot metal and scrap. It exhibits a band gap three times wider, a critical electric field strength ten times higher, and a thermal conductivity three times greater than those of silicon [12].

In recent years, SiC has been gaining increasing popularity in mechanical engineering due to the mass production of electric vehicles, renewable energy technologies, and power electronics. According to the International Energy Agency (IEA), more than 17 million electric vehicles were sold in 2024, 3 million more than in the first quarter of 2023. SiC - based devices are in high demand in electric vehicles, as they improve vehicle efficiency while reducing overall weight and size [13].

The consumption of SiC in the aerospace and defense industries is also growing rapidly, as in recent years the global military sector, combined with the development of electric and hybrid-electric aircraft, has become an important driver of SiC market growth [14].

Depending on the production method, the following SiC synthesis processes are segmented:

Acheson method

This is the classical synthesis method, in which silica (SiO_2) and carbon (coke) react at high temperatures (around 2000-2500 °C) in an electric furnace. The furnace is equipped with transformers rated at 4000–4500 kVA and consumes 7500-7600 kWh of electricity per hour [15]. The process proceeds according to the reaction:

$$SiO_2 + 3C = SiC + 2CO \qquad \text{(Eq. 1)}$$

This method is used to obtain silicon carbide for applications in the abrasive and semiconductor industries [16].

Lely method

The Lely method is a high-temperature synthesis process that enables the production of high-quality SiC crystals. In this process, silica (SiO_2) and carbon (e.g., coal dust or coke) react at temperatures of about 1500-2000 °C in a gaseous atmosphere. The advantages of this method include the high purity of the obtained material and its suitability for manufacturing semiconductors and other high-performance components [17].

SiC produced by the Lely method is characterized by a high degree of purity and a minimal amount of impurities, making it ideal for use in semiconductor devices such as diodes and transistors. An important aspect of this process is also the growth of large SiC crystals, which enables the use of this material in high-tech industries such as aerospace [18].

Chemical Vapor Deposition (CVD) method

In this method, SiC is deposited onto a substrate from the gas phase, where gaseous silicon and carbon compounds interact at high temperatures. This technique is primarily used to produce thin SiC films for microelectronic devices and nanotechnologies [19].

Despite the variety of synthesis methods, traditional industrial technologies for producing SiC generally rely on natural quartz raw materials and carbonaceous reducing agents [20]. All of the above-mentioned SiC synthesis methods are characterized by high energy consumption and reliance on costly natural raw materials. Despite the significant number of studies dedicated to SiC synthesis, the development of a resource-efficient, highly effective technology for SiC production using technogenic waste as a raw material base remains highly relevant.

In this context, particular importance is attached to studying the effects of mechanical activation, particle size of the initial components, and high-temperature sintering conditions on phase formation processes and the morphology of the final product. Therefore, the present work aims to investigate the synthesis of SiC under laboratory conditions using technogenic wastes and to identify the patterns of how technological parameters influence the phase composition and yield of SiC.

MATERIALS AND METHODS

Microsilica is a byproduct of ferroalloy production, particularly during the smelting of silicon-rich ferroalloys. It is a dark gray powder containing silicon dioxide. During ferroalloy smelting, the reduction of quartz raw materials results in the formation of finely dispersed silicon dioxide (SiO2) in the form of micro particles with a size of 0.1-1 μm. The composition and properties of microsilica provide the basis for its use in various industries [21].

As in many countries worldwide, the ferroalloy industry is also a key sector of Kazakhstan's metallurgical industry. In terms of quality, product range, and production volume, Kazakhstan's ferroalloy industry ranks among the leading globally. It is on par with technologically advanced nations, making it among the largest exporters. Kazakhstan, with its truly vast reserves of various mineral resources, fully meets the needs of its industry.

At present, ferroalloy production in Kazakhstan is carried out by enterprises such as «JSC Donskoy Mining and Processing Plant», «JSC «Aksu Ferroalloy Plant», and «JSC Aktobe Ferroalloy Plant», which are part of the «Transnational company Kazchrome»; «LLP A & K» (Ekibastuz); «LLP KSP Steel» (Pavlodar); «LLP KazFerroGroup» (Shymkent); «Karaganda Ferroalloy Plant» of «LLP YDD Corporation»; «LLP Asia FerroAlloys» (Karaganda), among many others. These industrial enterprises produce large volumes of various grades of ferrosilicon and metallurgical silicon. During the smelting of silicon-rich ferroalloys, microsilica is generated as a byproduct [22-31].

In this study, microsilica was used as the silica-containing raw material. It represents a technogenic byproduct of technical silicon production at «LLP Tau-Ken Temir». The composition of microsilica is shown in **Fig. 2** [32].

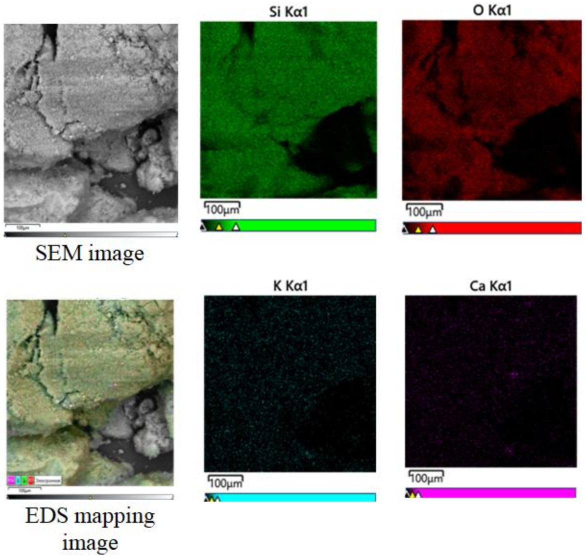


Fig. 2 Energy-Dispersive Spectroscopy (EDS) results of microsilica

To reduce silicon dioxide in microsilica, petroleum coke is used as a reducing agent. Petroleum coke is a solid substance formed as a byproduct during oil refining. It serves as a source of relatively high-purity carbon. In this study, petroleum coke is of particular interest for SiC synthesis due to its low ash content and high carbon concentration (91.17%) [33]. The technical composition of petroleum coke is presented in **Table 1.**

Table 1 Technical composition of petroleum coke, wt.%

A	W	C	V	S
0,12	0,70	91,17	8,08	3,27

Rice husk is an agro-industrial byproduct generated during rice processing, accounting for about 20% of the total grain mass. It is a lightweight, porous material mainly composed of cellulose, silicates, and organic substances such as lignin and cellulose [34]. The primary disposal methods are combustion and landfilling, which are inefficient and lead to additional environmental problems, including carbon dioxide emissions and soil contamination by ash residues after burning [35].

Rice cultivation in Kazakhstan is concentrated mainly in the Kyzylorda region, which accounts for 89.5% of the total sown area of this crop. The remaining 10.5% is distributed among the Almaty, Turkistan, and Zhetysu regions. In other areas of the country, rice is not cultivated due to unsuitable climatic conditions. In 2024, Kazakhstan experienced a decline in rice production. According to data from August 2024, about 450 thousand tons of rice are expected to be harvested, compared to 503.3 thousand tons in 2023 [37]. The main reason for the reduction in sown areas is a shortage of irrigation water, which leads to lower yields and final production. By 2024, around 90 thousand tons of rice husk had already accumulated in Kazakhstan [36].

Before pyrolysis, rice husk is preliminarily cleaned from foreign impurities, dried, and prepared for processing. The elemental composition of rice husk is: C – 39.3–41.2%, H – 5.6–6.3%, O – 0.5–0.6%, N – 36.7–37.5%. Thermo-oxidative pyrolysis of rice husk occurs at temperatures between 600 and 750 °C. During pyrolysis, the organic matter of rice husk (mainly cellulose and other carbon-containing components) decomposes into a carbonaceous residue (bio char) and volatile compounds. In contrast, silicate compounds form silicon dioxide (SiO_2). The composition of the pyrolysis products of rice husk is shown in **Fig. 3**.

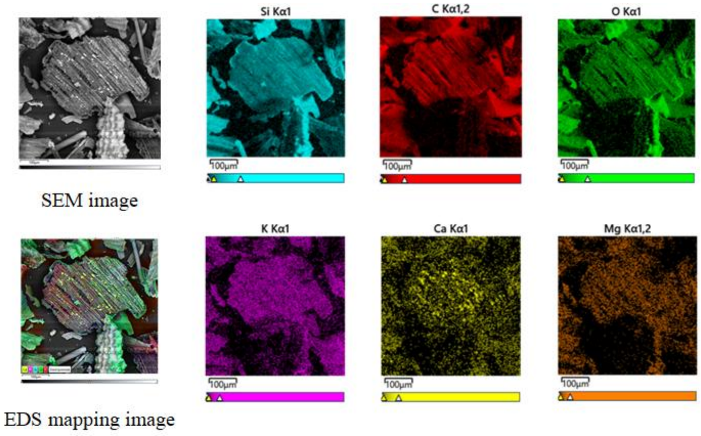


Fig. 3 Energy-Dispersive Spectroscopy (EDS) results of rice husk pyrolysis

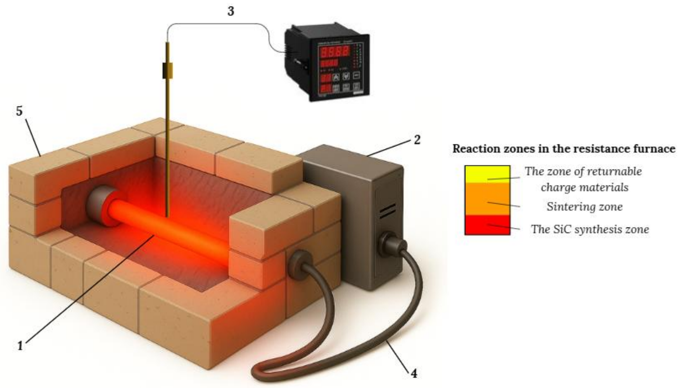
According to EDS analysis of rice husk after pyrolysis, the product mainly consists of SiO_2 and C, with additional volatile components. As a result of pyrolysis, a sample of material with the technical and chemical composition presented in **Table 2** was obtained.

Table 2 Technical and chemical composition of rice husk pyrolysis product

Technical composition, %			Chemical composition of ash, %				
W	V	A	SiO_2	Al_2O_3	Fe	CaO	MgO
3,96	3,74	55,14	94,17	0,21	0,43	0,72	0,19

The solid carbon content in the obtained product is 37.16%, which fully meets the requirements for further processing. The production of SiC involves the reaction of silica (SiO_2) and carbon (C) at high temperatures (>1600 °C), resulting in the formation of SiC and carbon monoxide. The industrial process of SiC synthesis is highly exothermic and requires careful control of temperature, pressure, and gas flow [16]. Microsilica and rice husk can replace quartz sand in the production of SiC. These materials have much smaller particle sizes than quartz sand, leading to more efficient reactions with carbon and a higher yield of SiC [39]. An important role in the study of SiC synthesis from waste materials is played by the preliminary mechanical activation of the initial charge mixture and the synthesis temperature regime. Moreover, microsilica and rice husk, after mechanical activation, acquire a larger surface area, which can promote the nucleation and growth of SiC crystals and improve the crystallinity and morphology of the final valuable product. During grinding, the charge components are subjected to the intense action of milling balls, which create impact and shear stresses. This leads to the breakdown of aggregates and the uniform distribution of the components within the mixture. The high rotation speed of the mill enables the production of much finer particles compared to conventional grinding methods, reaching fractions on the order of several micrometers. Such a fine state increases the contact area between silica and carbon, thereby enhancing the system's reactivity [38].

For subsequent SiC synthesis, a laboratory resistance furnace of in-house design was developed, following the operating principle of the Acheson furnace. A 100 kVA transformer was used as a power source. The setup enables high-temperature synthesis via electrical heating, with current supplied to a graphite electrode, similar to the industrial process. The furnace design ensures uniform temperature distribution in the reaction zone and creates conditions for effective interaction between the initial components, thereby facilitating the formation of SiC. The schematic of the laboratory furnace is shown in **Fig. 4.**

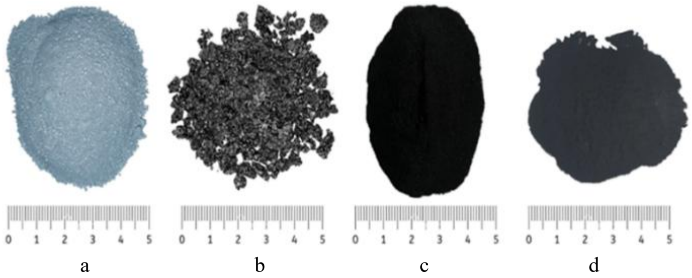


1 – graphite electrode; 2 – furnace transformer; 3 – thermocouple;
4 – current lead; 5 – fireclay bricks; 6 – reaction zones.

Fig. 4 Cross-section of the laboratory furnace for SiC synthesis

The furnace body is made of fireclay bricks (5). A graphite electrode with a diameter of 10 mm (1) is placed along the perimeter of the working chamber. An electric current of 180–200 A is supplied to the electrode via the furnace transformer (2). The furnace is fully sealed; the charge mixture is loaded into the working zone and compacted by ramming. A VR-5/20 thermocouple (6) is installed in the upper part of the furnace to monitor the temperature, being immersed in the electrode surface. Three zones are distinguished in the furnace's working space: the main reaction zone (2), the sintering zone (3), and the return charge zone (4). The SiC synthesis process takes 40 minutes at temperatures of 1600–1700°C.

The initial appearance of microsilica is shown in **Fig. 5a**, and petroleum coke is presented in **Fig. 5b**.



a b c d

Fig. 5 Charge mixture of microsilica and petroleum coke

The charge mixture of microsilica and petroleum coke was prepared by manual mixing in a mortar (**Fig. 5c**), followed by milling in a planetary ball mill (**Fig. 5d**).

The charge mixture of rice husk is shown in **Fig. 6**: image (a) presents its initial appearance, image (b) shows the appearance after pyrolysis, and image (c) shows the appearance after milling in a planetary centrifugal mill (PCM).

a b c

Fig. 6 Charge mixture of rice husk

The charge mixtures for SiC synthesis were loaded into the working space of the resistance furnace. During heating, the silica in the charge reacted with carbon to form SiC, releasing gaseous products. After the process was completed, the furnace was gradually cooled, and SiC was extracted from the reaction zone, while the residual materials were reused. The obtained samples were analyzed to determine the required characteristics of SiC. The synthesis process in the self-designed laboratory resistance furnace is shown in **Fig. 7.**

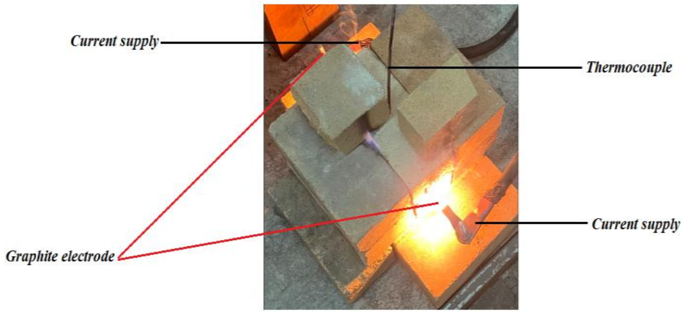


Fig. 7 Illustration of the resistance furnace operation for SiC synthesis

Grinding the charge mixture in a planetary centrifugal mill (PCM) is a key stage in the preparation process, ensuring high dispersion of the components, their mechanical activation, and homogeneity. This process is critically important for subsequent SiC synthesis reactions, where maximum contact between silica and carbon is required. For analysis of the obtained SiC samples, a Zeiss ZEM20 SEM equipped with an Oxford EDS attachment was used, enabling the acquisition of high-magnification microstructural images and the investigation of morphological and structural features. Standard imaging conditions were employed, including the use of backscattered electrons (BSE) for surface imaging and energy-dispersive spectroscopy (EDS) for elemental analysis. The SEM images clearly show SiC crystals with a characteristic structure. The samples obtained formed small rhombohedral crystals. Two samples were studied: the first synthesized from microsilica and petroleum coke, and the second obtained from rice husk. The samples were mounted on aluminum holders using carbon tape. Imaging was performed at magnifications of ×1000 and ×10,000 under high vacuum. The analysis allowed identification of the morphological features, shapes, and particle sizes depending on the raw material used.

RESULTS AND DISCUSSION

During milling, it was observed that carbon particles were evenly distributed and partially adsorbed on the silica surface. This interaction promotes the formation of a "coating" of the reducing agent, creating favorable conditions for the reduction of SiO_2 at high temperatures.

In the laboratory resistance furnace, optimal conditions for SiC formation are achieved at 1700 °C with a 40-minute holding time. **Fig. 8** shows: (a) the loaded charge; (b) the furnace with the installed thermocouple in the closed state; (c) the obtained material – top view; and (d) the obtained material – side view.

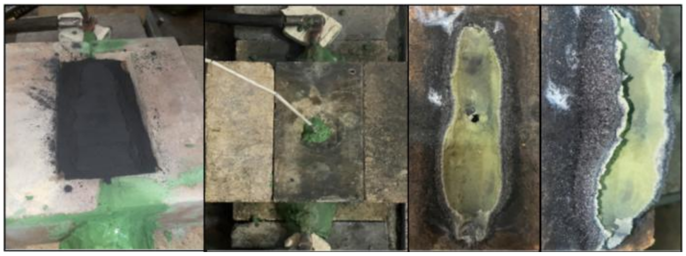
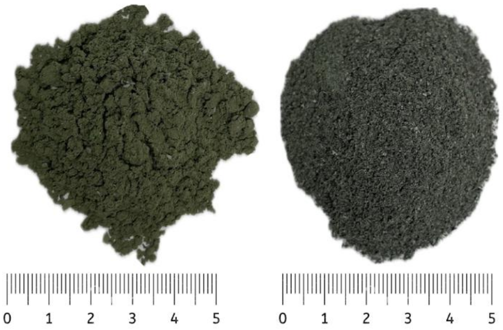

a b c d

Fig. 8 Appearance of the obtained SiC samples and the working space of the furnace

The obtained SiC samples are shown in **Fig. 9**: (9a) SiC synthesized from microsilica and petroleum coke; (9b) SiC obtained from rice husk.



a b

Fig. 9 Obtained SiC samples: (a) SiC from microsilica and petroleum coke; (b) SiC from rice husk

Both types of SiC exhibit a characteristic greenish tint, indicating their high purity. As is well known, green SiC has a low impurity content and is used in abrasive materials and the semiconductor industry [39].

Differences in the morphology and structure of the obtained products were observed. SiC synthesized from microsilica and petroleum coke is characterized by a denser structure and a high degree of crystallinity, which is associated with the uniform distribution of carbon in the charge after mechanical activation. In the case of rice husk, SiC formation is accompanied by preservation of a porous structure, which is determined by the biomass's natural morphology and its pyrolytic transformations. Such porosity may be of interest for applications as sorbents or catalyst supports.

Thus, this study demonstrates the fundamental possibility of producing high-purity SiC from both industrial waste (microsilica) and agro-industrial waste (rice husk). Comparative analysis shows that the choice of raw material enables targeted control over the morphological characteristics of silicon carbide, opening prospects for its use in various industries—from abrasive production to the creation of functional materials with well-developed surface areas.

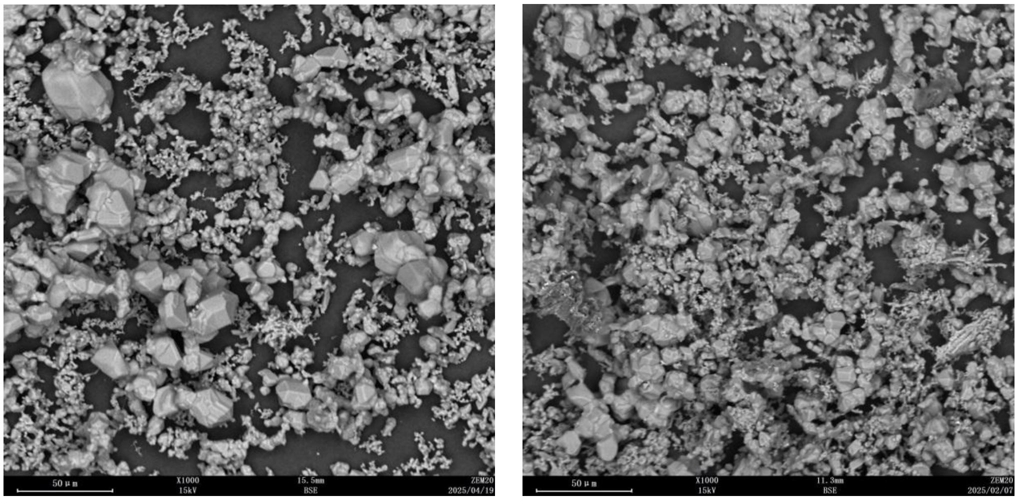
Elemental analysis using EDS confirmed that the samples' primary composition is silicon and carbon, indicating the presence of silicon carbide. Small regions that are not part of the main crystalline material were also observed, possibly indicating phase impurities or residual material.

Fig. 10 shows the particle morphologies of SiC obtained from microsilica and rice husk. SEM results reveal the characteristics of SiC crystals. Since these crystals exhibit a similar shape in both cases, EDS analysis was conducted, confirming the formation of SiC. The images show the presence of silicon (Si) and carbon (C), with possible traces of other elements depending on impurities in the starting materials.

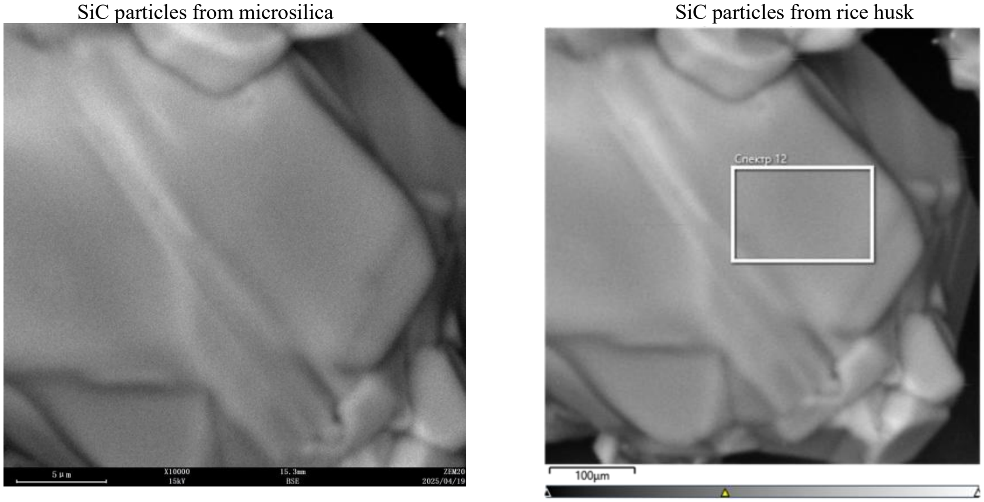
After obtaining the morphology images of the samples using scanning electron microscopy (SEM), elemental microanalysis was performed using energy-dispersive spectroscopy (EDS). The studies were carried out for both SiC samples synthesized from different starting materials. The crystal morphology at ×10,000 magnification showed a similar shape and structure, indicating comparable phase-forming conditions in both cases.

EDS analysis results confirmed an almost identical elemental composition of the analyzed crystals. According to the spectral data, the SiC crystals contain, on average, 53.83 at. % carbon, 45.5 at. % silicon, and 0.66 at. % oxygen. The presence of oxygen may be associated with slight surface oxidation or residual impurities.

SEM allowed the acquisition of detailed images of the SiC structure. The morphology of the material is characterized by clearly distinguishable SiC crystals. Elemental analysis confirmed that the sample consists of silicon and carbon. However, the presence of impurities may indicate specific aspects of the synthesis process that could affect the material's mechanical and electrical properties. For a comprehensive analysis of the obtained SiC samples, X-ray diffraction (XRD) was performed using a DRON-2 diffractometer, which is designed to identify the phase composition and crystalline structure of materials. SiC samples obtained from microsilica, petroleum coke, and rice husk were studied in powdered form. The investigation was conducted over a 2θ range, measuring diffraction peak intensities and subsequently comparing them with database references. **Fig. 11** shows the XRD pattern of SiC synthesized from microsilica and petroleum coke.



SiC particles from microsilica SiC particles from rice husk



X10000 EDS results of the SiC samples

Fig 10. SEM morphology of SiC particles obtained in the resistance furnace

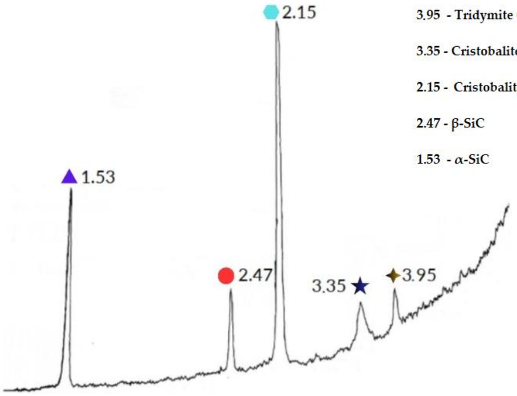


Fig. 11 XRD pattern of SiC synthesized from microsilica and petroleum coke

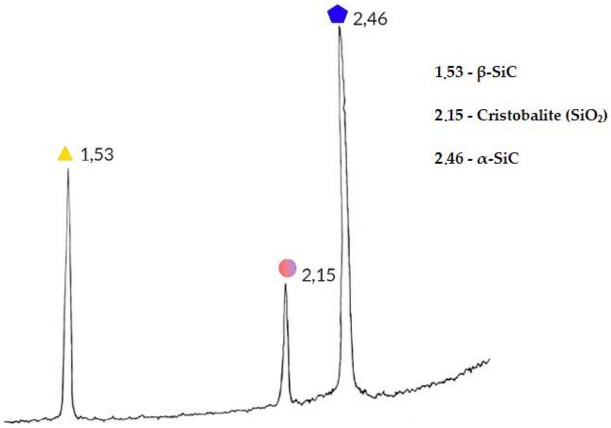


Fig. 12. XRD pattern of SiC synthesized from rice husk

Fig. 12 shows the XRD pattern of SiC obtained from rice husk. In the diffractogram, peaks at 1.53 and 2.46 are characteristic of SiC. These peaks confirm the presence of SiC in the sample and its crystalline structure. The diffractogram shows two main components: SiC and silicon dioxide (SiO_2). The peaks at 1.53 (6H-SiC) and 2.46 (4H-SiC) confirm the presence of SiC, while the peak at 2.15 corresponds to cristobalite, a modification of SiO_2. This indicates that the carbothermal process of rice husk produces both SiC and SiO_2, which is a typical feature of this material. The XRD pattern of the obtained SiC from rice husk reveals the formation of cubic 3C and hexagonal 4H polytypes of SiC.

The results of X-ray diffraction analysis confirm the presence of SiC in the samples, indicating the successful formation of the main reaction product, which was subsequently used to study its morphological and structural characteristics by SEM. The presence of cristobalite and tridymite is also an important indicator of the processes occurring during synthesis. These forms of silicon dioxide may appear as by-products at high temperatures, and their presence can be associated with the thermal decomposition of silica or its phase transformations. Importantly, their presence confirms the high temperature achieved during the SiC synthesis process.

The experimental results showed that both the duration of mechanical activation and the sintering temperature significantly influence the SiC synthesis process. Increasing the milling time in the planetary centrifugal mill (PCM) enhanced the batch's reactivity, resulting in more intensive SiC formation.

CONCLUSION

Silicon carbide (SiC) was successfully synthesized from industrial and agricultural wastes - microsilica and rice husk. Mechanical activation of the charge-enhanced component increased its reactivity and promoted the formation of dense SiC. XRD, SEM, and EDS analyses confirmed the formation of β-SiC with high crystallinity and minimal impurities. SiC from microsilica exhibited a thicker, more uniform structure, whereas that from rice husk exhibited a porous morphology. The results demonstrate the feasibility of producing high-purity SiC from waste materials, offering potential applications in metallurgy, electronics, and advanced functional materials.

Acknowledgements: This research is funded by the Science Committee of the Ministry of Education and Science of the Republic of Kazakhstan (Grant No. AP27510125)

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