ISSN: 1991-346X (Print)
ISSN: 2518-1726 (Online)

ACADEMIC JOURNAL OF PHYSICAL AND CHEMICAL SCIENCES

№3
2025

ISSN 2518-1483 (Online),
ISSN 2224-5227 (Print)

2025 • 3

CENTRAL ASIAN ACADEMIC RESEARCH CENTER

ACADEMIC JOURNAL OF PHYSICAL AND CHEMICAL SCIENCES

PUBLISHED SINCE JANUARY 1944

ALMATY, NAS RK

ACADEMIC JOURNAL OF PHYSICAL AND CHEMICAL SCIENCES.
ISSN 2518-1483 (Online), ISSN 2224-5227 (Print)
Owner: «Central Asian Academic Research Center» LLP (Almaty).
The certificate of registration of a periodical printed publication in the Committee of Information of the Ministry of Information and Social Development of the Republic of Kazakhstan **№ KZ93VPY00121157** issued **05.06.2025**
Thematic scope: *physics and chemistry.*
Periodicity: 4 times a year.
http://reports-science.kz/index.php/en/archive

ACADEMIC JOURNALOF PHYSICAL AND
CHEMICAL SCIENCES **2025 • 3**

ACADEMIC JOURNAL OF PHYSICAL AND CHEMICAL SCIENCES
ISSN 2518-1483 (Online), ISSN 2224-5227 (Print)
Меншіктеуші: «Орталық Азия академиялық ғылыми орталығы» ЖШС (Алматы қ.).
Ақпарат агенттігінің мерзімді баспасөз басылымын, ақпарат агенттігін және желілік басылымды қайта есепке қою туралы ҚР Мәдениет және Ақпарат министрлігі «Ақпарат комитеті» Республикалық мемлекеттік мекемесі **05.06.2025 ж.** берген **№ KZ93VPY00121157** Куәлік.
Тақырыптық бағыты: *физика, химия.*
Мерзімділігі: жылына 4 рет.
http://reports-science.kz/index.php/en/archive

ACADEMIC JOURNAL OF PHYSICAL AND CHEMICAL SCIENCES

ISSN 2518-1483 (Online), ISSN 2224-5227 (Print)

Собственник: ТОО «Центрально-азиатский академический научный центр» (г. Алматы).

Свидетельство **№ KZ93VPY00121157** о повторной регистрации периодического печатного издания информационного агентства, информационного агентства и сетевого издания, выданное Республиканским государственным учреждением «Комитет информации» Министерства культуры и информации Республики Казахстан **05.06.2025**

Тематическая направленность: *физика, химия.*

Периодичность: 4 раза в год.

http://reports-science.kz/index.php/en/archive

CONTENTS

PHYSICS

CHEMISTRY

МАЗМҰНЫ

ФИЗИКА

ХИМИЯ

СОДЕРЖАНИЕ

ФИЗИКА

ХИМИЯ

ACADEMIC JOURNAL OF PHYSICAL AND CHEMICAL SCIENCES
ISSN 2224-5227
Volume 3.
Number 355 (2025), 258–273

https://doi.org/10.32014/2025.2518-1483.378

UDC 661.183.2:661.666.3:620.92

© A.Zh. Mutushev*, A.B. Seisenova, O.S. Kapizov, A.M. Nuraly, D.K. Mukhanov, 2025.
Scientific Center for New Technologies, AlAkSan, Almaty Technological University, Almaty, Kazakhstan;
Center for Advanced Science and Technology, Almaty, Kazakhstan.
E-mail: alibek_090@mail.ru

INTEGRATED PROCESS FOR THE SYNTHESIS OF CARBON–SILICON NANOCOMPOSITES FROM BIOWASTE AND METALLURGICAL SLUDGE

Mutushev Alibek Zhumabekovich — PhD, Principal Research Scientist, Scientifi c Center for New Technologies, AlAkSan, Assistant Professor, Department of Chemistry, Chemical Technology and Ecology, Almaty Technological University, Almaty, Kazakhstan,
E-mail: alibek_090@mail.ru, ORCID ID: https://orcid.org/0000-0002-5047-5608;
Seisenova Aknur Berdibayevna — PhD, Principal Investigator (PI), Scientific Center for New Technologies, AlAkSan, Almaty, Kazakhstan,
E-mail: z_aknura@mail.ru , ORCID ID: https://orcid.org/0000-0002-8981-307X;
Kapizov Omirzak Sembiuly — Master of Technics and Technology, Senior Research Scientist, Scientific Center for New Technologies, AlAkSan, Almaty, Kazakhstan,
E-mail: ok-sir@mail.ru , ORCID ID: https://orcid.org/0000-0002-1979-7625;
Nuraly Assiya Mambetkyzy — PhD, Principal Research Scientist, Scientific Center for New Technologies, Center for Advanced Science and Technology, Assistant Professor, Department of Chemistry, Chemical Technology and Ecology, Almaty Technological University, Almaty, Kazakhstan,
E-mail: assiya488@gmail.com, ORCID ID: https://orcid.org/0000-0002-2323-0365;
Mukhanov Dauren Kabdrakimovich — Senior Research Scientist, Scientific Center for New Technologies, AlAkSan, Almaty, Kazakhstan,
E-mail: dd_511@mail.ru , ORCID ID: https://orcid.org/0009-0002-1645-8625.

Abstract. This study presents an integrated and sustainable technology for producing nanostructured carbon–silicon composite materials from renewable biomass feedstock (rice husk) combined with industrial waste residues. The proposed approach aims to address both waste valorization and the development of advanced functional materials for next-generation energy storage systems. The synthesis process involves consecutive stages of carbonization, activation, and demineralization, resulting in the formation of a highly porous structure with a well-developed specific surface area. Carbonization was carried out in an inert CO_2 atmosphere at temperatures ranging from 300 to 1000 °C, followed by physical activation in the range of 600 to 1200 °C. Subsequent removal of mineral impurities was achieved using a modified Soxhlet extraction technique, which ensured effective purification of the final product. The obtained silicon dioxide was predominantly in an X-ray amorphous state, exhibiting a diffuse peak maximum at 2θ = 24°, with a specific surface area of 120–150 m²/g and a pore volume of 0.5–0.8 cm³/g. XRF elemental analysis confirmed a SiO_2 purity level of up to 99.7%. SEM imaging revealed spherical particles with an average diameter of ~50 nm and a uniform distribution, while FTIR spectroscopy confirmed the preservation of characteristic siloxane (Si–O–Si) bonds. The developed approach demonstrates a promising route for converting low-cost biomass and waste resources into high-value carbon–silicon materials suitable for use in electrochemical energy storage and other advanced technological applications.

Keywords: carbon–silicon materials, rice husk, carbonization, activation, nanostructuring, amorphous silica, waste utilization, energy storage devices

Funding: *This research has been/was/is funded by the Committee of Science of the Ministry of Science and Higher Education of the Republic of Kazakhstan (Grant No. BR28713383)*

©А.Ж. Мутушев*, А.Б. Сейсенова, Ө.С. Капизов, Ә.М. Нұралы, Д.К. Муханов, 2025.

Scientific Center for New Technologies, AlAkSan, Алматы технологиялық университеті, Алматы, Қазақстан;

Center for Advanced Science and Technology, Алматы, Қазақстан.

E-mail: alibek_090@mail.ru

БИОҚАЛДЫҚТАР МЕН МЕТАЛЛУРГИЯЛЫҚ ШЛАМНАН КӨМІРТЕК–КРЕМНИЙ НАНОКОМПОЗИТТЕРІН СИНТЕЗДЕУДІҢ ИНТЕГРАЦИЯЛАНҒАН ӘДІСІ

Мутушев Алибек Жумабекович — PhD, Жетекші ғылыми қызметкер, Scientific Center for New Technologies, AlAkSan, ассистент-профессор, Химия, химиялық технология және экология кафедрасы, Алматы технологиялық университеті, Алматы, Қазақстан,
E-mail: alibek_090@mail.ru h, ORCID ID: https://orcid.org/0000-0002-5047-5608;

Сейсенова Ақнұр Бердибаевна — PhD, Жоба жетекшісі (PI) , Scientific Center for New Technologies, AlAkSan, Алматы, Қазақстан,
E-mail: z_aknura@mail.ru, ORCID ID: https://orcid.org/0000-0002-8981-307X;

Капизов Өмірзақ Сембіұлы — техника және технология магистрі, аға ғылыми қызметкер, Scientific Center for New Technologies, AlAkSan, Алматы, Қазақстан,
E-mail: ok-sir@mail.ru , ORCID ID: https://orcid.org/0000-0002-1979-7625;

Нұралы Әсия Мамбетқызы — PhD, жетекші ғылыми қызметкер, Scientific Center for New Technologies, Center for Advanced Science and Technology, ассистент-профессор, Химия, химиялық технология және экология кафедрасы, Алматы технологиялық университеті, Алматы, Қазақстан,
E-mail: assiya488@gmail.com, ORCID ID: https://orcid.org/0000-0002-2323-0365;

Муханов Даурен Кабракимович — аға ғылыми қызметкер, Scientific Center for New Technologies, AlAkSan, Алматы, Қазақстан,
E-mail: dd_511@mail.ru , ORCID ID: https://orcid.org/0009-0002-1645-8625.

Аннотация: Ұсынылған жұмыста жаңартылатын өсімдік тектес шикізаттан (күріш қауызынан) және өнеркәсіптік қалдықтардан наноқұрылымды көміртек-кремний композиттік материалдарын алудың интеграцияланған әрі экологиялық бағдарланған технологиясы сипатталады. Мұндай тәсіл биомассаны және қалдық түзуші шикізатты кешенді кәдеге жарату міндеттерін шешу тұрғысынан, сондай-ақ қазіргі заманғы энергетикалық және технологиялық қолданбаларда сұранысқа ие жаңа функционалдық материалдарды әзірлеу үшін өзекті болып табылады. Ұсынылған әдістеме карбонизация, активация және деминерализацияның бірізді сатыларын қамтиды, олар кеуектілігі дамыған құрылым мен жоғары меншікті беттің қалыптасуын қамтамасыз етеді. Карбонизация CO_2 инертті атмосферасында 300–1000 °C температурада жүргізілді, ал активация процесі 600–1200 °C диапазонында орындалды. Минералды қоспаларды тиімді жою үшін модификацияланған Сокслет аппараты қолданылды, бұл өнімнің тазарту дәрежесін едәуір арттыруға мүмкіндік берді. Алынған кремний диоксиді рентгеноаморфты күйде болды, оның $2\theta = 24°$ мәнінде диффуздық шыңы байқалды, меншікті бетінің ауданы 120–150 м²/г, ал кеуек көлемі 0,5–0,8 см³/г құрады. РФА әдісімен элементтік талдау SiO_2 тазалығын 99,7%-ға дейін растады. СЭМ микроскопиясы орташа мөлшері шамамен 50 нм болатын бөлшектердің біркелкі таралуын көрсетті, ал ИҚ-спектроскопия силоксандық байланыстардың (Si–O–Si) сақталуын дәлелдеді. Дамытылған технология арзан әрі қолжетімді ресурстарды жаңа материалдарына айналдырудың жоғары тиімділігін және өміршеңдігін көрсетеді, олар электрохимиялық энергия жинақтағыштарында, каталитикалық процестерде және басқа да озық технологиялық салаларда қолдануға жарамды. Осылайша, ұсынылған тәсіл экологиялық міндеттерді шешуді және жоғары қосылған құны бар функционалдық наноматериалдарды жасауды үйлестіреді, бұл оның қазіргі заманғы химия және энергетика өнеркәсібінің орнықты дамуы үшін маңыздылығын айқындайды.

Түйін сөздер: көміртек–кремний материалдары, күріш қауызы, көмірлендіру, белсендіру, наноқұрылымдандыру, аморфты кремний диоксиді, қалдықтарды кәдеге жарату, энергия сақтау құрылғылары

© А.Ж. Мутушев*, А.Б. Сейсенова, О.С. Капизов, А.М. Нуралы, Д.К. Муханов, 2025.
Scientific Center for New Technologies, AlAkSan, Алматинский технологический университет, Алматы, Казахстан;
Center for Advanced Science and Technology, Алматы, Казахстан.
E-mail: alibek_090@mail.ru

ИНТЕГРИРОВАННАЯ ТЕХНОЛОГИЯ ПОЛУЧЕНИЯ УГЛЕРОДНО-КРЕМНИЕВЫХ НАНОКОМПОЗИТОВ ИЗ БИООТХОДОВ И МЕТАЛЛУРГИЧЕСКИХ ШЛАМОВ

Мутушев Алибек Жумабекович — PhD, Ведущий научный сотрудник, Scientific Center for New Technologies, AlAkSan, ассистент-профессор, кафедра Химии, химической технологии и экологии, Алматинский технологический университет, Алматы, Казахстан,

E-mail: alibek_090@mail.ru h, ORCID ID: https://orcid.org/0000-0002-5047-5608;
Сейсенова Ақнұр Бердибаевна — PhD, Руководитель проекта (PI), Scientific Center for New Technologies, AlAkSan, Алматы, Казахстан,
E-mail: z_aknura@mail.ru, ORCID ID: https://orcid.org/0000-0002-8981-307X;
Капизов Омирзак Сембиулы — магистр техники и технологии, старший научный сотрудник, Scientific Center for New Technologies, AlAkSan, Алматы, Казахстан,
E-mail: ok-sir@mail.ru , ORCID ID: https://orcid.org/0000-0002-1979-7625;
Нуралы Асия Мамбеткызы — PhD, ведущий научный сотрудник, Scientific Center for New Technologies, Center for Advanced Science and Technology, ассистент-профессор, кафедра Химии, химической технологии и экологии, Алматинский технологический университет, Алматы, Казахстан,
E-mail: assiya488@gmail.com , ORCID ID: https://orcid.org/0000-0002-2323-0365;
Муханов Даурен Кабракимович — старший научный сотрудник, Scientific Center for New Technologies, AlAkSan, Алматы, Казахстан,
E-mail: dd_511@mail.ru , ORCID ID: https://orcid.org/0009-0002-1645-8625.

Аннотация: В представленной работе описана интегрированная и экологически ориентированная технология получения наноструктурированных углеродно-кремниевых композитных материалов из возобновляемого сырья растительного происхождения (шелуха риса) и промышленных отходов. Такой подход является актуальным с точки зрения решения задач комплексной утилизации биомассы и отходообразующего сырья, а также разработки новых функциональных материалов, востребованных в современных энергетических и технологических приложениях. Предлагаемая методика включает последовательные стадии карбонизации, активации и деминерализации, обеспечивающие формирование развитой пористой структуры и высоких значений удельной поверхности. Карбонизацию проводили в инертной атмосфере CO_2 при температурах 300–1000 °C, процесс активации осуществлялся в диапазоне 600–1200 °C. Для эффективного удаления минеральных примесей применялся модифицированный аппарат Сокслета, что позволило значительно повысить степень очистки продукта. Полученный диоксид кремния находился в рентгеноаморфном состоянии с максимумом диффузного пика при $2\theta = 24°$, обладая удельной поверхностью 120–150 м²/г и поровым объёмом 0,5–0,8 см³/г. Элементный анализ методом РФА подтвердил чистоту SiO_2 до 99,7%. СЭМ-микроскопия показала равномерное распределение частиц со средним размером порядка 50 нм, а ИК-спектроскопия подтвердила наличие силоксановых связей (Si–O–Si). Разработанная технология демонстрирует высокую эффективность и перспективность преобразования дешёвых и доступных ресурсов в материалы, пригодные для применения в электрохимических накопителях энергии, каталитических процессах и других передовых технологических областях. Таким образом, представленный подход сочетает в себе решение экологических задач и создание функциональных наноматериалов с высокой добавленной стоимостью, что подчёркивает его значимость для устойчивого развития современной химической и энергетической промышленности.

Ключевые слова: углеродно-кремниевые материалы, рисовая шелуха, карбонизация, активация, наноструктурирование, аморфный кремнезем, утилизация отходов, накопители энергии

Introduction. In the 21st century, the problem of industrial and biological waste utilization has acquired strategic importance in the context of sustainable development. According to international analytical agencies, more than 700 million tons of metallurgical waste are generated annually worldwide, including blast furnace and steelmaking slags, sludges, and dust fractions containing significant amounts of silicon, aluminum, iron, and other elements. A large share of these materials is either inefficiently processed or stockpiled, forming technogenic landscapes that pose a threat of soil, water, and air pollution with heavy metals and fine particulate matter. Equally urgent is the issue of biomass processing — agricultural residues, wood waste, and organic by-products of the food industry. The global volume of biological waste exceeds 3 billion tons per year, and its uncontrolled burning or decomposition is accompanied by emissions of greenhouse gases, including methane and carbon dioxide, thereby exacerbating global climate change.

At the same time, there is a rapid increase in demand for energy storage systems (ESS), driven by the development of renewable energy, electric transport, portable electronics, and distributed power grids. According to BloombergNEF forecasts, by 2035 the global ESS market will grow more than 15-fold compared to 2020. This will require a substantial increase in the production of batteries with high specific capacity, long service life, and the availability of raw materials.

According to estimates, between 7 and 10 billion tons of waste are generated globally each year, of which approximately 2 billion tons account for municipal solid waste (MSW) (Bioenergy International). More recent data confirm that the global MSW volume in 2019 amounted to between 2.29 and 3.13 billion tons, which is 30–50% higher compared to 2004–2019 levels (PMC). Other projections suggest that by 2050 this volume could nearly double, reaching almost 4 billion tons if the trend continues (Statista).

Agricultural residues alone constitute a significant fraction of waste: over 80% of the biomass from cultivated crops remains unused. For example, about 998 million tons of agricultural waste are generated annually, including 709.2 million tons of straw and 673.3 million tons of rice straw.

For every ton of steel produced, about 0.15 tons of slag are generated — regardless of the production technology (Wikipedia). In China, the world's largest steel producer, the annual volume exceeds 120 million tons (ScienceDirect). In developed countries, slag recycling rates exceed 90%, whereas in China they are around 20%.

The United Nations Environment Programme (UNEP) projects that by 2050, waste-related costs — including biodiversity loss, greenhouse gas emissions, and mortality — will reach USD 640 billion annually, representing a 75% increase compared to 2020.

Relevance of Waste Recycling for Si–C Material Production

In recent years, increasing attention has been paid to the integrated recycling of biological and metallurgical waste for the production of carbon–silicon (Si–C) composites for energy storage systems (ESS). Biomass serves as a sustainable source of carbon matrices and SiO_2 precursors, while metallurgical slags and sludges act as concentrates of SiO_2 and associated metals. Studies have shown that controlled microand mesoporosity, optimal interlayer spacing in carbon structures, and core–shell architectures (Si@C) can enhance reversible capacity, high-rate retention, and structural stability (Chen et al., 2025; Abe et al., 2022; Wang et al., 2022).

Classical and modern approaches include:

Conversion of $SiO_2 \rightarrow Si$ followed by the formation of $C/SiO_x/Si$ composites;

Use of gradient structures and elastic binders to compensate for volumetric expansion;

Post-treatment and doping to optimize electrical conductivity and stabilize the solid–electrolyte interphase (SEI) (Wang et al., 2025; Jin et al., 2025).

Research Gaps and Challenges

Despite a significant number of studies dedicated to the processing of biomass and metallurgical waste separately, the integration of these technologies into a unified process chain remains underdeveloped. Treating the production of carbon and silicon-containing materials as independent processes limits the potential for the combined utilization of heterogeneous feedstocks and hinders the implementation of a closed-loop production concept.

Aim and Objectives of the Study

The aim of this work is to develop and scale up an integrated technology for processing biological and metallurgical waste to obtain high-performance carbon–silicon materials for energy storage systems, in accordance with international standards and sustainability principles.

The research objectives are to:

Analyze and select promising types of biological and metallurgical feedstocks with high carbon and silicon content.

Develop a laboratory-scale technological process for synthesizing Si–C materials, including preparation, modification, and composite formation.

Optimize synthesis parameters to achieve high electrochemical performance.

Scale up from laboratory setups to pilot production, assessing process scalability.

Evaluate the industrial potential and economic feasibility of implementing the technology, including environmental indicators and compliance with international standards.

Methods and materials.

2.1 Raw Materials

Various types of biowaste with significant potential for recycling and subsequent industrial application were used as raw materials.

Nut shells (hazelnut, walnut, and others) represent a hard fruit shell with a developed porous structure and high mechanical strength, making them promising feedstocks for the production of sorbents and activated carbons. According to research data, after low-temperature pyrolysis, hazelnut shells exhibit a specific surface area of approximately 19.3 m^2/g with a bulk density of 0.40 g/cm^3, while walnut shells have a specific surface area of 19.7 m^2/g with a density of 0.47 g/cm^3. The use of this type of raw material enables environmentally safe disposal of biodegradable components, reduces the strain on non-renewable natural resources, and yields effective adsorbent materials (https://apni.ru/article/1670-svojstva-produktov-retsiklinga-rastitelnikh?utm_source)

Rice husk is the hard outer shell of the rice grain and consists primarily of amorphous silica and lignin. The mass fraction of the husk is about 20% of the grain weight, and the annual global volume of rice husk generation amounts to hundreds of millions of tons. The high silica content leads to low biodegradability under natural conditions and renders thermal destruction ineffective, resulting in waste accumulation and environmental risks. At the same time, rice husk has high technological value: during processing, an amorphous silica-based nanoporous structure is formed, which can be used in the production of building materials, fertilizers, sorbents, composites, and activated carbons. Pyrolysis products include a solid silicon–carbon phase (~50–55% carbon, ~40–45% silicon dioxide) used as a filler, sorbent, and feed additive, as well as an organic condensate and a gaseous phase containing valuable chemical compounds suitable for further utilization.

Wood sawdust is a by-product of the woodworking industry, consisting of small particles of wood containing approximately 70% polysaccharides (cellulose and hemicellulose) and about 27% lignin. The elemental composition of sawdust includes approximately 50% carbon, 6% hydrogen, 44% oxygen, and about 0.1% nitrogen. Sawdust is a sought-after raw material for the production of biofuels (briquettes and pellets), serves as a substrate for mushroom cultivation, is used as mulch, animal bedding, organic fertilizer, and as a feedstock for the manufacture of pressed products.

Table 1- Comparison table

Type of biowaste	Main components	Key Features	Possible application
Nut shells	Organic matrix, carbon structure	Specific surface area ~19–20 m^2/g, density 0.4–0.5 g/cm^3	Sorbents, activated carbon, adsorption materials
Rice husk	Amorphous silicon dioxide, lignin	Nanoporous structure, high silicon content	Fillers, sorbents, composites, fertilizers, bioenergy
Wood waste	Cellulose, hemicellulose, lignin, carbon	Polymer composition, biodegradation, organic matrix	Fuel, fertilizers, mycelial substrates, pressed products

Metallurgical Waste

Metallurgical slag is a by-product of iron and steel production, representing a complex mixture of silicate and oxide compounds formed during metal smelting processes. The chemical composition of slag is determined by the production technology and the raw materials used.

Blast furnace slags are characterized by a calcium oxide (CaO) content of 29–47%, silicon dioxide (SiO_2) of 27–43%, aluminum oxide (Al_2O_3) of 4–14%, magnesium oxide (MgO) of 5–15%, iron oxide (FeO) of 0.2–0.6%, manganese oxide (MnO) of 0.1–9%, and sulfur of 0.6–2.2%.

Steelmaking (converter) slags contain approximately CaO ~40–55%, SiO_2 ~7–18%, Al_2O_3 ~2–6%, MgO ~6–10%, FeO/Fe_2O_3 ~12–28%, MnO ~13–14%, and sulfur ~1–1.9%.

The physical characteristics of slags include a true density of about 2900–3000 kg/m^3 and bulk density of 2200–2800 kg/m^3. They exhibit increased porosity and frost resistance. Slag is a multiphase silicate system with variable chemical and mineralogical composition, often containing metallic inclusions (up to 5%). Steelmaking slags have relatively low hydraulic activity.

Slags are classified according to their basicity based on the CaO/SiO_2 ratio: values below 1 indicate acidic slags, while values above 1 indicate basic slags.

Applications of metallurgical slags include:

Road and civil construction (crushed stone, base layers, and road surfacing);

Cement industry (granulated blast furnace slag as a component of slag Portland cement, with a share of up to 35% in standard Portland cement and up to 80% in special blends);

Production of dry building mixes, silicate concretes, and for soil stabilization;

Metallurgical processes (e.g., in open-hearth production) to reduce lime consumption and lower CO_2 emissions.

Table 2 - Comparison table

Waste	Composition	Main characteristics	Application
Metallurgical slag	CaO, SiO_2, Al_2O_3, MgO, Fe/Mn oxides, S	Density ~2200–3000 kg/m^3; porosity, complex composition	Roads, cement, building mixtures, soil stabilization
Quartz concentrates	Almost pure SiO_2, impurities ≤30 ppm	Size 100–300 µm, ultra-low impurities	Semiconductor, glass, crystal and quartz technology
Silicon-containing sludge	SiO_2 + Fe_2O_3, Al_2O_3 etc., sometimes >98% Si	High Si content, porous structure	Agglomerate-blast raw material component; moisture regulation; charge; valuable metals

2.2. Pretreatment Methods

The preliminary preparation of raw materials is an important stage of technological processes, ensuring uniform composition, improved physicochemical properties of the materials, and increased efficiency of their subsequent processing.

Mechanical preparation.

Drying reduces the moisture content of the raw materials to the target level, preventing caking, improving grinding efficiency, and facilitating material transport (https://burondt.ru/files/TextEtk/EtkDocsFile2228.pdf). Comminution (crushing and milling) is carried out in crushers, mills, and other equipment to increase the specific surface area and enhance reactivity (Wills et al., 2016). Screening is performed using sieve analysis or air classification to separate fractions with a specified particle size distribution (https://publications.rwth-aachen.de/record/444642/files/59_Innovative_approaches.pdf ; https://en.wikipedia.org/wiki/Mineral_processing).

Removal of impurities and dechlorination (for metallurgical waste).

Dechlorination is a thermal treatment of the material in an inert or reducing atmosphere to remove chlorine-containing compounds, which may form during interactions with fluxes and salts (https://chm.pops.int/Portals/0/docs/from_old_website/documents/meetings/cop_1/meetingdocs/langs/inf1_7/INF-7r.pdf ; Höber et al., 2022). Removal of impurities (heavy metals, iron, aluminum, etc.) is achieved using magnetic separation, flotation, or hydrometallurgical processing, including reduction roasting followed by leaching (Binnemans et al., 2020; Faisal et al.,2025; Devi et al., 2021).

Table 3 – Main pretreatment methods for waste

Processing stage	Purpose and benefits
Drying	Reduction of moisture, improvement of grinding, prevention of caking and spoilage
Crushing (grinding)	Increase of specific surface area, increase of reaction activity
Sorting	Optimization of granulometric composition, improvement of homogeneity of the mixture
Dechlorination	Removal of chlorine-containing contaminants, reduction of toxicity
Removal of impurities	Purification from metals and undesirable components, improvement of the quality of the final raw materials

2.3 Methods for Synthesizing Carbon–Silicon Materials

The synthesis of carbon–silicon (C–Si) materials encompasses a variety of technologies aimed at producing composites and nanostructures with tailored properties, such as high porosity, specific functionality, and improved thermal conductivity.

Carbonization / Pyrolysis of Biomass

This method is based on the thermal treatment of organic feedstocks (biomass) in an oxygen-free environment to obtain a carbon matrix. The processes include pyrolysis, hydrothermal carbonization (HTC), and fast carbonization, enabling the production of carbon materials with controlled porosity and structure (https://en.wikipedia.org/wiki/Hydrothermal_carbonization?ysclid=meqny1isfl175057245), (Simonenko et al., 2013). In particular, hydrothermal carbonization (HTC) is carried out under pressure at around 180 °C, allowing the processing of wet biomass without prior drying (https://en.wikipedia.org/wiki/Sol%E2%80%93gel_process?ysclid=meqo6ake2g520290754).

Reduction Reactions and Silicon Modification

C–Si composites can be obtained via carbothermal reduction, in which silica (SiO_2) is reduced with carbon to form silicon carbide (SiC) or silicon-containing structures. Hybrid approaches are particularly effective, combining preliminary sol–gel preparation of the initial SiO_2–C mixture with carbothermal synthesis at 1200–1500 °C in vacuum (Molkenova et al., 2015).

Nanostructuring (Sol–Gel, Mechanochemistry, Plasma Chemistry)

Sol–gel method — a wet-chemical technology in which hydrolysis and polycondensation of alkoxide precursors (e.g., TEOS) form a granular or network gel structure. Subsequent carbonization and heat treatment can yield nanocrystalline SiC structures (Bapat et al., 2004).

Mechanochemical activation — involves the use of high-energy ball milling, resulting in ultra-fine particle size reduction, enhanced chemical reactivity of mixtures, and further C–Si structure formation without the use of a liquid phase.

Plasma-chemical methods — enable the generation of silicon nanoparticles with controlled sizes (20–80 nm) and crystalline orientation through low-temperature plasma discharges.

Table 4 – Comparison of methods for synthesizing carbon–silicon materials

Method	Description
Carbonization / HTC	Production of carbon matrix from bio-raw materials. HTC allows working with wet biomass
Carbothermic reduction	Synthesis of SiC from SiO_2 and carbon source at high temperatures
Sol-gel + carbothermy	Methods are combined: gel formation and carbonization to control the structure of the Si–C product
Mechanochemistry	High-energy milling accelerates reactions, reduces size and activates the material
Plasmachemistry	Generation of silicon nanoparticles with a narrow size distribution in plasma

2.4 Methods of Analysis and Testing

A comprehensive investigation of carbon–silicon materials requires elemental, structural, surface, and electrochemical analysis techniques. These methods enable the assessment of composition, morphology, texture, and functional properties of synthesized products.

Chemical Analysis

X-Ray Fluorescence (XRF) — a rapid, non-destructive technique for qualitative and quantitative determination of the elemental composition of samples. Particularly useful for the analysis of metals, glass, ceramics, and construction materials.

Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES, ICP-AES) — a highly sensitive method with a broad dynamic range, applicable to various media (liquids, solids after dissolution), commonly used in mining, metallurgy, and materials science.

Structural Analysis

X-Ray Diffraction (XRD) — a key method for phase identification, crystallite size determination, and detection of structural changes in materials.

Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) — enable visualization of morphology, nanostructures, and, in the case of SEM–EDS, provide elemental composition at the microscale.

Surface Analysis

Brunauer–Emmett–Teller (BET) method — determines the specific surface area and porosity of a material based on gas adsorption isotherms.

Fourier-Transform Infrared Spectroscopy (FTIR) and Raman Spectroscopy — sensitive to chemical bonds and functional groups, complementing each other in identifying structural and electronic characteristics.

Electrochemical Testing

Cyclic Voltammetry (CV) — used to study electrochemical activity, reaction kinetics, and determine material capacitance.

Charge–discharge tests, including those at specific currents and potentials, are employed to assess capacitance, cycling stability, and energy storage efficiency.

Electrochemical Impedance Spectroscopy (EIS) — analyzes system impedance to evaluate resistances, capacitance, and kinetics in electrode and interfacial processes.

Table 5 — Analytical Methods and Their Applications

Method	Application
XRF	Rapid elemental analysis, non-destructive, suitable for a wide range of samples
ICP-OES	High sensitivity, broad dynamic range, requires sample dissolution
XRD	Phase identification, crystalline structure determination
SEM / TEM	Morphology, nanostructure, elemental composition (SEM–EDS)
BET	Specific surface area and porosity measurements
FTIR / Raman	Identification of functional groups and chemical bonds
CV, charge–discharge, EIS	Electrochemical activity, capacitance, kinetics, and resistance

Results.

The synthesis process involves three treatment stages.

Carbonization is the first stage, during which carbon–silicon composites (CSCMs) are typically formed via pyrolysis of the raw material in the temperature range of 600–900 °C under an inert atmosphere (usually nitrogen). The main objective of carbonization is the removal of volatile components, maximization of the specific content of carbon and silicon, and the production of a material with sufficiently high specific surface area and porosity.

Activation is the second stage, involving the exposure of the carbon material to oxidizing gases such as CO_2 or steam in the temperature range of 600–1200 °C. This process removes the more disordered carbon fraction and leads to the formation of a well-developed porous structure.

To remove mineral impurities, demineralization is carried out using a modified Soxhlet extractor.

The combination of carbonization, activation, and demineralization processes ensures the formation of the required structural and physicochemical properties of the final products.

The synthesis was performed by carbonizing and activating plant-based materials in a custom-designed furnace at temperatures ranging from 300 to 1000 °C for 30–90 minutes under a CO_2 atmosphere, without air access. Figures 2 and 3 show the pilot unit for producing carbonized material from plant feedstock (5 kg h^{-1}).

The reactor is designed for the sequential execution of feedstock dehydration, accompanied by depolymerization and partial decomposition in the temperature range of 650–800 °C. This stage includes water removal (up to 280 °C) and decarboxylation with the formation of pyrolysis tars through concurrent dehydration processes.

X-Ray Diffraction (XRD) Analysis

The results of the sample study are presented in Figure 1. The obtained silica powder is in an X-ray amorphous state, as confirmed by XRD analysis. The X-ray diffraction patterns exhibit a single broad diffuse peak at $2\theta = 24°$, characteristic of the amorphous structure of rice husk silica, whereas for amorphous silicon dioxide the maximum of the diffuse peak is typically observed at $2\theta = 30°$. The observed broad curve (Fig. 1) with a maximum intensity at 24.0° / 2θ further confirms the amorphous structure of the obtained silica.

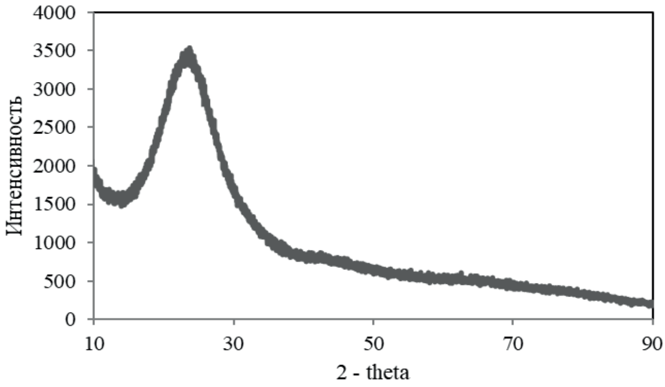
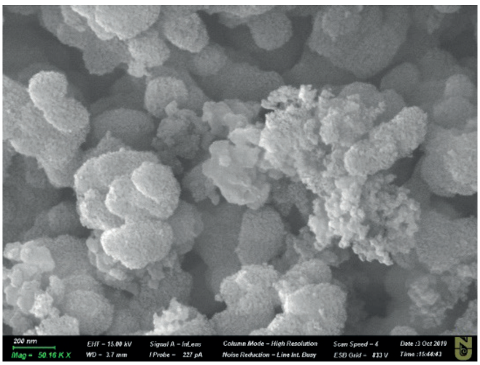


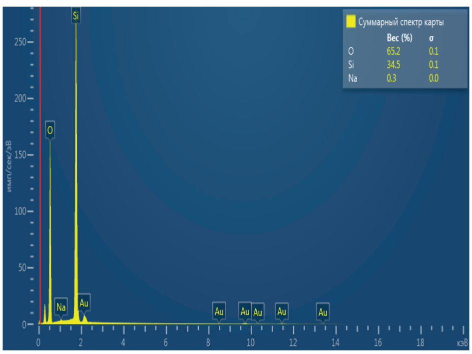
Figure 1 – XRD pattern of SiO_2 obtained from rice husk

Scanning Electron Microscopy with Energy Dispersive Spectroscopy (SEM-EDS)

The SEM-EDS results for SiO_2 derived from rice husk are presented in Figure 2.

The morphology of the silica obtained from pretreated rice husk, as shown in the SEM images, demonstrates complete transformation of the pretreated husk into an amorphous nanomaterial with an average particle size of approximately 50 nm. According to the EDS analysis, the silica contains 34.5 wt.% silicon and 65.2 wt.% oxygen.





Element	wt. %
Si	34,5
O	65,2
Na	0,3

Figure 2 – SEM micrographs of SiO_2 and EDS elemental analysis

Low-temperature nitrogen adsorption

The synthesized SiO_2 from rice husk exhibited a high specific surface area and significant pore volume.

The results obtained via low-temperature nitrogen adsorption measurements revealed that the specific surface area of the silica samples was 120 and 150 m²/g, respectively.

The pore volume of the SiO_2-RH1 and SiO_2-RH3 samples increased from 0.5 cm³/g to 0.8 cm³/g, while the average pore diameter decreased from 26.4 nm to 18.4 nm.

The specific surface areas, pore diameters, and pore volumes of the silica samples are summarized in Table 6.

Table 6 – Surface area and pore characteristics of silica samples

Sample	Surface area Sspecific., m2/g	Pore characteristics (DFT method)	
		Vpore, cm3/g	Dпор, нm
SiO2 RH 1	120	0.5	26.4
SiO2 RH 3	150	0.8	18.4

Results and Analysis of Samples by XRF

The elemental composition of the initial rice husk ash (RHA) and the purified SiO_2, determined by X-ray fluorescence (XRF), is presented in Table 7.

The content of all elements is given in weight percent (%).

Table 7 – X-ray fluorescence analysis results for rice husk ash and silica samples

Elemental composition	Rice husk ash			Pure SiO2 sample		
	RHA1	RHA2	RHA3	SiO21	SiO22	SiO23
SiO2	83.8	73.9	84.4	98.2	99.1	99.7
Cl	–	3.1	–	1.8	0.8	–
K2O	8.6	18.5	4.8	–	–	0.1
CaO	6.7	2.8	8.3	–	–	0.2
MnO	0.2	0.6	–	–		–
Fe2O3	0.6	0.6	2.5	–	0.02	–
ZnO	0.2	0.5	–	–	0.1	0.04

Fourier-Transform Infrared Spectroscopy (FTIR) Results

The functional groups in the sample were identified using a Thermo Scientific FTIR Nicolet 6700 equipped with an attenuated total reflectance (ATR) accessory. Spectra were recorded with a resolution of 4 cm^{-1} over the range of 4000–400 cm^{-1}.

Changes in the functional groups of the samples were studied by analyzing the FTIR spectra (Figures 3). The absorption peaks located at 1055 cm^{-1} and 1058 cm^{-1} are attributed to the stretching vibrations of C–OH and siloxane (Si–O–Si) bands, which were observed in the FTIR spectra of untreated rice husk (RH) samples. The peak at 2921 cm^{-1} corresponds to the symmetric and asymmetric stretching vibrations of aliphatic C–H bonds in $–CH_3$ and $–CH_2$ groups, originating from cellulose, hemicellulose, and lignin structures, respectively.

The FTIR spectra of the extracted silica after preliminary acid washing showed the same absorption peak at 1055 cm^{-1} as in the raw RH, indicating that the pretreatment method did not significantly alter the surface properties of the silica.

Discussion. Synthesis of Carbon–Silicon Materials from Biomass, Specifically Rice Husk

The synthesis of carbon–silicon materials from biomass, particularly rice husk, has demonstrated high efficiency when employing sequential stages of carbonization, activation, and demineralization.

In the first stage, carbonization at 600–900 °C in a nitrogen atmosphere removed volatile components and concentrated carbon and silicon in the product, resulting in the formation of a developed porous structure. The second stage, activation at 600–1200 °C in a CO_2 atmosphere, further enhanced porosity by removing amorphous carbon and generating a more ordered microporous architecture. The final stage, demineralization using a modified Soxhlet extractor, effectively removed mineral impurities, including potassium, calcium, iron, and manganese compounds, as confirmed by XRF analysis.

X-ray diffraction (XRD) revealed that the obtained silica is X-ray amorphous, as evidenced by a single broad diffuse peak at approximately $2\theta \approx 24°$, characteristic of amorphous SiO_2 derived from plant-based raw materials. This result is consistent with literature reports indicating that silica from rice husk is predominantly amorphous in its native form.

Scanning electron microscopy (SEM-EDS) showed that the synthesized silica possesses a nanostructured morphology with particle sizes of about 50 nm. EDS analysis indicated a silicon content of 34.5 wt% and an oxygen content of 65.2 wt%, corresponding to the stoichiometry of silicon dioxide. The presence of sodium and trace amounts of other elements is attributed to residual minerals not fully removed during demineralization.

BET surface area analysis demonstrated that the SiO_2 samples achieved a specific surface area of up to 150 m^2/g, with a pore volume of 0.8 cm^3/g and an average pore diameter of about 18 nm. The reduction in average pore diameter compared with less-activated samples indicates the development of a more advanced mesoporous structure, beneficial for use as a matrix for silicon-containing composites in lithium-ion battery anodes.

XRF analysis confirmed that after acid purification, the silica purity reached 99.7%. The significant reduction in K_2O, CaO, and Fe_2O_3 contents compared with rice husk ash demonstrates the effectiveness of the pretreatment process.

FTIR spectroscopy identified the main functional groups. Absorption peaks in the range of 1055–1058 cm^{-1} correspond to the stretching vibrations of Si–O–Si bonds typical for amorphous silica. The persistence of this peak after acid washing indicates that chemical purification does not disrupt the primary SiO_2 structure. The disappearance or attenuation of peaks associated with C–H vibrations (2921 cm^{-1}) indicates the removal of organic components.

Overall, these results demonstrate that the proposed technological scheme enables the production of high-purity, nanostructured, amorphous silica with a developed porous structure, making it promising for the fabrication of carbon–silicon composites in electrochemical energy storage devices, as well as in adsorption and catalytic applications.

Conclusion. In the course of the present study, a technological scheme for producing high-purity amorphous silica from rice husk was developed and implemented, employing sequential processes of carbonization, activation, and demineralization. The following findings were established:

Carbonization in the temperature range of 600–900 °C under an inert atmosphere effectively removed volatile components, increased the relative content of carbon and silicon, and facilitated the formation of a well-developed porous structure in the initial material. Activation with carbon dioxide at 600–1200 °C promoted the development of micro- and mesoporous structures through the removal of disordered carbon. Demineralization using a modified Soxhlet extractor ensured the removal of alkali and alkaline earth metal impurities, enabling an increase in silica purity to 99.7%.

X-ray diffraction confirmed the amorphous state of the obtained SiO_2, while SEM-EDS analysis revealed a nanoscale particle size (~50 nm) and a silicon content of 34.5 wt%.

Low-temperature nitrogen adsorption measurements indicated a specific surface area of 120–150 m^2/g, a pore volume of 0.5–0.8 cm^3/g, and an average pore diameter of 18.4–26.4 nm.

The practical significance of the developed technology lies in the potential for scaling up the processes to pilot-scale units (5 kg/h) and integrating them into industrial lines for agricultural waste processing. This opens the way for comprehensive waste utilization with the production of valuable nanostructured materials.

Future research prospects include:

• modification of the obtained silica to improve its electrochemical properties;

• integration of the material into hybrid electrodes for supercapacitors and lithium-ion batteries;

• optimization of the energy efficiency of the technological processes.

References

Chen Y., Cui J., Wang S., Xu W., Guo R. (2025) Biomass-Derived Hard Carbon Materials for High-Performance Sodium-Ion Battery. Coatings, 15, 156. https://doi.org/10.3390/coatings15020156 (in Eng.)

Abe Y., Tomioka M., Kabir M. et al. (2022) Role of SiOx in rice-husk-derived anodes for Li-ion batteries. Sci Rep 12, 975 https://doi.org/10.1038/s41598-022-04979-5 (in Eng.)

Wang L., Chen L., Liu W. et al. (2022) Recovery of titanium, aluminum, magnesium and separating silicon from titanium-bearing blast furnace slag by sulfuric acid curing—leaching. Int J Miner Metall Mater 29. — P.1705–1714 https://doi.org/10.1007/s12613-021-2293-3 (in Eng.)

Wang K., Zheng H., Li S. et al. (2025) Study on carbonation behavior and carbon footprint of steel slag-calcium carbide slag-desulfurization gypsum composite system. Sci Rep 15, 15199 https://doi.org/10.1038/s41598-025-99803-1 (in Eng.)

Jin B., Liao L., Shen X., Mei Z., Du Q., Liang L., Lei B., Du J. Advancement in Research on Silicon/Carbon Composite Anode Materials for Lithium-Ion Batteries. Metals 2025, 15, 386. https://doi.org/10.3390/met15040386 (in Eng.)

Svojstva produktov reciklinga rastitelnyh othodov, poluchennyh metodom nizkotemperaturnogo piroliza i gazifikacii [Elektronnyj resurs]. https://apni.ru/article/1670-svojstva-produktov-retsiklinga-rastitelnikh?utm_source (in Eng.)

«ITS 15-2021 utilizaciya i obezvrezhivanie othodov (krome termicheskih sposobov)». [Elektronnyj resurs]. https://burondt.ru/files/TextEtk/EtkDocsFile2228.pdf (in Eng.)

Wills B.A., Finch J. Mineral Processing Technology. 8th ed., Elsevier, 2016. https://shop.elsevier.com/books/wills-mineral-processing-technology/wills/978-0-08-097053-0 (in Eng.)

RWTH Aachen University. Innovative approaches for an optimized processing of metallurgical slags, 2014. [Elektronnyj resurs]. https://publications.rwth-aachen.de/record/444642/files/59_Innovative_approaches.pdf (in Eng.)

Wikipedia. "Mineral processing." https://en.wikipedia.org/wiki/Mineral_processing (in Eng.)

Stockholm Convention. Technical Guidelines on the Environmentally Sound Management of Wastes with POPs, 2005. https://chm.pops.int/Portals/0/docs/from_old_website/documents/meetings/cop_1/meetingdocs/langs/inf1_7/INF-7r.pdf (in Eng.)

Höber L.; Witt K.; Steinlechner S. Selective Chlorination and Extraction of Valuable Metals from Iron Precipitation Residues. Appl. Sci. 2022, 12, 3590. https://doi.org/10.3390/app12073590 (in Eng.)

Binnemans K., Jones P.T., Manjón Fernández, Á. et al. Hydrometallurgical Processes for the Recovery of Metals from Steel Industry By-Products: A Critical Review. J. Sustain. Metall. 6. — P. 505–540 (2020). https://doi.org/10.1007/s40831-020-00306-2 (in Eng.)

Faisal Mahmoo, Mujahid Ali, Mustafa Khan, Christian Fabrice Magoua Mbeugang, Yusuf Makarfi Isa, Alexander Kozlov, Maxim Penzik, Xing Xie, Haiping Yang, Shihong Zhang, Bin Li,A review of biochar production and its employment in synthesizing carbon-based materials for supercapacitors, Industrial Crops and Products,Volume 227, 2025, 120830,ISSN0926-6690, https://doi.org/10.1016/j.indcrop.2025.120830 (in Eng.)

A Mamta Devi, Sachin Rawat, Swati Sharma, A comprehensive review of the pyrolysis process: from carbon nanomaterial synthesis to waste treatment, Oxford Open Materials Science, Volume 1, Issue 1, 2021, itab014, https://doi.org/10.1093/oxfmat/itab014

Hydrothermal carbonization. Wikipedia Википедия; https://en.wikipedia.org/wiki/Hydrothermal_carbonization?ysclid=meqny1isfl175057245 (in Eng.)

Simonenko E.P., Simonenko N.P., Derbenev A.V. et al. Synthesis of nanocrystalline silicon carbide using the sol-gel technique. Russ. J. Inorg. Chem. 58, - P.1143–1151 (2013). https://doi.org/10.1134/S0036023613100215 (in Eng.)

Sol–gel processWikipedia, https://en.wikipedia.org/wiki/Sol%E2%80%93gel_ process?ysclid= meqo6ake2g520290754

Anara Molkenova, Izumi Taniguchi,Preparation and characterization of SiO2/C nanocomposites by a combination of mechanochemical-assisted sol–gel and dry ball milling processes,Advanced Powder Technology,Volume 26, Issue 2,2015. — P. 377-384, ISSN 0921-8831, https://doi.org/10.1016/j.apt.2014.11.013. (in Eng.)

Ameya Bapat, Curtis Anderson, Christopher R Perrey, C Barry Carter, Stephen A. (2004) Campbell and Uwe Kortshagen. Plasma synthesis of single-crystal silicon nanoparticles for novel electronic device applications. Plasma Phys. Control. Fusion 46 B97. DOI 10.1088/0741-3335/46/12B/009. (in Eng.)

Publication Ethics and Publication Malpractice in the journals of the Central Asian Academic Research Center LLP

For information on Ethics in publishing and Ethical guidelines for journal publication see http://www.elsevier.com/publishingethics and http://www.elsevier.com/journal-authors/ethics.

Submission of an article to the journals of the Central Asian Academic Research Center LLP implies that the described work has not been published previously (except in the form of an abstract or as part of a published lecture or academic thesis or as an electronic preprint, see http://www.elsevier.com/postingpolicy), that it is not under consideration for publication elsewhere, that its publication is approved by all authors and tacitly or explicitly by the responsible authorities where the work was carried out, and that, if accepted, it will not be published elsewhere in the same form, in English or in any other language, including electronically without the written consent of the copyright-holder. In particular, translations into English of papers already published in another language are not accepted.

No other forms of scientific misconduct are allowed, such as plagiarism, falsification, fraudulent data, incorrect interpretation of other works, incorrect citations, etc. The Central Asian Academic Research Center LLP follows the Code of Conduct of the Committee on Publication Ethics (COPE), and follows the COPE Flowcharts for Resolving Cases of Suspected Misconduct (http://publicationethics. org/files/u2/New_Code.pdf). To verify originality, your article may be checked by the Cross Check originality detection service http://www.elsevier.com/editors/plagdetect.

The authors are obliged to participate in peer review process and be ready to provide corrections, clarifications, retractions and apologies when needed. All authors of a paper should have significantly contributed to the research.

The reviewers should provide objective judgments and should point out relevant published works which are not yet cited. Reviewed articles should be treated confidentially. The reviewers will be chosen in such a way that there is no conflict of interests with respect to the research, the authors and/or the research funders.

The editors have complete responsibility and authority to reject or accept a paper, and they will only accept a paper when reasonably certain. They will preserve anonymity of reviewers and promote publication of corrections, clarifications, retractions and apologies when needed. The acceptance of a paper automatically implies the copyright transfer to the Central Asian Academic Research Center LLP.

The Editorial Board of the Central Asian Academic Research Center LLP will monitor and safeguard publishing ethics.

Правила оформления статьи для публикации в журнале смотреть на сайте:
www:nauka-nanrk.kz
ISSN 2518-1483 (Online), ISSN 2224-5227 (Print)
http://reports-science.kz/index.php/en/archive

Ответственный редактор *А. Ботанқызы*
Редакторы: *Д.С. Аленов, Т. Апендиев*
Верстка на компьютере *Г.Д. Жадырановой*

Подписано в печать 3.09.2025.
Формат 60x88 1/8.
18,0 п.л. Заказ 3.

«Central Asian Academic Research Center» LLP
Алматы, Қонаев к-сі, 142