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MICROSTRUCTURAL AND ELEMENTAL CHARACTERISTICS OF CLAY AND MINERAL ADDITIVES FOR POTENTIAL USE IN CERAMIC MATERIALS

КЕРАМИКАЛЫҚ МАТЕРИАЛДАРДА ПОТЕНЦИАЛДЫ ҚОЛДАНУҒА АРНАЛҒАН САЗ ЖӘНЕ МИНЕРАЛДЫ ҚОСПАЛАРДЫҢ МИКРОҚҰРЫЛЫМДЫҚ ЖӘНЕ ЭЛЕМЕНТТІК СИПАТТАМАЛАРЫ

МИКРОСТРУКТУРНЫЕ И ЭЛЕМЕНТНЫЕ ОСОБЕННОСТИ ГЛИНЫ И МИНЕРАЛЬНЫХ ДОБАВОК ДЛЯ ПОТЕНЦИАЛЬНОГО ИСПОЛЬЗОВАНИЯ КЕРАМИЧЕСКИХ МАТЕРИАЛАХ

Z. Zhumadilova [1], **Ye. Orynbekov** [1], **M. Zhuginissov** [1], **A. Tolegenova** [1*], **A. Zhagifarov** [1], **R. Nurlybayev** [1]
[1]Satbayev University, Almaty, Kazakhstan
*Corresponding author: Aigerim Tolegenova, e-mail: a.tolegenova@satbayev.university

keywords:
clay, fly ash, silica fume, waste glass powder, microstructure, sem–eds analysis, ceramic materials.

ABSTRACT
This study investigates the microstructural and elemental characteristics of raw mineral materials used as potential additives in ceramic production. Clay, waste glass powder, silica fume, and fly ash were analyzed using scanning electron microscopy (SEM) coupled with energy-dispersive X-ray spectroscopy (EDS). The materials were ground and sieved to obtain particles smaller than 74 µm prior to analysis. SEM observations revealed distinct morphological features: clay particles exhibited irregular plate-like structures, waste glass showed angular fractured particles, silica fume consisted of extremely fine agglomerated particles, and fly ash displayed both spherical and irregular particles typical of combustion by-products. EDS analysis confirmed that silica and oxygen are the dominant elements in all materials, while aluminum, magnesium, potassium, and calcium were present in smaller amounts. The results indicate that these materials contain significant silica and aluminosilicate components, suggesting their potential use as mineral additives in ceramic systems and sustainable construction materials.

Түйінді сөздер:
балшық, ұшпа күл, микрокремнезем, шыны ұнтағы, микроқұрылым, SEM-EDS талдауы, керамикалық материалдар

ТҮЙІНДЕМЕ
Бұл жұмыста керамикалық материалдар өндірісінде қолдануға болатын минералдық қоспалардың микроқұрылымы мен элементтік құрамы зерттелді. Балшық, шыны ұнтағы, микрокремнезем және ұшпа күл сканерлеуші электрондық микроскопия (SEM) және энергиядисперсиялық рентгендік талдау (EDS) әдістерін қолдану арқылы зерттелді. Талдауға дейін материалдар ұсақталып,

74 мкм електен өткізілді. SEM нәтижелері бөлшектердің әртүрлі морфологиясын көрсетті: балшық пластинкалы құрылыммен сипатталады, шыны ұнтағы бұрышты сынған бөлшектерден тұрады, микрокремнезем өте ұсақ агломерацияланған бөлшектерден тұрады, ал ұшпа күл жану өнімдеріне тән сфералық және әртүрлі пішінді бөлшектерден тұрады. EDS талдауы зерттелген материалдардың негізгі элементтері кремний мен оттегі екенін, ал алюминий, магний, калий және кальций аз мөлшерде кездесетінін көрсетті. Алынған нәтижелер бұл материалдардың құрамында кремний және алюмосиликат компоненттерінің жоғары мөлшерде бар екенін көрсетеді және оларды керамикалық әрі экологиялық тұрақты құрылыс материалдарын өндіруде минералдық қоспа ретінде қолдану мүмкіндігін дәлелдейді.

Ключевые слова:
глина, зола уноса, микрокремнезем, стеклобой, микроструктура, SEM-EDS анализ, керамические материалы

АННОТАЦИЯ

В данной работе исследованы микроструктурные и элементные характеристики минеральных материалов, рассматриваемых как потенциальные добавки для производства керамических изделий. Глина, стеклобой, микрокремнезем и зола уноса были исследованы с использованием сканирующей электронной микроскопии (SEM) в сочетании с энергодисперсионным рентгеновским анализом (EDS). Перед анализом материалы были измельчены и просеяны через сито с размером ячеек 74 мкм. SEM-наблюдения показали различную морфологию частиц: глина характеризуется пластинчатой структурой, стеклобой представлен угловатыми фрагментированными частицами, микрокремнезем состоит из ультрадисперсных агломерированных частиц, а зола уноса содержит сферические и неправильной формы частицы. EDS-анализ показал, что основными элементами исследуемых материалов являются кремний и кислород, тогда как алюминий, магний, калий и кальций присутствуют в меньших количествах. Полученные результаты свидетельствуют о высоком содержании кремнеземных и алюмосиликатных компонентов, что подтверждает перспективность использования данных материалов в качестве минеральных добавок при производстве керамических и устойчивых строительных материалов.

INTRODUCTION

The increasing demand for sustainable construction materials has led to growing interest in the utilization of industrial by-products and waste materials in ceramic production. The recycling of industrial waste not only reduces environmental pollution but also promotes the conservation of natural resources and the development of environmentally friendly building materials (Ahmaruzzaman, 2010; Mohajerani et al., 2017). In recent years, the construction industry has been actively seeking alternative raw materials capable of partially replacing natural resources traditionally used in building materials production. The use of industrial by-products in ceramic technology is considered an effective approach to achieving circular economy principles by transforming waste streams into valuable secondary resources. It has been reported that the global production of fly ash exceeds 750 million tons per year, while a significant portion of this waste still remains unused or disposed of in landfills, creating environmental concerns (Ahmaruzzaman, 2010).

Among various industrial by-products, fly ash has received considerable attention due to its high content of silica and alumina, which typically account for 60–80% of its chemical composition. These oxides are similar to the mineral components of clay materials and therefore allow fly ash to be successfully incorporated into ceramic bodies. Previous studies have shown that the incorporation of 10–30% fly ash into clay bricks can improve compressive strength and durability while reducing firing energy consumption (Abbas et al., 2017; Sutcu et al., 2019; Yuan et al., 2022). In addition, the presence of fine spherical particles in fly ash contributes to improved packing density and microstructural development during sintering. This results in enhanced densification and reduced porosity of fired ceramic products, which ultimately improves their mechanical and durability properties.

Silica fume is another industrial by-product widely used in ceramic systems due to its extremely fine particle size and high amorphous silica content, which usually exceeds 85–95% SiO_2. Because of its ultrafine particles and high pozzolanic reactivity, silica fume can significantly influence the microstructure formation of ceramic materials during thermal processing. The addition of silica fume can influence sintering behavior and improve the physico-mechanical properties of fired clay materials depending on the firing conditions and additive content (Baspinar et al., 2010; Elmaghraby & Ismail, 2016). Previous studies have reported that the incorporation of 5–15% silica fume may enhance the densification of ceramic bodies, reduce pore size distribution, and increase compressive strength (Kumar et al., 2021). Moreover, the presence of highly reactive silica may promote the formation of additional silicate phases during firing, which contributes to the improvement of the overall structural integrity of ceramic products.

Waste glass is also considered a promising secondary raw material for ceramic production. Due to its amorphous structure and high silica content, which typically ranges from 65–75% SiO_2, crushed glass can act as a fluxing agent during firing and promote liquid phase formation at temperatures of approximately 900–1050 °C (Demir, 2009). The formation of a liquid phase during firing facilitates particle bonding and contributes to the densification of the ceramic matrix. Several studies have demonstrated that the incorporation of 10–25% waste glass in clay bricks contributes to waste recycling and improves the physical properties of ceramic products, including strength and water resistance (Akinwumi et al., 2019; Mao et al., 2020; Lesovik et al., 2024). Furthermore, the combined use of fly ash and waste glass in ceramic materials has been investigated as a sustainable approach for recycling industrial wastes and improving the overall performance of ceramic products (Karayannis et al., 2017; Yadav et al., 2024).

Despite numerous studies on the use of industrial by-products in ceramic brick production, the microstructural characteristics and elemental composition of the raw materials play a crucial role in determining their behavior during processing and firing. The morphology of particles, distribution of chemical elements, and interaction between mineral components significantly influence sintering mechanisms, phase formation, and the final properties of ceramic materials.

Therefore, the aim of this study is to investigate the microstructure and elemental composition of clay, crushed waste glass, silica fume, and fly ash using scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS) in order to evaluate their potential application in clay-based ceramic materials.

MATERIALS AND METHODS

The clay used in this study was obtained from the Burunday brick plant located in Almaty, Kazakhstan. Waste glass powder was sourced from recycled construction and demolition glass waste collected in Almaty, Kazakhstan. Silica fume was provided by LLP "QAZAQ Innotec" (Almaty, Kazakhstan), and its physical and chemical characteristics were determined based on the technical documentation supplied by the manufacturer.

Fly ash was obtained from the Pavlodar CHP-1 (Pavlodar, Kazakhstan). Fly ash is a by-product of coal combustion and is widely used as a pozzolanic material due to its high content of reactive aluminosilicate phases. Its chemical composition is characterized by a significant amount of silica (SiO_2), which allow it for use as an active mineral additive in cementitious and ceramic systems (Kuldeyev et al., 2025).

For the SEM and EDS investigations, the raw materials – clay, waste glass powder, silica fume, and fly ash – were first finely ground and then sieved through a 200-mesh sieve to obtain powder particles smaller than 74 μm (0.074 mm). The appearance of the raw materials used in the study is shown in Figure 1.

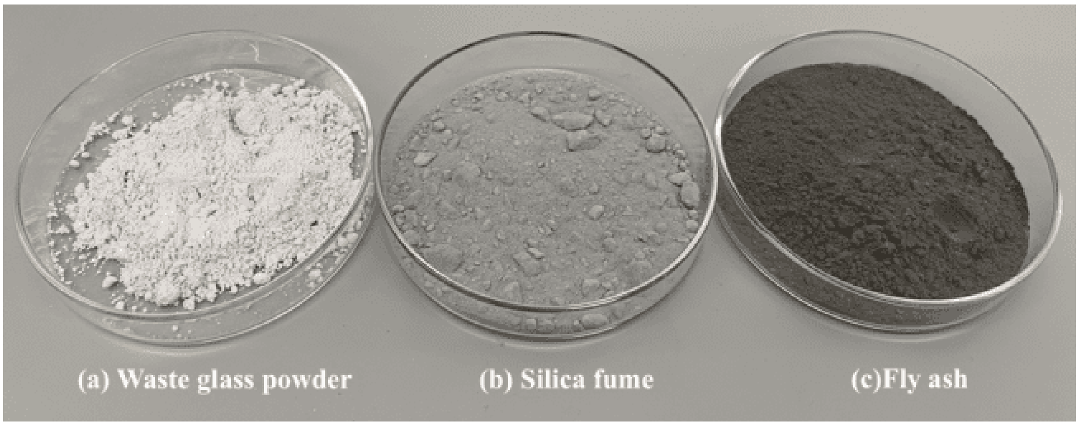

Figure 1. Raw mineral additive materials used in the study
Note – compiled by the authors

The microstructural characteristics of the materials were examined using a JEOL JCM-7000 scanning electron microscope (Japan) at magnification 3000× and accelerating voltage 10kV with a working distance of 14 mm in high-vacuum mode. Elemental composition was determined by energy-dispersive X-ray spectroscopy (EDS) integrated with Scanning electron microscopy (SEM) system.

RESULTS AND DISCUSSION

The microstructure and elemental composition of the investigated raw materials were studied using scanning electron microscopy (SEM) coupled with energy-dispersive X-ray spectroscopy (EDS). SEM images together with elemental mapping and EDS spectra are presented in Figures 2–5.

3.1 Microstructure of clay, waste glass powder, silica fume and fly ash

The SEM micrograph of clay (Figure 2) reveals irregular plate-like particles forming dense agglomerates. Such morphology is typical for natural aluminosilicate clay minerals, which generally consist of layered crystalline structures. The particles exhibit a flaky structure and rough surfaces, indicating the presence of fine clay platelets that tend to aggregate during drying and grinding.

The EDS spectrum confirms that the main elements present in the clay are oxygen, silicon and aluminum, which are characteristic components of aluminosilicate minerals. Minor amounts of magnesium, potassium and calcium are also detected. The elemental mapping images demonstrate a relatively uniform distribution of Al, Si, O and Mg throughout the analyzed region, confirming the homogeneous composition of the clay material.

The observed morphology and elemental composition are consistent with typical natural aluminosilicate clays used in ceramic production. Similar microstructural characteristics of clay minerals and their aluminosilicate composition have been reported in previous studies investigating raw materials for fired brick manufacturing (Ahmaruzzaman, 2010; Sutcu et al., 2019).

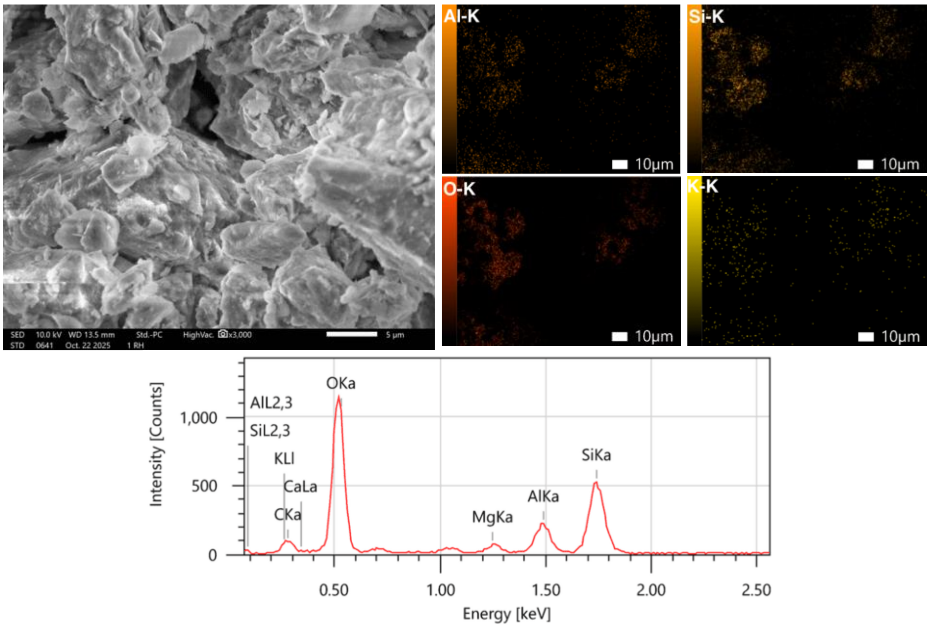

Figure 2. Clay: SEM micrograph with corresponding EDS elemental distribution maps (Al, Si, O, K) and the EDS spectrum showing the elemental composition

Note – compiled by the authors

The SEM image of waste glass powder (Figure 3) shows irregular and angular particles with sharp edges. Such morphology is typical for mechanically crushed glass and reflects its brittle fracture behavior. The particles appear relatively dense and smooth compared to clay particles, which is consistent with the amorphous structure of glass.

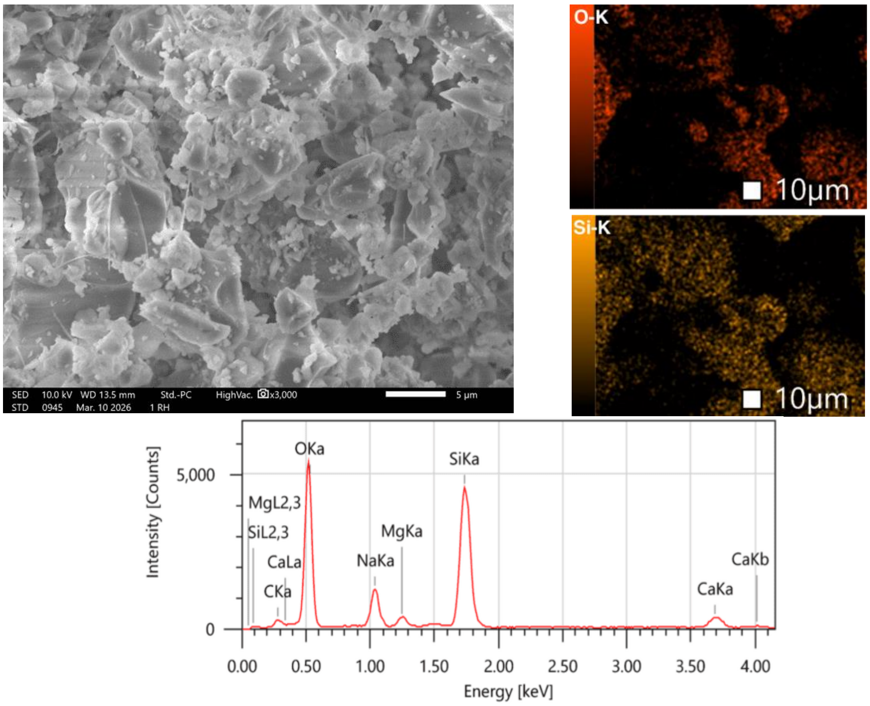

Figure 3. Waste glass: SEM micrograph with corresponding EDS elemental distribution maps (O, Si) and the EDS spectrum showing the elemental composition

Note – compiled by the authors

EDS analysis indicates that silicon and oxygen are the dominant elements in the waste glass powder, confirming its silica-rich composition. Small amounts of sodium, calcium and magnesium are also detected, which correspond to typical components of soda-lime glass. The presence of these elements suggests that the material may exhibit fluxing properties during high-temperature processing.

The irregular angular morphology and silica-rich composition of the waste glass powder are consistent with the characteristics of mechanically crushed glass reported in previous studies. Waste glass particles have been widely investigated as a fluxing additive in ceramic and brick production due to their high silica content and ability to promote vitrification during firing (Demir, 2009; Mohajerani et al., 2017; Mao et al., 2020).

The SEM micrograph of silica fume presented in Figure 4 demonstrates extremely fine particles forming agglomerated clusters. The particles are significantly smaller than those observed in clay and glass waste, which is characteristic of silica fume produced as a by-product of silicon and ferrosilicon alloy production.

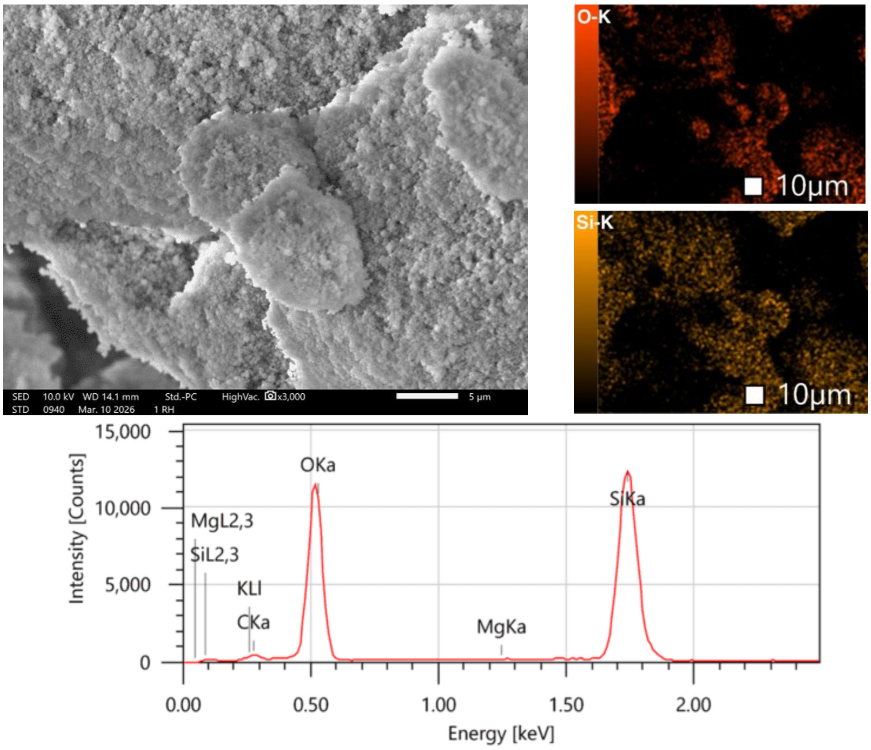

Figure 4. Silica fume: SEM micrograph with corresponding EDS elemental distribution maps (O, Si) and the EDS spectrum showing the elemental composition

Note – compiled by the authors

EDS results demonstrate that silicon and oxygen are the predominant elements in the material, confirming the high silica content of the silica fume. Elemental mapping shows a uniform distribution of these elements across the analyzed area. The high specific surface area and fine particle size indicate that silica fume may exhibit high reactivity and can potentially enhance the microstructure of ceramic materials.

The extremely fine particle size and high silicon content observed in silica fume are typical characteristics of this industrial by-product. Previous studies have reported that silica fume can improve the microstructure and densification of ceramic materials due to its high reactivity and ultrafine particle size (Baspinar et al., 2010; Elmaghraby & Ismail, 2016; Kumar et al., 2021).

The SEM image of fly ash in Figure 5 reveals the presence of both spherical and irregularly shaped particles. The spherical morphology is typical for fly ash particles formed during coal combustion processes. Such particles are often hollow or solid microspheres that originate from the melting and rapid cooling of mineral components in coal.

EDS analysis indicates that the main elements in fly ash are silicon, aluminum and oxygen, which confirms its aluminosilicate nature. Minor quantities of magnesium, calcium and potassium are also detected. Elemental mapping demonstrates that these elements are distributed relatively uniformly across the particle surfaces.

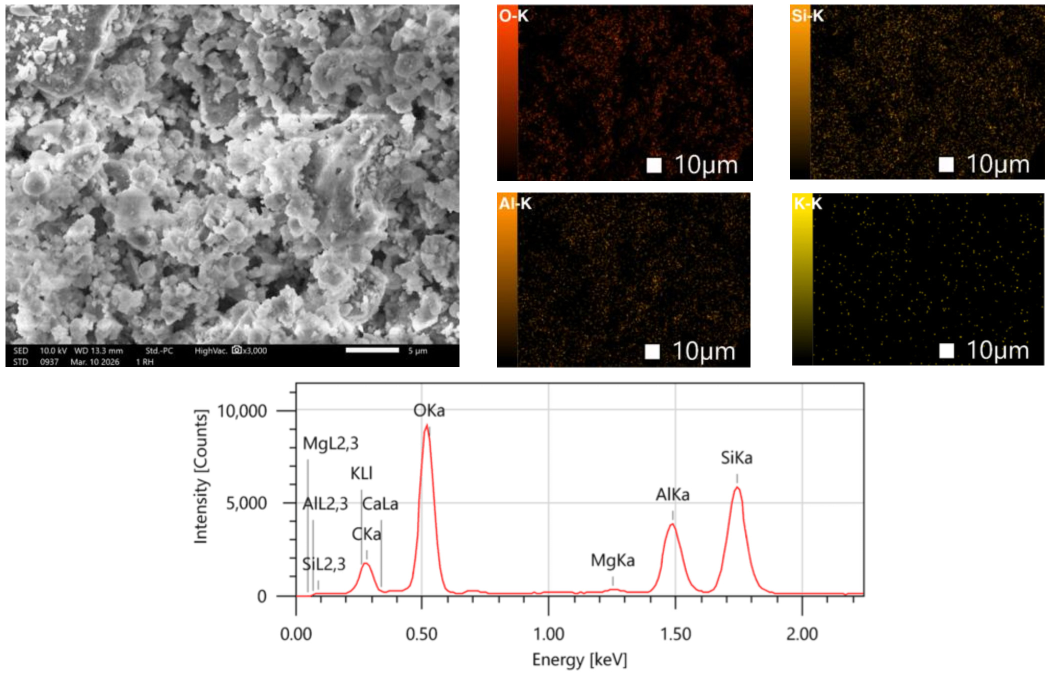

Figure 5. Fly ash: SEM micrograph with corresponding EDS elemental distribution maps (O, Si, K, Al) and the EDS spectrum showing the elemental composition

Note – compiled by the authors

The spherical morphology and aluminosilicate composition observed in fly ash are consistent with the typical characteristics of coal combustion by-products reported in the literature. Fly ash has been widely used as a supplementary raw material in ceramic and brick production due to its silica and alumina content and potential to improve the sintering process (Abbas et al., 2017; Sutcu et al., 2019; Yuan et al., 2022).

The elemental composition of the investigated materials determined by EDS analysis is presented in Table 1. The results confirm that the studied raw materials are mainly composed of silicon and oxygen, which are characteristic elements for aluminosilicate and silica-based materials.

Table 1. Elemental composition of the studied materials determined by EDS analysis, %

Material	C	O	Mg	Al	Si	K	Ca	Na	Total
Clay	10.18	49.53	1.97	7.07	20.12	2.95	8.18		100
Glass	10.18	49.53	7.07	-	20.12	-	2.95	1.97	100
Silica fume	6.73	44.85	0.18	-	47.83	0.41	-	-	100
Fly	22.58	39.46	0.39	12.10	22.75	1.19	1.52	-	100
Note – compiled by the authors									

The obtained results are consistent with previous studies reporting the successful utilization of industrial by-products such as fly ash, waste glass and silica-rich materials in ceramic production. These materials contribute to improved sintering behavior, densification and mechanical performance of fired clay products while simultaneously reducing the consumption of natural raw materials (Karayannis et al., 2017; Yadav et al., 2024; Yoon & Yun, 2005).

Overall, the SEM observations and EDS analysis confirm that the investigated materials are rich in silica and aluminosilicate phases. Clay and fly ash mainly contain aluminosilicate components, whereas waste glass powder and silica fume are characterized by a high silica content. These characteristics indicate that the studied materials can serve as promising raw materials and mineral additives in ceramic compositions, potentially enhancing densification and microstructural stability during firing and contributing to the development of sustainable ceramic construction products (Karayannis et al., 2017; Yadav et al., 2024; Yuan et al., 2022).

3.2 Practical implementation

The SEM and EDS analyses reveal that the studied materials exhibit distinct microstructural and chemical characteristics. Clay and fly ash are dominated by aluminosilicate components (Si, Al and O), while waste glass powder and silica fume contain a high proportion of silica.

Based on the obtained microstructural and elemental analysis, the investigated materials can be considered promising components for ceramic production. Clay can serve as the primary aluminosilicate matrix, while fly ash may act as an additional aluminosilicate source improving sintering behavior. Silica fume and waste glass powder, due to their high silica content and fine particle size, may function as mineral additives that enhance densification and microstructural stability during firing. Therefore, the use of these materials can contribute to the development of ceramic products based on industrial by-products and recycled materials, supporting sustainable construction practices.

CONCLUSION

The microstructural and elemental characteristics of clay, waste glass powder, silica fume, and fly ash were investigated using scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS). The SEM analysis revealed distinct morphological features of the studied materials. Clay particles exhibited irregular plate-like structures typical of aluminosilicate minerals, while waste glass powder showed angular fractured particles. Silica fume consisted of extremely fine agglomerated particles, whereas fly ash contained both spherical and irregular particles characteristic of coal combustion products.

The EDS results confirmed that silicon and oxygen are the dominant elements in all investigated materials, while aluminum, magnesium, potassium, and calcium were detected in smaller amounts. The obtained results indicate that the studied materials are rich in silica and aluminosilicate components.

Overall, the investigated materials can be considered promising raw materials and mineral additives for ceramic compositions. Their chemical composition and microstructural characteristics may contribute to improved sintering behavior and the development of sustainable ceramic construction materials.

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INFORMED CONSENT STATEMENT: Written informed consent was obtained from all participants prior to their involvement in the study.

DATA AVAILABILITY STATEMENT: The data supporting the findings of this technical study are available from the corresponding author upon reasonable request.

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Авторлар туралы мәліметтер
Информация об авторах
Information about authors

Жумадилова Жанар Оразбековна – PhD, Сәтбаев Университеті, Алматы қ., Қазақстан
Жұмаділова Жанар Оразбекқызы – PhD, Сатпаев Университет, г. Алмты, Казахстан
Zhumadilova Zhanar – PhD, Satbayev University, Almaty, Kazakhstan,
e-mail: z.zhumadilova@satbayev.university,
ORCID: https://orcid.org/0000-0001-9487-2018,

Орынбеков Елжан Серикович – Техника ғылымдарының кандидаты, Сәтбаев Университеті, Алматы қ., Қазақстан
Орынбеков Елжан Серікұлы – Кандидат технических наук, Сатпаев Университет, г. Алматы, Казахстан
Orynbekov Yelzhan – Candidate of technical sciences, Satbayev University, Almaty, Kazakhstan,
e-mail: eljan_79@mail.ru,
ORCID: https://orcid.org/0000-0003-2131-6293,

Жугинисов Маратбек Турабайулы – Техника ғылымдарының докторы, Сәтбаев Университеті, Алматы қ., Қазақстан
Жүгінісов Маратбек Турабайұлы – Доктор технических наук, Сатпаев Университет, г. Алматы, Казахстан
Zhuginissov Maratbek – Doctor of technical sciences, Satbayev University, Almaty, Kazakhstan,
e-mail: m.zhuginissov@satbayev.university,
ORCID: https://orcid.org/0000-0001-5594-3653,

Төлегенова Әйгерім Қайратқызы – PhD, Сәтбаев Университеті, Алматы қ., Қазақстан
Толегенова Айгерим Кайратовна – PhD, Сатпаев Университет, г. Алматы, Казахстан
Tolegenova Aigerim – PhD, Satbayev University, Almaty, Kazakhstan,
e-mail: a.tolegenova@satbayev.university,
ORCID: https://orcid.org/0000-0003-1312-4101,

Жагифаров Адлет Музафарұлы – PhD, Сәтбаев Университеті, Алматы қ., Қазақстан
Жагифаров Адлет Музафарович – PhD, Сатпаев Университет, г. Алмты, Казахстан
Zhagifarov Adlet – PhD, Satbayev University, Almaty, Kazakhstan,
e-mail: a.zhagifarov@satbayev.university,
ORCID: https://orcid.org/0000-0001-8046-4697,

Нұрлыбаев Руслан Ерғалиұлы – PhD, Сәтбаев Университеті, Алматы қ., Қазақстан
Нурлыбаев Руслан Ергалиевич – PhD, Сатпаев Университет, г. Алмты, Казахстан
Nurlybayev Ruslan – PhD, Satbayev University, Almaty, Kazakhstan,
e-mail: rusya_nre@mail.ru,
ORCID: https://orcid.org/0000-0003-0161-6256