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RESEARCH ARTICLE

## SILICA FUME EFFECTS ON PHYSICAL AND MECHANICAL PROPERTIES OF ASH-SLAG WASTE CERAMIC BODIES

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**Abstract.** *Ash-slag waste from thermal power plants can be used as a technogenic aluminosilicate component of ceramic bodies; however, increasing its content requires an assessment of material properties under specified forming and firing conditions. This study aimed to evaluate the feasibility of incorporating 50 wt.% fresh ground ash-slag waste from Pavlodar TPP and to determine the effect of silica fume on the properties of fired specimens. The scientific novelty lies in experimentally establishing, for this raw-material system, the effect of adding 3.0 wt.% silica fume to a ceramic body with a high content of fresh ash-slag waste: strength increases without the formation of additional identifiable crystalline phases or significant changes in water absorption, bulk density, and shrinkage. A reference composition containing equal mass fractions of clay and ground ash-slag waste and a modified composition containing 3.0 wt.% silica fume were investigated. Cylindrical specimens were formed, dried to constant mass, and fired at 950 °C. The particle-size distribution, chemical composition, and phase composition of the raw materials were determined. Linear shrinkage, compressive strength, water absorption, bulk density, and the elemental and phase compositions of the fired specimens were evaluated. Silica fume increased the compressive strength from 14.60 to 16.85 MPa, corresponding to an increase of 15.4%. No significant differences were observed in water absorption, bulk density, forming moisture content, or linear shrinkage. After firing, both compositions exhibited similar crystalline phase assemblages. The results confirm the feasibility of incorporating fresh ground ash-slag waste at a content of 50 wt.% as a partial replacement for natural clay and of increasing material strength through the addition of 3.0 wt.% silica fume without adversely affecting the investigated properties.*

**Keywords:** *technogenic aluminosilicate raw material; coal-combustion residues; clay-based ceramic body; partial replacement of natural clay; firing temperature; compressive strength; water absorption.*

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ҒЫЛЫМИ МАҚАЛА

## МИКРОКРЕМНЕЗЕМНІҢ КҮЛ-ҚОЖ ҚАЛДЫҚТАРЫ НЕГІЗІНДЕГІ КЕРАМИКАЛЫҚ МАССАЛАРДЫҢ ФИЗИКА- МЕХАНИКАЛЫҚ ҚАСИЕТТЕРІНЕ ӘСЕРІ

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**Аңдатпа.** Жылу электр станцияларының күл-қож қалдықтарын керамикалық массалардың техногендік алюмосиликатты құрамдасы ретінде пайдалануға болады, алайда олардың мөлшерін арттыру белгіленген қалыптау және күйдіру жағдайларында алынатын материалдардың қасиеттерін бағалауды талап етеді. Зерттеудің мақсаты Павлодар қаласындағы ЖЭО-дің 50 мас.% мөлшеріндегі жаңа ұнтақталған күл-қож қалдықтарын пайдалану мүмкіндігін бағалау және микрокремнеземнің күйдірілген үлгілердің қасиеттеріне әсерін анықтау болды. Жұмыстың ғылыми жаңалығы осы шикізат жүйесі үшін құрамында жаңа күл-қож қалдықтарының мөлшері жоғары керамикалық массаға 3,0 мас.% микрокремнезем енгізудің әсерін тәжірибелік жолмен анықтаудан тұрады: беріктік қосымша сәйкестендірілетін кристалдық фазалар түзілмей және су сіңіргіштік, орташа тығыздық пен шөгу көрсеткіштері елеулі өзгермей артады. Саз бен ұнтақталған күл-қож қалдықтарының массалық үлестері тең базалық құрам және 3,0 мас.% микрокремнезем қосылған модификацияланған құрам зерттелді. Цилиндрлік үлгілер қалыптатып, тұрақты массаға дейін кептірілді және 950 °С температурада күйдірілді. Бастапқы құрамдастардың гранулометриялық, химиялық және фазалық құрамы анықталды. Күйдірілген үлгілердің сызықтық шөгуі, сығылу кезіндегі беріктік шегі, су сіңіргіштігі, орташа тығыздығы, сондай-ақ элементтік және фазалық құрамы бағаланды. Микрокремнеземді енгізу сығылу кезіндегі беріктік шегін 14,60тан 16,85 МПа-ға дейін, яғни 15,4%-ға арттырды. Құрамдардың су сіңіргіштігі, орташа тығыздығы, қалыптау ылғалдылығы және сызықтық шөгуі бойынша елеулі айырмашылықтар анықталған жоқ. Күйдірілгеннен кейін екі құрам да кристалдық фазалардың ұқсас жиынтығымен сипатталды. Алынған нәтижелер жаңа ұнтақталған күл-қож қалдықтарын табиғи сазды ішінара алмастырушы ретінде 50 мас.% мөлшерінде пайдалану және зерттелген қасиеттерді нашарлатпай, 3,0 мас.% микрокремнезем қосу арқылы материалдың беріктігін арттыру мүмкіндігін растайды.

**Түйінді сөздер:** техногендік алюмосиликатты шикізат; көмір жағу өнімдері; саз негізіндегі керамикалық масса; табиғи сазды ішінара алмастыру; күйдіру температурасы; қысымға беріктік; су сіңіргіштік.

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НАУЧНАЯ СТАТЬЯ

## ВЛИЯНИЕ МИКРОКРЕМНЕЗЁМА НА ФИЗИКО-МЕХАНИЧЕСКИЕ СВОЙСТВА КЕРАМИЧЕСКИХ МАСС НА ОСНОВЕ ЗОЛОШЛАКОВЫХ ОТХОДОВ

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**Аннотация.** Золошлаковые отходы тепловых электростанций могут использоваться как техногенный алюмосиликатный компонент керамических масс, однако увеличение их содержания требует оценки свойств материалов при заданных условиях формования и обжига. Цель исследования заключалась в оценке возможности использования свежих измельчённых золошлаковых отходов ТЭЦ г. Павлодара в количестве 50 мас.% и определении влияния микрокремнезёма на свойства обожжённых образцов. Научная новизна работы состоит в экспериментальном установлении для данной сырьевой системы эффекта введения 3,0 мас.% микрокремнезёма в керамическую массу с высоким содержанием свежих золошлаковых отходов: прочность повышается без образования дополнительных идентифицируемых кристаллических фаз и заметного изменения водопоглощения, плотности и усадки. Исследовали базовый состав с равными массовыми долями глины и измельчённых золошлаковых отходов и модифицированный состав с 3,0 мас.% микрокремнезёма. Цилиндрические образцы формовали, высушивали до постоянной массы и обжигали при 950 °С. Для исходных компонентов определяли гранулометрический, химический и фазовый состав; для обожжённых образцов - линейную усадку, предел прочности при сжатии, водопоглощение, среднюю плотность, элементный и фазовый состав. Введение микрокремнезёма повысило предел прочности при сжатии с 14,60 до 16,85 МПа, или на 15,4%. Водопоглощение, средняя плотность, формовочная влажность и линейная усадка составов существенно не различались. После обжига оба состава характеризовались близким набором кристаллических фаз. Полученные результаты подтверждают возможность замены природной глины измельчёнными золошлаковыми отходами в количестве 50 мас.%, а также повышения прочности материала добавлением 3,0 мас.% микрокремнезёма без ухудшения исследованных показателей.

**Ключевые слова:** техногенное алюмосиликатное сырьё; продукты сжигания угля; глинистая керамическая масса; частичная замена природной глины; температура обжига; прочность при сжатии; водопоглощение.

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## 1 INTRODUCTION

Coal-combustion residues, including fly ash and ash-slag waste (ASW), are increasingly considered as secondary aluminosilicate raw materials for construction ceramics. Their incorporation into fired clay products can reduce the consumption of natural clay and promote the diversion of industrial residues from long-term storage or disposal [1-4]. The properties of ash-containing ceramic bodies are governed by the chemical and mineralogical composition of the raw materials, particle size distribution, waste content, forming conditions, and firing regime [3-6].

Previous studies have demonstrated the feasibility of using coal fly ash and ash-slag residues in porous ceramics, structural ceramics, and illite-based ceramic bodies [1, 4, 5, 7, 8]. However, the effects of ash incorporation on shrinkage, compressive strength, water absorption, bulk density, and phase composition depend strongly on the origin and properties of the ash, the clay-based formulation, and the firing temperature [3-8, 15].

Recent studies have confirmed the feasibility of using ash-slag waste, local clay raw materials, phosphorus slag, waste glass, diatomite, and fly ash in the production of ceramic materials [16-20]. The reported results show that the composition of mineral components and firing conditions affect sintering, density, water absorption, porosity, and compressive strength of fired ceramics.

Silica fume is a highly dispersed silica-rich by-product that has been investigated as an additive in fired clay bricks. Earlier studies have shown that its effect on sintering behaviour, density, water absorption, and strength depends on the dosage and firing temperature [6, 9-11]. In particular, silica fume may locally enhance sintering because of its reactive amorphous silica component, although the magnitude and direction of the effect vary with the ceramic body composition and thermal treatment [9, 10].

The use of other technogenic mineral raw materials, including volcanic ash and coal-mining overburden, has also been investigated for the production of fired clay bricks and ceramic composites [12-14]. These studies highlight the importance of raw-material characterisation, particle-size control, composition design, and firing conditions in achieving the required physical and mechanical performance of fired construction ceramics. In Kazakhstan, ceramic bricks incorporating thermal-power-plant ash and agricultural residues have also been investigated, confirming the relevance of local industrial by-products as components of ceramic building materials [14].

Accordingly, this study investigated the feasibility of incorporating fresh ground ASW from Pavlodar TPP-1 as a partial replacement for clay from the Pavlodar Region in fired ceramic bodies. Two compositions were evaluated: a clay-ASW body containing equal mass fractions of clay and ASW, and a comparable composition containing 3.0 wt.% silica fume. The study examined the particle-size, chemical, and phase characteristics of the raw materials, as well as forming moisture content, shrinkage, compressive strength, water absorption, bulk density, and crystalline phase composition of specimens fired at 950 °C.

## 2 MATERIALS AND METHODS

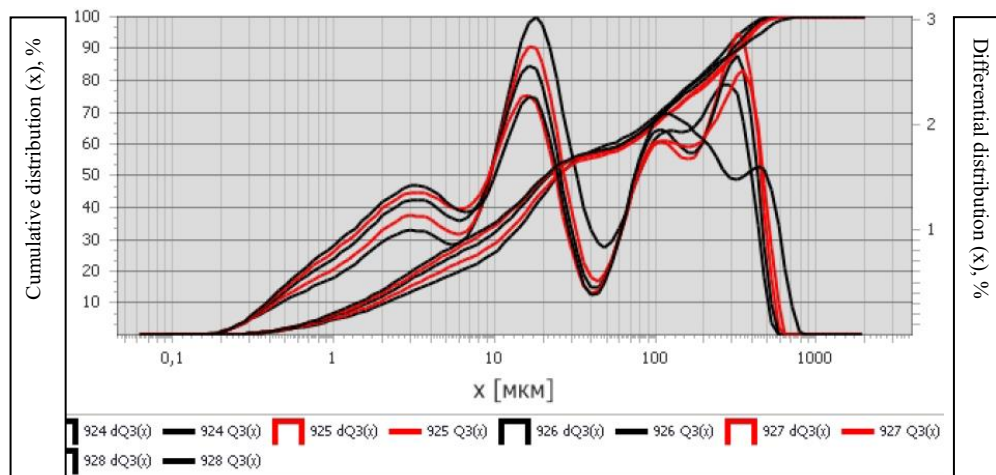
### 2.1. Raw Materials

Clay from the Pavlodar Region, fresh ground ASW from Pavlodar TPP-1, and silica fume were used as raw materials. The clay served as the plastic component of the ceramic bodies. On a dry basis, the clay content was 50.0 wt.% in composition 1 and 48.5 wt.% in composition 2.

The clay had a bulk density of 1110 kg/m<sup>3</sup> and a plasticity index of 16.5. According to GOST 21216-2014, it was classified as a medium-plasticity clay raw material. Before preparation of the ceramic bodies, the clay was ground and passed through a 1 mm sieve; the residue retained on the sieve did not exceed 5%.

The particle-size distribution of the clay was measured by laser diffraction using an Analysette 22 MicroTec plus particle-size analyser (Fritsch, Germany) with the Fraunhofer optical model over a measurement range of 0.01-2100 µm (Figure 1). Based on five measurements, the volume-weighted

mean particle diameter,  $D[4,3]$ , ranged from 10.99 to 15.99  $\mu\text{m}$ , whereas the modal particle size ranged from 88.1 to 102.4  $\mu\text{m}$ .



**Figure 1** – Cumulative and differential particle-size distribution curves of the clay measured by laser diffraction [Author's material]

The chemical composition of the clay was determined by X-ray fluorescence (XRF) analysis. Silicon dioxide ( $\text{SiO}_2$ ) and aluminium oxide ( $\text{Al}_2\text{O}_3$ ) were the predominant oxide components, accounting for 61.21 and 19.06 wt.%, respectively. The  $\text{Fe}_2\text{O}_3$  content was 7.53 wt.% (Table 1).

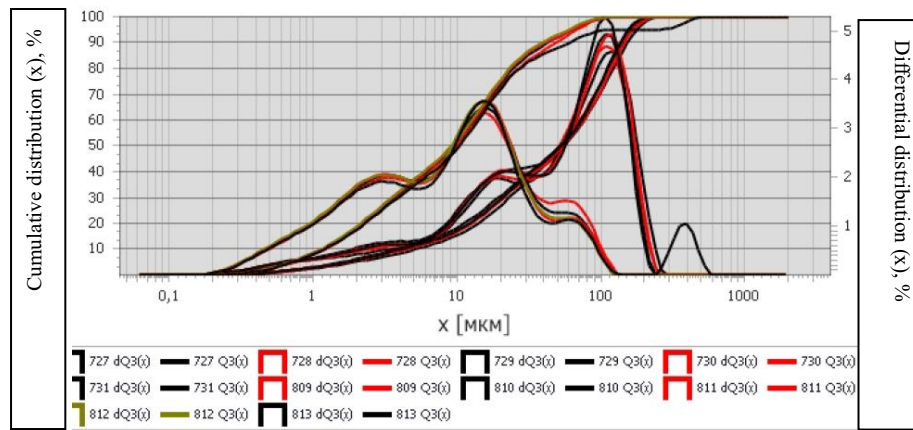
**Table 1** – Chemical composition of the clay determined by X-ray fluorescence analysis [Author's material]

| Name of the raw material component | Oxide composition, mass % |                         |                         |              |              |                       |                |                      |               |              |                        |
|------------------------------------|---------------------------|-------------------------|-------------------------|--------------|--------------|-----------------------|----------------|----------------------|---------------|--------------|------------------------|
|                                    | $\text{SiO}_2$            | $\text{Al}_2\text{O}_3$ | $\text{Fe}_2\text{O}_3$ | $\text{CaO}$ | $\text{MgO}$ | $\text{Na}_2\text{O}$ | $\text{TiO}_2$ | $\text{K}_2\text{O}$ | $\text{SO}_3$ | $\text{MnO}$ | $\text{P}_2\text{O}_5$ |
| Clay                               | 61,21                     | 19,06                   | 7,53                    | 3,12         | 3,60         | 0,01                  | 0,91           | 3,12                 | 0,54          | 0,6          | 0,04                   |

The mineralogical composition of the clay was determined by powder X-ray diffraction (XRD). Kaolinite, illite, quartz, calcite, and iron-containing phases were identified in the clay sample.

Fresh ash-slag waste (ASW), generated during coal combustion at Pavlodar TPP-1 of Aluminium of Kazakhstan JSC, was used as a technogenic aluminosilicate component of the ceramic bodies. Before use in the ceramic bodies, the ASW was ground in a laboratory ball mill.

The particle-size distribution of the ground ASW was measured by laser diffraction using an ANALYSETTE 22 MicroTec plus particle-size analyser (Fritsch, Germany) with the Fraunhofer optical model over a measurement range of 0.01-2100  $\mu\text{m}$  (Figure 2). In the first measurement series, the modal particle size ranged from 62.4 to 68.6  $\mu\text{m}$ , while the volume-weighted mean particle diameter,  $D[4,3]$ , ranged from 2.49 to 2.69  $\mu\text{m}$ . In the second measurement series, the modal particle size ranged from 15.5 to 33.9  $\mu\text{m}$ , whereas  $D[4,3]$  ranged from 4.12 to 5.64  $\mu\text{m}$ .



**Figure 2** – Cumulative and differential particle-size distribution curves of ground ASW measured by laser diffraction [Author's material].

The chemical composition of the ground ASW was determined by XRF analysis. Silicon dioxide ( $\text{SiO}_2$ ) and aluminium oxide ( $\text{Al}_2\text{O}_3$ ) were the predominant oxide components, accounting for 61.1 and 18.6 wt.%, respectively. The  $\text{Fe}_2\text{O}_3$  content was 7.6 wt.% (Table 2).

**Table 2** – Chemical composition of ash-slag waste (ASW) determined by X-ray fluorescence analysis [Author's material]

| Name of the raw material component | Oxide composition, mass % |                         |                         |              |              |                       |                |                      |               |              |                        |
|------------------------------------|---------------------------|-------------------------|-------------------------|--------------|--------------|-----------------------|----------------|----------------------|---------------|--------------|------------------------|
|                                    | $\text{SiO}_2$            | $\text{Al}_2\text{O}_3$ | $\text{Fe}_2\text{O}_3$ | $\text{CaO}$ | $\text{MgO}$ | $\text{Na}_2\text{O}$ | $\text{TiO}_2$ | $\text{K}_2\text{O}$ | $\text{SO}_3$ | $\text{MnO}$ | $\text{P}_2\text{O}_5$ |
| Fresh ASW from Pavlodar TPP-1      | 61,1                      | 18,6                    | 7,6                     | 3,8          | 3,5          | 0,1                   | 1,0            | 3,6                  | 0,4           | 0,1          | 0,2                    |

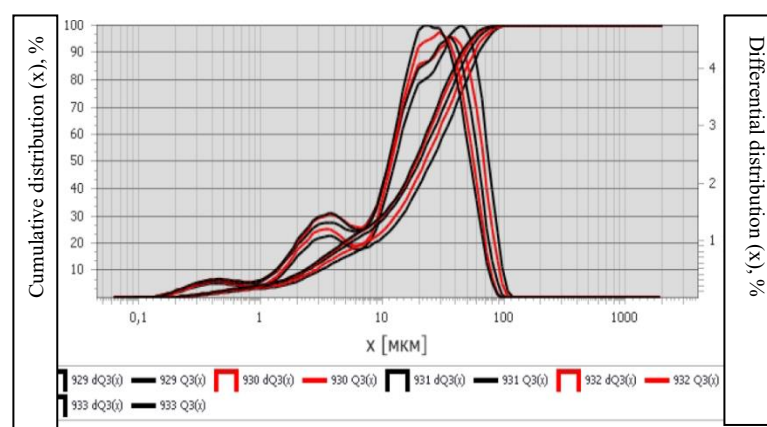
The mineralogical composition of the ground ASW was determined by powder XRD.  $\alpha$ -Quartz, mullite, akermanite, muscovite, and enstatite were identified in the sample. A weak amorphous halo was observed in the diffraction pattern over the  $2\theta$  range of 15–35° (Table 3).

**Table 3** – Semi-quantitative phase composition of the crystalline fraction of ground ASW [Author's material]

| № | Mineral phase    | Content, wt.% |
|---|------------------|---------------|
| 1 | $\alpha$ -Quartz | 43,2          |
| 2 | Mullite          | 29,1          |
| 3 | Akermanite       | 12,2          |
| 4 | Muscovite        | 8,4           |
| 5 | Enstatite        | 7,1           |

Silica fume, a by-product generated during the production of metallurgical-grade silicon with a purity of 99.9% in submerged arc furnaces at TauKenTemir LLP, was used as a fine silica-rich additive. The material was collected from process off-gases by the plant's gas-cleaning system. It consists predominantly of amorphous, ultrafine silicon dioxide. Silica fume has previously been investigated as a reactive silica-rich additive in fired clay bricks, where its influence on sintering and mechanical performance was found to depend on both dosage and firing temperature [9-11].

The particle-size distribution of the silica fume was measured by laser diffraction using an ANALYSETTE 22 MicroTec plus particle-size analyser (Fritsch, Germany) with the Fraunhofer optical model over a measurement range of 0.01-2100  $\mu\text{m}$  (Figure 3). Based on five measurements, the volume-weighted mean particle diameter,  $D[4,3]$ , ranged from 2.31 to 2.42  $\mu\text{m}$ , whereas the modal particle size ranged from 21.2 to 29.7  $\mu\text{m}$ .



**Figure 3** – Cumulative and differential particle-size distribution curves of silica fume measured by laser diffraction [Author's material].

The chemical composition of the silica fume is presented in Table 4. The SiO<sub>2</sub> content was 94.64 wt.%.

**Table 4** – Chemical composition of silica fume [Author's material]

| Name of the raw material component                  | Oxide composition, mass % |                                |                                |      |      | pH of the aqueous extract | H <sub>2</sub> O | Loss on ignition (LOI), wt. % |
|-----------------------------------------------------|---------------------------|--------------------------------|--------------------------------|------|------|---------------------------|------------------|-------------------------------|
|                                                     | SiO <sub>2</sub>          | Al <sub>2</sub> O <sub>3</sub> | Fe <sub>2</sub> O <sub>3</sub> | CaO  | Cl   |                           |                  |                               |
| Silica fume (metallurgical-grade silicon, grade 99) | 94,64                     | 0,07                           | 0,10                           | 0,51 | 0,29 | 8,38                      | 0,33             | 4,57                          |

## 2.2. Ceramic body compositions and experimental procedure

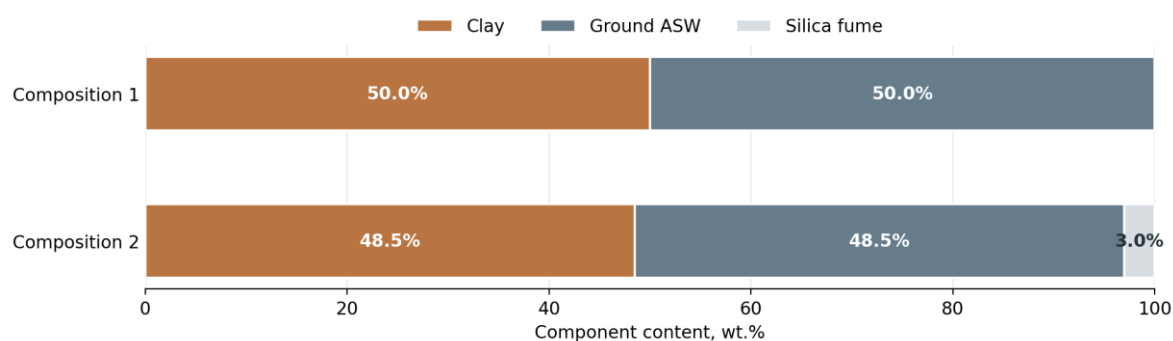
Two ceramic body compositions incorporating clay, fresh ground ASW from Pavlodar TPP-1, and silica fume were prepared for this study. Composition 1 comprised equal mass fractions of clay and ground ASW. In Composition 2, 3.0 wt.% silica fume was introduced, while the contents of clay and ASW were proportionally reduced to 48.5 wt.% each. The compositions of the ceramic bodies are summarised in Table 5.

**Table 5** – Compositions of the investigated ceramic bodies [Author's material]

| № | Composition | Clay, wt. % | Ground ASW, wt. % | Silica fume, wt. % |
|---|-------------|-------------|-------------------|--------------------|
| 1 | 1           | 50,0        | 50,0              | 0,0                |
| 2 | 2           | 48,5        | 48,5              | 3,0                |

The comparison between Compositions 1 and 2 enabled evaluation of the effect of incorporating 3.0 wt.% silica fume on the properties of the fired specimens while maintaining a similar clay-to-ASW ratio.

The proportions of the raw-material components in the investigated ceramic bodies are shown graphically in Figure 4.



**Figure 4** – Proportions of clay, ground ash–slag waste (ASW), and silica fume in the investigated ceramic bodies  
[Author's material]

Before batch preparation, the clay was ground and passed through a sieve with a 1 mm mesh opening; the retained fraction did not exceed 5 wt.%. The ASW was ground in a laboratory ball mill. The components were weighed according to the proportions specified in Table 5 and dry-mixed until a homogeneous mixture was obtained. Water was then added to obtain a target forming moisture content of 22 wt.%, followed by further mixing.

Cylindrical specimens, 50 mm in diameter and 50 mm in height, were formed by compaction in laboratory moulds. After demoulding, the specimens were dried at 105 °C to constant mass. Firing was carried out in a laboratory electric muffle furnace at 950 °C. The selected firing temperature was used to evaluate the investigated bodies under a moderate laboratory firing regime and to facilitate comparison with earlier studies of clay-based ceramic bodies containing coal-combustion residues [4, 5, 7, 8]. The furnace was heated automatically at an average rate of approximately 7 °C min<sup>-1</sup>, followed by a 30 min dwell at the maximum temperature. After firing, the specimens were cooled in the furnace to room temperature over approximately 24 h.

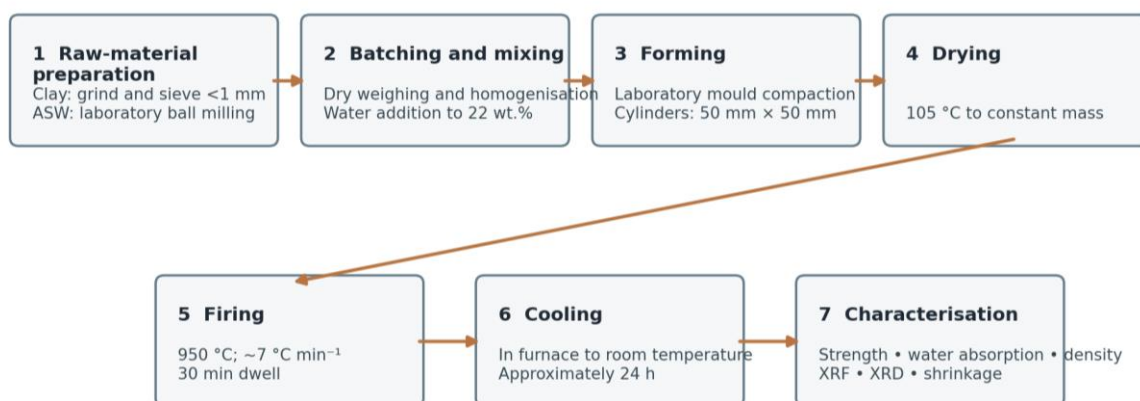
The selected firing temperature of 950 °C lies within the range commonly considered for laboratory studies of ash-containing ceramic products and enables comparison with previous work on fired ceramics incorporating ash-based residues [4, 6, 14].

Compressive strength was measured using a Controls testing machine. The load was increased continuously until failure, with the failure occurring within 20–60 s. Testing was performed at an air temperature of approximately 21 °C and a relative humidity of approximately 64%. Water absorption was determined after immersion of pre-dried specimens in water at 20±5 °C. Bulk density was calculated from the mass and geometric dimensions of the dried specimens. Six specimens were tested for each composition and for each measured property.

The procedures used to determine compressive strength, water absorption, and bulk density were based on GOST 8462-85 and GOST 7025-91, adapted for laboratory-scale cylindrical specimens. The measured compressive-strength and bulk-density values were assessed against the classification requirements specified in GOST 530-2012. GOST 8462-85 establishes methods for determining the ultimate compressive and flexural strengths of wall materials, whereas GOST 7025-91 specifies methods for determining the water absorption and density of ceramic and calcium silicate bricks and stones. GOST 530-2012 sets out general requirements, including strength grades and bulk-density classes, for ceramic bricks and stones.

The complete sequence of specimen preparation, thermal treatment, and characterisation is summarised in Figure 5.

### Experimental workflow and key processing parameters



**Figure 5** – Experimental workflow and key processing parameters used in the study [Author's material]



**Figure 6** – Principal laboratory stages: (a) raw-material components before mixing; (b) prepared ceramic body after mixing; (c) cylindrical specimens after firing [Author's material]

## 3 RESULTS AND DISCUSSION

Considering the differences in the particle sizes of the raw components, the effect of silica fume incorporation on the elemental and phase compositions, as well as on the physical and mechanical properties of the fired ceramic specimens, was evaluated. Compositions 1 and 2, prepared under identical forming, drying, and firing conditions at 950 °C, were compared.

### 3.1. Elemental and phase compositions of the fired specimens

To assess the effect of silica fume on the composition of the fired material, specimens of Composition 1, containing 50.0 wt.% clay and 50.0 wt.% ground ASW, and Composition 2, containing 48.5 wt.% clay, 48.5 wt.% ground ASW, and 3.0 wt.% silica fume, were investigated. The specimens were fired at 950 °C.

The elemental composition was determined by XRF analysis of pressed powder pellets. The results are presented in Table 6 as normalised elemental mass fractions. XRF is appropriate here because it provides bulk elemental-composition data, whereas XRD is used to identify crystalline phases.

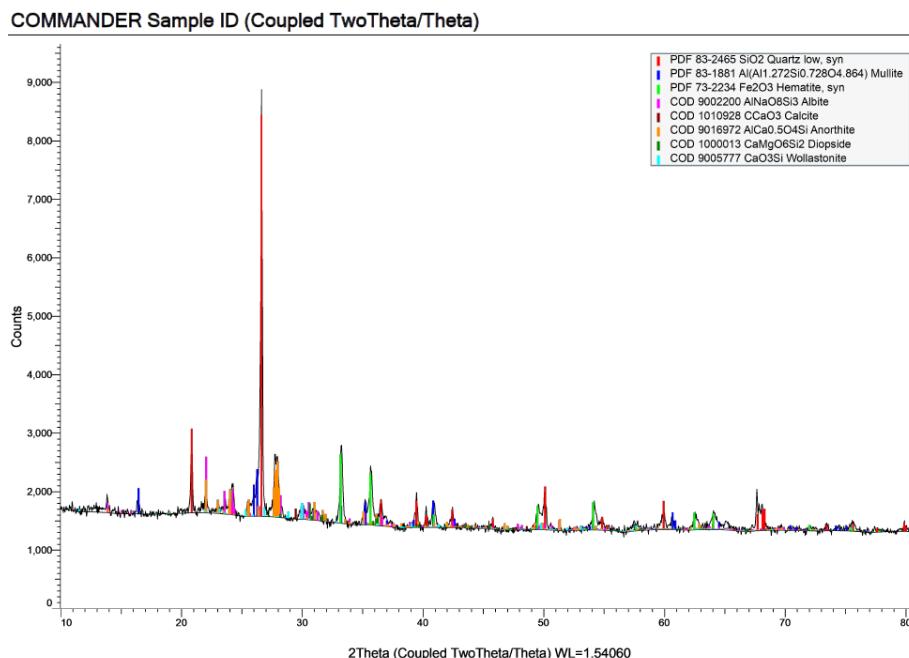
**Table 6** – Elemental composition of the fired ceramic specimens determined by XRF, wt.%

| № | Element | Composition 1: | Composition 2 |
|---|---------|----------------|---------------|
| 1 | Si      | 47,25          | 49,57         |
| 2 | Al      | 21,35          | 18,83         |

|    |                |       |       |
|----|----------------|-------|-------|
| 3  | Fe             | 19,33 | 19,33 |
| 4  | Ca             | 3,91  | 3,98  |
| 5  | Mg             | 2,88  | 2,77  |
| 6  | K              | 2,07  | 1,84  |
| 7  | Ti             | 0,95  | 0,82  |
| 8  | S              | 0,76  | 1,04  |
| 9  | Mn             | 0,43  | 0,38  |
| 10 | Cl             | 0,48  | 0,73  |
| 11 | Na             | 0,12  | 0,04  |
| 12 | P              | 0,12  | 0,14  |
| 13 | Sr             | 0,09  | 0,13  |
| 14 | Ba             | <0,10 | 0,18  |
| 15 | Other elements | <0,10 | <0,10 |

Si, Al, and Fe were the predominant elements in both specimens. In Composition 1, their normalised mass fractions were 47.25, 21.35, and 19.33 wt.%, respectively, whereas the corresponding values for Composition 2 were 49.57, 18.83, and 19.33 wt.%. The incorporation of 3.0 wt.% silica fume increased the normalised Si mass fraction by 2.32 percentage points and decreased the Al mass fraction by 2.52 percentage points. The Fe content remained unchanged within the precision of the reported results. Since the data were normalised, they were used for comparative characterisation of the elemental compositions of the specimens and do not represent the absolute contents of the components.

The XRD patterns of fired Compositions 1 and 2 are shown in Figures 4 and 5, respectively, and were used to identify the crystalline phases present in the fired specimens. The following crystalline phases were identified in Composition 1: quartz, mullite, hematite, albite, anorthite, diopside, wollastonite, and calcite. A semi-quantitative assessment of the crystalline phases in Composition 1 is presented in Table 7.



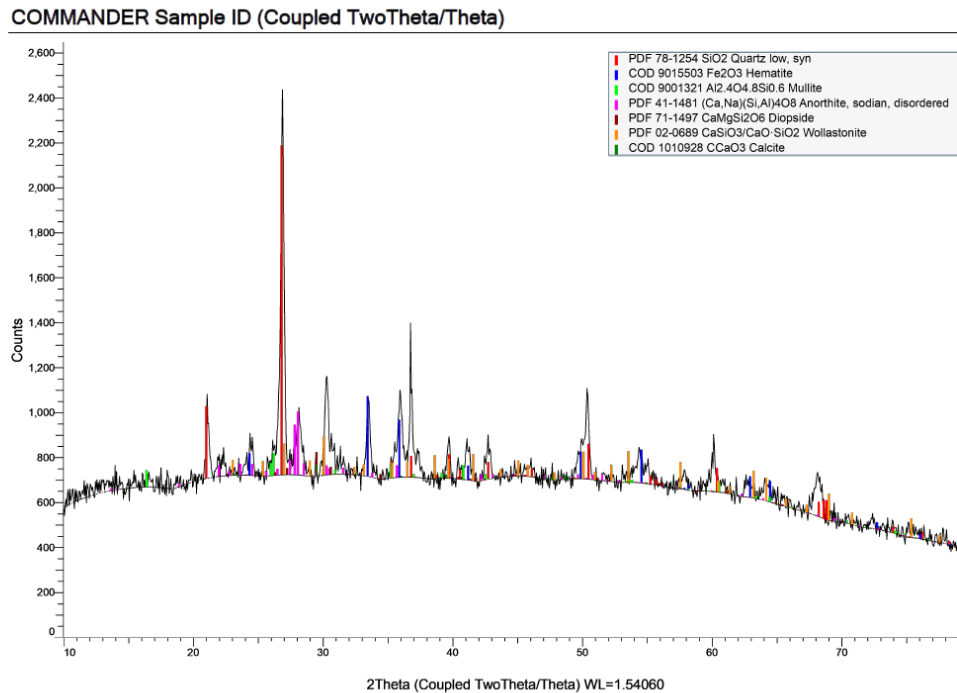
**Figure 7** – XRD pattern of fired Composition 1 containing 50.0 wt.% clay and 50.0 wt.% ground ASW; firing temperature: 950 °C [Author's material]

**Table 7** – Semi-quantitative phase composition of fired Composition 1 determined by XRD [Author's material]

| № | Phase     | Formula                                          | Content, % |
|---|-----------|--------------------------------------------------|------------|
| 1 | Quartz    | SiO <sub>2</sub>                                 | 28,2       |
| 2 | Anorthite | CaAl <sub>2</sub> Si <sub>2</sub> O <sub>8</sub> | 24,4       |

|   |              |                                       |      |
|---|--------------|---------------------------------------|------|
| 3 | Albite       | $\text{NaAlSi}_3\text{O}_8$           | 21,9 |
| 4 | Mullite      | $\text{Al}_6\text{Si}_2\text{O}_{13}$ | 11,9 |
| 5 | Hematite     | $\text{Fe}_2\text{O}_3$               | 4,9  |
| 6 | Wollastonite | $\text{CaSiO}_3$                      | 4,3  |
| 7 | Diopside     | $\text{CaMgSi}_2\text{O}_6$           | 3,5  |
| 8 | Calcite      | $\text{CaCO}_3$                       | 0,9  |

In Composition 2, quartz, mullite, hematite, a plagioclase phase within the anorthite-albite series, diopside, wollastonite, and calcite were identified (Figure 5). The incorporation of 3.0 wt.% silica fume did not result in the appearance of any additional crystalline phases that could be reliably identified within the detection limits of the XRD analysis.



**Figure 8** – XRD pattern of fired Composition 2 containing 48.5 wt.% clay, 48.5 wt.% ground ASW, and 3.0 wt.% silica fume; firing temperature: 950 °C [Author's material]

The identification of quartz, mullite, feldspars, diopside, and wollastonite is consistent with the presence of Si, Al, Ca, Mg, Na, and K in the normalised elemental compositions of the fired materials. The presence of hematite is consistent with the detected Fe content. Both compositions exhibited a similar set of crystalline phases after firing at 950 °C.

The occurrence of quartz, mullite, feldspar-related phases, hematite, diopside, wollastonite, and calcite is generally consistent with phase assemblages reported for fired clay-based ceramics containing aluminosilicate and coal-combustion residues [4, 5, 8]. The absence of additional crystalline phases detectable by XRD after the addition of 3.0 wt.% silica fume suggests that, under the applied firing conditions, the additive influenced the relative elemental composition and physical performance of the specimens without producing a separate crystalline phase detectable by the applied method.

The phase assemblage identified by XRD is plausible for fired clay-based ceramics containing quartz, feldspars, hematite, mullite, diopside, and wollastonite.

### 3.2. Forming moisture content and shrinkage of the ceramic bodies

The forming moisture content of Compositions 1 and 2 was 22 wt.%, which enabled the production of specimens without modifying the adopted forming procedure. The results of the determination of mass change and linear shrinkage after drying and firing are presented in Table 8.

**Table 8** – Forming moisture content, mass change, and linear shrinkage of the ceramic specimens [Author's material]

| № | Property                        | Composition 1 | Composition 2 |
|---|---------------------------------|---------------|---------------|
| 1 | Forming moisture content, wt. % | 22,0          | 22,0          |
| 2 | Mass loss after drying, %       | 18,4          | 18,6          |
| 3 | Mass loss after firing, %       | 21,9          | 21,7          |
| 4 | Linear shrinkage in diameter, % | 6,0           | 6,4           |
| 5 | Linear shrinkage in height, %   | 5,3           | 5,2           |

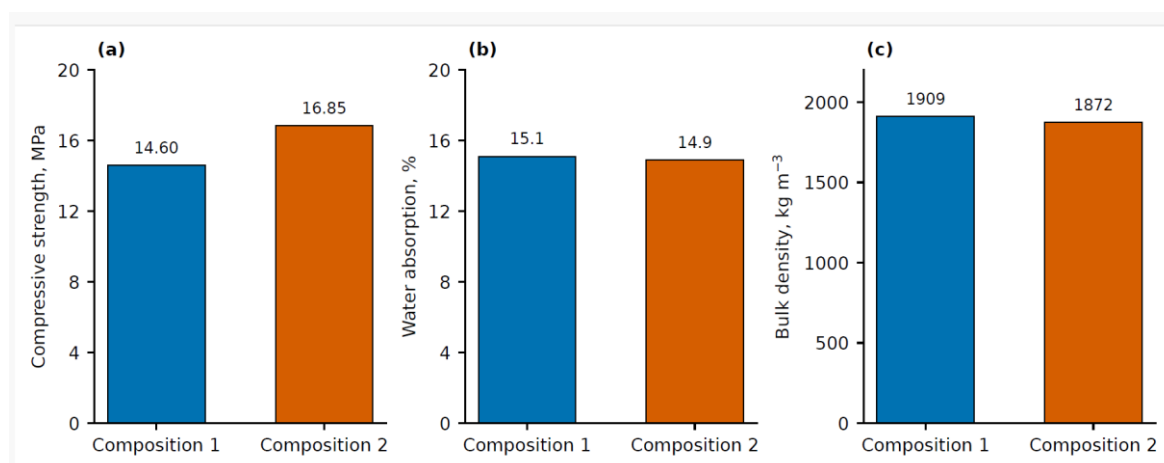
The close values of forming moisture content and linear shrinkage indicate that the addition of 3.0 wt.% silica fume did not substantially alter the forming behaviour of the clay-ASW bodies under the adopted processing conditions. This observation is relevant because ash-containing ceramic formulations can exhibit changes in drying and firing shrinkage depending on the mineralogical composition and particle-size characteristics of the incorporated waste [4, 5].

### 3.3. Compressive strength, water absorption, and bulk density

The physical and mechanical properties of the fired ceramic specimens are presented in Table 9. For a clearer comparison of the compressive strength, water absorption, and bulk density of Compositions 1 and 2, the corresponding values are also shown in Figure 6. These properties are routinely used to characterise the performance of fired ceramic materials.

**Table 9** – Physical and mechanical properties of the fired ceramic specimens [Author's material]

| № | Property                                      | Composition 1 | Composition 2 |
|---|-----------------------------------------------|---------------|---------------|
| 1 | Compressive strength, MPa                     | 14,60         | 16,85         |
| 2 | Strength grade according to GOST 530-2012     | M125          | M150          |
| 3 | Water absorption, %                           | 15,1          | 14,9          |
| 4 | Bulk density, kg m <sup>-3</sup>              | 1909          | 1872          |
| 5 | Bulk-density class according to GOST 530-2012 | 2,0           | 2,0           |

**Figure 9** – Comparison of the compressive strength (a), water absorption (b), and bulk density (c) of the fired ceramic specimens [Author's material]

The compressive strength increased from 14.60 MPa for Composition 1 to 16.85 MPa for Composition 2. The difference was 2.25 MPa, corresponding to a 15.4% increase relative to Composition 1. When compared with the strength-grade criteria of GOST 530-2012, the measured values corresponded to M125 for Composition 1 and M150 for Composition 2.

The water absorption values of Compositions 1 and 2 were 15.1% and 14.9%, respectively; the difference did not exceed 0.2 percentage points. The bulk density decreased from 1909 kg m<sup>-3</sup> for Composition 1 to 1872 kg m<sup>-3</sup> for Composition 2, a difference of 37 kg m<sup>-3</sup>. Both values fall within bulk-density class 2.0, corresponding to the range of 1410-2000 kg m<sup>-3</sup> according to GOST 530-2012.

Thus, under the applied forming, drying, and firing conditions, Composition 2 exhibited higher compressive strength while maintaining similar water absorption values and bulk-density class 2.0.

The observed increase in compressive strength is consistent with previous reports that the effect of silica-rich fine additives in fired clay products depends on the composition of the ceramic body and the firing temperature [6, 9-11]. However, the mechanisms responsible for the strength increase in the investigated compositions require further microstructural examination.

#### 4 CONCLUSIONS

The feasibility of using fresh ground ASW from Pavlodar TPP-1 in clay-based ceramic bodies was investigated. The effect of incorporating 3.0 wt.% silica fume on the properties of specimens fired at 950 °C was also evaluated. The main findings are as follows:

1. Fresh ground ASW exhibited an aluminosilicate composition, containing 61.1 wt.% SiO<sub>2</sub>, 18.6 wt.% Al<sub>2</sub>O<sub>3</sub>, and 7.6 wt.% Fe<sub>2</sub>O<sub>3</sub>. The crystalline fraction of ASW comprised  $\alpha$ -quartz, mullite, akermanite, muscovite, and enstatite, while a weak amorphous contribution was also observed in the XRD pattern. These characteristics indicate that the ASW can serve as a technogenic aluminosilicate component in ceramic bodies.

2. The ceramic body containing 50.0 wt.% clay and 50.0 wt.% ASW exhibited a forming moisture content of 22.0 wt.% and linear shrinkage values of 6.0% in diameter and 5.3% in height after firing at 950 °C. The fired specimens showed a compressive strength of 14.60 MPa, water absorption of 15.1%, and a bulk density of 1909 kg m<sup>-3</sup>.

3. When compared with the relevant classification requirements of GOST 530-2012, these values corresponded to strength grade M125 and bulk-density class 2.0.

4. Replacing equal proportions of clay and ASW with 3.0 wt.% silica fume, resulting in 48.5 wt.% clay, 48.5 wt.% ASW, and 3.0 wt.% silica fume, increased the compressive strength to 16.85 MPa. This represents an increase of 15.4% relative to the silica-fume-free composition. Water absorption and bulk density were 14.9% and 1872 kg m<sup>-3</sup>, respectively. The measured properties corresponded to strength grade M150 and bulk-density class 2.0 when compared with GOST 530-2012.

5. After firing at 950 °C, both compositions exhibited similar crystalline phase assemblages comprising quartz, mullite, hematite, feldspars, diopside, wollastonite, and calcite. The addition of silica fume did not result in the formation of additional crystalline phases that could be reliably identified within the detection limits of the applied XRD analysis.

6. Under the investigated laboratory conditions, the incorporation of 50.0 wt.% fresh ground ASW from Pavlodar TPP-1 enabled partial replacement of natural clay in ceramic bodies. This approach may reduce the consumption of natural clay and promote the utilisation of ash-slag waste in ceramic building materials rather than its storage. Before industrial application can be confirmed, further work should include testing of specimens with standardised dimensions, assessment of frost resistance and leaching behaviour, and pilot-scale validation under production conditions. Such assessments are necessary to confirm both the durability and environmental suitability of ceramic products containing industrial mineral residues [6, 12-15].

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## CONFLICT OF INTEREST

The authors state that there is no conflict of interest.

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During the preparation of this manuscript, the authors used artificial intelligence tools (ChatGPT) solely for editorial assistance, such as improving phrasing and checking grammar, spelling, and punctuation. All ideas, interpretations, and conclusions are the responsibility of the authors, who take full accountability for the content of the article.

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*Vladimir Larichkin* – Methodology, Validation, Interpretation of Results, Writing – Review and Editing, Supervision.

*Aliya Aldungarova* – Methodology, Validation, Writing – Review and Editing, Scientific Consultation.

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