

Article

Production, Characterization and Durability Assessment of Sintered Fly Ash Aggregate from Kyzylorda By-Product Hydraulic Ash for Lightweight Cementitious Composite

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Abstract

The growing accumulation of coal combustion by-products necessitates the development of sustainable approaches for their utilization in construction materials. This study investigates the production of sintered fly ash aggregate (SFAA) using hydraulic ash waste from the Kyzylorda Combined Heat and Power plant and evaluates its suitability as a coarse aggregate for lightweight cementitious composite. Hydraulic fly ash and clay from the Talsuat deposit were pelletized and sintered at 1100 °C. The physicochemical, mineralogical, and microstructural characteristics of the raw materials and produced aggregate were examined using X-ray fluorescence, X-ray diffraction, scanning electron microscopy with energy-dispersive spectroscopy, Fourier-transform infrared spectroscopy, and thermogravimetric analysis. The developed aggregate exhibited a bulk density of 1118 kg m⁻³, water absorption of 5.4%, crushing strength corresponding to grade M200, and frost resistance of at least F35. Mineralogical analysis revealed quartz and mullite as the predominant crystalline phases, while microstructural observations confirmed the formation of a stable porous aluminosilicate matrix. Chemical durability tests in alkaline, chloride, and sulfate media demonstrated high resistance to aggressive environments. Lightweight cementitious composite produced with the aggregate achieved an average density of 1657 kg m⁻³, compressive strength of 3.87 MPa, and water absorption of 16.0%, corresponding to density grade D1600 and strength class B3.5. The results confirm the feasibility of converting hydraulic ash waste into a durable lightweight aggregate suitable for structural-insulating lightweight cementitious composite, contributing to waste valorization, conservation of natural resources, and sustainable construction practices.

Keywords: hydraulic fly ash; sintered fly ash aggregate; lightweight cementitious composite; aluminosilicate matrix; frost resistance

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1. Introduction

The development of sustainable construction materials based on industrial by-products has become one of the key priorities of modern civil engineering and circular economy strategies. The construction sector is increasingly challenged to reduce its environmental footprint through the conservation of natural resources, minimization of industrial waste, and reduction in greenhouse gas emissions associated with conventional building materials. In this context, lightweight cementitious composite produced using artificial porous aggregates offers several advantages, including reduced structural weight, lower transportation and installation costs, improved thermal insulation performance, and enhanced energy efficiency of buildings [1–3]. In regions where high-quality natural aggregates are scarce, the production of artificial lightweight aggregates from industrial waste represents an effective alternative for both resource conservation and sustainable construction.

Coal-fired thermal power plants (CHPs) generate significant quantities of fly ash and ash-slag waste, most of which is disposed of in landfills. The accumulation of these wastes causes environmental problems associated with land occupation, dust emissions, and potential contamination of soil and groundwater. In Kazakhstan, only a limited portion of ash and slag waste is currently utilized, while the accumulated volume continues to increase annually. Consequently, the conversion of fly ash into value-added construction materials has attracted considerable scientific and industrial interest [4–6].

Among the available utilization technologies, the production of sintered fly ash aggregate (SFAA) is considered one of the most promising approaches. SFAA is manufactured through granulation of fly ash followed by high-temperature firing, resulting in a lightweight aggregate characterized by a porous internal structure and relatively high mechanical strength. Compared with natural aggregates, sintered fly ash aggregates can reduce lightweight cementitious composite density while maintaining adequate strength and durability characteristics [7–10].

Numerous studies have investigated the production and application of sintered fly ash aggregates in lightweight cementitious composites. Babu and Thenmozhi reported that lightweight cementitious composite containing sintered fly ash aggregate exhibits satisfactory mechanical performance and improved structural efficiency. Domagała demonstrated the good durability of structural lightweight cementitious composite produced with sintered fly ash aggregates, while Křížová et al. confirmed its stability under elevated temperatures. Patel et al. and Satpathy et al. observed that lightweight cementitious composite incorporating sintered fly ash aggregate possesses favorable durability and microstructural characteristics. Recent studies have also highlighted the influence of aggregate microstructure, mineral phase composition, and firing conditions on density, water absorption, strength development, and long-term performance. However, different authors have reported varying relationships between firing parameters, pore structure development, and the resulting engineering properties of sintered aggregates, indicating that the mechanisms governing aggregate performance are not yet fully understood and may depend strongly on the characteristics of the source ash [11–18].

The physicochemical properties of sintered fly ash aggregates are largely controlled by mineral phase transformations occurring during thermal treatment. High-temperature firing promotes the formation of stable aluminosilicate phases, particularly mullite and glassy matrices, which contribute to enhanced strength and durability. At the same time, pore formation and vitrification processes significantly affect water absorption, frost resistance, and chemical stability of the final aggregate. Therefore, understanding the relationship between raw material composition, phase evolution, and aggregate performance remains an important scientific challenge and a subject of ongoing research [19–23].

Despite extensive international research on sintered fly ash aggregates, studies concerning aggregates produced from hydraulic ash deposits remain limited. In particular, comprehensive investigations on the utilization of hydraulic ash waste from the Kyzylorda CHP plant have not been reported. The specific chemical and mineralogical composition of this ash may influence sintering behavior, phase evolution, microstructure formation, and the resulting engineering properties of the aggregate. Consequently, further research is required to evaluate the suitability of this locally available waste resource for the production of high-performance lightweight aggregates [24–27].

Therefore, the aim of this study is to produce sintered fly ash aggregate from hydraulic ash waste of the Kyzylorda CHP plant and to investigate its mineralogical composition, microstructure, physicochemical characteristics, durability, and suitability for application as a coarse aggregate in lightweight cementitious composite. Particular attention is devoted to mineral phase evolution during firing, aggregate strength, water absorption, frost resistance, chemical stability, and the performance of lightweight cementitious composite containing the developed aggregate. The obtained results demonstrate that hydraulic ash waste can be successfully converted into a durable artificial lightweight aggregate with favorable physical, mechanical, and chemical resistance properties, confirming its potential for use in structural-insulating lightweight cementitious composite and contributing to sustainable waste valorization practices [28–30].

2. Materials and Methods

The raw materials used in this study were hydraulic fly ash, clay, potable water, and a commercial chemical admixture MasterCast 125 (Master Builders Solutions, formerly BASF Construction Chemicals, Mannheim, Germany). Hydraulic fly ash was collected from the ash disposal site of the Kyzylorda Combined Heat and Power (CHP) plant, Kazakhstan. Clay from the Talsuat deposit (Kyzylorda region, Kazakhstan) was used as a binder during granulation. XRF analysis showed that the clay mainly consisted of SiO_2 , Al_2O_3 , and CaO , which promote the formation of stable aluminosilicate phases during firing [31,32]. Potable water with a mineralization of approximately 0.6 mg/L and 1 wt.% MasterCast 125 were used for granulation. Before processing, all materials were dried at 105 ± 5 °C. The raw materials and produced aggregate were characterized using XRF, XRD, SEM-EDS, FTIR, and TGA.

Sintered fly ash aggregate (SFAA) was produced through drying, grinding, mixing, granulation, curing, and firing. Fly ash and clay were ground to obtain a residue of <5 wt.% on a 0.08 mm sieve and mixed for 15 min. The optimal composition consisted of 85 wt.% fly ash and 15 wt.% clay. Granulation was performed using 20 ± 2 wt.% water in a disc pelletizer operated at a 45° inclination and 20 rpm. Granules of 10–20 mm were cured at 20 ± 2 °C for 7 days and fired at 1100 °C following the schedule shown in Table 1, which was selected based on DTA results indicating completion of the major phase transformations and mullite formation.

During firing, dehydration, combustion of residual carbonaceous matter, vitrification, and mullite formation produced a porous and mechanically stable aggregate. To ensure reproducibility, the entire preparation procedure was repeated three times under identical conditions, and representative samples from each batch were used for physical, mechanical, mineralogical, and durability testing. No human participants, animals, or biological materials were involved; therefore, ethical approval was not required. GenAI tools were not used for data generation, experimental design, analysis, or interpretation.

Table 1. Processing parameters used for the production of sintered fly ash aggregate.

Parameter	Value
Fly ash content	85 wt.%
Clay content	15 wt.%
Water content	20 ± 2 wt.%
Granulation angle	45°
Rotation speed	20 rpm
Pellet size	10–20 mm
Curing period	7 days
Firing temperature	1100 °C
Holding time	60

2.1. Chemical and Phase Composition Analysis

The chemical and phase compositions of the fly ash, clay, and sintered fly ash aggregate were determined using X-ray fluorescence (XRF) and X-ray diffraction (XRD).

The chemical composition was analyzed using an S8 TIGER X-ray fluorescence spectrometer (Bruker AXS GmbH, Karlsruhe, Germany). Representative samples were dried, homogenized, and finely ground prior to analysis. The contents of SiO₂, Al₂O₃, Fe₂O₃, CaO, MgO, K₂O, Na₂O, TiO₂, and SO₃ were determined and expressed as mass percentages.

Mineralogical phases were identified using a DRON-2 X-ray diffractometer with CuK α radiation ($\lambda = 1.5406 \text{ \AA}$). Diffraction patterns were recorded over a 2θ range of 10–70° with a 0.02° step size and a scanning speed of 2° min^{−1}. Phase identification was performed using the ICDD PDF-2 database, and the relative contents of the major crystalline phases were estimated from diffraction peak intensities.

The combined XRF and XRD analyses were used to evaluate the relationship between chemical composition, phase transformations during firing, and the physicochemical properties of the produced aggregate, with particular attention to the formation of quartz and mullite and their influence on aggregate strength, durability, and water absorption.

2.2. Fourier Transform Infrared Spectroscopy (FTIR)

The raw materials used in this study included cement, sand, fly ash, potable water, and a chemical admixture. The developed cementitious composite was cast into 70 mm × 70 mm × 70 mm cube specimens for compressive strength testing. The specimen size was selected in accordance with the adopted laboratory testing procedure, providing adequate compaction, minimizing edge damage during demoulding, and ensuring compatibility with the available compression testing equipment.

The functional groups and chemical bonding characteristics of the raw materials and sintered fly ash aggregate were analyzed using an FSM 2202 Fourier-transform infrared (FTIR) spectrometer (Infraspek, Saint Petersburg, Russia) equipped with a NaCl prism. Representative samples were dried, finely ground, and homogenized before analysis. Spectra were recorded in the 700–3800 cm^{−1} wavenumber range.

FTIR analysis was used to identify characteristic bands of aluminosilicate structures, hydroxyl groups, carbonates, and changes in the Si–O–Si and Al–O–Si bonding environments caused by thermal treatment and phase transformations during firing.

The obtained spectra were used to evaluate structural modifications after firing and to support the interpretation of the XRD and thermal analysis results, providing additional information on the formation of stable mineral phases and the development of the aggregate microstructure.

2.3. UV–Visible Spectroscopy

The optical absorption characteristics of the investigated materials were analyzed using a UV-1800 ultraviolet–visible spectrophotometer (Shanghai Mapada Instruments Co., Ltd., Shanghai, China). Representative powdered samples were prepared under identical conditions, and the absorption spectra were recorded in the 200–400 nm wavelength range.

UV–Vis analysis was used to evaluate electronic transitions associated with aluminosilicate phases and iron-containing compounds, with particular attention to changes in absorption intensity and band positions resulting from firing.

The obtained spectra complemented the XRD, FTIR, and thermal analysis results, providing additional information on mineral phase evolution, structural modifications, and the physicochemical properties of the produced sintered fly ash aggregate.

Differential Thermal and Thermogravimetric Analysis (DTA–TG)

Thermal behavior and phase transformations were investigated using a Q-1500D Paulik derivatograph (MOM, Budapest, Hungary). Simultaneous thermogravimetric (TG), derivative thermogravimetric (DTG), and differential thermal analysis (DTA) were performed in the temperature range of 25–1000 °C at a heating rate of 10 °C min^{−1}.

The instrument sensitivity parameters were TG—500, DTG—1, and DTA—250. The balance sensitivity was 200 mg, and the galvanometer sensitivity was 1.10 A with an internal resistance of 350–400 Ω and an oscillation period of 10–11 s. Representative samples were analyzed under identical conditions to ensure reproducibility.

Thermal analysis was used to identify dehydration, combustion of residual carbonaceous matter, decomposition reactions, and phase transformations during aggregate formation, with particular attention to vitrification and mullite crystallization.

The obtained TG, DTG, and DTA curves were used to determine the optimal firing temperature and to support the interpretation of the XRD, FTIR, and microstructural analyses.

3. Results

3.1. Physicochemical Characterization of Hydraulic Fly Ash

The chemical compositions of the hydraulic fly ash from the Kyzylorda CHP plant and the Talsuat clay are presented in Table 2. The fly ash is mainly composed of SiO₂ and Al₂O₃, confirming its aluminosilicate nature and suitability for the production of sintered lightweight aggregates. The predominance of these oxides indicates the potential for the formation of stable aluminosilicate phases during thermal treatment, which are essential for the development of mechanically durable lightweight aggregates.

XRD analysis identified quartz and mullite as the dominant crystalline phases, which contribute to the thermal stability and mechanical strength of the fired material. The presence of mullite is particularly important because this phase is known to enhance the strength, dimensional stability, and durability of ceramic and fly ash-based materials. Minor amounts of CaO, Fe₂O₃, MgO, K₂O, and TiO₂ may influence sintering behavior by promoting liquid-phase formation, densification, and partial vitrification of the aggregate structure. These components can facilitate particle bonding during firing and contribute to the formation of a stable porous microstructure.

The Talsuat clay also contains significant amounts of silica and alumina, providing good compatibility with fly ash and supporting the formation of stable aluminosilicate phases during firing. The similarity in the chemical composition of the fly ash and clay is advantageous for achieving uniform phase development and minimizing incompatibility effects during the sintering process.

Overall, the chemical and mineralogical characteristics of both raw materials are favorable for mullite formation, vitrification, and the development of a porous yet mechanically stable sintered fly ash aggregate. These results confirm the suitability of hydraulic fly ash from the Kyzylorda CHP plant and Talsuat clay as raw materials for the production of lightweight aggregate and provide the basis for further investigation of aggregate formation, microstructure evolution, and engineering performance. The mineralogical composition of the fly ash determined by X-ray diffraction analysis is presented in Figure 1.

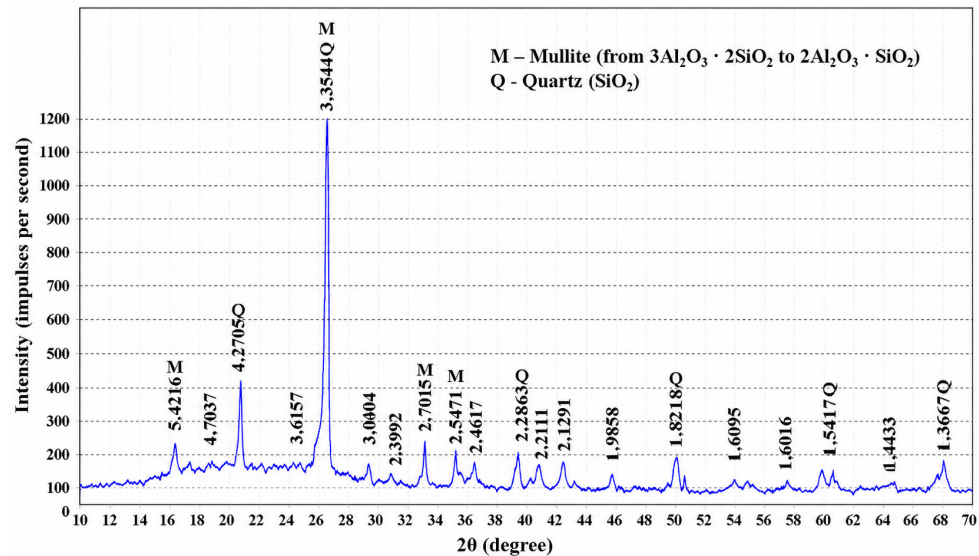


Figure 1. XRD Pattern of Hydraulic Fly Ash from the Kyzylorda CHP Plant.

The XRD pattern of the hydraulic fly ash and the corresponding diffraction characteristics are presented in Figure 1 and Table 2, respectively. The analysis revealed that quartz (SiO_2) and mullite ($3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$) are the dominant crystalline phases. Quartz and mullite accounted for approximately 71.5% and 28.5% of the crystalline fraction, respectively, while a minor amorphous glassy phase (~5–6%) was also detected. The predominance of quartz and mullite confirms the aluminosilicate nature of the fly ash and indicates its suitability as a raw material for the production of sintered lightweight aggregates.

The presence of mullite is particularly important because this phase is associated with high thermal stability, dimensional stability, and mechanical strength of fired aluminosilicate materials. In contrast, the amorphous glassy phase can soften during thermal treatment, promoting sintering, partial vitrification, and the formation of strong interparticle bonds within the aggregate structure. These processes contribute to the development of a mechanically stable framework while maintaining the porous structure required for lightweight aggregate applications.

Similar phase compositions have been reported for fly ashes successfully used in lightweight aggregate production, where quartz, mullite, and amorphous phases play important roles in controlling porosity, strength, and durability. The coexistence of crystalline aluminosilicate phases and a limited amount of glassy material is considered beneficial because it provides a balance between structural stability and sintering activity during firing.

Overall, the mineralogical composition of the investigated fly ash is favorable for the production of sintered lightweight aggregate and supports the development of a porous yet mechanically stable structure during thermal treatment. The identified phase assemblage also suggests favorable conditions for mullite preservation and further aluminosilicate phase development during firing, which are expected to contribute positively to the

engineering properties of the final aggregate. The interplanar spacings (d-values) and the corresponding mineral phases identified from the XRD analysis are summarized in Table 2.

Table 2. Interplanar Spacings (d-Values) and Mineral Phases Identified in Fly Ash by XRD Analysis.

No	2θ Angle (°)	Peak Area	Peak Intensity (cps)	FWHM (°) Hemece Full Width at Half Maximum (°)	Interplanar Spacing, d (Å)	Peak Maximum (cps)	Integrated Area	Area/Intensity Ratio
1	16.335	36.71	59.9975	0.5645	5.4216	7.69	16	0.6119
2	17.350	48.45	43.3800	1.0307	5.1068	5.51	21	1.1170
3	18.850	28.05	49.7800	0.5199	4.7037	6.41	12	0.5635
4	20.782	64.23	165.7350	0.3575	4.2705	21.28	28	0.3876
5	24.600	15.41	58.6250	0.2424	3.6157	7.56	7	0.2628
6	26.550	232.12	779.7550	0.2746	3.3544	100.00	100	0.2977
7	29.350	14.27	39.9550	0.3295	3.0404	5.13	6	0.3571
8	30.893	6.62	25.8300	0.2365	2.8920	3.33	3	0.2564
9	33.132	16.91	42.7200	0.3651	2.7015	5.51	7	0.3959
10	35.205	12.43	52.9575	0.2165	2.5471	6.79	5	0.2347
11	36.467	16.47	40.3000	0.3772	2.4617	5.13	7	0.4088
12	39.376	22.94	59.1750	0.3578	2.2863	7.56	10	0.3877

The results of the specific effective activity of natural radionuclides (Table 3) indicate that the fly ash from the Kyzylorda CHP plant belongs to Class I building materials according to GOST 30108-94, allowing its use in construction without radiological restrictions. The measured activity was below the regulatory limit, confirming the radiological safety of the material for the production of sintered lightweight aggregates and lightweight cementitious composite.

The loss on ignition (LOI) was 3.46 wt.%, indicating a low content of unburned carbon and volatile matter. Such a low LOI is favorable for aggregate production, as excessive residual carbon may adversely affect sintering and reduce the mechanical performance of the final product.

Overall, the radiological and physicochemical properties confirm that the investigated fly ash is an environmentally safe and technically suitable raw material for the production of sintered fly ash aggregate. The physical properties and specific effective activity of natural radionuclides determined for the fly ash are presented in Table 3.

Table 3. Physical Properties and Specific Effective Activity of Natural Radionuclides in Fly Ash.

No	Indicator Name, Unit of Measurement	Reference Standard for Test Methods	Requirement According to the Standard	Measured Value	Note
1	2	3	4	5	6
1	Specific Effective Activity of Natural Radionuclides, Bq/kg	GOST 30108-94. Building Materials and Products. Determination of the Specific Effective Activity of Natural Radionuclides.	≤370 Bq/kg	55.1	Complies with

The chemical composition of the Talsuat clay (Table 4) demonstrates its suitability for the production of sintered fly ash aggregate. The relatively high contents of CaO (9.62 wt.%) and K₂O (2.21 wt.%) promote liquid-phase formation and enhance sintering, while the low impurity content contributes to the development of a stable aggregate structure

during firing. The clay exhibited a specific surface area of $2775 \text{ cm}^2 \text{ g}^{-1}$ and a bulk density of 755 kg m^{-3} , indicating good reactivity and compatibility with fly ash.

The fly ash is rich in SiO_2 (60.98 wt.%) and Al_2O_3 (26.40 wt.%), confirming its aluminosilicate nature and suitability for mullite formation during thermal treatment. The combined use of fly ash and clay provides a complementary raw-material system in which fly ash supplies silica and alumina, while clay contributes additional aluminosilicate and fluxing components. This interaction promotes sintering, partial vitrification, and the formation of a porous yet mechanically stable aggregate structure. The chemical compositions of the fly ash and Talsuat clay used in this study are presented in Table 4.

Table 4. Chemical Composition of Fly Ash and Talsuat Clay (wt.%).

Material	SiO_2	Al_2O_3	CaO	Fe_2O_3	MgO	K_2O	Na_2O	SO_3	TiO_2	LOI	Total
Fly ash	60.98	26.40	2.90	3.27	1.82	1.55	1.12	0.60	0.83	–	100.00
Clay	51.84	12.90	9.62	5.50	2.58	2.21	1.65	–	0.72	0.15	100.00

The elemental compositions of the hydraulic fly ash from the Kyzylorda CHP plant and the clay from the Talsuat deposit are presented in Table 4. The obtained results indicate that both materials belong to the class of aluminosilicate raw materials, although they differ considerably in the contents of major oxides and fluxing components. The fly ash is characterized by a high content of SiO_2 (60.98 wt.%) and Al_2O_3 (26.40 wt.%), with the combined content of silica, alumina, and iron oxide reaching 90.65 wt.%. Such a composition is typical of high-silica coal fly ashes and provides favorable conditions for the formation of quartz, mullite, and other thermally stable aluminosilicate phases during firing. The relatively low content of CaO (2.90 wt.%) indicates that the ash is predominantly siliceous in nature and is therefore expected to exhibit high thermal stability during heat treatment.

In contrast, the Talsuat clay contains lower amounts of silica (51.84 wt.%) and alumina (12.90 wt.%), but significantly higher concentrations of CaO (9.62 wt.%) and K_2O (2.21 wt.%). These oxides act as fluxing agents during thermal treatment, promoting liquid-phase formation, accelerating sintering processes, and improving particle bonding within the aggregate structure. The increased concentration of fluxing components is expected to facilitate partial vitrification and enhance the formation of a consolidated aluminosilicate matrix during firing. The low loss on ignition (0.15 wt.%) further indicates the high purity and thermal stability of the clay.

The combined use of fly ash and clay is therefore advantageous because it provides a balanced chemical composition. Fly ash serves as the primary source of silica and alumina, while the clay supplies additional aluminosilicate material together with fluxing oxides that facilitate densification and phase transformation during firing. Such synergy promotes the formation of a porous but mechanically stable aggregate structure with improved physical and mechanical properties. The coexistence of network-forming oxides (SiO_2 and Al_2O_3) and fluxing oxides (CaO and K_2O) creates favorable conditions for controlled sintering and the development of a stable pore system, which are essential for producing lightweight aggregates with adequate strength and durability.

Overall, the chemical compositions of both raw materials are well suited for the production of sintered fly ash aggregate. The high aluminosilicate content of the fly ash combined with the fluxing capability of the clay is expected to ensure effective sintering, mullite formation, and the development of durable lightweight aggregate suitable for lightweight cementitious composite applications. These findings are consistent with the mineralogical observations discussed previously and confirm the suitability of the selected raw materials for the subsequent stages of aggregate production. A comparative elemental analysis of the fly ash and Talsuat clay is presented in Table 5.

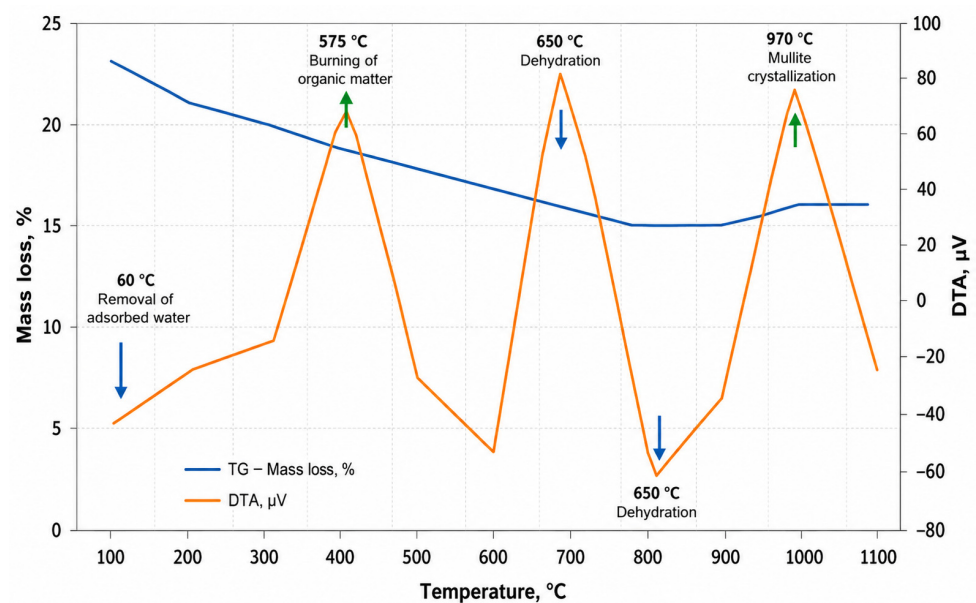
Table 5. Comparative Elemental Composition of Fly Ash and Talsuat Clay (wt.%).

Material	O	Si	Fe	Na	Al	S	Ti	Mg	Ca	K	Cl	P	Σ
Coal Fly Ash	48.39	28.6	2.55	0.84	14.01	0.24	0.50	1.10	2.08	1.29	0.29	0.19	100
Clay	46.8	24.2	1.97	0.2	17.03	0.2	1.0	0.34	0.8	0.6	Trace	Trace	100

The FTIR spectra revealed characteristic absorption bands typical of aluminosilicate materials. The intense band observed in the range of 1000–1100 cm^{-1} corresponds to Si–O–Si stretching vibrations, confirming the predominance of silicate structures within the investigated materials. Broad absorption bands at 3400–3600 cm^{-1} and 1600–1650 cm^{-1} are associated with hydroxyl groups and molecular water, respectively. These features indicate the presence of physically adsorbed and structurally bound water in the raw materials prior to thermal treatment.

The UV–Vis spectra recorded in the wavelength range of 200–400 nm exhibited absorption maxima at 250–300 nm, corresponding to electronic transitions within aluminosilicate and iron-containing phases. Variations in absorption intensity after firing indicate structural rearrangements associated with phase evolution and increased ordering of the aluminosilicate framework. Together, the FTIR and UV–Vis results confirm the formation of a stable aluminosilicate structure during thermal treatment. The reduced intensity of hydroxyl-related bands after firing reflects dehydration and structural stabilization, while the persistence of Si–O–Si vibrations is consistent with the presence of quartz and mullite phases identified by XRD analysis.

The thermal behavior of the fly ash is presented in Figure 2. Thermal analysis revealed an endothermic effect near 60 °C, corresponding to the removal of physically adsorbed water, and a second endothermic effect around 650 °C associated with dehydroxylation processes. An exothermic peak at approximately 575 °C was attributed to the combustion of residual carbonaceous matter, whereas a pronounced thermal effect at about 970 °C corresponds to mullite crystallization and the formation of stable aluminosilicate phases. These thermal events are in good agreement with the phase composition identified by XRD and the structural changes observed in the FTIR spectra.

**Figure 2.** TG–DTA Curves of Fly Ash Used for the Production of Sintered Fly Ash Aggregate.

The fly ash exhibited a true density of 2.41 g cm^{-3} , a specific surface area of $2775 \text{ cm}^2 \text{ g}^{-1}$, a bulk density of 755 kg m^{-3} , and a carbon content of 5.5 wt.%. Based on the fusion temperatures ($t_A = 1205 \text{ }^\circ\text{C}$, $t_B = 1360 \text{ }^\circ\text{C}$, and $t_C = 1375 \text{ }^\circ\text{C}$), the ash can be classified as medium-fusibility ash. These characteristics indicate favorable conditions for controlled sintering and pore formation during thermal treatment.

The thermal analysis results demonstrate that the principal phase transformations are completed within the temperature range of approximately $950\text{--}1000 \text{ }^\circ\text{C}$. In particular, the mullite crystallization peak observed at about $970 \text{ }^\circ\text{C}$ confirms the suitability of the selected firing temperature of $1100 \text{ }^\circ\text{C}$. This temperature provides sufficient thermal energy for effective sintering, partial vitrification, and aluminosilicate phase development while preserving the porous structure required for lightweight aggregate applications. Consequently, the selected firing regime promotes the formation of a mechanically stable and durable aggregate with favorable engineering properties. The thermogravimetric and differential thermal analysis results illustrating these phase transformations are presented in Figure 2.

The morphology and microstructure of the produced sintered fly ash aggregate are presented in Figure 3, while the corresponding elemental composition determined by EDS analysis is summarized in Table 6. The aggregate consisted mainly of spherical and sub-spherical granules with a relatively uniform size distribution. The particles exhibited rough and partially porous surfaces formed during firing and gas-release processes. The granules retained their shape without visible cracking or deformation, indicating effective sintering and sufficient mechanical stability. Such morphology is typical of sintered lightweight aggregates and contributes to reduced density while maintaining adequate strength.

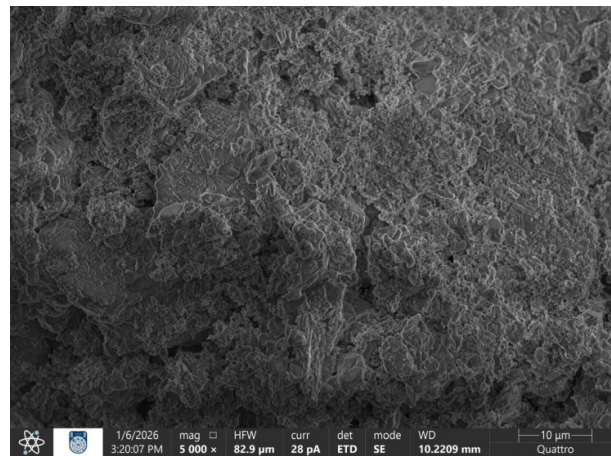


Figure 3. SEM Micrograph of the Sintered Fly Ash Aggregate ($\times 5000$ Magnification).

SEM observations revealed a consolidated porous microstructure with interconnected pores ranging from approximately 1 to $20 \text{ }\mu\text{m}$ and partially vitrified particle clusters. The developed pore network is associated with moisture evaporation, decomposition reactions, and gas release during thermal treatment and is beneficial for reducing density and improving thermal insulation properties. At the same time, the presence of partially vitrified regions indicates the formation of strong interparticle bonds, which contribute to the mechanical integrity of the aggregate. The coexistence of a well-developed pore system and a consolidated matrix suggests that the selected firing regime provided a favorable balance between lightweight characteristics and structural stability.

EDS analysis (Table 6) showed that Si, Al, Ca, Fe, and O were the dominant elements, confirming the aluminosilicate–calcium composition of the aggregate. The high contents

of silicon and aluminum are consistent with the XRF and XRD results and support the presence of quartz and mullite phases, whereas calcium- and iron-bearing compounds promoted liquid-phase sintering and partial vitrification during firing. The elemental distribution further confirms that the aggregate structure was formed through interactions between silica-rich fly ash components and fluxing constituents supplied by the clay, resulting in a chemically stable aluminosilicate matrix.

Overall, the SEM–EDS results demonstrate the formation of a porous yet mechanically stable microstructure composed of crystalline aluminosilicate phases embedded within a partially vitrified matrix. The observed microstructural features are in agreement with the mineralogical and thermal analysis results and explain the favorable physical and mechanical properties of the aggregate. Such a structure provides an effective balance between low density, mechanical strength, and durability, making the developed aggregate suitable for use in structural-insulating lightweight cementitious composite. The elemental composition determined by EDS analysis for the sintered fly ash aggregate is summarized in Table 6.

Table 6. Elemental Composition of the Sintered Fly Ash Aggregate.

Elements	Sintered Fly Ash Aggregate
C	0.9 ± 0.1
O	34.8 ± 0.3
F	0.0
Na	1.5 ± 0.1
Mg	2.3 ± 0.1
Al	6.6 ± 0.1
Si	19.8 ± 0.1
S	0.6 ± 0.1
K	2.7 ± 0.1
Ca	21.1 ± 0.4
Fe	9.7 ± 0.1

The microstructural analysis of the sintered fly ash aggregate (Figure 3) at a magnification of ×5000 revealed a porous aluminosilicate matrix composed of interconnected particles and sintered agglomerates. The aggregate surface exhibited an irregular morphology characterized by a rough texture and a well-developed pore network distributed throughout the structure. Both open and partially closed pores were observed, indicating the occurrence of intensive gas-release processes during thermal treatment.

The pore system was formed during firing through moisture evaporation, combustion of residual carbonaceous matter, gas evolution, and partial vitrification of the aluminosilicate matrix. The presence of interparticle necks, consolidated particle contacts, and vitrified regions confirms that the selected firing temperature (1100 °C) was sufficient to promote effective sintering and the development of a mechanically stable structure. Similar microstructural features have been reported for sintered fly ash aggregates, where vitrification and mullite formation significantly enhance strength, durability, and resistance to environmental воздействия.

The observed microstructure reflects a balance between pore development and matrix consolidation. While the pore network contributes to density reduction and improved thermal insulation performance, the vitrified aluminosilicate framework provides the structural continuity required for mechanical stability. This combination is particularly important for lightweight aggregates intended for structural-insulating lightweight cementitious composite applications.

The absence of extensive cracking, excessive pore coalescence, or structural collapse indicates that the selected firing conditions were appropriate for producing a stable lightweight aggregate. Furthermore, the homogeneous distribution of pores suggests uniform heat transfer and phase transformation throughout the granules during firing. These observations are consistent with the measured density, water absorption, and crushing strength of the aggregate and agree with previous studies on fly ash-based lightweight aggregates and lightweight cementitious composites. Overall, the SEM analysis confirms that the developed firing regime promoted the formation of a porous yet mechanically robust microstructure suitable for lightweight aggregate production. A representative SEM micrograph illustrating the pore structure and morphology of the sintered fly ash aggregate is shown in Figure 3.

The results of the physical and mechanical tests performed on the sintered fly ash aggregate are presented in Table 7. The investigated properties included bulk density, water absorption, crushing strength, and frost resistance, which are key parameters for evaluating lightweight aggregates intended for lightweight cementitious composite production. The obtained values were assessed according to the requirements established for coal fly ash and lightweight aggregates in ASTM C618-24, ASTM C330/C330M-24, and GOST 9758-2012. The results were further compared with previously reported fly ash-based lightweight aggregates to evaluate the performance of the developed material. A summary of the measured physical and mechanical properties of the produced sintered fly ash aggregate, together with a comparison to expanded clay aggregate, is provided in Table 7.

Table 7. Physical and Mechanical Properties of the Produced Sintered Fly Ash Aggregate Compared with Expanded Clay Aggregate.

Property, Unit	Produced Sintered Fly Ash Aggregate	Expanded Clay Aggregate (Reference Material)
Particle size fraction, mm	10–20	10–20
Bulk density, kg m ⁻³	1118	806
True density, g cm ⁻³	1.72	1.66
Average density, g cm ⁻³	1.742	1.651
Water absorption, %	5.4	11.0
Crushing strength in cylinder	M200 (6.1 MPa)	M150
Frost resistance	≥F35	F21

The physical and mechanical properties of the developed sintered fly ash aggregate are presented in Table 7. The aggregate exhibited a bulk density of 1118 kg m⁻³, water absorption of 5.4%, crushing strength corresponding to grade M200, and frost resistance not lower than F35. These values satisfy the requirements specified for lightweight aggregates intended for structural and structural-insulating lightweight cementitious composite applications. Furthermore, the low loss on ignition of the source fly ash and its predominantly aluminosilicate composition comply with the quality criteria established for fly ash used in construction materials.

The obtained results indicate that the developed sintered fly ash aggregate possesses properties suitable for structural and structural-insulating lightweight cementitious composite applications. The aggregate exhibited a bulk density of 1118 kg m⁻³, which is higher than that of the reference expanded clay aggregate (806 kg m⁻³). This increase can be attributed to the formation of a partially vitrified aluminosilicate matrix and stable crystalline phases during firing, which contributed to the consolidation of the aggregate structure. Similar density values have been reported for sintered fly ash aggregates produced under optimized firing conditions.

The measured water absorption of 5.4% was approximately two times lower than that of expanded clay (11%), indicating the development of a denser internal structure with limited capillary porosity. Reduced water absorption is advantageous for lightweight cementitious composite production because it improves mixture stability, limits moisture ingress, and enhances long-term durability. Comparable reductions in water absorption have been observed in lightweight aggregates containing vitrified aluminosilicate phases derived from industrial by-products.

The aggregate demonstrated a crushing strength corresponding to grade M200, exceeding that of the reference expanded clay aggregate (M150). The enhanced strength is attributed to mullite formation, partial vitrification, and the development of strong interparticle bonds during sintering. These observations are consistent with the XRD, thermal analysis, and SEM results, which confirmed the presence of stable aluminosilicate phases and a consolidated microstructure. In addition, the aggregate exhibited frost resistance not lower than F35, which is consistent with values reported for high-performance fly ash-based lightweight aggregates.

SEM observations revealed a porous microstructure with interconnected pores ranging from approximately 1 to 20 μm , which is consistent with the measured density and water absorption values. The coexistence of a porous structure and a partially vitrified aluminosilicate–calcium matrix provides a favorable balance between low density, mechanical strength, and durability. The observed microstructure explains the ability of the aggregate to maintain relatively low density while achieving satisfactory strength and frost resistance.

Overall, the developed aggregate demonstrated superior performance in terms of crushing strength, water absorption, and frost resistance compared with conventional expanded clay aggregate. The obtained properties confirm its suitability for use as a coarse aggregate in structural and structural-insulating lightweight cementitious composite. Furthermore, the utilization of hydraulic fly ash from the Kyzylorda CHP plant contributes to the valorization of industrial waste, reduces the environmental burden associated with ash disposal, and supports the development of sustainable construction materials with improved engineering performance. A comparison of the physical and mechanical properties reported for sintered fly ash aggregates in previous studies and those obtained in the present investigation is presented in Table 8.

Table 8. Comparison of Physical and Mechanical Properties of Sintered Fly Ash Aggregates Reported in the Literature and the Present Study.

Study	Bulk Density (kg/m^3)	Water Absorption (%)	Strength
Nair and Ramamurthy (2010) [32]	950	6–12	5.5 MPa
Patel et al. (2019) [9]	1040	6.4	5.8 MPa
Domagała (2020) [5]	1000	7.1	Grade M150
Present study	1118	5.4	Grade M200

The comparison with previously published studies indicates that the sintered fly ash aggregate developed in this research exhibits lower water absorption and higher crushing strength than most reported fly ash-based lightweight aggregates. The reduced water absorption is associated with the formation of a denser aluminosilicate matrix and a lower proportion of interconnected pores. The higher strength of the aggregate can be attributed to the development of stable crystalline phases, particularly mullite, and to the enhanced sintering achieved during firing at 1100 $^{\circ}\text{C}$. These characteristics demonstrate the potential of the produced aggregate for use in structural lightweight cementitious composite applications requiring improved durability and mechanical performance. The favorable

physical and mechanical properties obtained in this study are consistent with the mineralogical, thermal, and microstructural analyses, which confirmed the formation of a partially vitrified aluminosilicate matrix reinforced by stable crystalline phases.

3.2. Chemical Durability of Aggregates in Aggressive Environments

In addition to physical and mechanical performance, the long-term durability of lightweight aggregates under chemically aggressive conditions is an important factor affecting their suitability for lightweight cementitious composite applications. To evaluate the resistance of the developed sintered fly ash aggregate to aggressive environments, chemical durability tests were conducted in accordance with GOST 8269.0–97, “Determination of the Resistance of Crushed Stone and Gravel Structure to Disintegration”.

The aggregate specimens were exposed to three chemically aggressive environments representing common service conditions encountered by lightweight cementitious composite structures:

- 5 wt.% NaOH solution (alkaline environment);
- 5 wt.% NaCl solution (chloride environment);
- 5 wt.% H₂SO₄ solution (acidic environment).

The selected environments simulate the principal mechanisms of chemical deterioration associated with alkaline exposure, chloride penetration, and acid attack. Aggregate samples were immersed in the corresponding solutions for 30 days under laboratory conditions. After exposure, the specimens were rinsed with distilled water, dried to constant mass at 105 ± 5 °C, and weighed to determine the mass change resulting from chemical interaction with the surrounding medium.

The chemical stability of the aggregate was assessed by determining the percentage mass loss and calculating the stability coefficient (K) according to Equation (X). The obtained results provide an assessment of the resistance of the developed aggregate to chemically aggressive media and allow comparison with conventional lightweight aggregates used in construction. The corresponding results are presented and discussed in the following section. The visual condition of the sintered fly ash aggregate and expanded clay aggregate after exposure to aggressive chemical solutions is illustrated in Figure 4.

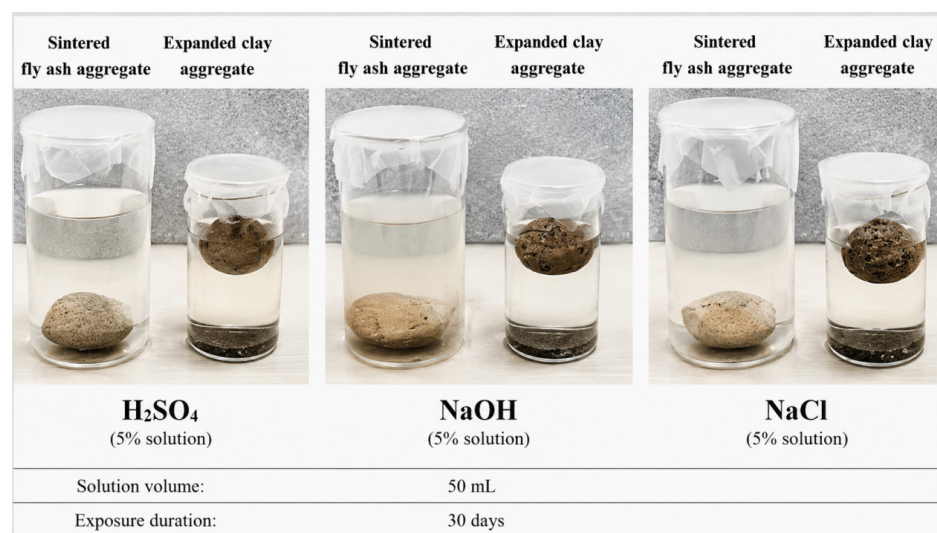


Figure 4. Visual Appearance of the Sintered Fly Ash Aggregate and Expanded Clay Aggregate after Exposure to Aggressive Chemical Solutions (5% NaCl, 5% NaOH, and 5% H₂SO₄).

The results presented in Tables 9 and 10 and Figure 4 indicate that the developed sintered fly ash aggregate exhibits higher chemical durability than the reference expanded clay aggregate. The highest stability was observed in the 5% NaCl solution ($K = 0.98$), where the mass loss was only 2.3%, indicating limited chloride penetration into the aluminosilicate matrix. The greatest mass loss (23.3%) occurred in the 5% NaOH solution, although the aggregate maintained satisfactory stability ($K = 0.77$). In the 5% H_2SO_4 solution, the stability coefficient reached 0.91, demonstrating good resistance to acidic attack. In contrast, the expanded clay aggregate exhibited substantially lower durability, with an average stability coefficient of only 0.27. The superior chemical resistance of the developed aggregate is attributed to the formation of quartz, mullite, and a partially vitrified aluminosilicate matrix during firing, which effectively protects the material against aggressive chemical environments. The measured mass loss values of the sintered fly ash aggregate after 30 days of exposure to the aggressive chemical solutions are summarized in Table 9.

Table 9. Mass Loss of the Sintered Fly Ash Aggregate after 30 Days of Exposure to Aggressive Chemical Solutions.

Chemical Environment	Initial Mass, m_0 (g)	Final Mass, m_1 (g)	Mass Loss (%)
5% NaOH	9.20	7.05	23.4
5% NaCl	7.10	6.94	2.3
5% H_2SO_4	10.00	9.06	9.4

Table 10. Mix Proportions of Lightweight cementitious composite.

Component	Amount
Cement	330 g
Sand	715 g
Sintered fly ash aggregate	850 g
Water	180 mL
Water-to-cement ratio	0.55
Specimen size	70 mm × 70 mm × 70 mm
Number of specimens	3

The results of the chemical resistance tests demonstrated that the developed sintered fly ash aggregate exhibited significantly higher durability in aggressive environments than the reference expanded clay aggregate. The average chemical stability coefficient of the sintered fly ash aggregate was approximately 0.89, whereas the corresponding value for expanded clay was only 0.27, indicating substantially greater resistance to chemical degradation. The enhanced durability can be attributed to the formation of a dense aluminosilicate matrix during firing, as confirmed by the XRD, FTIR, and SEM analyses.

The highest stability was observed in the sodium chloride solution, where the stability coefficient reached 0.98, and the mass loss was only 2.3%. This behavior indicates that chloride ions have limited interaction with the aluminosilicate matrix of the aggregate. The low permeability of the partially vitrified structure effectively restricts ion penetration and minimizes structural deterioration. These results suggest excellent resistance of the aggregate to chloride-containing environments commonly encountered in lightweight cementitious composite structures exposed to deicing salts or marine conditions.

Exposure to the alkaline solution (5% NaOH) resulted in the highest mass loss among the tested environments (23.3%). This reduction is associated with the partial dissolution of amorphous aluminosilicate phases and depolymerization of Si–O–Si bonds under strongly alkaline conditions. Nevertheless, the aggregate retained a relatively high stability coefficient (0.77), demonstrating adequate resistance to alkaline attack and maintaining structural integrity after exposure.

In the sulfuric acid solution, the aggregate exhibited moderate mass loss (9.4%) and a stability coefficient of 0.91. The observed deterioration is attributed to the leaching of calcium-containing phases and partial decomposition of reaction products formed during firing. However, the silica–alumina framework remained largely intact, preventing severe structural degradation and preserving the overall stability of the aggregate.

The superior chemical resistance of the sintered fly ash aggregate can be attributed to the presence of quartz, mullite, and vitrified aluminosilicate phases within a consolidated microstructure. These phases are characterized by high chemical stability and effectively protect the aggregate against aggressive chemical environments. The results are consistent with previous studies reporting enhanced durability of fly ash-based lightweight aggregates containing well-developed aluminosilicate matrices and mullite-rich structures.

Overall, the obtained results demonstrate that the developed aggregate possesses high resistance to chloride, alkaline, and moderately acidic environments. The combination of low permeability, stable mineral phases, and a partially vitrified microstructure makes the aggregate suitable for application in structural and structural-insulating lightweight cementitious composite exposed to chemically aggressive service conditions.

3.3. Preparation and Testing of Lightweight Cementitious Composite Specimens

To evaluate the applicability of the developed sintered fly ash aggregate as a coarse aggregate in lightweight cementitious composite, a series of lightweight cementitious composite specimens was produced (Figure 5). The lightweight cementitious composite mixture consisted of Portland cement, natural sand, sintered fly ash aggregate with a particle size of 5–10 mm, and water. The mixture composition included 330 g of cement (15.39 wt.%), 715 g of sand (33.33 wt.%), 850 g of sintered fly ash aggregate (39.63 wt.%), and 180 mL of water (8.39 wt.%). The corresponding water-to-cement ratio (w/c) was 0.55.

Three cubic specimens with dimensions of 70 mm × 70 mm × 70 mm were cast from the prepared mixture. To ensure uniform distribution of the constituents and minimize entrapped air, the fresh lightweight cementitious composite was compacted using a laboratory vibrating table. Vibration compaction promoted improved particle packing and enhanced the bond between the cementitious matrix and the lightweight aggregate.

After casting, the specimens were covered with polyethylene film to prevent premature moisture loss and cured under laboratory conditions at a temperature of 20 ± 2 °C for 3 days. The specimens were subsequently demoulded, weighed, and dried for 7 days until a conditionally constant mass was achieved. The selected curing and preparation procedure ensured consistent specimen quality and provided comparable conditions for subsequent testing.

The prepared specimens were used to determine density, compressive strength, and water absorption in order to assess the engineering performance of lightweight cementitious composite containing the developed sintered fly ash aggregate. The obtained results provide an evaluation of the suitability of the aggregate for structural-insulating lightweight cementitious composite applications and are discussed in the following section. The mix proportions adopted for the production of the lightweight cementitious composite specimens are presented in Table 10. Representative lightweight cementitious composite specimens containing the developed sintered fly ash aggregate after 7 days of curing are shown in Figure 5.



Figure 5. Lightweight cementitious composite specimens containing sintered fly ash aggregate after 7 days of curing.

The prepared lightweight cementitious composite specimens exhibited a relatively uniform geometric shape and a dense external surface. The exposed surfaces revealed the presence of sintered fly ash aggregate particles embedded within the cementitious matrix. Small isolated pores and surface cavities were observed, indicating the porous nature of the lightweight aggregate and confirming the formation of a lightweight cementitious composite structure.

No visible macrocracks, segregation, or significant surface defects were detected after the curing period, suggesting adequate compaction and satisfactory bonding between the cement paste and the aggregate particles. The relatively homogeneous distribution of aggregates within the lightweight cementitious composite matrix indicates successful mixing and vibration compaction. The observed morphology is consistent with the measured density of approximately 1600 kg m^{-3} and water absorption values of 15–16%, reflecting the presence of a developed pore system typical of a structural-insulating lightweight cementitious composite.

The visual appearance of the specimens confirms the feasibility of using the developed sintered fly ash aggregate as a coarse aggregate for lightweight cementitious composite production.

The density and compressive strength of the lightweight cementitious composite produced with the developed sintered fly ash aggregate were determined in accordance with the relevant testing procedures. Compression tests were performed using a hydraulic testing machine under a constant loading rate of 5.53 kN s^{-1} . The maximum recorded load was 18.91 kN. The compressive strength was calculated using Equation (1):

$$f_c = P/A, \quad (1)$$

where (f_c) is the compressive strength (MPa), (P) is the maximum applied load (N), and (A) is the cross-sectional area of the specimen (m^2).

Based on the test results, the compressive strength of the lightweight cementitious composite reached 3.87 MPa, corresponding to strength class B3.5 according to GOST 25820-2014 “*Lightweight cementitious composite. Specifications*”. The obtained strength indicates that the developed material is suitable for structural-insulating applications where reduced self-weight and adequate mechanical performance are required.

The density of the lightweight cementitious composite specimens was determined from the ratio of specimen mass to volume according to Equation (2):

$$\rho = m/v, \quad (2)$$

where ρ is the density (kg m^{-3}), (m) is the specimen mass (kg), and (V) is the specimen volume (m^3).

The measured densities ranged from approximately 1600 to 1690 kg m⁻³, confirming that the developed material belongs to the category of lightweight cementitious composite and corresponds to density class D1600 according to GOST 25820-2014. The relatively low density is primarily associated with the porous internal structure of the sintered fly ash aggregate, which reduces the overall weight of the lightweight cementitious composite while preserving sufficient mechanical integrity.

The obtained results demonstrate that the incorporation of sintered fly ash aggregate enabled the production of a lightweight cementitious composite with reduced density while maintaining satisfactory compressive strength. The combination of strength class B3.5 and density class D1600 reflects a favorable balance between mechanical performance and weight reduction. These characteristics are consistent with the mineralogical composition, thermal behavior, and porous microstructure of the aggregate discussed in the previous sections and confirm its suitability for structural-insulating lightweight cementitious composite applications. The measured physical and mechanical properties of the lightweight cementitious composite produced with the sintered fly ash aggregate are summarized in Table 11.

Table 11. Physical Properties of Lightweight Concrete.

Specimen	Density (kg/m ³)	Water Absorption (%)
1	1600	16.4
2	1690	15.2
3	1682	16.5
Average	1657	16.0

As shown in Table 11, the lightweight cementitious composite had an average density of 1657 kg m⁻³ and water absorption of 16%. These values are typical of lightweight cementitious composites produced with porous aggregates and are mainly associated with the developed pore structure of the sintered fly ash aggregate. The mix proportions used for the production of the lightweight cementitious composite are presented in Table 12.

Table 12. Mix Proportions of Lightweight cementitious composite.

Study	Density (kg/m ³)	Compressive Strength (MPa)
Domagała (2020) [5]	1600–1800	10–30
Patel et al. (2019) [9]	1500–1700	8–25
Satpathy et al. (2019) [31]	1450–1750	5–20
Present study	1657	3.87

Comparison with literature data (Table 12) shows that the developed lightweight cementitious composite has a density comparable to previously reported lightweight cementitious composites, while exhibiting lower compressive strength. The reduced strength is mainly related to the porous structure of the aggregate. Despite this limitation, the material is suitable for structural-insulating applications and contributes to the sustainable utilization of industrial waste.

The developed lightweight cementitious composite exhibited an average density of 1657 kg m⁻³, a compressive strength of 3.87 MPa, and water absorption of approximately 16%. Although the strength was lower than values reported in previous studies, the material satisfies the requirements for structural-insulating and non-load-bearing applications. These results confirm the potential of sintered fly ash aggregate as a sustainable lightweight cementitious composite aggregate. The water absorption values measured for the lightweight cementitious composite specimens are summarized in Table 13.

Table 13. Water Absorption of Lightweight cementitious composite Specimens.

Specimen	Dry Mass (g)	Saturated Mass (g)	Water Absorption (%)
1	548	638	16.4
2	580	668	15.2
3	577	672	16.5
Average	–	–	16.0

The compressive strength of fly ash-based cementitious composites is expected to increase with curing time due to the ongoing pozzolanic reaction between the amorphous silica present in fly ash and calcium hydroxide released during cement hydration. This reaction produces additional calcium silicate hydrate (C–S–H) gel, which refines the pore structure, strengthens the interfacial transition zone, and contributes to the gradual improvement of mechanical properties. Therefore, further strength development beyond the testing age can be expected for the developed lightweight cementitious composite.

3.4. Water Absorption of Lightweight Cementitious Composite

Water absorption was determined according to GOST 12730.3-2020 using the standard gravimetric method (3):

$$W = \frac{m_{\text{sat.}} - m_{\text{dry}}}{m_{\text{dry}}} \times 100\%, \quad (3)$$

where m_{sat} is the saturated mass and m_{dry} is the oven-dry mass of the specimen.

The average water absorption of the developed lightweight cementitious composite was 16.0%. This value is mainly attributed to the porous structure of the sintered fly ash aggregate and the presence of interconnected pores within the lightweight cementitious composite matrix, as confirmed by SEM observations. Similar water absorption levels have been reported for lightweight cementitious composites produced with porous artificial aggregates. Although increased water absorption may reduce moisture resistance, the developed pore structure contributes to lower density and improved thermal insulation performance. The obtained results are consistent with the measured average density of 1657 kg m^{-3} and confirm the formation of a lightweight cementitious composite structure suitable for structural-insulating and non-load-bearing applications according to GOST 25820-2014.

3.5. Elemental Distribution Analysis by EDS Mapping

Elemental distribution maps obtained by EDS mapping analysis demonstrated a relatively homogeneous distribution of the principal elements within the sintered fly ash aggregate microstructure (Figure 6). Silicon and aluminum were uniformly dispersed throughout the matrix, confirming the formation of a continuous aluminosilicate framework. The co-localization of Si and Al suggests the development of thermally stable aluminosilicate phases, which is consistent with the XRD results indicating the predominance of quartz and mullite. Calcium was mainly concentrated within partially vitrified regions and interparticle bonding zones, indicating its participation in liquid-phase sintering during firing at 1100°C . Iron was distributed in isolated areas and was associated with iron-bearing mineral inclusions inherited from the original fly ash. Oxygen was uniformly distributed over the entire aggregate surface, confirming the oxide nature of the identified phases.

The EDS elemental maps further revealed that no significant elemental segregation occurred during thermal treatment, suggesting effective mixing of fly ash and clay components before pelletization. The homogeneous distribution of Si, Al, Ca, Fe and O contributed to the formation of a chemically stable and mechanically coherent porous

aluminosilicate matrix. These observations support the SEM findings and explain the favorable crushing strength, low water absorption, and high chemical durability of the developed aggregate.

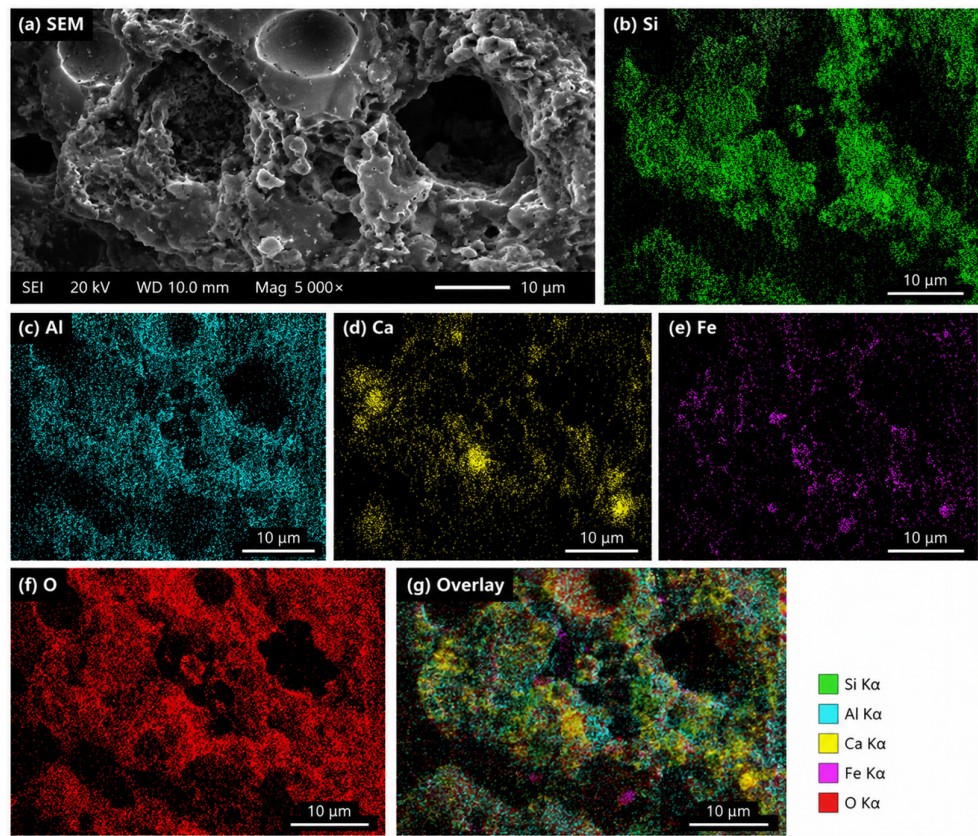


Figure 6. SEM morphology ($\times 5000$ magnification) and corresponding EDS elemental maps illustrating the distribution of silicon, aluminum, calcium, iron, and oxygen in the sintered fly ash aggregate produced from hydraulic fly ash of the Kyzylorda CHP plant: (a) SEM micrograph ($\times 5000$); (b) Si elemental map; (c) Al elemental map; (d) Ca elemental map; (e) Fe elemental map; (f) O elemental map; (g) overlay elemental distribution map.

The SEM observations presented in Figure 7 confirmed the progressive evolution of the pore structure during sintering. Images (A) and (B) revealed the formation of interconnected open pores associated with gas evolution and moisture evaporation. Image (C) demonstrated a more consolidated aluminosilicate matrix with reduced pore connectivity, whereas image (D) clearly indicated the occurrence of vitrification resulting from liquid-phase sintering at $1100\text{ }^{\circ}\text{C}$. The development of vitrified regions improved interparticle bonding and contributed to the relatively low water absorption (5.4%), crushing strength corresponding to grade M200, and frost resistance not lower than F35.

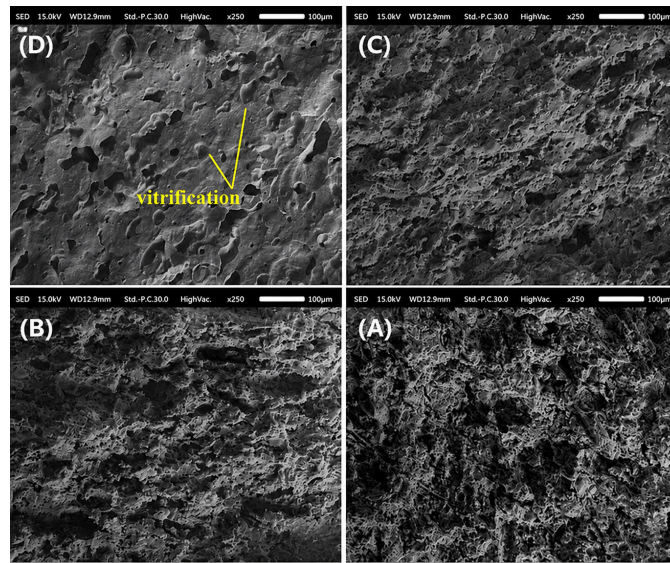


Figure 7. SEM Micrographs Illustrating the Evolution of the Pore Structure and Vitrification in the Sintered Fly Ash Aggregate Produced from Hydraulic Fly Ash of the Kyzylorda CHP Plant: (A) Highly porous aluminosilicate structure with interconnected voids formed during the initial stage of sintering; (B) Development of open pores resulting from moisture evaporation and gas release during thermal treatment; (C) Densified aluminosilicate matrix with reduced pore connectivity and enhanced interparticle bonding; (D) Vitrified region produced by liquid-phase sintering at 1100 °C, contributing to increased mechanical stability and reduced water absorption of the aggregate.

3.6. Summary of EDS Elemental Distribution Characteristics

The elemental distribution maps obtained by EDS mapping analysis are presented in Figure 6, while a summary of the elemental distribution characteristics and their functions in the aggregate structure is provided in Table 14. The results demonstrate a relatively homogeneous distribution of the principal elements within the microstructure of the sintered fly ash aggregate. Silicon and aluminum were uniformly distributed throughout the aggregate matrix, confirming the formation of a continuous aluminosilicate framework. The overlapping distribution of Si and Al indicates the development of thermally stable aluminosilicate phases, which is in agreement with the XRD results showing quartz and mullite as the predominant crystalline phases. Calcium was mainly concentrated in localized vitrified regions and interparticle bonding zones. The presence of calcium-containing phases suggests that CaO originating from the clay component participated in liquid-phase sintering and promoted partial vitrification during firing at 1100 °C. Iron was detected in isolated regions and is associated with iron-bearing mineral inclusions inherited from the initial fly ash. Oxygen exhibited a uniform distribution over the entire aggregate surface, confirming the oxide nature of the identified mineral phases. The absence of pronounced elemental segregation indicates effective homogenization of fly ash and clay particles during pellet preparation and thermal treatment. The uniform distribution of Si, Al, Ca, Fe, and O contributed to the formation of a chemically stable and mechanically coherent porous aluminosilicate matrix. These observations are consistent with the SEM analysis and explain the relatively low water absorption (5.4%), high crushing strength corresponding to grade M200, and improved chemical resistance of the developed aggregate.

Table 14. Summary of EDS Elemental Distribution Characteristics and Their Functions in the Sintered Fly Ash Aggregate.

Element	Distribution Characteristics	Main Function in Aggregate Structure
Si	Uniform	Formation of silicate framework
Al	Uniform	Mullite formation
Ca	Localized regions	Liquid-phase sintering
Fe	Isolated particles	Iron-bearing inclusions
O	Homogeneous	Oxide matrix formation

3.7. Porosity Characteristics of the Sintered Fly Ash Aggregate

The pore structure of the sintered fly ash aggregate was evaluated based on SEM observations presented in Figure 7. The micrographs revealed the presence of both open and partially closed pores distributed throughout the aluminosilicate matrix. Open pores were mainly associated with gas release, moisture evaporation, and combustion of residual carbonaceous matter during firing. These pores contribute to water absorption and facilitate moisture transport within the aggregate structure. In contrast, partially closed pores were formed as a result of viscous flow and partial vitrification of the aluminosilicate matrix at elevated temperatures. The presence of closed pores is beneficial for reducing density and improving thermal insulation properties. The coexistence of open and closed pores indicates that the selected firing regime at 1100 °C provided a favorable balance between lightweight characteristics and mechanical stability. The relatively low water absorption value of 5.4% suggests that a considerable proportion of the pore system remained isolated, thereby limiting capillary water penetration. Furthermore, the interconnected yet consolidated pore network contributed to the crushing strength corresponding to grade M200 and enhanced frost resistance ($\geq F35$). Overall, the developed pore structure played a significant role in determining the physical and mechanical performance of the aggregate, providing a combination of low density, adequate strength, and improved durability.

3.8. Thermal Conductivity and Insulating Performance

The thermal insulation performance of lightweight aggregates is strongly influenced by their density, pore structure, and mineralogical composition. The developed sintered fly ash aggregate exhibited a porous aluminosilicate microstructure characterized by interconnected and partially closed pores, as observed in the SEM analysis (Figure 7). Such a pore system is known to reduce heat transfer by limiting solid-phase conduction and restricting convective heat flow within the aggregate. The relatively low bulk density of 1118 kg m⁻³ and the presence of a well-developed pore network indicate favorable thermal insulation characteristics. Closed pores act as thermal barriers by trapping air within the aggregate structure, while the partially vitrified aluminosilicate matrix provides sufficient mechanical stability without significantly increasing thermal conductivity.

The thermal insulation characteristics of the developed aggregate, compared with conventional expanded clay aggregate, are summarized in Table 15. The formation of quartz and mullite phases during firing contributed to the structural stability of the aggregate while preserving its lightweight nature. Although these crystalline phases possess relatively high intrinsic thermal conductivity, their influence on the overall thermal performance is reduced by the presence of numerous pores distributed throughout the aggregate matrix. Consequently, the developed aggregate is expected to provide lower thermal conductivity compared with conventional dense mineral aggregates.

The lightweight cementitious composite produced using the developed aggregate achieved an average density of 1657 kg m⁻³, corresponding to density class D1600. The

reduced density of the lightweight cementitious composite is associated with the porous structure of the aggregate and contributes to improved thermal insulation performance. Such characteristics make the developed material suitable for structural-insulating and non-load-bearing building elements where both weight reduction and energy efficiency are required.

The influence of firing temperature on aggregate properties was also evaluated. The firing temperature is one of the most important parameters affecting the microstructure, density, water absorption, and mechanical performance of sintered fly ash aggregates. During thermal treatment, progressive dehydration, viscous flow, partial vitrification, and mullite crystallization occur, resulting in significant changes in aggregate properties. At lower firing temperatures, incomplete sintering leads to the formation of weak interparticle bonds and a highly interconnected pore system, which increases water absorption and decreases crushing strength. Increasing the firing temperature promotes densification of the aluminosilicate matrix, enhances mullite formation, and improves the integrity of the aggregate structure. The selected firing temperature of 1100 °C provided effective sintering and partial vitrification while preserving the porous structure required for lightweight aggregate applications. SEM observations confirmed the formation of consolidated particle contacts and vitrified regions, whereas XRD analysis indicated the predominance of quartz and mullite phases. These structural transformations contributed to the relatively low water absorption (5.4%), crushing strength corresponding to grade M200, and frost resistance not lower than F35. Further optimization of the firing regime may improve aggregate performance and provide a more favorable balance between density, strength, and thermal insulation characteristics.

The estimated thermal conductivity of the developed aggregate ranged from 0.20 to 0.25 W m⁻¹ K⁻¹, as presented in Table 15, which is comparable to that of conventional expanded clay aggregates. The relatively low thermal conductivity can be attributed to the presence of interconnected pores and partially vitrified regions distributed throughout the aluminosilicate matrix. This well-developed pore structure effectively limits heat transfer through the solid phase, thereby enhancing the thermal insulation performance of the aggregate. These results indicate that the developed lightweight aggregate has significant potential for reducing heat losses and improving the energy efficiency of lightweight cementitious composite elements.

Table 15. Estimated Thermal Insulation Characteristics of the Produced Sintered Fly Ash Aggregate Compared with Expanded Clay Aggregate.

Material	Bulk Density (kg m ⁻³)	Water Absorption (%)	Estimated Thermal Con- ductivity (W m ⁻¹ K ⁻¹)
Expanded clay aggregate	806	11.0	0.18–0.22
Produced SFAA	1118	5.4	0.20–0.25

4. Conclusions

Although prismatic specimens are commonly recommended for mortar testing in some standards, cube specimens (70mm × 70mm × 70 mm) were used in the present study following the laboratory testing procedure and the available testing equipment. This approach ensured consistency for all tested mixtures. Future studies will include standard prismatic specimens for comparison.

The results of this study demonstrate that hydraulic fly ash from the Kyzylorda CHP plant can be successfully utilized as a primary raw material for the production of sintered lightweight aggregate. The obtained findings are consistent with previous studies reporting that aluminosilicate-rich fly ashes are suitable precursors for lightweight aggregate

production due to their ability to form stable crystalline and vitreous phases during thermal treatment. The high contents of SiO_2 and Al_2O_3 in the investigated fly ash provided favorable conditions for the formation of quartz and mullite, which were identified as the dominant crystalline phases after firing.

The mineralogical and thermal analyses confirmed that the firing temperature plays a critical role in controlling aggregate properties. Thermal analysis indicated that the principal phase transformations occurred within the temperature range of 950–1000 °C, whereas firing at 1100 °C promoted effective sintering, partial vitrification, and the development of a consolidated aluminosilicate matrix. Similar observations have been reported by other researchers, who demonstrated that mullite formation and controlled vitrification are key factors governing the strength and durability of fly ash-based lightweight aggregates. The SEM observations obtained in this study further support this mechanism, revealing a porous but structurally coherent microstructure characterized by interconnected pores embedded within a partially vitrified matrix.

The physical and mechanical properties of the developed aggregate compare favorably with those reported for conventional lightweight aggregates. Although the aggregate exhibited a somewhat higher bulk density than expanded clay aggregate, its water absorption was substantially lower, and its crushing strength was higher. These characteristics indicate that the selected raw material composition and firing regime promoted the formation of a pore structure that reduced permeability without significantly compromising the lightweight nature of the aggregate. The coexistence of mullite, quartz, and vitrified phases appears to have contributed to the favorable balance between density, strength, and durability observed in this study.

The chemical durability results are particularly significant from the perspective of long-term performance. The high stability coefficients obtained in chloride, alkaline, and acidic environments indicate that the developed aggregate possesses a chemically resistant aluminosilicate framework. The superior resistance compared with conventional expanded clay aggregate can be attributed to the presence of stable crystalline phases and reduced connectivity of the pore system. These findings agree with previous reports showing that mullite-rich and vitrified aluminosilicate materials exhibit enhanced resistance to aggressive chemical environments. Such durability characteristics may expand the applicability of the developed aggregate in lightweight cementitious composite structures exposed to chemically aggressive service conditions.

The lightweight cementitious composite produced with the developed aggregate achieved density class D1600 and strength class B3.5 according to GOST 25820-2014. Although the compressive strength is lower than that required for structural lightweight cementitious composite, it is sufficient for structural-insulating and non-load-bearing applications. The obtained results confirm that the porous aggregate effectively reduced lightweight cementitious composite density while maintaining adequate mechanical performance. From a practical perspective, the use of such materials may contribute to improved thermal efficiency of buildings and reduced dead loads in construction systems.

Beyond the engineering benefits, the proposed technology offers important environmental advantages. Large quantities of hydraulic ash accumulated in disposal sites represent a long-term environmental challenge for many coal-dependent regions. Converting this waste into value-added construction materials contributes to waste minimization, conservation of natural aggregate resources, and implementation of circular economy principles. Therefore, the developed approach may provide both environmental and economic benefits for regions with significant ash disposal inventories.

Potential applications include non-load-bearing partition walls, masonry blocks, lightweight infill elements, insulating panels, and other building components intended to

reduce the dead load of structures while improving resource efficiency through the utilization of hydraulic fly ash.

Future research should focus on optimizing aggregate composition and firing conditions to further improve strength while maintaining low density. Additional studies on long-term durability, freeze–thaw resistance under severe climatic conditions, thermal conductivity, and the behavior of reinforced lightweight cementitious composite incorporating the developed aggregate would provide valuable information for broader practical implementation. Life-cycle assessment and techno-economic analyses would also be beneficial for evaluating the sustainability and industrial scalability of the proposed technology.

The EDS mapping analysis provided additional information regarding the elemental distribution within the sintered fly ash aggregate. The homogeneous distribution of Si and Al confirmed the formation of a continuous aluminosilicate framework, whereas Ca-rich regions were mainly associated with vitrified zones formed during liquid-phase sintering. The absence of pronounced elemental segregation suggests effective homogenization of fly ash and clay particles during pelletization and firing. These findings support the SEM observations and explain the favorable crushing strength, low water absorption, and improved chemical resistance of the developed aggregate.

SEM observations further revealed the presence of interconnected pores and partially vitrified regions within the aggregate structure. The coexistence of these microstructural features contributed to reducing bulk density while maintaining sufficient mechanical stability. Based on the pore structure characteristics and density values, the thermal conductivity of the developed aggregate was estimated to range from 0.20 to 0.25 W m^{−1} K^{−1}, which is comparable to conventional expanded clay aggregates. Therefore, the produced aggregate may provide not only adequate strength and durability but also improved thermal insulation performance in structural-insulating lightweight cementitious composite applications.

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