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Article

Effect of waste glass and silica fume additions on the properties of fired clay ceramics

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Abstract. This study investigates the influence of crushed waste glass (GL) and silica fume (SF) on the densification and microstructural characteristics of fired clay ceramics. Ceramic specimens were prepared by partially replacing clay with 10, 15, and 20 wt.% of GL and SF. They fired at 1050, 1100, and 1125 °C to evaluate the effect of temperature on bulk density and to determine the optimal firing regime. Based on density measurements and visual examination, the samples fired at 1125 °C were identified as the most effective and selected for further evaluation. Water absorption testing and microstructural characterization were conducted for specimens fired at 1125 °C. Increasing firing temperature enhanced densification for all compositions. Glass-modified ceramics exhibited the highest bulk density and lowest water absorption due to intensified liquid-phase sintering and pore filling. In contrast, silica fume additions resulted in comparatively higher porosity and water absorption under identical conditions. Microstructural analysis confirmed a denser and more homogeneous matrix in glass-containing specimens with uniformly distributed Si-, Al-, and Ca-rich phases. The results demonstrate that both additives can modify the properties of fired clay ceramics; however, crushed glass provides a more pronounced improvement in densification and water resistance at 1125 °C.
Keywords: fired clay ceramics, crushed glass, silica fume, bulk density, firing temperature, microstructure, water absorption.

1. Introduction

Fired clay ceramic materials, such as ceramic bricks, tiles, and construction ceramic products, are manufactured by shaping clay raw materials followed by high-temperature firing. During firing, sintering, and vitrification (glass formation) occur, which determine the final bulk density, porosity, and water absorption (key parameters governing the durability and service performance of ceramic products) [1], [2]. As a rule, increasing the firing temperature in the range of 900-1150 °C leads to higher density and reduced open porosity; however, it is accompanied by increased energy consumption and production costs [3], [4]. Therefore, the development of compositions that enable control of sintering and structure formation through mineral additives, including those derived from industrial waste, is of particular importance.

A promising approach involves the use of silica-rich waste, particularly silica fume, which is a highly dispersed amorphous silicon dioxide with a typical particle size of less than 1 μm, capable of influencing the sintering kinetics and phase formation during firing [5]. Incorporation of silica fume in amounts up to 10 wt.% has been shown to increase the strength and density of fired bricks at temperatures of 1000-1100 °C, whereas at lower firing temperatures a tendency toward reduced density is observed [6]. In several studies, silica fume has also been investigated as part of complex waste-containing systems with a total waste content of up to 90 wt.%, highlighting its technological potential while simultaneously indicating a strong sensitivity of properties to firing temperature [7].

In parallel with silica fume, waste glass has been extensively studied as an additive for clay ceramics. Waste glass has been demonstrated to act as a flux, forming a liquid phase and intensifying liquid phase sintering at temperatures as low as 900-1000 °C, thus promoting structural densification and reducing porosity [8]. Experimental studies confirm that the incorporation of glass waste at levels of 5-10 wt.% leads to increased density and strength and reduced water absorption of fired clay bricks; moreover, at 900 °C, properties comparable to those of reference samples fired at 1000 °C can be achieved [9].

The properties of ceramic materials are influenced not only by composition and additive type but also by firing conditions, including temperature and furnace atmosphere [10]. Variations in firing conditions (oxidization or reduction atmosphere) at temperatures of approximately 1100-1170 °C have been established to significantly alter the phase composition and physical and mechanical properties of ceramic products, including increases in density and strength of 10-20% [11]. Related studies also indicate that glass waste retains a positive influence on the properties of cementitious and mineral materials under thermal exposure up to 600 °C, confirming its stability at elevated temperatures [12].

Several studies demonstrated the feasibility of using silica fume not only in clay-based ceramics but also in specialized high-temperature ceramic systems. For example, effective recycling of silica fume waste in the synthesis of β-eucryptite ceramics with the formation of a stable crystalline structure at 1000 °C has been reported, confirming its high reactivity during firing [13].

For clay ceramics, a more consistent positive effect has been observed with the use of waste glass. Increased glass content has been shown to be 30-40 wt.%, and raising the firing temperature to 1100 °C results in higher density and strength and a reduction in water absorption to levels below 6%, with glass particle size playing a decisive role [14]. Similar conclusions were obtained when waste glass was used in combination with sedimentary materials, where a sintering temperature of 1050 °C or higher was identified as the key factor controlling structural densification [15].

In a broader context of waste recycling in brick production, it is emphasized that the use of secondary raw materials can reduce the consumption of natural resources and energy demand [16]. However, large-scale industrial implementation remains limited, despite the strength values achieved of 15-20 MPa and water absorption below 10% [17]. Reviews of waste glass recycling practices in clay bricks also highlight the need to optimize compositions and firing regimes to ensure stable and reproducible properties [18]. At the same time, studies focusing on the combined use of silica fume with other mineral wastes indicate that brick properties are largely governed by firing temperature in the range of 700-800 °C, whereas the influence of additives may be secondary compared to the temperature factor [19].

Despite the substantial body of research on the use of industrial wastes in clay ceramics, existing studies mainly address either waste glass or silica fume individually, or as components of complex multi-waste systems. Moreover, organic and inorganic additives exert different effects on porosity (increases of up to 30-35%) and mechanical properties, underscoring the need for a clear differentiation of their roles in structure formation processes [20]. In addition, the influence of waste glass on the durability and transport properties of ceramic products, including water permeability and frost resistance, requires further systematization [21].

Considering the identified trends, it can be hypothesized that silica fume and waste glass modify the sintering behavior of clay ceramics through different mechanisms. Due to its high dispersity, silica fume exhibits pronounced temperature sensitivity and can enhance densification or increase porosity depending on firing regime [22]. In contrast, waste glass at elevated temperatures is expected to provide more stable liquid-phase sintering and pore filling, which can result in reduced water absorption and increased bulk density [23].

The objective of this study is to systematically evaluate the effects of silica fume and waste glass as partial replacements of clay at levels of 10, 15, and 20 wt.% on the densification behavior and microstructural evolution of fired clay ceramics. The specimens were fired at temperatures of 1050, 1100, and 1125 °C to assess the influence of firing temperature on bulk density and to identify a rational firing regime. Based on density results, 1125 °C was selected for detailed analysis of water absorption and microstructure using scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS) techniques. The investigated ceramics are intended for structural and non-structural clay bricks for masonry applications, including load-bearing and facade units where controlled density and reduced water absorption are required. The scientific novelty of this work lies in the direct comparison of two widely available silica-rich wastes under identical dosages and firing conditions, enabling the establishment of microstructure-property relationships governing densification and water resistance of fired clay ceramics and expanding the potential for resource-efficient ceramic production [24].

2. Methods

2.1. Raw materials

For this study, clay obtained from the Almaty deposit (Burunday district, Almaty, Kazakhstan) served as the primary raw material. The clay is characterized by a plasticity index of 9.0, which corresponds to low plasticity, and is classified as an easy-to-melt material based on its refractoriness. According to its technological characteristics, the clay is appropriate for the production of frost-resistant ceramic bricks of grades M100 and M125 by plastic molding, in accordance with the standard requirements [25]. According to X-ray diffraction analysis, clay is a polymineral material dominated by quartz, with the presence of calcite and feldspar (plagioclase). The mineral assemblage also includes phyllosilicate phases such as mica (phlogopite) and chlorite, along with minor amounts of dolomite. The chemical composition is characterized by a predominance of SiO_2 (approximately 55-60 wt.%) and Al_2O_3 (about 15-18 wt.%), with minor contents of CaO, MgO, and Fe_2O_3, as well as Na_2O (1.88 wt.%) and SO_3 (0.74 wt.%). The loss on ignition is about 10.02%, indicating the presence of bound water and carbonate components.

As mineral additives, crushed glass (GL) and silica fume (SF) were used in combination with the clay raw material. The silica fume was supplied by “Qazaq Innotec” LLP (Almaty, Kazakhstan) and is a highly dispersed by-product of ferroalloy production, characterized by high pozzolanic activity. Due to its fine particle size, silica fume was used as a microfiller and reactive additive to improve the packing density and microstructural homogeneity of clay-based mixtures [26]. Silica fume consisted of ultrafine spherical particles (<1 μm), while crushed glass particles were irregular in shape.

Crushed glass was obtained from recycled construction and demolition glass waste (Almaty, Kazakhstan). Before using, the glass waste was ground in a planetary ball mill (XQM-4, Changsha Tianchuang Powder Technology Co., Ltd., Changsha, China) to obtain a fine powder. No additional chemical or mineralogical analysis was conducted for the crushed glass; its role was limited to that of a finely dispersed silica additive introduced into the clay matrix. Both additives were mixed with clay in doses of 10, 15, and 20 wt.% relative to the clay mass.

2.2 Preparation of ceramic specimens

Before specimen preparation, the clay was dried in a drying oven SHS 80-01 SPU (SHS 80-01 SPU, Smolensk SKTB SPU, Smolensk, Russia) at 105-110 °C, ground, and sieved through a 1 mm mesh. Silica fume and crushed glass were additionally milled until all particles passed through a 0.315 mm sieve. Two types of ceramic compositions were investigated.

After the required proportions (10, 15, and 20 wt.% relative to the clay mass) were weighed, the raw materials were first homogenized in a dry state. Subsequently, water was gradually added until a plastic and moldable mass was obtained. Cylindrical specimens with a diameter of 50 mm and a height of 55 mm were produced for further testing. The specimens were formed using a hydraulic press PGM-100MG4A (SKB Stroypribor, Chelyabinsk, Russia) under a compaction load of 6-10 kN.

The formed samples were dried in an SHS 80-01 SPU drying oven at 105-110 °C for 2 h, after which their mass and dimensions were recorded. Thermal treatment was carried out in an SNOL 1.6/1300 muffel furnace (SNOL ThermoLab, Utena, Lithuania) at temperatures of 1050, 1100, and

1125 °C for 1 hour under slightly oxidizing conditions, following the procedure described in [11] for fusible ceramic raw materials.

Two series of ceramic compositions were examined. The first series consisted of clay-silica fume mixtures, whereas the second series included clay-crushed glass systems. Based on preliminary experimental studies [11], [23], mineral additives were introduced at replacement levels of 10% (SF10 and GL10), 15% (SF15 and GL15), and 20% (SF20 and GL20) by weight of clay, resulting in ceramic charges containing 90/10, 85/15, and 80/20 clay-to-additive ratios, respectively. The reference specimen (C0) with 100% of clay has been prepared. For each composition, two specimens have been prepared. The proportions are selected based on previous research by the authors [11], [23].

Firing shrinkage was not determined quantitatively, as the study focused on densification and microstructural behavior. The specimens maintained geometric integrity at 1125 °C, while higher temperatures led to noticeable deformation due to excessive vitrification.

Figure 2 presents photographs of representative specimens fired at 1125 °C, corresponding to the temperature at which the maximum values of bulk density were achieved.

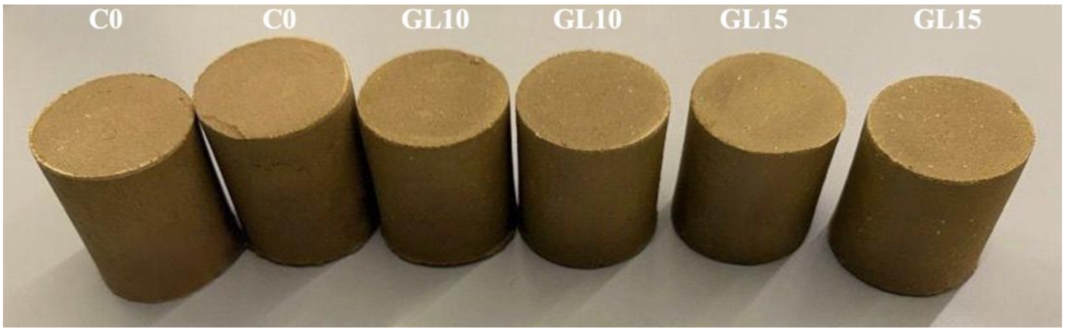


Figure 2 – Specimens with crushed glass after firing at a temperature of 1125 °C

2.2. Microstructural study

Microstructural characterization was carried out using Scanning Electron Microscopy (SEM) and Energy-Dispersive X-ray Spectroscopy (EDS). The phase composition was investigated by X-ray diffraction using a DRON-3.0 diffractometer (Bourevestnik, Saint Petersburg, Russia) equipped with CuKα radiation and a β-filter. Data were collected in θ-2θ scanning mode at an accelerating voltage of 35 kV, a current of 20 mA, and a scanning rate of 2°/min. Phase identification and semi-quantitative analysis were performed using the ICDD PDF-2 database (International Centre for Diffraction Data, Newtown Square, PA, USA). Elemental composition and microstructural features of the specimens were examined using a scanning electron microscope (JEOL JSM-7000, JEOL Ltd., Tokyo, Japan). Observations were conducted at an accelerating voltage of 10 kV, with a working distance of 14 mm, under low-vacuum conditions at ×1000 magnification.

2.3. Physical and mechanical properties

The average density of the ceramic specimens was calculated from the mass-to-volume ratio of cylindrical samples with a diameter of 50 mm and a height of 55 mm. Density values were determined in accordance with the methodology described in [23].

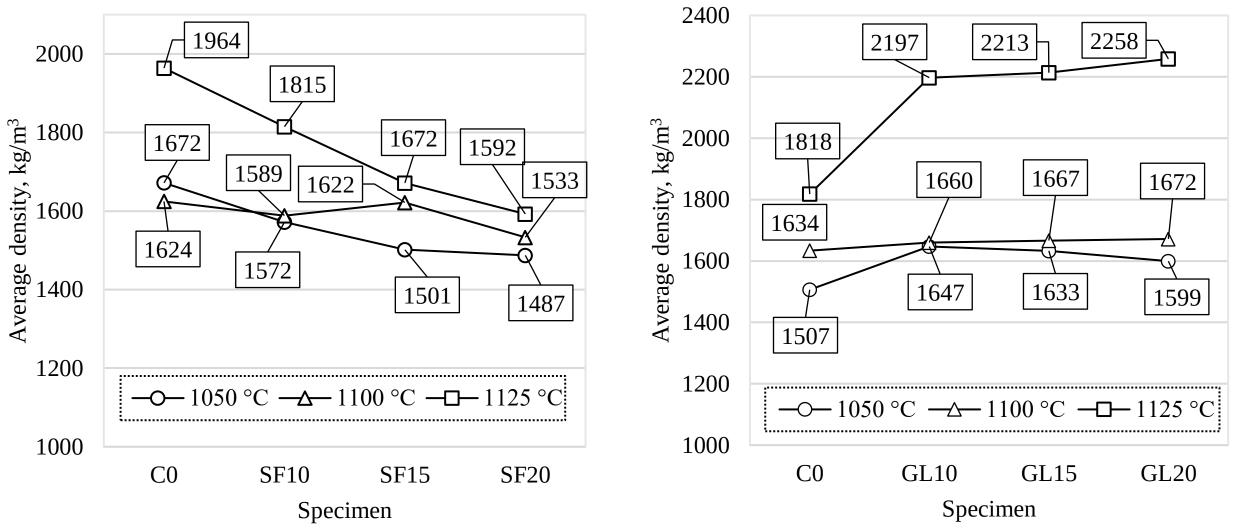
Water absorption was evaluated as an indicator of the degree of moisture uptake by the ceramic material after prolonged immersion in water. The test procedure followed the standard requirements [27].

3. Results and Discussion

This section presents the key experimental results on the effect of crushed waste glass and silica fume on the densification and microstructural characteristics of fired clay ceramics. The influence of additive type, replacement level, and firing temperature on bulk density, water absorption, and microstructure is analyzed with respect to ceramic materials intended for brick production. Each additive was evaluated independently to enable a direct comparison of its effects on sintering behavior and structure formation.

3.1. Effect of firing temperature on bulk density, visual appearance, and water absorption

The effect of firing temperature on the bulk density of the ceramic specimens containing silica fume and crushed glass is illustrated in Figure 1, demonstrating the pronounced influence on the compaction behavior of all investigated compositions.



a) Silica fume (SF) b) Crushed glass (GL)

Figure 1 – Effect of firing temperature on the bulk density of ceramic specimens with SF and GL

As shown in Figure 1a, for the specimens containing silica fume (SF series), an increase in firing temperature from 1050 °C to 1125 °C led to a noticeable increase in bulk density for all additive contents. The most significant compaction was observed at 1125 °C, indicating enhanced sintering and improved consolidation of the ceramic matrix at this temperature [28]. At lower firing temperatures (1050 and 1100 °C), the density values remained comparatively lower, suggesting incomplete sintering of the ceramic body. A gradual decrease in density with an increase in the silica fume content was observed at all firing temperatures, which can be attributed to the influence of the additive on pore formation and vitrification behavior. A similar temperature-dependent trend was observed for the compositions containing crushed glass (GL series), as shown in Figure 1b. However, at 1125 °C, the highest values of bulk density among all investigated compositions were achieved for specimens with crushed glass. This pronounced compaction can be associated with the softening and partial melting of the glass phase, which promotes liquid-phase sintering and effective pore filling. In contrast, specimens fired at 1050 °C and 1100 °C exhibited reduced densification, indicating limited activation of the glass phase at lower temperatures.

The reduced bulk density observed in silica fume-modified specimens can be attributed to the ultrafine particle size and high specific surface area of silica fume [29], which increase water demand during shaping and may promote the development of a more open pore structure during firing. Similar effects of silica fume on porosity and density of clay-based ceramics have been reported in [6], [19], [22]. In contrast to crushed glass, silica fume does not contain significant amounts of fluxing oxides and therefore does not effectively promote liquid-phase sintering at the investigated temperatures [9], [14]. Consequently, densification is less pronounced compared to glass-modified compositions. The silica fume used in this study contains negligible residual carbon, and carbon burnout is not considered a governing factor affecting density [6], [13].

In general, the results confirm that 1125 °C is an effective firing temperature to achieve sufficient compaction of the ceramic compositions investigated. In particular, the specimens demonstrated the highest density values without visible deformation or structural defects. Therefore, specimens C0, GL10, and GL15 fired at 1125 °C were selected as the optimal firing temperature for subsequent water absorption measurements and microstructural characterization. A similar densification trend has been reported in previous studies on waste glass-modified clay bricks [30]. Researchers reported that the incorporation of recycled glass led to an increase in bulk density and mechanical performance due to liquid-phase sintering, particularly at elevated firing temperatures above 1000 °C. Comparable results were also observed by [31], who attributed the enhanced densification to the fluxing action of glass waste, promoting pore closure and matrix consolidation during firing. In our case, the temperatures above 1125 °C resulted in visible geometric deformation of the specimens due to excessive vitrification, suggesting that 1125 °C represents the upper practical firing limit for the investigated compositions.

Water absorption results for specimens fired at 1125 °C are shown in Figure 3.

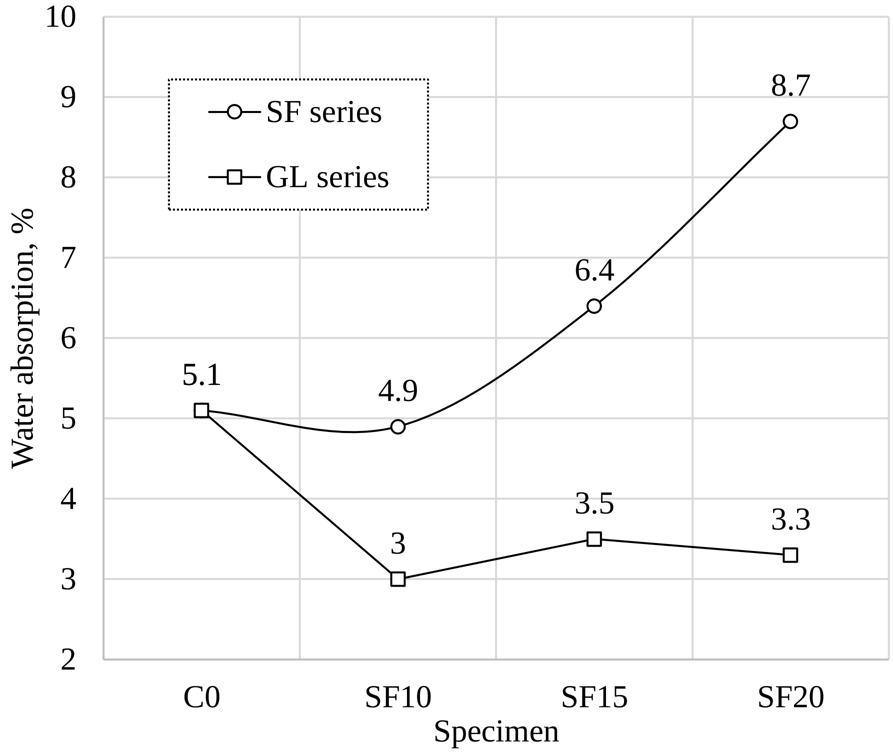
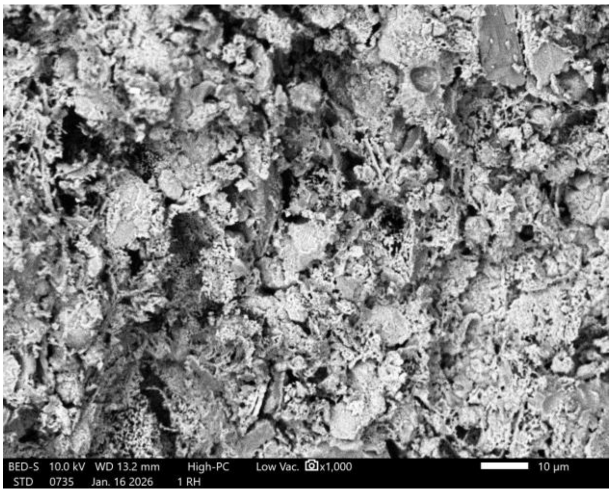


Figure 3 – Water absorption of ceramic specimens modified with SF and GL fired at 1125 °C

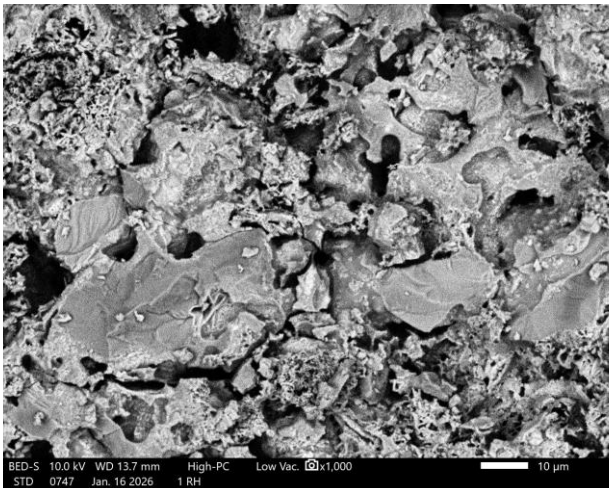
As shown in Figure 2, for the silica fume-modified compositions (SF series), water absorption increases with increasing additive content and exceeds 6% for SF15 and SF20, indicating the formation of a more open pore structure at higher silica fume contents. In contrast, the crushed glass-modified compositions (GL series) exhibit significantly lower water absorption compared to both the reference specimen and the SF series. The reduced water absorption of glass-containing ceramics is attributed to improved vitrification and effective pore filling during firing, resulting in improved water resistance, which is consistent with findings reported in [32], [33]. Findings [32] demonstrated that the incorporation of glass waste into clay ceramics significantly reduced water absorption due to enhanced vitrification and sealing of open pores. Similar trends were reported by [33], who showed that crushed waste glass contributes to the formation of a dense glassy phase, leading to improved water resistance of fired clay bricks.

3.2. Results of microstructural analysis using SEM and EDS

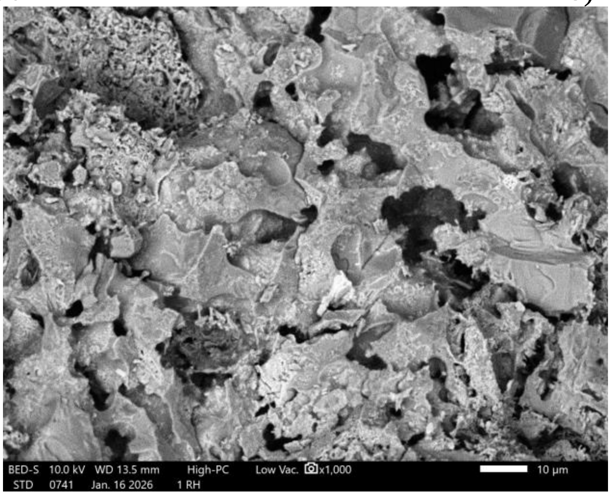
Figure 4 presents SEM micrographs of ceramic specimens fired at 1125 °C, including the reference composition and compositions containing crushed glass.



a) C0



b) GL10



c) GL15

Figure 4 – SEM micrographs of ceramic specimens fired at 1125 °C

It is seen from Figure 4a that the reference specimen (C0) exhibits a relatively heterogeneous microstructure with irregularly shaped particles and a noticeable amount of open porosity, indicating incomplete compaction of the ceramic matrix. Incorporation of 10 wt.% crushed glass (GL10) led to a more compact microstructure formation characterized by smoother particle surfaces and improved interparticle bonding, as is seen from Figure 4b. The presence of partially vitrified regions suggests the activation of liquid-phase sintering, which contributes to pore filling and enhanced matrix consolidation. Further increase in the crushed glass content to 15 wt.% (GL15) resulted in a denser and more homogeneous microstructure with reduced pore size and improved particle integration, as is seen from Figure 4c. The observed microstructural refinement is consistent with the higher bulk density values obtained for glass-containing specimens fired at 1125 °C (Figure 1b). These observations suggest the beneficial role of crushed glass in developing vitrification and compaction during high-temperature firing. Comparable microstructural features have been reported in earlier SEM investigations of glass-containing clay ceramics. Findings in [34] observed smoother particle surfaces and reduced open porosity in glass-modified bricks, which were associated with partial melting of the glass phase and enhanced interparticle bonding. Similar vitrified regions and pore-filling effects were also reported by [35] in fired ceramics produced from waste glass mixtures.

To further clarify the influence of crushed glass on the phase distribution and elemental composition of the ceramic matrix, Figure 5 shows the elemental EDS maps of the specimen containing 15 wt.% crushed glass fired at 1125 °C, as it exhibited the most favorable compaction behavior observed in Figure 4. The maps indicate a relatively uniform distribution of Si and Al throughout the ceramic matrix, confirming the formation of a continuous aluminosilicate framework.

The presence of Ca and Na is associated with the glass additive and suggests the development of a glassy phase that contributes to matrix vitrification. The corresponding EDS spectrum confirms the dominance of the Si-Al-O components with minor alkali and alkaline-earth elements, supporting the enhanced compaction observed at this firing temperature.

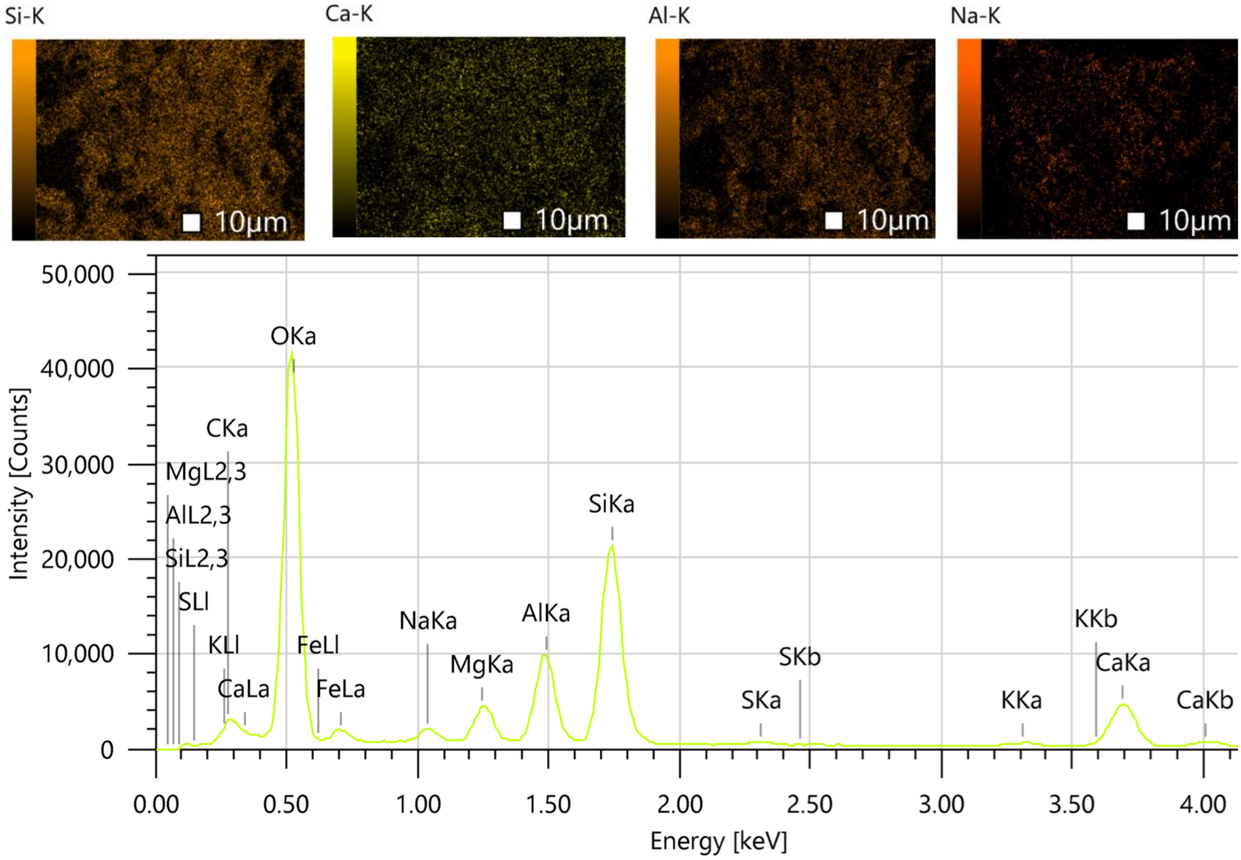


Figure 5 – EDS elemental maps and corresponding EDS spectrum of GL15 (scale bar: 10 µm)

The detected minor alkali components are consistent with the fluxing role of waste glass reported by [32], who noted that Na_2O in the glass additive promotes vitrification and contributes to denser fired clay bricks. The predominance of the Si-Al-O matrix in GL15 also agrees with the results of [34], who associated waste-glass incorporation with vitrification and glassy-phase formation in fired clay bricks. A similar glassy-phase response in glass-containing ceramics was reported by [33].

4. Conclusions

Based on the experimental results, the following conclusions can be drawn:

- Increasing the firing temperature from 1050 to 1125 °C led to a significant improvement in densification for all investigated compositions, with the highest bulk density obtained at 1125 °C, identifying this temperature as the optimal firing condition.

- The additive type strongly influenced compaction behavior. At replacement levels of 10-20 wt.%, ceramics containing crushed waste glass consistently exhibited higher bulk density than those modified with silica fume, indicating a more effective densification mechanism associated with liquid-phase sintering.

- Water absorption measurements at 1125 °C showed a clear reduction in open porosity with increasing densification. Glass-modified ceramics demonstrated the lowest water absorption, confirming the pore-filling effect of the glassy phase formed during firing.

- SEM and EDS analyses revealed that crushed glass addition produced a denser and more homogeneous ceramic matrix with reduced pore size and uniform distribution of Si-, Al-, and Ca-rich phases, explaining the enhanced compactness and reduced water absorption.

Overall, the study demonstrated that the crushed waste glass fired at 1125 °C performs as an effective mineral additive for improving the density, porosity-related properties, and microstructural characteristics of fired clay ceramics, while further research is required to assess long-term durability and industrial applicability.

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