

## Article

# Influence of Waste Glass and Silica Fume Additives on the Properties and Microstructure of Clay-Based Ceramic Materials

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## Abstract

This study investigates the effects of waste glass (GL) and silica fume (SF) on the physical, mechanical, and microstructural properties of clay-based ceramic materials produced from low-grade calcite-rich clay loam. Waste glass and silica fume were incorporated at 10–20 wt.%, and the specimens were fired at temperatures ranging from 1050 to 1150 °C. The average density, water absorption, compressive strength, phase composition (XRD), and microstructure (SEM) were evaluated. The results showed that waste glass significantly enhanced the sintering behavior of the ceramic body through liquid-phase formation at elevated temperatures. The average density increased from approximately 1650 to 2200 kg/m<sup>3</sup>, while water absorption decreased from 6.5% to 3.2%. The optimum firing temperature was 1125 °C, at which the ceramic compositions containing 10 wt.% and 15 wt.% waste glass exhibited no visible deformation and achieved compressive strengths of 32–36 MPa. In contrast, silica fume was less effective as a fluxing additive, resulting in lower strength, increased deformation, and cracking at temperatures above 1100 °C. SEM observations confirmed the formation of a denser, more homogeneous microstructure in the waste glass-modified specimens, while XRD analysis revealed a reduction in quartz content, accompanied by enhanced formation of the diopside and anorthite phases. Among all investigated compositions, the ceramic material containing 10 wt.% waste glass and fired at 1125 °C exhibited the most balanced combination of density, water absorption, and compressive strength, demonstrating its potential for producing high-performance clay-based ceramic materials.

**Keywords:** clay ceramics; waste glass; silica fume; firing temperature; compressive strength; density; water absorption; microstructure



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## 1. Introduction

The construction industry is one of the largest consumers of natural resources and simultaneously a major generator of industrial and municipal waste [1]. The accumulation of mineral waste, including fly ash, slags, waste glass, and silica-rich by-products, has intensified interest in circular-economy approaches aimed at reducing landfill disposal and promoting sustainable construction materials [2].

One of the most effective approaches to addressing this issue is incorporating waste materials into the production of construction products [3]. As noted by Fugazzotto et al. [4]

and Hossain and Roy [5], the recycling of industrial and solid wastes into ceramic materials simultaneously contributes to waste management, reduces landfill disposal, and decreases the consumption of primary raw materials. Fugazzotto et al. [4] emphasized that industrial ceramic waste can be reused as aluminosilicate precursors for building blocks and binders, including alkali-activated materials, thereby enabling the production of consolidated materials with lower CO<sub>2</sub> emissions than traditional cement- and ceramic-based products. Similarly, Hossain and Roy [5] highlighted those various industrial by-products, such as fly ash, rice husk ash, blast-furnace slag, sludge, waste glass, and other residues, can be incorporated into ceramic systems as alternative ingredients, thereby providing value-added products while conserving natural resources. Ceramic technology offers unique advantages because high-temperature firing promotes the formation of a stable mineral matrix that can encapsulate various components without compromising the material's long-term performance [6,7].

An additional advantage is the ability to utilize a wide range of waste materials, including fly ash, slags, waste glass, and fine silica-rich additives. As demonstrated in several review studies, the proportion of secondary components in ceramic composites may reach several tens of percent and, in some cases, nearly completely replace natural raw materials, thereby opening new prospects for the development of environmentally efficient construction materials [8]. According to García [9] and co-authors, the recycling of ceramic and mineral waste into construction materials significantly reduces environmental burden while simultaneously providing alternative raw material sources. Their study demonstrated that clay and mineral waste can be effectively reused in construction-related applications due to their ability to encapsulate other minerals, interact with chemical species, and improve material functionality through thermal activation or sintering. Similar conclusions were reported in the review studies by Muñoz Velasco [10], Monteiro [11], and Zhang [12], who emphasized that incorporating waste into fired brick production is one of the most realistic routes for waste valorization in countries with developed ceramic industries. Furthermore, Buravchuk [13] and co-authors reported that the use of technogenic materials, such as ash and slag waste, promotes sintering and the formation of mullite-like phases, thereby improving the strength and frost resistance of ceramic products. However, the influence of waste materials is not always straightforward and strongly depends on their chemical composition and dosage.

Clay remains the primary raw material for ceramic production due to its abundance and technological versatility. As noted by Vasić [14] and co-authors, the chemical composition of clays plays a crucial role in determining ceramic properties: a content of 15–20% Al<sub>2</sub>O<sub>3</sub>, 3–9% Fe<sub>2</sub>O<sub>3</sub>, and the presence of fluxing oxides provide favorable conditions for sintering and the formation of a dense structure. Studies by Arianpour [15] and co-authors demonstrated that firing clay materials at temperatures above 1000 °C leads to the formation of mullite and anorthite, thereby increasing compressive strength by 24.84–32.85 MPa. However, the phase assemblage after firing depends on the mineralogical composition of the raw clay. In calcite-rich clays, diopside and anorthite may form preferentially instead of mullite. Similarly, Rat [16] and co-authors found that increasing the firing temperature to 1100 °C resulted in a density of approximately 1.67 g/cm<sup>3</sup> and flexural strength up to 34.47 MPa, accompanied by reduced porosity and water absorption.

Sintering processes involve both solid-state and liquid-phase mechanisms. According to Garzón [17] and co-authors, vitrification of clay systems occurs within the temperature range of 800–1350 °C, with activation energies ranging from 45 to 151 kJ/mol. As the firing temperature increases, a liquid phase forms, accelerating diffusion and densification. However, as emphasized by El Ouahabi [18] and co-authors, excessive development of these processes may lead to deformation of ceramic products and deterioration of their

service performance. Additional studies by Abubakar [19] and co-authors demonstrated that increasing firing temperature results in higher density and shrinkage, while porosity decreases. Similar findings were reported by Sutcu [20] and Abouloifa [21], who observed increased strength and reduced water absorption with higher firing temperatures.

Silica fume is an ultrafine amorphous silicon dioxide by-product that is widely used as a pozzolanic material in cementitious composites due to its high reactivity and micro-filling effect. In cement-based systems, silica fume reacts with calcium hydroxide released during cement hydration, promoting the formation of additional calcium silicate hydrate (C–S–H), reducing porosity, and improving the mechanical performance and durability of the hardened matrix. Recent studies have also shown that silica fume can be alkali-activated, further enhancing its reactivity and mechanical performance in cement mortars [22].

In ceramic materials, silica fume has also attracted increasing attention as a silica-rich additive. Kumar et al. [23] reported that the incorporation of 4 wt.% silica fume increased the compressive strength of fired clay bricks by 27.55% while reducing water absorption. Abdel Hamid et al. [24] demonstrated that the mechanical performance of silica fume-containing bricks strongly depends on firing temperature, whereas Baspinar et al. [25] observed that silica fume promotes densification and vitrification at elevated temperatures. However, contradictory results have also been reported. Elmaghraby and Ismail [26] found that insufficient liquid-phase formation may lead to increased porosity and reduced strength, indicating that the effectiveness of silica fume strongly depends on both the raw material composition and the firing regime.

In high-temperature systems, silica fume exhibits different behavior. Khattab [27] and co-authors demonstrated that at firing temperatures of 1300–1400 °C, cordierite and mullite phases form, resulting in a density of approximately 2.112 g/cm<sup>3</sup> and a porosity of about 10%. Similar observations were reported by Zawrah [28] and co-authors, who found that increasing silica fume content promotes mullite formation and alters mechanical properties. In addition, Zhou [29] and co-authors showed that silica fume may accelerate sintering by forming a transient liquid phase; however, excessive amounts may lead to grain growth and strength reduction. Therefore, the effect of silica fume is governed by the balance between improved particle packing and the development of liquid-phase sintering.

Waste glass represents one of the most effective modifiers for ceramic systems due to the presence of alkali oxides. In the study by De Faria Busch [30] and co-authors, incorporating up to 30% waste glass reduced water absorption to approximately 17% while ensuring strength values that met standard requirements. Darweesh [31] demonstrated that the optimal glass content was approximately 25%, at which the maximum density and strength were achieved, whereas further increases adversely affected these properties. Flores Nicolás [32] and co-authors showed that the addition of glass reduces the firing temperature to 800 °C and shortens the sintering duration to 8 h. Similar conclusions were reported by Silva [33] and co-authors, who demonstrated that waste glass can replace natural raw materials while improving physical and mechanical properties and reducing energy consumption.

Recent studies by Coulon [34] and co-authors confirmed that the firing temperature can be lowered by approximately 100 °C through the incorporation of waste glass, which is highly significant for the decarbonization of the ceramic industry. The literature indicates that waste glass serves as an effective fluxing agent that promotes liquid-phase sintering, reduces porosity, and increases density [35]. In contrast, silica fume exerts a more complex influence that strongly depends on firing temperature and system composition. At lower temperatures, silica fume may deteriorate material properties, whereas at higher temperatures it contributes to the formation of new crystalline phases and structural improvement [36,37].

Despite the considerable number of studies on incorporating silica fume and waste glass into clay-based ceramic materials, most investigations examine these additives separately across different raw material systems, firing regimes, and forming conditions [35,38,39]. Furthermore, limited attention has been paid to comparing silica fume and waste glass within the same clay system under identical firing conditions, despite their fundamentally different mechanisms of action during sintering [40,41]. This makes direct comparison of their influence on sintering behavior, phase formation, and the development of engineering properties difficult. In particular, there is limited understanding of how silica fume and waste glass behave within the same clay matrix under identical firing temperatures [42]. However, such comparative analysis is essential for understanding the differences between an additive that primarily affects particle packing and reactivity and one that promotes liquid-phase formation.

Moreover, previous studies have often focused on individual parameters such as strength, water absorption, or density, whereas the comprehensive relationships among density, shrinkage, water absorption, strength, phase composition, and microstructure remain insufficiently explored [35,38,43]. The experimental methodology adopted in this study was based on the authors' previous investigations of clay-based ceramics containing diatomite and bentonite clay [44,45]. However, unlike those studies, which focused on diatomite, bentonite clay and firing conditions, the present work systematically compares waste glass and silica fume under identical processing conditions. Furthermore, physical, mechanical, mineralogical, and microstructural analyses were integrated to clarify their influence on sintering behavior and ceramic performance. In addition, most published studies have investigated either waste glass or silica fume separately, while direct comparisons of their performance under identical processing conditions are scarce. The combined influence of additive type and firing temperature on phase evolution, densification behavior, and the resulting ceramic properties has also not been systematically evaluated, particularly for low-grade calcite-rich clay loam. Therefore, a systematic investigation is required to establish the relationships between ceramic properties, phase transformations, and microstructural evolution under varying additive types and firing temperatures.

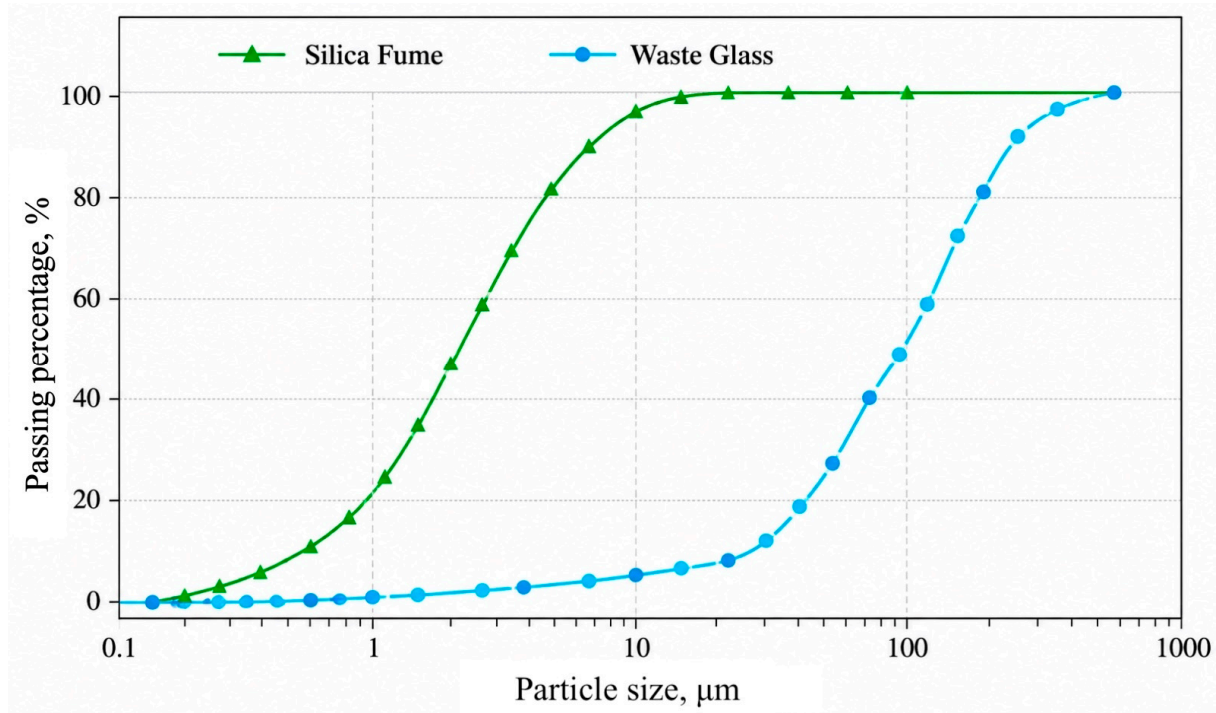
The present study aims to evaluate the effects of silica fume and waste glass on the densification, water absorption, compressive strength, phase evolution, and microstructural characteristics of clay ceramics fired at different temperatures. Particular attention is given to identifying the optimal additive type and firing temperature, and to clarifying the relationship among phase evolution, sintering behavior, and the development of dense, durable, and dimensionally stable ceramic materials.

## 2. Materials and Methods

### 2.1. Raw Materials

The primary raw material used in this study was clay loam obtained from the Almaty deposit (Burundai district, Almaty, Kazakhstan). The clay loam is characterized by a high calcium carbonate content and a plasticity index of 9.0, indicating a low-plasticity material. Based on its refractory properties, it belongs to the group of low-melting clays. Based on its technological characteristics, clay loam is suitable for producing ceramic bricks of strength grades M75 and M100 by the plastic forming method in accordance with the requirements of the relevant standards [46]. XRD analysis of the raw clay loam revealed that it consisted mainly of quartz (46%) and calcite (25%), together with albite, mica, microcline, and chlorite. The high calcite content indicates that the raw material belongs to the group of calcium-rich clays, which is expected to favor the formation of calcium aluminosilicate phases during firing.

Waste glass (GL) and silica fume (SF) were used as mineral additives. Silica fume, supplied by “Qazaq Innotec” LLP (Almaty, Kazakhstan), is a highly dispersed by-product of ferroalloy production with high pozzolanic activity. Due to its ultrafine particle size ( $<1\ \mu\text{m}$ ), silica fume acts as a microfiller and reactive additive, improving packing density and microstructural homogeneity in clay-based compositions [47]. The particle size distribution of silica fume and waste glass is presented in Figure 1. The materials consist predominantly of fine particles, confirming their suitability for improving particle packing during sintering.



**Figure 1.** Particle size distribution of the silica fume and waste glass.

Waste glass was obtained from recycled construction and demolition waste (Almaty, Kazakhstan). Prior to use, it was ground in a planetary ball mill (XQM-4, Changsha Tianchuang Powder Technology Co., Ltd., Changsha, China). Owing to the high hardness of the glass, prolonged milling was required; approximately 5–6 h were needed to produce 0.5 kg of powder that passed completely through a 0.315 mm sieve. All waste glass compositions were prepared using the same grinding procedure, ensuring identical processing conditions throughout the study. Due to its high content of amorphous silica and alkali oxides, waste glass acts as a fluxing additive during firing, promoting liquid-phase formation and enhancing densification of the fired ceramic body.

The morphology, elemental composition, and elemental distribution of raw clay loam, silica fume, and waste glass were examined using SEM–EDS. The results are presented and discussed in Section 3.1.

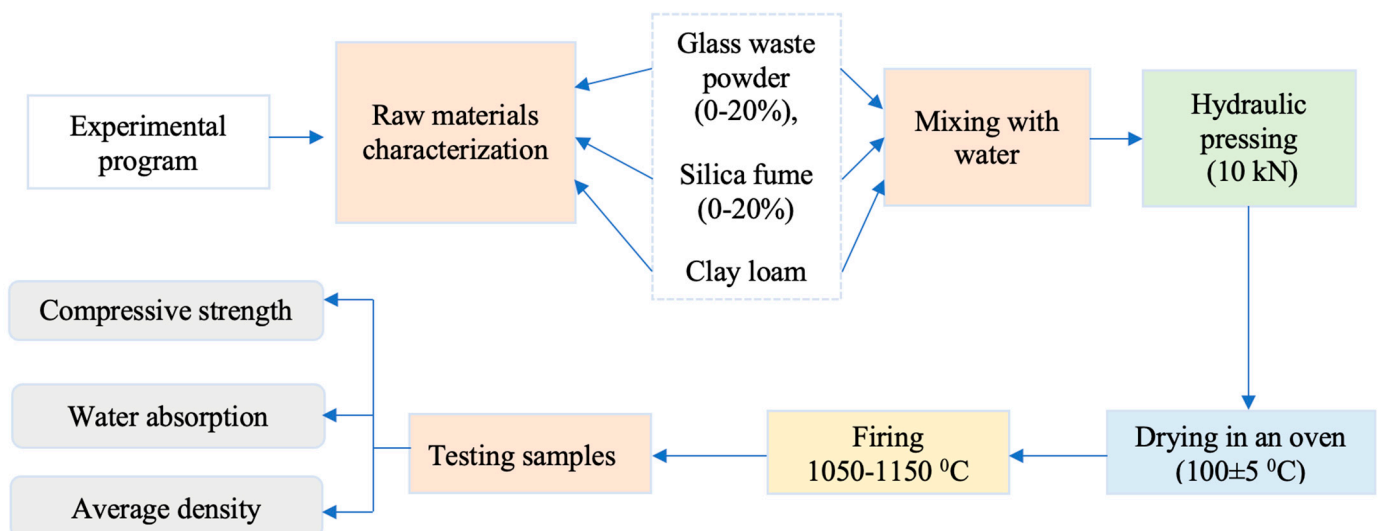
## 2.2. Preparation of Raw Materials and Manufacture of Ceramic Specimens

Before specimen preparation, the clay loam was dried in an SHS 80-01 SPU drying oven (Smolensk SKTB SPU, Smolensk, Russia) at 105–110 °C, then ground and sieved through a 1 mm mesh. Silica fume and waste glass were ground until they completely passed through a 0.315 mm sieve. Two ceramic compositions were prepared for specimen production: clay loam–waste glass and clay loam–silica fume. The raw materials were combined at loam-to-additive ratios of 90:10, 85:15, and 80:20 by weight. The selected additive contents (10–20 wt.%) were based on preliminary experiments and previous

studies [45,46], which indicated that this range is sufficient to evaluate the influence of mineral additives on ceramic properties while avoiding excessive deformation associated with higher replacement levels. For convenience, the compositions containing 10, 15, and 20 wt.% waste glass were designated as GL10, GL15, and GL20, respectively, while the corresponding silica fume compositions were designated as SF10, SF15, and SF20.

After weighing according to the specified proportions, the raw materials were dry-mixed to ensure homogeneous distribution of the components. Water was then gradually added until a plastic, moldable ceramic mass was obtained. Cylindrical specimens with a diameter of 50 mm and a height of 55 mm were produced using a PGM-100MG4A hydraulic press (SKB Stroypribor, Chelyabinsk, Russia) under a compaction load of 10 kN. A compaction load of 10 kN was selected based on preliminary trials. Loads above 10 kN caused extrusion of the plastic ceramic mass from the mold, resulting in distortion of the specimen geometry, whereas 10 kN provided stable specimen formation and adequate compaction. The dimensions of the specimens were selected for laboratory evaluation of the influence of mineral additives under identical processing conditions and were not intended to reproduce the dimensions of commercial ceramic products. All compositions were prepared and tested using identical specimen geometry, ensuring a reliable comparative assessment. The control specimen (C0), consisting of 100% clay loam, was also prepared. Three specimens were formed for each composition. The selected additive contents and specimen preparation procedure were based on the authors' previous studies [44,45].

The formed samples were dried in an SHS 80-01 SPU drying oven at 100–105 °C for 2 h, after which their mass and dimensions were recorded. The temperature was increased at a constant heating rate of 5 °C/min until the target firing temperatures of 1050, 1100, 1120, 1125, and 1150 °C were reached. The selected firing temperatures cover the onset of intensive sintering and the vitrification range of the investigated clay, enabling determination of the optimum firing temperature while avoiding severe deformation at higher temperatures. The specimens were held at the target temperature for 1 h and then allowed to cool naturally inside the switched-off furnace to room temperature. The overall experimental program, including raw material characterization, sample preparation, firing, and testing, is presented in Figure 2.



**Figure 2.** Schematic representation of the experimental program, including raw material characterization, sample preparation, firing, and testing procedures.

### 2.3. Microstructural and Phase Characterization

The phase composition of the ceramic specimens was determined using X-ray diffraction (XRD) with a DRON-3.0 diffractometer (Bourestnik, Inc., Saint Petersburg, Russia) equipped with CuK $\alpha$  radiation and a  $\beta$ -filter. Data were collected in the  $2\theta$  range of  $5\text{--}70^\circ$  at an accelerating voltage of 35 kV and a current of 20 mA, with a scanning rate of  $2^\circ/\text{min}$ . Phase identification and semi-quantitative analysis were performed using the ICDD PDF-2 database.

The microstructure and morphology of the samples were examined using a scanning electron microscope, JEOL JSM-7000 (JEOL Ltd., Tokyo, Japan). Observations were carried out at an accelerating voltage of 10 kV, with a working distance of approximately 14 mm, under low-vacuum conditions. The microstructural analysis was performed at magnifications up to  $\times 3000$  to evaluate pore structure, particle bonding, and densification.

### 2.4. Physical and Mechanical Properties

Average density, defined as the mass per unit volume of a material in its natural state, was determined and calculated according to the procedure described in [47]. The measurements were performed on cylindrical specimens with a diameter of 50 mm and height of 55 mm.

Water absorption was determined after 48 h of water saturation following the standard procedure described in [48].

Compressive strength was measured using a hydraulic testing press in accordance with [47,49]. The tests were conducted on cylindrical specimens with dimensions of 50 mm  $\times$  55 mm (diameter  $\times$  height). For each composition, at least three specimens were tested, and the average value was reported.

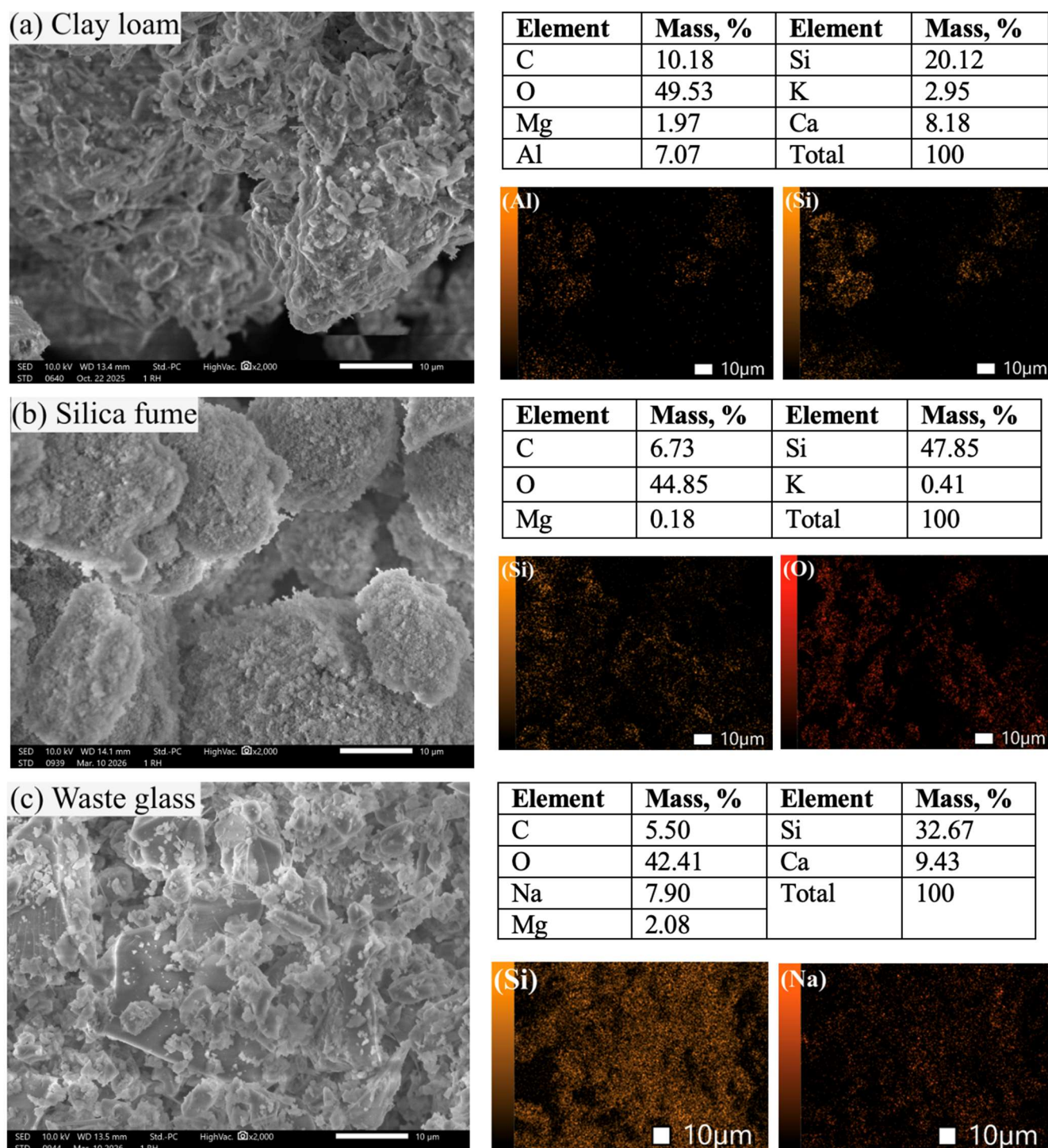
All physical and mechanical properties were determined using a minimum of three specimens for each test. All measurements were performed on three independently prepared specimens, and the results are reported as the mean  $\pm$  standard deviation.

## 3. Results and Discussion

### 3.1. Characterization of Raw Materials

According to X-ray diffraction (XRD) analysis, the clay loam is a polymineral material predominantly composed of quartz (46%), with calcite (25%) and albite feldspar (11%). The mineral assemblage also includes phyllosilicate phases such as mica (12%) and chlorite (2%), along with minor amounts of potassium feldspar (microcline, 4%).

Scanning electron microscopy coupled with energy-dispersive spectroscopy (SEM-EDS) was used to characterize the morphology, elemental composition, and elemental distribution of the clay loam, silica fume, and waste glass. The corresponding SEM micrographs, quantitative EDS elemental compositions, and elemental mapping results are presented in Figure 3. The analyses confirmed that clay loam is mainly composed of O, Si, Al, and Ca, silica fume is dominated by Si and O, whereas waste glass contains high amounts of Si, O, Na, and Ca, consistent with their expected compositions and functions during the firing process.



**Figure 3.** SEM microstructures, EDS quantitative elemental composition, and elemental distribution maps of the raw materials: (a) clay loam, (b) silica fume, and (c) waste glass.

### 3.2. Evaluation of Shape Stability of Fired Specimens

Visual examination of specimens fired at 1050, 1100, and 1150 °C revealed that samples fired at 1150 °C exhibited noticeable deformation. To determine the optimum firing temperature, additional specimens were subsequently fired at 1120 and 1125 °C. Specimens containing silica fume showed significant deformation above 1100 °C, attributed to the formation of excessive liquid phase during firing. Therefore, SF-containing compositions fired above 1100 °C were excluded from further investigation.

Visual examination of waste-glass-modified specimens fired between 1050 and 1125 °C indicated that compositions containing 20 wt.% waste glass experienced pronounced deformation at 1100, 1120, and 1125 °C. Consequently, only the control composition (C0) and the compositions containing 10 wt.% and 15 wt.% waste glass (GL10 and GL15) were selected for subsequent analyses. Based on the preliminary assessment of shape stability, only compositions that maintained sufficient geometric integrity after firing were considered for further study.

A comparative evaluation of the waste glass-modified specimens was then conducted, focusing on key physical and mechanical properties, including average density, water absorption, and compressive strength. The appearance of the selected fired specimens is shown in Figure 4. All selected specimens retained their cylindrical shape and showed no visible macroscopic defects, confirming adequate dimensional stability under the applied firing conditions.



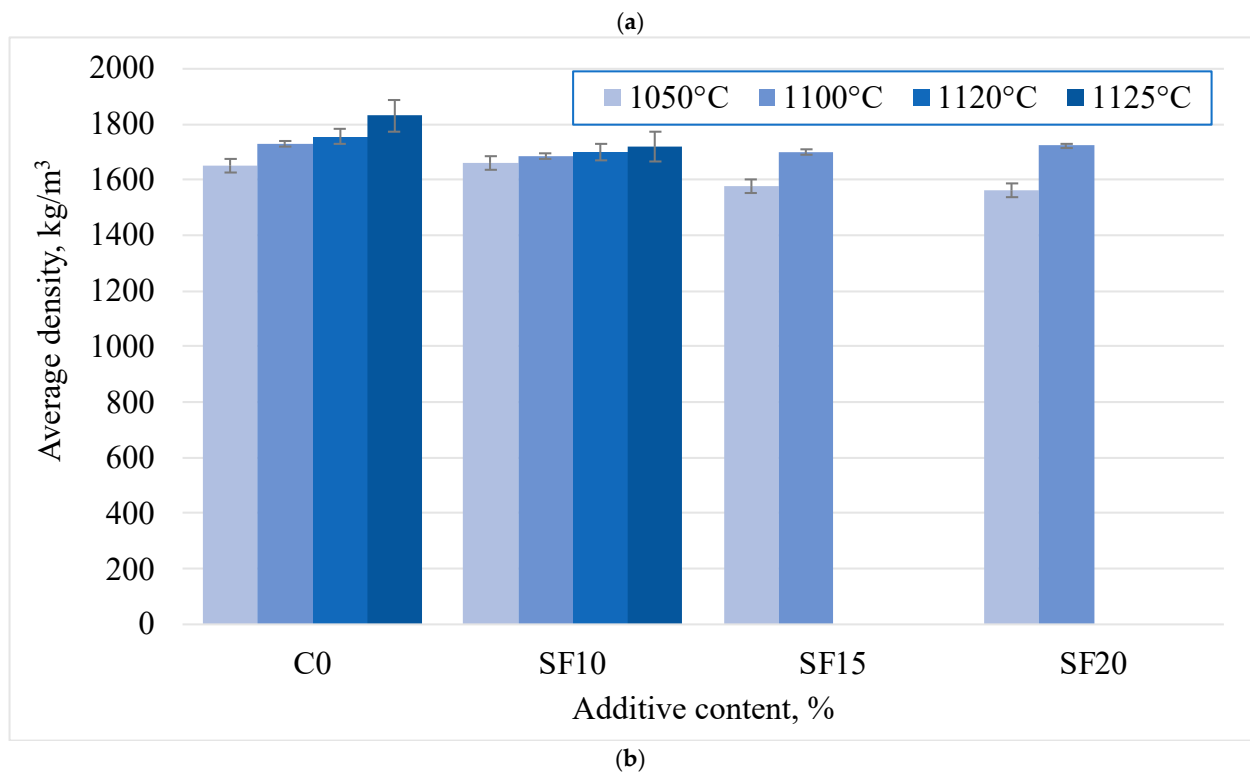
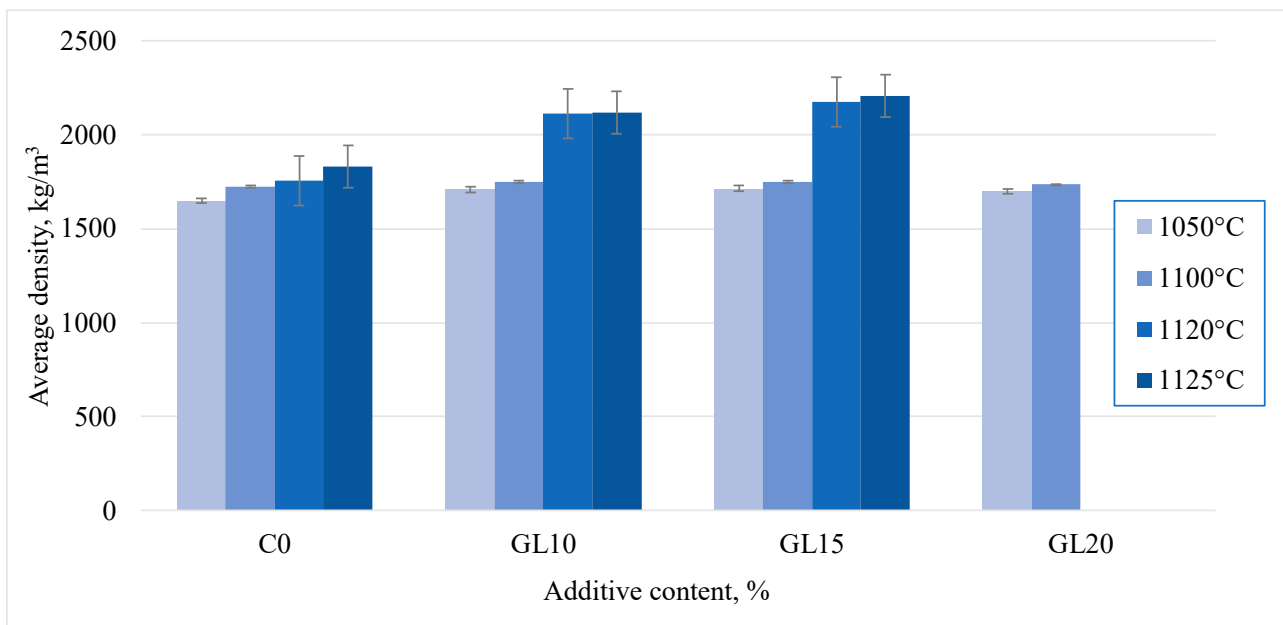
**Figure 4.** Appearance of the fired ceramic specimens after firing at 1125 °C, illustrating the influence of mineral additives on dimensional stability and deformation behavior.

### 3.3. Average Density of Fired Ceramic Specimens

The average density of the fired products significantly depends on the type of additive and the firing temperature (Figure 5a,b).

The average density of the control specimens fired at 1050 °C was 1650 kg/m<sup>3</sup>. Increasing the firing temperature to 1100, 1120, and 1125 °C resulted in a gradual increase in density to 1727, 1756, and 1831 kg/m<sup>3</sup>, respectively. This trend indicates enhanced densification of the ceramic body due to improved sintering and increased liquid-phase formation at elevated temperatures.

For the silica fume-modified compositions (Figure 5b), the average density at 1050 °C generally decreased with increasing silica fume content, from 1650 kg/m<sup>3</sup> for the control specimen (C0) to 1564 kg/m<sup>3</sup> for SF20. An exception was observed for SF10, which exhibited a slightly higher density of 1660 kg/m<sup>3</sup>, suggesting a moderate densification effect. However, further increases in silica fume content resulted in lower densities of 1576 and 1564 kg/m<sup>3</sup> for SF15 and SF20, respectively. It is assumed that higher firing temperatures are required to achieve adequate densification in compositions containing 15–20 wt.% silica fume. This assumption is supported by the results shown in Figure 5b, where the densities of SF15 and SF20 specimens fired at 1100 °C were 124 and 158 kg/m<sup>3</sup> higher, respectively, than those of the corresponding specimens fired at 1050 °C.



**Figure 5.** Effect of additive content and firing temperature on the average density of ceramic specimens: (a) silica fume-modified compositions, (b) waste glass-modified compositions. Values are presented as mean  $\pm$  standard deviation ( $n = 3$ ). Error bars represent the standard deviation.

In addition, the SF15 and SF20 specimens exhibited rough surface textures and a pronounced tendency to crack after firing. Due to their poor structural integrity and reduced performance, these compositions were excluded from further investigation.

The analysis of average density results showed that waste glass significantly enhanced the densification of the ceramic specimens. Average density increased with both higher waste glass content and higher firing temperature (Figure 4). For the GL10 composition, density increased from 1710 kg/m<sup>3</sup> to 2120 kg/m<sup>3</sup> as the firing temperature increased, while for GL15, the corresponding values increased from 1714 kg/m<sup>3</sup> to 2206 kg/m<sup>3</sup>.

The relatively small standard deviations indicate good repeatability of the experimental measurements. In all cases, the differences between the investigated compositions were substantially greater than the corresponding experimental deviations, confirming that the observed trends are attributable to additive type and firing temperature.

A particularly pronounced increase in density was observed at firing temperatures of 1120 and 1125 °C. At these temperatures, the average density of GL10 reached 2112–2120 kg/m<sup>3</sup>, whereas GL15 achieved 2179–2206 kg/m<sup>3</sup>. This increase indicates intensive densification of the ceramic body, resulting from the melting of waste glass and the formation of a substantial glassy phase. Notably, the GL10 and GL15 specimens maintained their geometric integrity and showed no visible deformation.

In contrast, the GL20 specimens deformed after firing at 1120 and 1125 °C due to excessive formation of the glassy phase within the ceramic body. These findings confirm that waste glass promotes densification at elevated temperatures through its fluxing effect and the generation of a liquid phase during firing.

The results are consistent with the general behavior of glass-containing ceramic systems, where moderate glass additions enhance densification, whereas excessive glass content or excessively high firing temperatures may lead to deformation caused by over-vitrification [50,51]. Overall, waste glass proved more effective than silica fume in improving the density and mechanical performance of the ceramic compositions, particularly at elevated firing temperatures, owing to its ability to intensify liquid-phase sintering and reduce porosity [41,52].

### 3.4. Water Absorption

Water absorption is a key indicator of the formation of a dense ceramic body. Because it reflects the degree of sintering and the porosity of clay-based ceramic materials, its variation was analyzed alongside changes in bulk density.

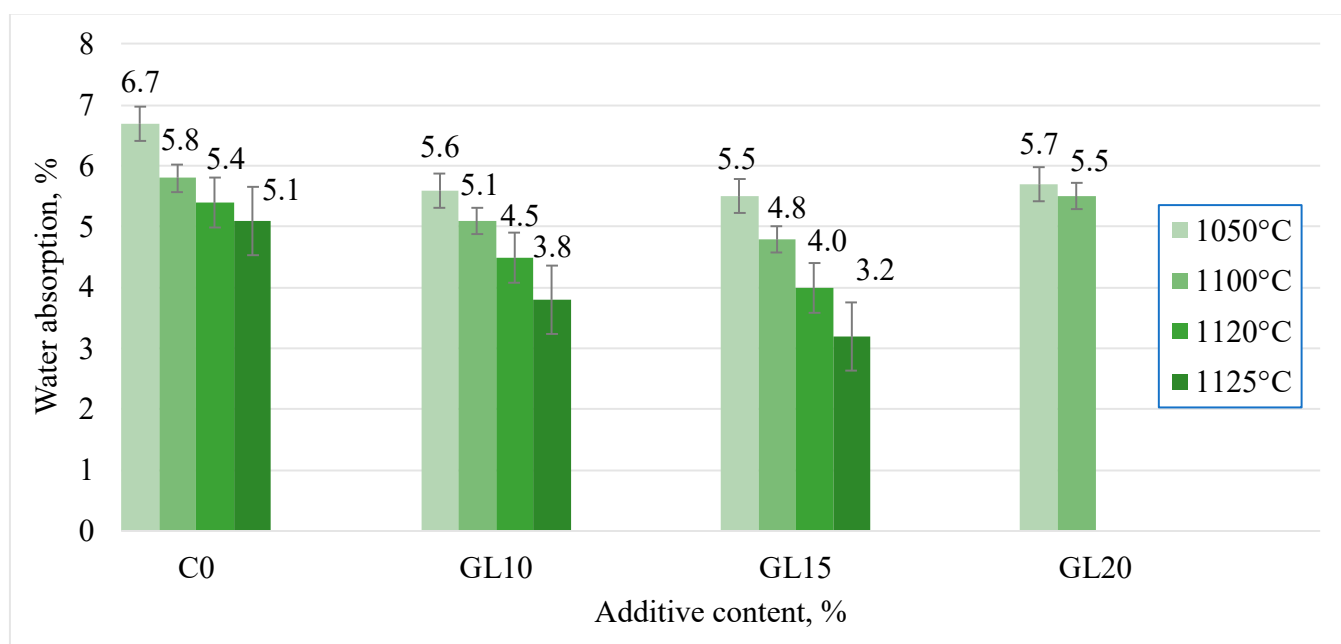
Water absorption values for the ceramic specimens, which depend on additive type, additive content, and firing temperature, are presented in Figure 6a,b.

The control specimens exhibited a water absorption of 6.5% after firing at 1050 °C. Increasing the firing temperature to 1100, 1120, and 1125 °C gradually reduced water absorption to 5.8%, 5.4%, and 5.1%, respectively. This decrease can be attributed to increased formation of a liquid phase at higher temperatures, which enhances sintering, promotes densification of the ceramic body, and reduces the volume of open pores available for water penetration.

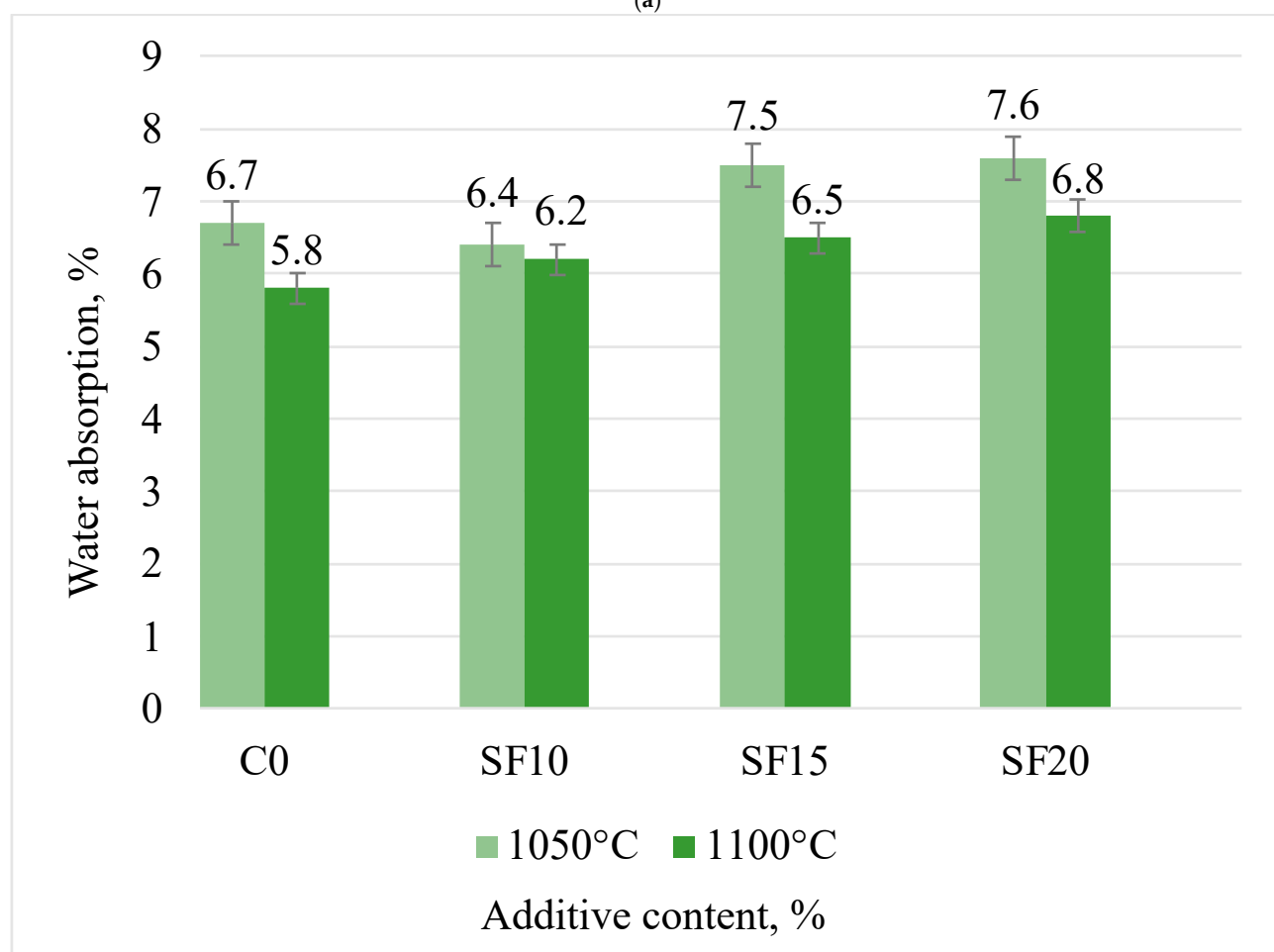
For the silica fume-modified compositions (Figure 6b), water absorption after firing at 1050 °C ranged from 6.4% to 7.6%, while specimens fired at 1100 °C exhibited values between 6.2% and 6.8%.

As shown in Figure 6b, the SF10 specimens exhibited water absorption values that were 0.3–0.5% lower than those of the control composition (C0) after firing at both 1050 and 1100 °C, indicating a moderate densification of the ceramic body. In contrast, the SF15 and SF20 specimens fired at 1050 °C showed water absorption values of 7.5% and 7.6%, respectively, which were 0.8–0.9% higher than that of the control specimen.

Although firing at 1100 °C reduced the water absorption of SF15 and SF20, the obtained values remained comparable to those of the control specimen fired at 1050 °C. These results indicate that silica fume exhibited limited fluxing efficiency under the investigated firing conditions and was less effective in promoting densification and pore reduction than waste glass.



(a)



(b)

**Figure 6.** Water absorption of ceramic specimens as a function of additive content and firing temperature: (a) silica fume-modified compositions (SF), (b) waste glass-modified compositions (GL).

The analysis of water absorption results showed that incorporating waste glass effectively reduced water absorption in the ceramic specimens. Water absorption decreased

with both higher waste glass content and higher firing temperature (Figure 6a,b). For the GL10 composition, water absorption decreased from 5.6% to 3.8% as the firing temperature increased, while for GL15 it decreased from 5.5% to 3.2%.

A particularly pronounced reduction was observed at firing temperatures of 1120 and 1125 °C. Under these conditions, the water absorption of GL10 decreased to 4.5–3.8%, whereas GL15 exhibited even lower values of 4.0–3.2%. The reduction in water absorption is attributed to enhanced liquid-phase sintering and densification of the ceramic body, which reduced the volume of open pores available for water penetration.

The observed changes in water absorption are in good agreement with the corresponding trends in average density. Compositions exhibiting higher average density generally showed lower water absorption, confirming a strong relationship among densification, pore-structure refinement, and water uptake in the fired ceramic specimens.

The results obtained are consistent with previous studies in which incorporating waste glass into clay-based ceramics led to a significant reduction in water absorption due to enhanced vitrification and pore sealing at elevated temperatures [41,53]. In contrast, silica-rich additives typically show a limited effect on water absorption at moderate firing temperatures, as their contribution to densification remains relatively weak [54].

### 3.5. Compressive Strength Development of Fired Ceramic Specimens

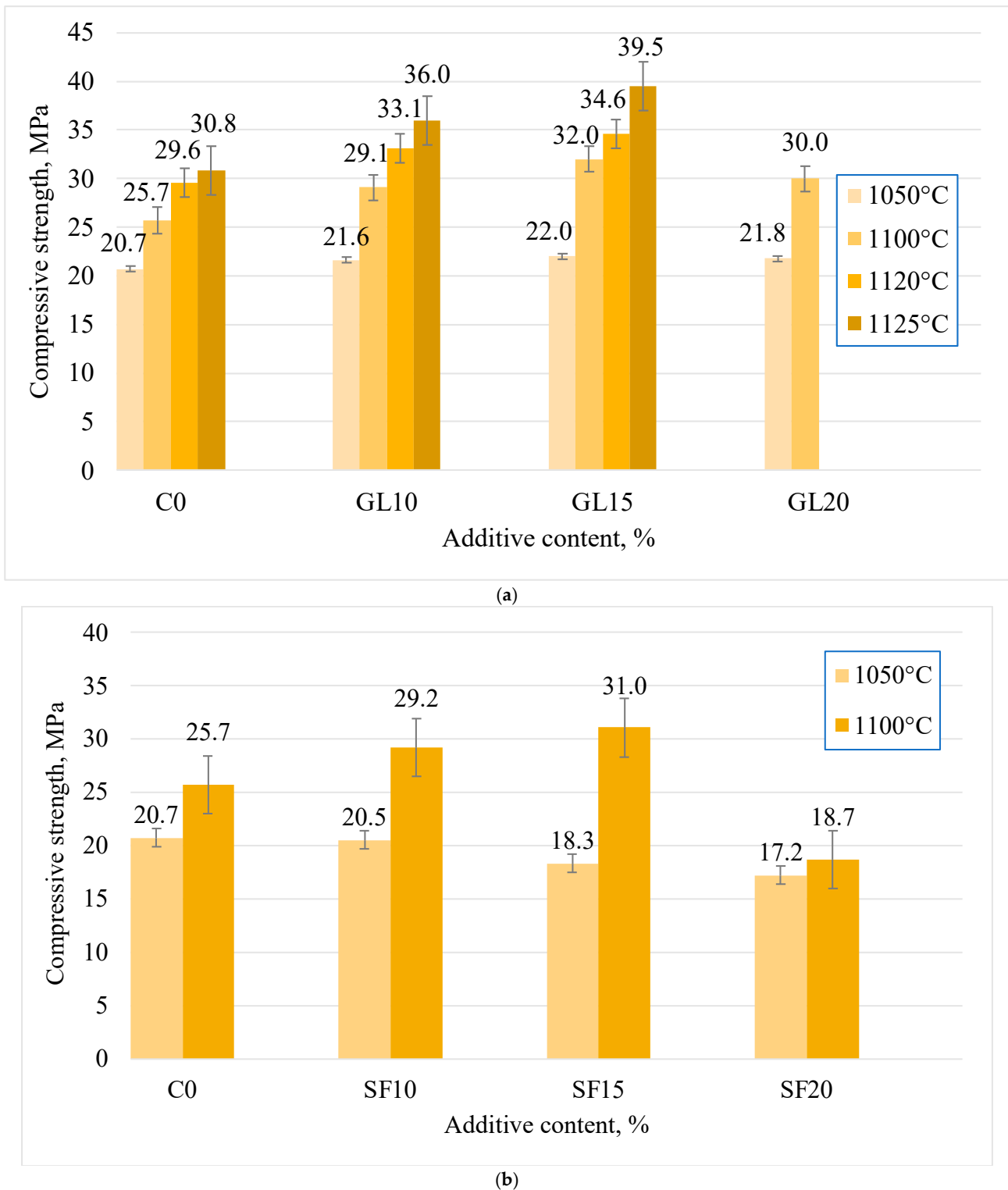
The dependence of the compressive strength of ceramic samples on the type of additive, its amount and the firing temperature is shown in Figure 7a,b. As shown in Figure 6a, the compressive strength of the waste-glass-modified specimens was influenced by both firing temperature and additive content.

At a firing temperature of 1050 °C, the control specimen (C0) exhibited a compressive strength of 20.7 MPa. Incorporating waste glass produced a moderate increase in strength, attributed to enhanced densification and the formation of a glassy phase during firing. In general, increasing the firing temperature substantially improved compressive strength across all compositions, including the control specimen.

The highest compressive strengths, ranging from 33.1 to 39.5 MPa, were recorded for the GL10 and GL15 compositions fired at 1120 and 1125 °C. In contrast, the GL20 specimens exhibited compressive strengths of 21.8 and 30.0 MPa after firing at 1050 and 1100 °C, respectively, which were slightly lower than those of the corresponding GL15 specimens fired under the same conditions. Although the compressive strength tests were conducted on laboratory cylindrical specimens, the measured strength values (20.7–39.5 MPa) are comparable to the strength range reported for structural fired clay products in standard [46]. Therefore, the developed waste-glass-modified compositions show promising potential for structural ceramic applications, particularly the GL10 and GL15 specimens fired at 1125 °C.

The relatively lower strength of GL20 may be attributed to pore formation from gas release during firing and to deformation associated with excessive glassy-phase formation. These effects can partially offset the benefits of liquid-phase sintering, resulting in reduced mechanical performance despite the higher waste glass content. The compressive strength results showed a strong correlation with the corresponding trends in average density (Figure 5) and water absorption (Figure 6).

Overall, the results demonstrate that waste glass improves the compressive strength of clay-based ceramic materials produced from low-grade calcite-rich clay loam [55]. The positive influence of waste glass was observed across all investigated firing temperatures. For example, the control composition (C0) exhibited a compressive strength of 20.7 MPa after firing at 1050 °C, whereas incorporating 10–15 wt.% waste glass increased the strength to 21.6–22.0 MPa, corresponding to an improvement of 5.3–6.3%.



**Figure 7.** Compressive strength of ceramic specimens as a function of additive content and firing temperature: (a) waste glass-modified compositions (GL), (b) silica fume-modified compositions (SF).

The control composition reached its maximum compressive strength of 30.8 MPa at 1125 °C. At the same firing temperature, adding 10–15 wt.% waste glass increased the compressive strength to 36.0–39.5 MPa, representing an enhancement of 16.8–28.2%. These improvements can be attributed to the fluxing action of waste glass, which promotes liquid-phase sintering, enhances densification, reduces porosity, and contributes to the formation of a stronger ceramic microstructure [40].

According to Figure 6b, the silica fume-modified specimens exhibited a distinct trend in strength development. Increasing silica fume content reduced compressive strength at all firing temperatures investigated. The limited effectiveness of silica fume may be attributed not only to its refractory nature but also to the absence of significant alkali fluxing oxides. Unlike waste glass, silica fume consists mainly of amorphous  $\text{SiO}_2$  and therefore does not generate sufficient liquid phase within the investigated firing temperature range. As a result, densification remained incomplete and the beneficial effect of liquid-phase sintering was considerably weaker [56].

Increasing the firing temperature to 1100 °C improved the compressive strength of the silica-fume-containing specimens, likely due to enhanced reactivity of the silica fume and its interaction with the loam matrix. These processes may have promoted the formation of a limited glassy phase, contributing to partial densification of the ceramic body. The highest compressive strength values, ranging from 18.3 to 31.0 MPa, were obtained for the SF15 composition. Nevertheless, the strength of all silica-fume-modified specimens remained lower than that of the corresponding waste-glass-containing compositions fired under the same conditions. These results indicate that, under the investigated firing conditions, silica fume was less effective than waste glass in promoting liquid-phase sintering and improving the mechanical performance of the clay-based ceramic materials.

To better evaluate the performance and novelty of the developed ceramic compositions, the results obtained in the present study were compared with those reported in previous studies on clay ceramics modified with waste glass and silica fume (Table 1). The comparison includes the optimum additive content, firing temperature, density, water absorption, mechanical properties, and the main outcomes reported in the literature.

**Table 1.** Comparison of the performance of the present study with similar literature.

| Study                             | Additive                     | Optimum Content (wt.%) | Firing Temp (°C) | Density (kg/m <sup>3</sup> ) | Water Absorption (%) | Mechanical Strength (MPa) | Key Outcome                                                                                                             |
|-----------------------------------|------------------------------|------------------------|------------------|------------------------------|----------------------|---------------------------|-------------------------------------------------------------------------------------------------------------------------|
| Shishkin et al. (2020) [40]       | Waste glass                  | 15                     | 1000             | 2200                         | 3                    | 240                       | Reduced sintering temperature and enhanced densification through liquid-phase sintering.                                |
| Busch et al. (2022) [30]          | Waste glass (WTP)            | 10                     | 1050             | ~1820                        | ~9                   | –                         | 10 wt.% glass improved vitrification and reduced water absorption.                                                      |
| Abdel Hamid (2021) [24]           | Silica fume + granite sludge | 5 SF + 25 GSW          | 800              | ~1700                        | ~10                  | 18.2                      | Silica fume enhanced sintering at elevated temperatures and improved compressive strength while reducing open porosity. |
| Flores Nicolas et al. (2024) [32] | Waste glass                  | 9.1                    | 800              | 1900                         | –                    | 43.55                     | Glass powder enabled low-temperature sintering and improved ceramic properties.                                         |
| Darweesh (2019) [31]              | Waste glass                  | 25                     | 1150             | ~2012                        | ≈3.1                 | –                         | Excessive glass (>25 wt.%) deteriorated ceramic properties                                                              |
| Kumar et al. (2021) [23]          | Silica fume                  | 4                      | 950              | 1501                         | 9.69                 | 15.65                     | Addition of 4 wt.% silica fume 4 wt.% silica fume improved strength and reduced water absorption.                       |
| Baspinar et al. (2010) [25]       | Silica fume                  | 10                     | 1100             | ~2560                        | <1                   | ≈104                      | High-temperature vitrification markedly increased density and strength.                                                 |
| Present study                     | Waste glass (GL10)           | 10                     | 1125             | 2120                         | 3.8                  | 39.5                      | GL10 provided the best balance between density, strength, water absorption and dimensional stability.                   |

As shown in Table 1, previous studies consistently reported that waste glass and silica fume improve the properties of clay ceramics by promoting densification and liquid-phase sintering. However, the optimum additive content and firing conditions varied depending on the raw materials used. In the present study, the addition of 10 wt.% waste glass produced a favorable combination of high density ( $2120 \text{ kg/m}^3$ ), low water absorption (3.8%), and compressive strength (39.5 MPa) without firing deformation. These results demonstrate that the moderate addition of waste glass is an effective approach to improving the performance of low-grade clay ceramics.

### 3.6. Phase Composition and Microstructural Characterization (XRD and SEM)

Because the waste-glass-modified compositions achieved the highest performance, further investigation of their phase composition was conducted. X-ray diffraction (XRD) analysis was performed on the control specimen (C0) and the GL10 specimens fired at 1120 and 1125 °C. The corresponding diffraction patterns are presented in Figure 8a–d. Figures 8a and 8b show the XRD patterns of the C0 specimens, while Figures 8c and 8d correspond to the GL10 specimens fired at 1120 and 1125 °C, respectively.

The qualitative and quantitative phase compositions of the specimens are summarized in Table 2. The XRD pattern of the control specimen fired at 1120 °C showed a mineralogical composition dominated by quartz (42%) and diopside (37%), with smaller amounts of moissanite (7%), albite (9%), and microcline (5%). After firing at 1125 °C, the quartz content decreased slightly to 40%, and the moissanite content decreased to 4%, accompanied by increases in diopside (38%), microcline (6%), and albite (11%). The increase in diopside content is particularly beneficial, as diopside contributes to the mechanical strength and densification of calcium-rich ceramic systems. The absence of mullite is attributed to the high calcite content of the raw clay, which favors the formation of diopside and anorthite during firing.

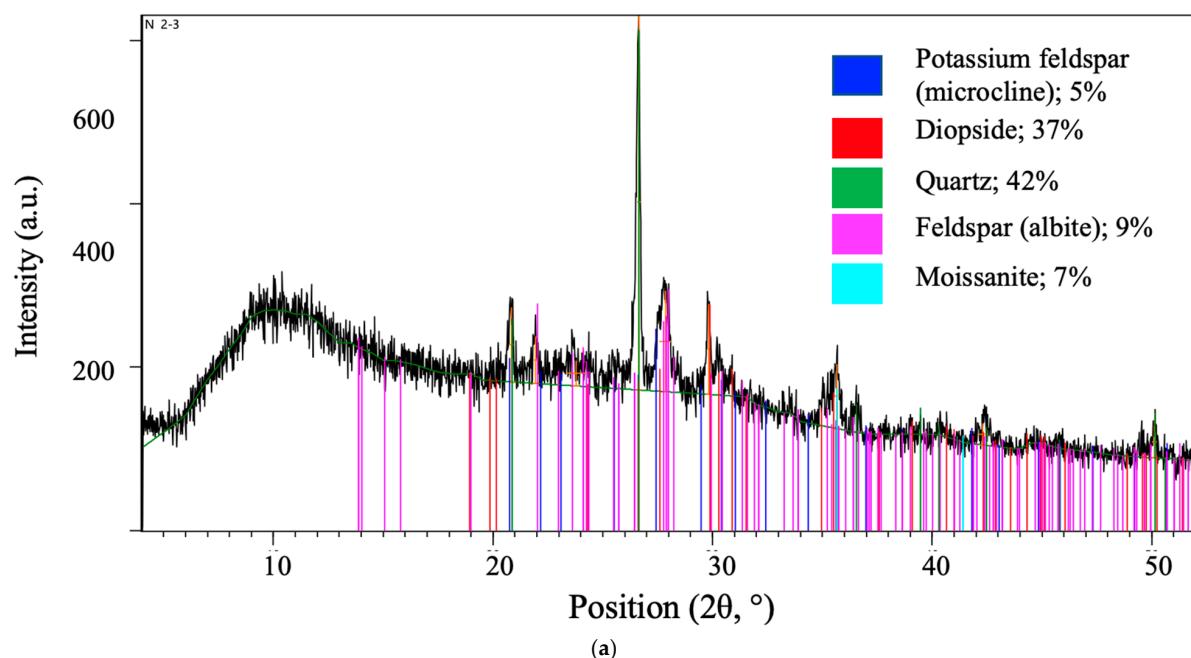
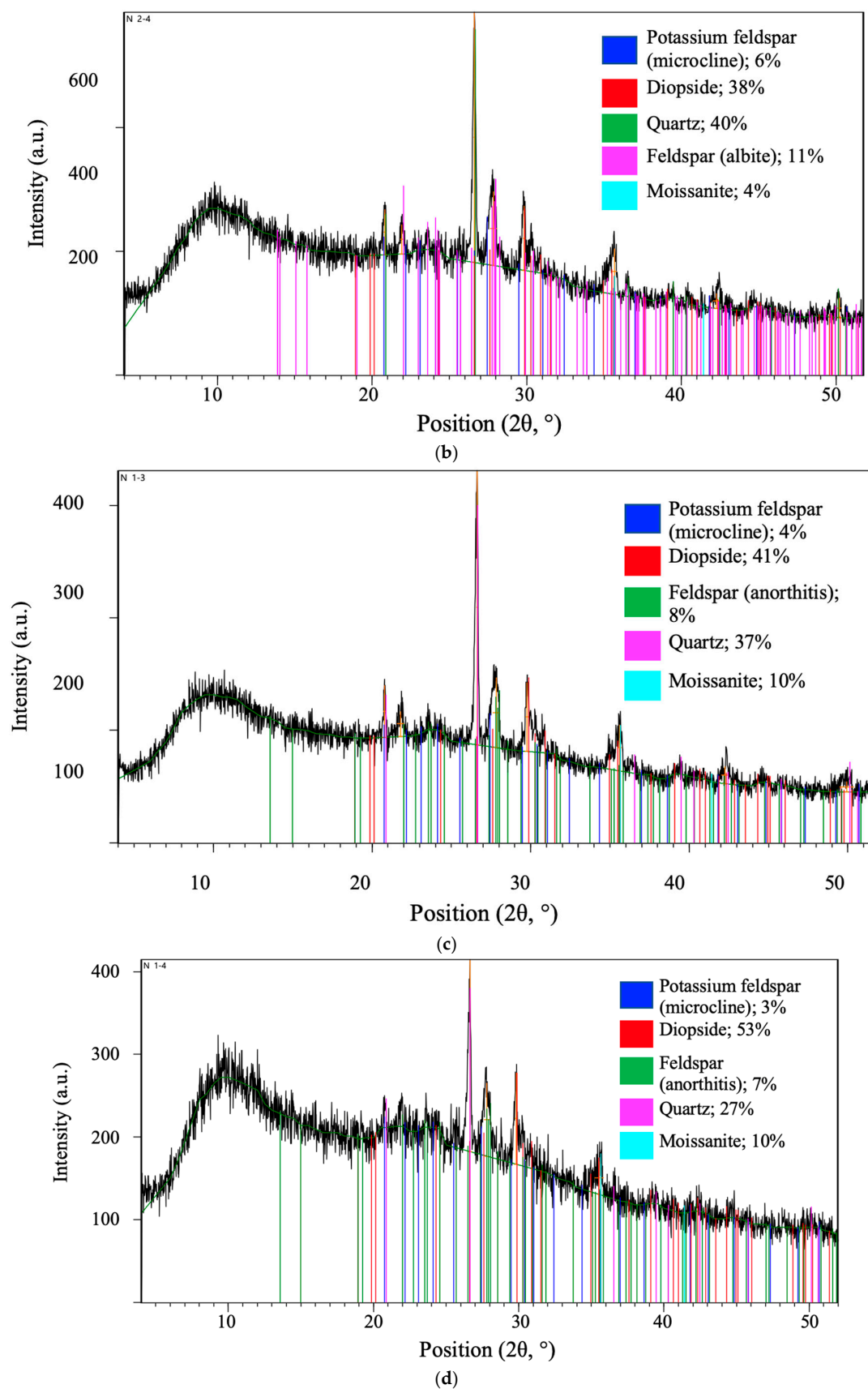


Figure 8. Cont.



**Figure 8.** XRD patterns of control and waste glass-modified (GL10) clay loam specimens fired at 1120 and 1125  $^{\circ}\text{C}$ : (a) control at 1120  $^{\circ}\text{C}$ ; (b) control at 1125  $^{\circ}\text{C}$ ; (c) GL10 at 1120  $^{\circ}\text{C}$ ; (d) GL10 at 1125  $^{\circ}\text{C}$ .

**Table 2.** Semi-quantitative phase composition of control and glass-modified clay loam-based compositions fired at 1120 and 1125 °C.

| Name of Composition | Phase Composition, %     |                 |                                                 |                                                                   |                                                                                                                 |                                                                        |
|---------------------|--------------------------|-----------------|-------------------------------------------------|-------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------|
|                     | Quartz, SiO <sub>2</sub> | Moissanite, SiC | Diopside, CaMg (SiO <sub>3</sub> ) <sub>2</sub> | Potassium Feldspar (Microcline) KAlSi <sub>3</sub> O <sub>8</sub> | Feldspar (Albite) (Na <sub>0.84</sub> Ca <sub>0.16</sub> ) Al <sub>1.16</sub> Si <sub>2.84</sub> O <sub>8</sub> | Feldspar (Anorthite), CaAl <sub>2</sub> Si <sub>2</sub> O <sub>8</sub> |
| C0 (1120 °C)        | 42                       | 7               | 37                                              | 5                                                                 | 9                                                                                                               |                                                                        |
| C0 (1125 °C)        | 40                       | 4               | 38                                              | 6                                                                 | 11                                                                                                              |                                                                        |
| GL10 (1120 °C)      | 37                       | 10              | 41                                              | 4                                                                 | n.d.                                                                                                            | 8                                                                      |
| GL10 (1125 °C)      | 27                       | 10              | 53                                              | 3                                                                 | n.d.                                                                                                            | 7                                                                      |

As the firing temperature increased, the intensity of quartz reflections gradually decreased, whereas the diopside diffraction peaks became more pronounced, particularly in the GL10 specimen fired at 1125 °C (Figure 8d). These changes indicate progressive phase transformation and enhanced sintering reactions at elevated temperatures.

According to the data in Table 2, incorporating waste glass significantly altered the phase composition of the fired ceramics. The quartz content decreased from 42% to 27%, while the proportion of diopside increased from 37% to 53%. In addition, albite, detected in the control composition, was no longer identified in the GL10 specimens, whereas anorthite appeared as a newly formed feldspar phase. These results indicate that the addition of waste glass promoted phase transformations during firing, including a reduction in quartz content, the decomposition of albite, and the formation of anorthite.

Similar phase evolution has been reported in prior studies of glass-modified clay ceramics, where incorporating waste glass promoted liquid-phase sintering, reduced residual quartz content, and enhanced the formation of calcium silicate and feldspar phases [57]. For example, earlier studies showed that glass-derived fluxing oxides intensify vitrification and facilitate reactions between SiO<sub>2</sub>-rich and CaO-bearing components, yielding compact ceramic matrices with reduced open porosity [58]. The present results are consistent with these findings, as the GL10 specimens exhibited lower quartz content, increased diopside formation, and the appearance of anorthite compared with the control samples.

At higher temperatures, the increased liquid-phase content may cause partial dissolution of crystalline phases and the formation of an amorphous glassy matrix, as indicated by the broader diffuse background in the GL10 patterns. The control samples were dominated by quartz and diopside, with minor amounts of moissanite, potassium feldspar (microcline), and albite. In contrast, the GL10 specimens had lower quartz content and higher diopside, especially at 1125 °C, where diopside reached 53% and quartz decreased to 27%.

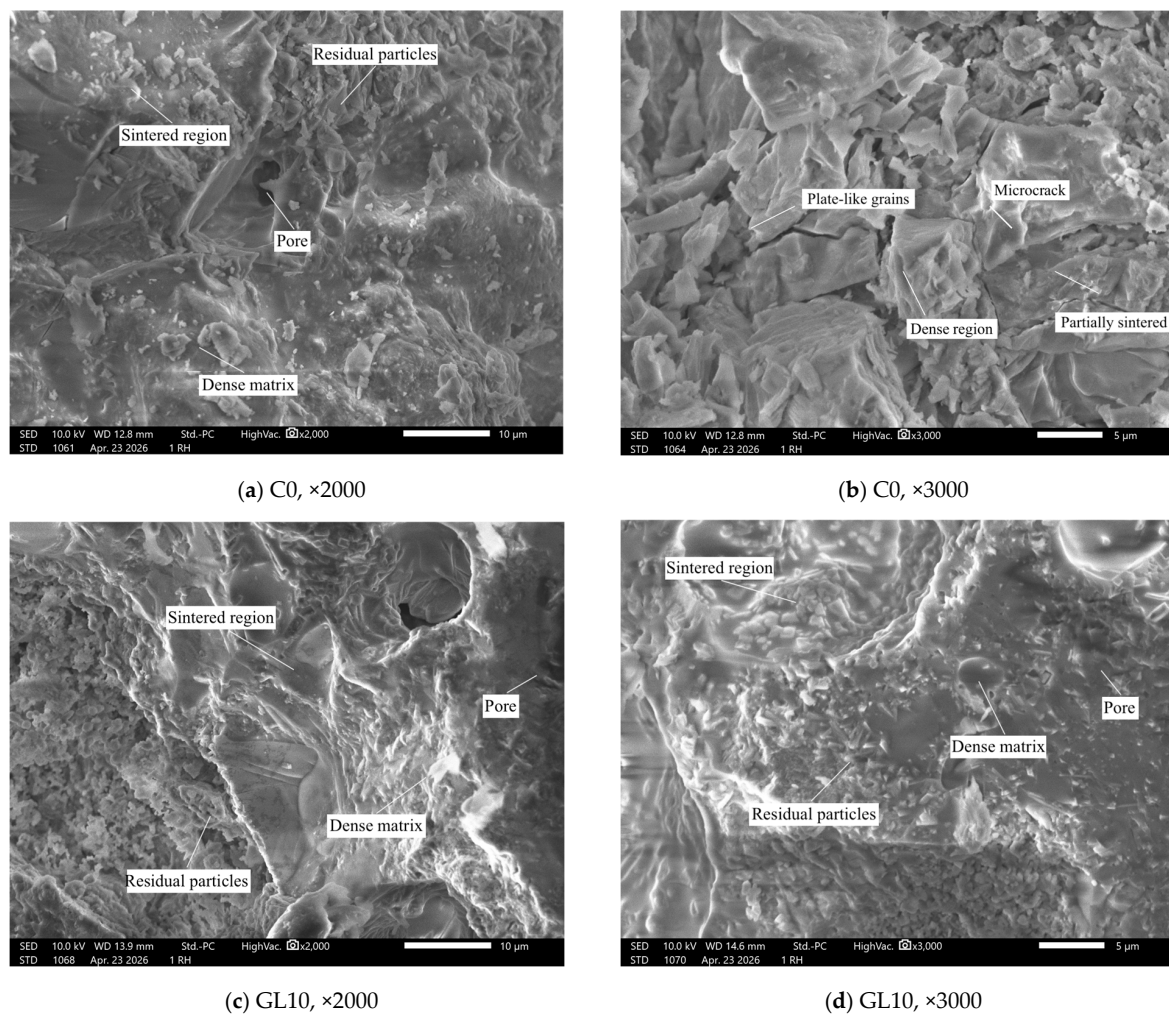
The observed phase transformations can be explained by the interaction between silica-rich glass and calcium-containing components of the clay loam during firing [59]. The presence of waste glass introduces fluxing oxides, which reduce the melting temperature and promote the formation of a liquid phase [60]. This enhances diffusion processes and facilitates the reaction between SiO<sub>2</sub> and CaO, leading to the formation of diopside and anorthite phases. These phase transformations are directly related to the improved densification observed in glass-modified compositions [61]. The formation of a liquid phase promotes particle rearrangement and pore filling, resulting in a more compact microstructure, as confirmed by SEM analysis. Consequently, the reduction in porosity leads to increased density, strength and decreased water absorption.

At higher temperatures, the increased amount of liquid phase may also lead to partial dissolution of crystalline phases and the formation of an amorphous matrix. While this enhances densification, excessive vitrification may adversely affect mechanical performance by forming microstructural defects.

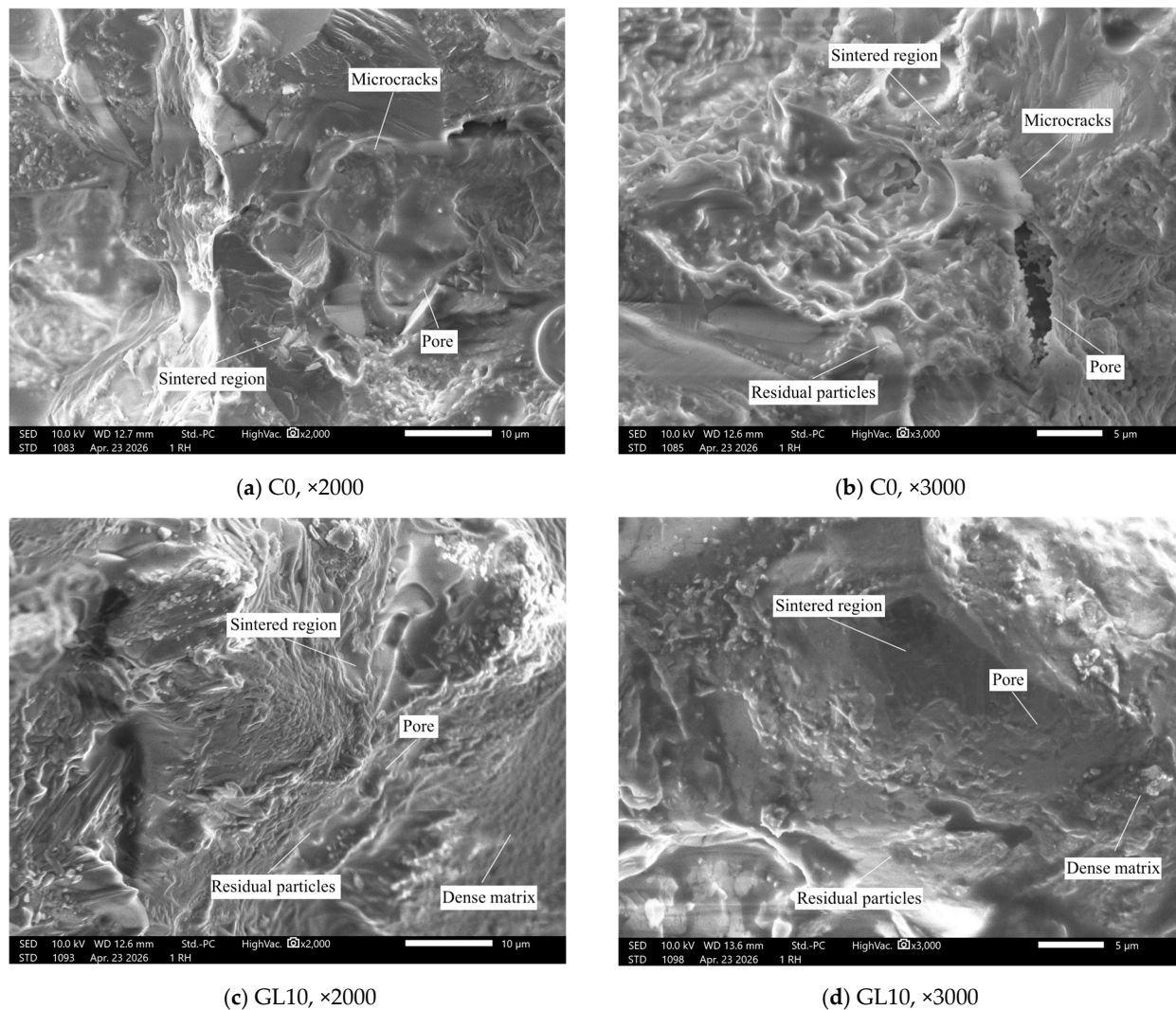
Overall, the XRD results confirm that the addition of waste glass significantly alters the phase composition and promotes liquid-phase sintering, which is the key mechanism governing the evolution of the microstructure and properties of the studied clay-based ceramic materials.

SEM micrographs of the ceramic specimens fired at 1120 and 1125 °C, Figures 9a–d and 10a–d, reveal a clear evolution of the microstructure as a function of both temperature and composition. At the lower magnification ( $\times 2000$ ), the overall structural arrangement and distribution of pores can be observed, whereas the higher magnification ( $\times 3000$ ) allows a more detailed assessment of pore morphology, interparticle bonding, and the development of sintered regions.

At 1120 °C, the control composition C0 (Figure 9a) exhibits a heterogeneous and relatively loosely bonded structure characterized by plate-like grains, microcracks, and a noticeable amount of residual porosity. These features are more distinctly visible at  $\times 3000$  (Figure 8b), where partially sintered regions and intergranular gaps indicate that densification is still governed predominantly by solid-state sintering. The absence of a sufficient viscous phase limits particle rearrangement and pore elimination, consistent with the moderate density values and relatively high water absorption observed in the control samples.



**Figure 9.** SEM micrographs of ceramic specimens fired at 1120 °C: (a,b) control composition (C0) at magnifications of  $\times 2000$  and  $\times 3000$ , (c,d) waste glass-modified composition (GL10) at magnifications of  $\times 2000$  and  $\times 3000$ .



**Figure 10.** SEM micrographs of ceramic specimens fired at 1125 °C: (a,b) control composition (C0) at magnifications of ×2000 and ×3000, (c,d) waste glass-modified composition (GL10) at magnifications of ×2000 and ×3000.

In contrast, the waste glass-modified composition (GL10) at 1120 °C demonstrates a more advanced stage of sintering. At ×2000 (Figure 9c), the formation of continuous sintered regions and a tighter matrix is evident, while at ×3000 (Figure 9d), residual particles and isolated pores can still be identified. This indicates the onset of liquid-phase-assisted sintering, where the softening of glass particles promotes interparticle bonding and enhances mass transport. However, the presence of remaining pores suggests that densification is not yet complete at this temperature.

A more pronounced microstructural transformation occurs at 1125 °C. The control composition C0 (Figure 10a,b) retains a relatively heterogeneous structure with visible pores and microcracks at both magnifications, confirming that densification remains limited in the absence of fluxing additives. In contrast, the GL10 composition exhibits a significantly more homogeneous and compact microstructure. At ×2000 (Figure 10c), a continuous and well-integrated matrix is observed, while at ×3000 (Figure 10d), the number and size of pores are noticeably reduced, and the connectivity between particles is substantially improved.

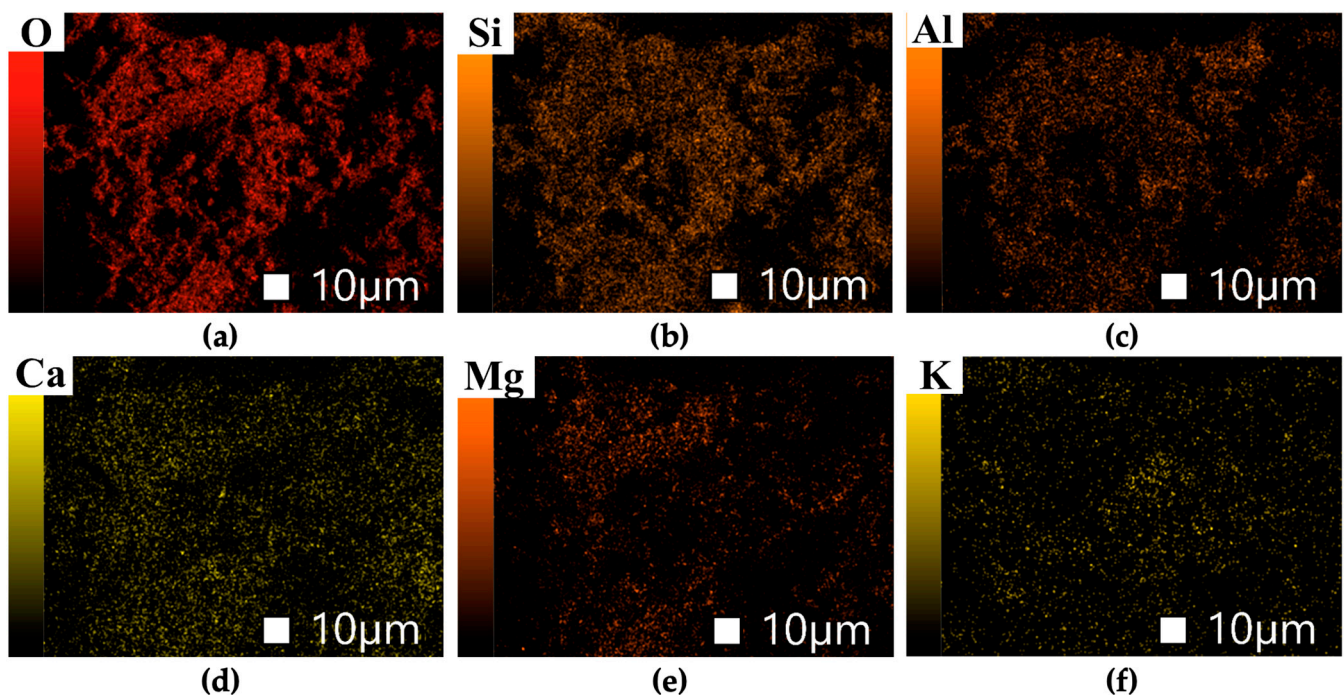
This microstructural refinement can be attributed to the formation of a viscous (sintered) phase, which enhances particle rearrangement, fills interparticle voids, and promotes the development of strong interparticle bonds. As a result, the pore structure becomes

less interconnected, and the overall porosity decreases. The reduction in interconnected porosity explains the improved compressive strength of GL10 compositions. These observations are in good agreement with the experimentally observed increase in average density and compressive strength, as well as the reduction in water absorption for the GL10 compositions.

At the same time, the SEM images also indicate the onset of localized over-sintering effects at higher temperatures. The increased amount of viscous phase may lead to pore coalescence, smoothing of grain boundaries, and the formation of isolated closed pores [62]. These features help explain the non-linear behavior of compressive strength at elevated temperatures, where excessive vitrification may partially reduce mechanical performance despite improved densification.

Overall, the combined SEM observations confirm that the dominant densification mechanism in glass-modified compositions is liquid-phase sintering. The presence of waste glass facilitates the formation of a transient viscous phase, which accelerates diffusion processes, enhances particle rearrangement, and promotes pore closure. This results in a dense and homogeneous microstructure compared to the control composition, where densification remains limited due to the absence of a fluxing component.

The Energy dispersive spectroscopy (EDS) elemental maps (Figure 11) confirm a relatively homogeneous distribution of the major elements within the ceramic matrix. Oxygen and silicon dominate the microstructure, reflecting the silicate framework formed during firing. Calcium and magnesium are uniformly distributed throughout the matrix, providing additional evidence for the formation of calcium–magnesium silicate phases, particularly diopside, identified by XRD. Likewise, aluminum is evenly distributed and participates in the formation of aluminosilicate phases, including anorthite. Potassium is mainly associated with residual potassium feldspar.



**Figure 11.** EDS elemental distribution maps of the GL10 ceramic specimen fired at 1125 °C showing the distribution of (a) O, (b) Si, (c) Al, (d) Ca, (e) Mg, and (f) K.

The absence of pronounced elemental segregation indicates that the liquid phase generated by softened waste glass enhanced elemental diffusion during firing. This transient viscous phase promoted mass transport between adjacent particles, accelerated the redistri-

bution of Ca, Mg, Al, and Si, and facilitated the nucleation and growth of newly formed crystalline phases. Consequently, pore filling and particle rearrangement became more effective, leading to the development of a dense and chemically homogeneous ceramic matrix. These observations are consistent with the XRD results and SEM micrographs, confirming that waste glass acts as an efficient fluxing additive by promoting liquid-phase sintering and enhancing the densification of the fired ceramic body.

The increase in diopside content and the appearance of anorthite in GL10 compositions at 1125 °C indicate enhanced phase reactions facilitated by the presence of glass. These transformations are directly associated with the formation of a viscous phase, which promotes particle rearrangement, pore filling, and stronger interparticle bonding. The combined XRD, SEM, EDS, density, water absorption, and compressive strength results consistently indicate that waste glass acts as an effective fluxing additive in calcite-rich clay ceramics. The formation of a liquid phase promoted the development of diopside- and anorthite-rich microstructures, resulting in enhanced densification and improved engineering performance. These findings confirm the strong relationship between phase evolution, microstructural refinement, and macroscopic properties of the fired ceramic body.

#### 4. Conclusions

The present study demonstrated that both the additive type and the firing temperature play critical roles in controlling the physical, mechanical, and microstructural properties of clay-based ceramic materials.

The results showed that waste glass is significantly more effective than silica fume in promoting densification of ceramic compositions. The incorporation of 10–15 wt.% waste glass led to a substantial increase in average density (up to 2200 kg/m<sup>3</sup>), a reduction in water absorption (down to ~3.0%) and improved compressive strength (up to 36–39.5 MPa at 1125 °C). In contrast, silica fume exhibited a limited influence on densification and caused structural instability at temperatures above 1100 °C.

Microstructural and phase analyses confirmed that the improved performance of waste glass-modified compositions is governed by liquid-phase sintering. XRD results showed a decrease in quartz content and increases in diopside and anorthite phases, while SEM observations revealed a less porous, more homogeneous matrix with reduced pore volume. These changes indicate enhanced particle bonding and pore elimination due to the formation of a viscous phase during firing. However, excessive glass content or elevated firing temperature led to over-vitrification, pore coalescence, and partial structural instability. This highlights the importance of balancing densification and structural integrity.

Among the investigated compositions, GL15 fired at 1125 °C demonstrated the best overall performance, providing an optimal combination of density, strength, and water absorption. Although GL15 exhibited the highest compressive strength, GL10 provided a more balanced combination of density, water absorption, dimensional stability and mechanical performance.

Overall, the results show that the additives' effects are governed by their distinct roles during firing. Waste glass acts primarily as a flux, promoting liquid-phase sintering, pore closure, and the formation of a less porous ceramic matrix. These phase and microstructural changes explain the higher bulk density and compressive strength, as well as the lower water absorption, observed in the GL10 and GL15 compositions at elevated firing temperatures. In contrast, silica fume, owing to its refractory nature, did not promote the formation of a sufficient amount of liquid phase at 1120 and 1125 °C to achieve effective densification of the ceramic body. Consequently, the superior sintering behavior of the waste-glass-modified compositions can be attributed to the fluxing action of waste glass, which provided a more favorable balance among densification, phase transformations,

and microstructural stability during firing. However, excessive glass content or firing temperature may cause over-vitrification and partial deformation, indicating that optimal ceramic performance is achieved only within a controlled additive–temperature range.

The results demonstrate that waste glass can be effectively used as an additive for low-grade calcium-rich clay raw materials in the production of building ceramics with reduced porosity, enhanced densification, and improved mechanical strength. The developed waste-glass-modified ceramics may be considered as promising materials for structural fired ceramic products, subject to further verification using full-scale specimens in accordance with product standards.

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