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Influence of fine aggregate type on geopolymer mortar performance in an elevated temperature environment

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Abstract: This paper focuses on developing a sustainable, high-temperature-resistant geopolymer mortar by selecting the most optimal fine aggregate type for high-temperature conditions. The developed building material has superior properties to conventional concrete and could possibly be significant in the context of fire-safety building materials. Ceramic brick waste (CBW) from open landfills and metakaolin waste (MKW) from an expanded glass production company (Lithuania) were used as precursor materials in a geopolymer system. The precursors were systematically substituted from 0 to 100% weight in five distinct compositions (F1-F5). The alkaline activator was a blend of 8M sodium hydroxide and sodium silicate (Na_2SiO_3/NaOH = 1.5). Five types of fine aggregates were examined: ordinary river sand (OSA), granite (GRA), basalt (BAS), ceramic waste (CBW), and corundum (COR), with a fraction size from 0 to 3 mm and the constant fine aggregate to precursor (FA/P) ratio of 1.5. Geopolymer mortar specimens were prepared to assess their compressive strength and density before and after exposure to high temperatures ranging from 200 to 800°C. A two-way analysis of variance (ANOVA) was conducted to evaluate the reliability of the residual compressive strength outcomes. The study showed that properly selected fine aggregates in an appropriate binder system improves high-temperature resistance. After 800 °C exposure, mortars with 75% CBW and 25% MKW precursors containing CBW and COR aggregates exhibited minimal microcracking and residual strengths of 34.7 MPa (78.7%) and 53.0 MPa (108.6%), respectively. This performance is attributed to partial reactivity, low thermal expansion, and inherent thermal stability.

Keywords: geopolymer mortar, ceramic brick waste, metakaolin waste, fine aggregate, thermal stability.

1. Introduction

The elevated level of urbanization on a global scale gives rise to the intensive necessity for new building and infrastructural development, which is unfeasible without fundamental building material – concrete. Concrete ranks second among the most widely used materials on the planet, exceeded in terms of consumption solely by water, with an average annual consumption of 1 m^3 per capita [1]. Concrete is regarded as a versatile material due to its role in climate-resilient infrastructure. However, a paradox emerges because ordinary Portland cement, an essential constituent of concrete, is also a significant contributor to climate change, owing to its association with elevated CO_2 levels. It is noteworthy that over 90% of countries worldwide are engaged in cement production, with China being responsible for over 50% of the total cement production [2]. Recent data indicates that global cement production is still growing; it reached 4.1 Gt in 2019 and 4.4 Gt in 2023 [3]. The total amount of CO_2 emissions across the globe in 2023 reached approximately 37 billion tonnes, with cement production contributing approximately 7-8% of this total [4]. The annual increase in carbon dioxide emissions from cement production sector represents a significant factor in the ongoing search for alternative building materials. Alkali-activated material or ‘geopolymer’ systems have attracted considerable attention as a potential replacement for traditional cementitious materials [5].

In the field of building materials, the fire resistance properties of a material are of paramount importance when evaluating its overall quality. It is widely acknowledged that cementitious materials frequently demonstrate substandard performance in extreme heat environments. Conversely, low-calcium alkali-activated materials (geopolymers) have been shown to exhibit stability under thermal treatment conditions. As demonstrated by Saavedra & Gutiérrez [6], cement-based concrete was shown to lose its strength following exposure to temperatures of 900°C, while geopolymer concrete was found to retain a strength of 15 MPa after exposure to temperatures of 1100°C. As posited by Zhao & Sanjayan [7], the retention of compressive strength at elevated temperatures of geopolymer conglomerates, in comparison to cementitious counterparts, can be attributed to the distinctive strength gain resulting from physio-chemical evolution during the heating process. This evolution leads to an increase in strength due to sintering and matrix densification. In general, geopolymers have been shown to gain strength within two distinct temperature ranges when exposed to elevated temperatures [8]. The initial enhancement in strength, attributable to secondary geopolymerization of unreacted precursor materials, appears from ambient temperature to an approximate temperature of 400°C. It has been demonstrated that the additional strength gain may be attributed to the viscous sintering effect, which has been shown to induce matrix densification at temperatures higher than 600°C. In the context of cementitious systems, it is noteworthy that a negligible enhancement in strength may be observed due to the reaction of unreacted particles at approximately 300°C. However, at temperatures ranging from 300 to 800°C, the reaction products begin to decompose, resulting in a significant loss of strength. Moreover, at temperatures exceeding 800°C, concrete can undergo a substantial loss of up to 100% of its initial strength, a phenomenon that may potentially lead to its structural collapse [9].

The temperature tolerance of geopolymer is influenced by several factors, including the Si/Al ratio, the precursor material, the activator, the aggregates, and the type and quantity of these materials. Researchers emphasize the pivotal role of the Si/Al ratio in the design of high-temperature-resistant geopolymer systems, as this ratio is a key parameter influencing the strength reductions at high temperatures. Kong et al. [10] demonstrated that at temperatures above 800°C, Si/Al ratios above 1.5 resulted in minimal strength reductions of up to 5%, attributable to the sintering of unreacted Si-rich precursor particles. A similar observation was made by Lahoti et al. [11] and He et al. [12], who found that geopolymers with higher Si/Al ratios exhibited superior performance in elevated-temperature environments.The performance of high-temperature resistant geopolymer composite is contingent upon the Si/Al ratio, which is determined by the selected type and quantity of precursor materials and alkaline activators. The precursor materials must be silicon and aluminum-rich, with minimal calcium content, to ensure optimal thermal stability [13]. These aluminosilicate sources are typically byproducts or wastes from various industrial or construction processes, including low calcium fly ash, metakaolin, silica fume, natural pozzolans, calcinated clays, or construction/demolition wastes [14]. The geopolymerization process is rendered unfeasible in the absence of an alkaline activator. This is particularly salient in the context of developing high-temperature resistant geopolymer systems, wherein the type and quantity of the alkali solution become pivotal factors [10]. The most usually used alkaline solutions are sodium or potassium-based hydroxides or silicates [15]. In the context of temperature resistance, K-based solutions (KOH or K_2SiO_3) are more favorable than Na-based solutions (NaOH or Na_2SiO_3). Hosan et al. [16] conducted a study in which they evaluated the performance of Na and K-based geopolymers at elevated temperatures. The experimental results indicate that K-based geopolymer composites demonstrate enhanced performance at elevated temperatures of up to 1000°C compared to their Na-based counterparts. This observation was attributed to the higher residual compressive strength, reduced thermal shrinkage, and fewer surface cracks. The enhanced performance of K-based geopolymers under high-temperature conditions can be attributed to the presence of leucite and kalsilite crystalline phases, which exhibit superior thermal stability compared to nepheline or sodaline phases that occur in Na-based geopolymers [17]. In the absence of elevated temperatures, Na-based geopolymers have been observed to demonstrate superior mechanical properties, particularly in the early stages of development. Additionally, sodium-based alkaline solutions are more economical and readily available than potassium-based alternatives [18]. Consequently, Na-based geopolymers are subjected to increasingly frequent testing in high-temperature conditions.

In cementitious or geopolymer systems, the use of aggregates is of paramount importance. The use of aggregates is necessary to reduce material costs and provide dimensional stability, strength, enhanced durability, and limit shrinkage and cracking. Aggregates typically comprise between 60% and 80% of the total conglomerate volume. The existing body of literature addressing the use of various fine aggregates in geopolymer systems under high-temperature conditions is limited. In the field of high-temperature resistant geopolymer systems, the selection of suitable aggregates is of critical importance. Pan et al. [19] have conducted research on the impact of elevated temperatures on the compressive strength of geopolymer mortar.

Their findings indicate that at high temperatures, the raw materials undergo decomposition due to thermal stress, which is a consequence of thermal incompatibility. This results in damage to the mortar. The presence of an uneven temperature field within the geopolymer mortar is attributable to the thermal conductivity and heat capacity of the constituent materials. The resultant temperature gradient consequently leads to displacement incoordination between the outer and inner components of the geopolymer mortar. Consequently, distinct aggregates possess disparate thermal expansion coefficients, thereby exerting varying effects on the internal structure of geopolymer mortar when subjected to high-temperature exposure. These thermal deformations, in turn, can precipitate either geopolymer mortar shrinkage or expansion, thereby diminishing the residual strength of the material. In the context of fine aggregates, natural river sand has emerged as the most commonly employed. Nevertheless, quartz aggregate exhibits an elevated thermal expansion coefficient, reaching a maximum at approximately 573°C [20], which may result in deleterious effects on geopolymer mortar under elevated temperatures. Additionally, limestone aggregates are a more preferable option for high-temperature geopolymer systems, although they decompose at 800–900°C, releasing CO_2 during the process [21]. As stated in the existing literature [22-26], the addition of alumina, silica fume, or other heat-resistant aggregates has been demonstrated to effectively mitigate thermal expansion/contraction and assist in preserving compressive strength at elevated temperature environment. The addition of 20 wt.% quartz or granite aggregate to metakaolin-based geopolymer was studied by Subaer and van Riessen [22], who found that it reduced total shrinkage by 50% in the temperature range of 23–500°C compared to geopolymer without aggregates. Furthermore, they found that increasing the aggregate content up to 40 wt.% yielded additional shrinkage reduction. A parallel observation is made in the study by Hassan et al. [23] that the incorporation of silicon-containing aggregates can reduce fly ash-based geopolymer thermal shrinkage by 40–60% at a temperature of 1000°C. According to Kovářík et al. [24], ceramic waste aggregates are a suitable option for high-temperature-resistant geopolymer mortars. The researchers recommend a fine aggregate-to-precursor (FA/P) ratio of 2.5, as well as a gradation design based on Fuller's model. These modifications led to a reduction in thermal shrinkage of geopolymer composite from 2.243% to 1.212% at a temperature of 1000°C, with minimal compressive strength loss. A research study was conducted by Şahin et al. [25] on aggregate types in metakaolin-based geopolymer composites. Six distinct types of aggregates were examined in this study, including silica sand, river sand, sandstone, Rilem sand, waste concrete aggregate, and basalt sand. The most favorable outcomes at 800°C were achieved with basalt aggregate due to its denser and more compact structure in comparison to other aggregates. As indicated by Li et al. [26], corundum aggregate exhibits superior potential in the context of high-temperature geopolymer development. Specifically, a volume fraction ranging from 50-55% has been identified as effective in mitigating mass loss, volume shrinkage, and crack formation. This approach has been shown to enhance mechanical properties and thermal stability, thereby promoting geopolymer development. Additionally, particle size distribution is a key factor when selecting the right fine aggregate. A study by Li et al. [27] analyzed the key fine aggregate parameters influencing geopolymer performance. The optimal range of strengths was achieved when 75–95% passed the 1.18 mm sieve and 15–25% passed the 0.15 mm sieve, with a fineness modulus between 2.2 and 2.6. Zhang et al. [28] analyzed how the ratio of fine aggregate-to-precursor (FA/P) ratio affects geopolymer mortar strength. They found that as the ratio increased from 1 to 5, the mortar's strength and impermeability decreased, while its crack resistance increased. However, Zailani et al. [29] state that the most optimal fine aggregate-to-precursor ratio for balancing robustness of bonding strength and favorable workability is 2:1. Additionally, it is recommended to avoid surpassing the FA/P ratio of 2:1, as this can result in an inadequate amount of paste to efficiently adhere to the aggregate, potentially leading to poor mixture workability and reduced structural integrity and strength.

Although numerous studies have examined the high-temperature behavior of geopolymers, the influence of fine aggregate type remains insufficiently explored. Existing research typically investigates single aggregates, uses inconsistent temperature regimes, or focuses on coarse aggregates. The present study provides a systematic, comparative evaluation of multiple fine aggregates under identical high-temperature exposure up to 800°C, using waste-derived precursors. By assessing residual compressive strength and microstructural changes within a unified experimental framework, this work identifies the most effective fine aggregate for high-temperature geopolymer mortar. This integrated approach has not been previously reported in the literature.

2. Experimental Framework

2.1. Testing setup and procedures

The elemental composition of the precursor material was established through the utilization of X-ray fluorescence (XRF) analysis. Apparatus: “Bruker X-ray S8 Tiger WD” XRF spectrometer (Bruker AXS, Karlsruhe, Germany).

The particle size distribution, density, and fineness of the precursor materials were established through the implementation of laser diffraction (dry method). Apparatus: “CILAS 1090 LD” laser scattering particle size analyzer (Cilas, Orléans, France).

The mineral compositions of precursor materials were established through the use of X-ray diffraction (XRD) analysis. Apparatus: “D8 Advance” diffractometer operating on 40 kV and 40 mA (Bruker AXS, Karlsruhe, Germany). The X-ray beam was filtered with a 0.02 mm Ni filter to select the CuKα wavelength. The powder X-ray diffraction patterns were identified in accordance with the recommendations of the PDF-2 database. The material's analyzed quantitatively using the Rietveld method (TOPAS 4.1).

The rheology of fresh geopolymer mortar mixture was established through a consistency (flow table) test of fresh geopolymer mortar according to EN 1015-3 [30]. Apparatus: a flow table with truncated conical mould (TESTING Bluhm & Feuerherdt, Berlin, Germany).

The mechanical performance of hardened geopolymer mortars was established through the application of compressive strength performance tests according to EN 12390-3 [31]. The compressive strength of geopolymer specimens was evaluated prior to and following exposure to elevated temperatures. Apparatus: “Zwick Z100” universal analysis machine (ZwickRoell, Ulm, Germany), with a loading rate of 0.6 ± 0.2 MPa/s.

The microstructure of geopolymerization reaction products was established through the use of scanning electron microscopy (SEM). Apparatus: high-tenacity SEM "FEI Quanta 200 FEG" microscope (FEI, Hillsboro, OR, USA) with a Schottky field emission gun (Hitachi, Tokyo, Japan). Chemical composition investigated with “Bruker Quad 5040 EDS detector (123 eV)” energy-dispersive X-ray spectroscopy (EDS) detector (Bruker, Karlsruhe, Germany).

The determination of the particle size distribution of the fine aggregate (sand) was executed in accordance with the European standard EN 933–1:2012 [32], while the fineness modulus (FM) was computed using the equation delineated in the standardized European norm EN 12620:2002+A1:2008 [33]. Apparatus: a series of sieves with square apertures ranging in size from 0.063 to 8.0 mm (TESTING Bluhm & Feuerherdt, Berlin, Germany).

The constant heating rate method was employed to simulate elevated temperature conditions. This method was selected to facilitate the expeditious attainment of the desired temperature. Safety was the top concern in choosing this method. To attain the desired temperatures of 200, 400, 600, and 800 ^{0}C, the temperature of the system was elevated from its ambient level at a rate of 5 ^{0}C per minute using an electric furnace. The targeted temperature was sustained for two hours to ensure the uniform distribution of heat across the specimens. The geopolymer specimens were then subjected to a gradual natural air cooling process within a furnace, continuing until the specimens reached ambient room temperature. A subsequent evaluation of the specimens was conducted after a 24-hour repose period, focusing on the post-heating characteristics.

2.2.. Constituent Materials and Their Characteristics

The precursor materials that were selected for the production of geopolymer paste were ceramic brick waste (CBW) and waste metakaolin (MKW). The selection of these waste materials was based on their high aluminosilicate content. Ceramic brick waste (see Fig. 1a) is identified as a predominant category of construction and demolition (C&D) debris. In this study, discarded ceramic bricks – originally ranging in size up to 35 centimeters – were first crushed to yield fragments between 0.5 and 4.0 centimeters. Subsequently, the fragments were submitted to a 2-hour mechanical milling procedure, resulting in a fine-ground powder with an increased specific surface area, thereby rendering it conducive to geopolymerization process. Metakaolin, an industrial by-product, is produced by the thermal activation of kaolinite clay and serves as an anti-agglomeration agent during the fabrication of expanded glass granules. The waste metakaolin (MKW) used in this study (see Fig. 1b) exhibits a modified oxide composition – particularly a higher Na_2O content – due to contamination with expanded glass particles, distinguishing it from conventional metakaolin. Ceramic brick waste and metakaolin waste were selected as precursor materials due to their divergent physicochemical characteristics. MKW, with its high amorphous content, has been shown to provide high reactivity and to promote geopolymerization, while CBW, having more crystalline nature resulting from prior thermal treatment, has been found to contribute to matrix stability and to improve performance at elevated temperatures.


(a) (b)

Fig. 1. Images of the precursor materials: (a) – CBW, (b) – MKW.

The effectiveness of the geopolymerization process was optimized through the implementation of two liquid alkaline solutions: sodium hydroxide and sodium silicate. The sodium hydroxide solution (NaOH) was prepared through the combination of sodium hydroxide pellets with a purity level of 97% and a specific volume of distilled water, resulting in a constant NaOH solution concentration of 8M. The sodium silicate solution (Na_2SiO_3) was supplied by a local chemical supplier and, according to the supplier specifications, has a silica modulus (Ms) of 3.0, with a composition of 9.3% Na_2O and 28.0% SiO_2, and a specific gravity of 1.38 g/cm^3.

The elemental composition of the precursor materials was ascertained through an X-ray fluorescence (XRF) investigation technique, the results of which are specified in Table 1. It was ascertained that select precursor materials correspond to the aluminosilicate material type, as their oxide composition is primarily SiO_2 and Al_2O_3. However, it was observed that MKW contains a relatively high percentage of Na_2O, a factor that must be considered when determining the appropriate dosage of alkaline solutions.

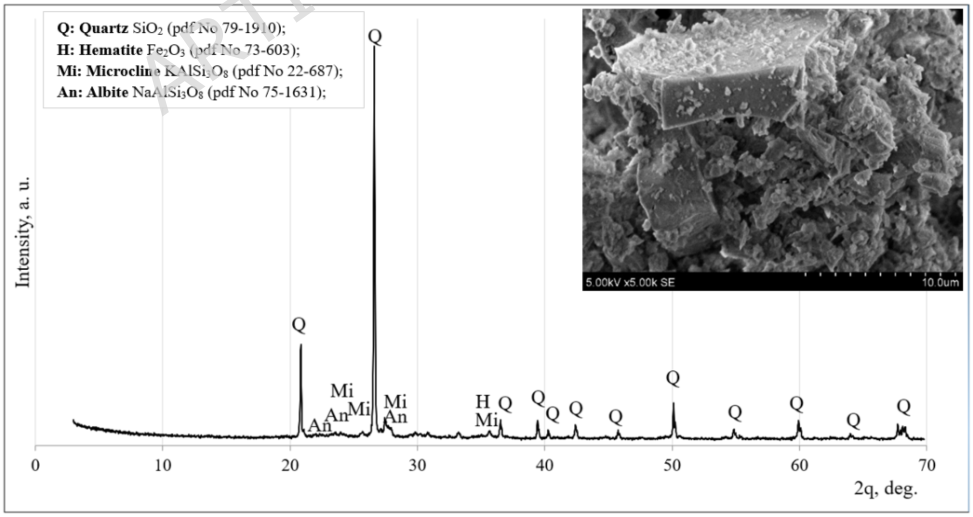
Table 1. Oxide composition and formed compounds (wt.%) of CBW and MKW precursor materials.

	Oxide/Compound quantity, %

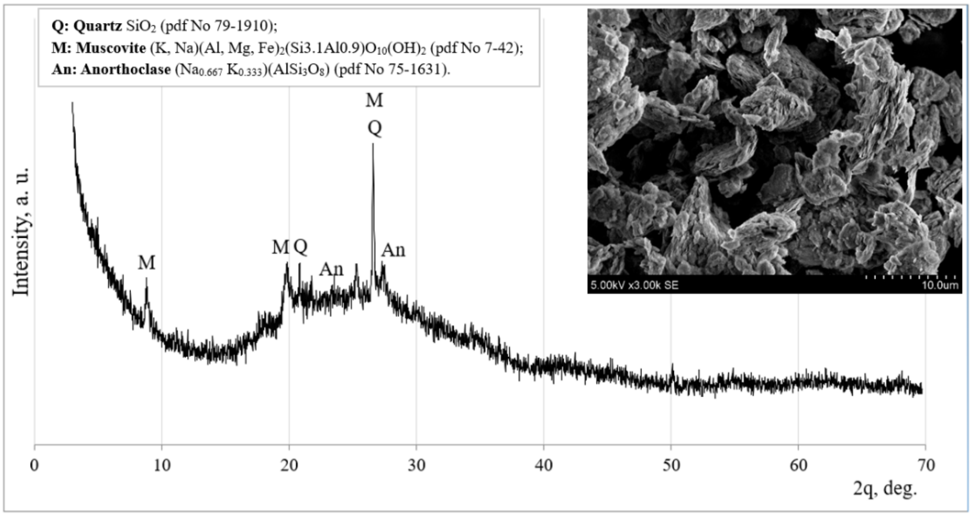
Precursor material	SiO_2	Al_2O_3	Fe_2O_3	K_2O	CaO	MgO	Na_2O	TiO_2	P_2O_5	SO_3	Cl	Other	LOI	Total
CBW	69.95	13.40	4.60	3.80	3.70	2.20	0.90	0.70	0.16	0.11	0.03	0.19	0.86	100
MKW	55.60	30.60	0.88	0.70	2.90	0.85	7.40	0.52	0.11	0.13	-	0.30	1.91	100

	Quartz	Microcline	Hematite	Albite	Muscovite	Kaolinite	Anorthite	Amorphous	Total
CBW	65.80	13.00	2.10	9.60	-	-	-	9.50	100
MKW	3.30	-	-	-	5.20	10.50	7.90	73.10	100

The selected CBW and MKW precursor materials had to be specified with a substantial presence of amorphous SiO_2 and Al_2O_3, in order to achieve an effective geopolymerization reaction. Their mineralogy was examined using X-ray diffraction (XRD). The XRD patterns are displayed in Figure 2, while the quantity analysis is presented in Table 1. It was observed that MKW precursor is significantly more suitable for geopolymerization reaction than CBW due to its higher reactivity (amorphous phase). The CBW precursor was characterized by a preponderance of quartz (65.80%) and microcline (13.00%) compounds, with a minimal amount of amorphous content (9.50%). In contrast, the MKW precursor exhibited a lower proportion of quartz (3.30%) and a higher proportion of kaolinite (10.50%) and anorthite (7.90%), along with a substantial content of amorphous phase (73.10%). Thus, the XRD patterns provide definitive confirmation of the predominant amorphous nature of MKW, whilst also indicating higher crystallinity in CBW. This supports their complementary roles in the geopolymer system, where MKW enhances reactivity and gel formation, while CBW contributes to structural stability and suitability for high-temperature applications.



(a)



(b)

Fig. 2. XRD pattern and SEM micrograph of precursor materials: (a) – CBW, (b) – MKW.

The microstructure of the precursor materials was subjected to a thorough analysis through scanning electron microscopy (SEM). The resulting SEM micrographs are displayed in Figure 2. The CBW precursor was identified as having a sharp-edged particle shape, while the MKW precursor demonstrated a plate-like stratified particle shape. Additionally, precursor materials were investigated with the technique of laser diffraction. The CBW precursor particle characteristics are as follows: a density of 2.77 g/cm^3, a specific surface area of 497.96 m^2/kg, a particle size distribution range of 0.1–100.0 μm, a maximum number of uniform particles of 2.01% at 1.4 μm, and a mean particle diameter of 12.89 μm. The MKW precursor is specified with the following parameters: a density of 2.59 g/cm^3, a specific surface area of 472.75 m^2/kg, a particle size distribution in range of 0.1 – 66 μm, a maximum number of uniform particles of 2.26% at 18.0 μm, and a mean particle diameter of 11.31 μm. A comparison of the two precursors reveals that they possess comparable fineness; however, the CBW precursor exhibits higher density.

The geopolymer system was enriched with five distinct types of fine aggregates (ordinary sand, pink granite, basalt, ceramic brick waste and corundum) to form a geopolymer mortar (see Fig. 3). The selected fine aggregate, ordinary sand (OSA) known as a natural river sand was utilized as a control aggregate for the purpose of comparing other aggregates, including pink granite (GRA), basalt (BAS), ceramic waste (CBW), and corundum (COR). The objective of this comparison was to ascertain their potential to enhance high-temperature resistance.

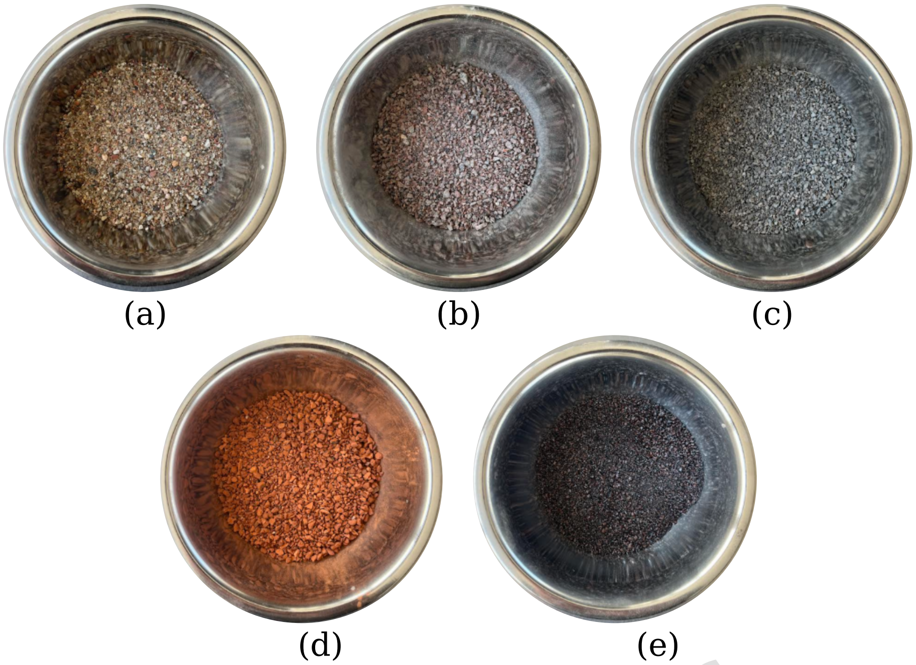

(a) (b) (c)
(d) (e)

Fig. 3. Variety of fine aggregates: (a) – ordinary river sand, (b) – pink granite, (c) – basalt, (d) – ceramic waste, (e) – corundum.

In this study, the selected natural fine aggregates (ordinary river sand, pink granite, basalt, and corundum) were obtained from a local Lithuanian market, and the ceramic waste fine aggregate was made by recycling old ceramic roof tiles. The tiles were first crushed and then sieved until a certain fine fraction was obtained. The essential parameters of the selected fine aggregates are provided by the manufacturer and are described in Table 2. According to the established parameters, the optimal performance under high-temperatures was predicted to be achieved in geopolymer compositions comprising corundum fine aggregates, attributable to their exceptional thermal stability and very high melting point, which makes corundum a suitable material for refractory and high-temperature applications [34, 35]. This enables evaluation of its potential use as a refractory fine aggregate in geopolymer composites exposed to elevated temperatures.

Table 2. Key properties of the selected fine aggregates.

Aggregate type	Key properties						
	Dominating oxides	Particle shape	Particle density, kg/m^3	Bulk density, kg/m^3	Melting temperature, ^{o}C	Thermal stability, ^{o}C	Water absorption, %
Ordinary (river) sand	SiO_2 >95%	Rounded/sub-rounded	~2650	~1550	~1710	≤500-573	0.8-1.5
Pink granite	SiO_2, Al_2O_3	Angular	2650-2700	~1400	1210-1260	≤700-750	0.3-0.6
Basalt	SiO_2, Al_2O_3 & Fe_2O_3	Angular	2900-3000	~1350	1250-1350	≤800-850	0.2-0.4
Ceramic waste	SiO_2, Al_2O_3	Angular/porous	2100-2300	~1000	~1300	≤900-1000	6.0-10.0

Corundum	Al_2O_3 >95%	Angular/sharp	3900-4000	~1600	~2050	≤1600	0.1-0.3

Additionally, the selected fine aggregates had a fraction size up to 3 millimeters. The distribution of particles is illustrated in Figure 4, with the cumulative percentage of passing sieves with square apertures presented. The analysis of fine aggregate granulometry has demonstrated that ordinary river sand, pink granite, and corundum are finer than ceramic brick and basalt aggregates. It has been demonstrated that the fraction distribution of ordinary river sand, pink granite, and corundum aggregates is quite similar, with a majority of their particles exhibiting uniform distribution across the 0.5, 1.0, and 2.0 mm sieves. In contrast, ceramic waste and basalt fine aggregates demonstrate a higher proportion of particles retained on the 1.0, 2.0, and 4.0 mm sieves. It can be concluded that basalt and ceramic waste fine aggregates lack finer particles, which could lead to better fluidity due to the small specific surface area. However, this could potentially result in inadequate filling of the geopolymer system matrix, which might lead to degraded mechanical performance, particularly at elevated temperatures. The hypothesis is supported by the findings of the fineness modules (FM) analysis, which revealed that basalt and ceramic waste exhibited the highest FM values of 4.16 and 4.17, respectively. Meanwhile, pink granite demonstrated a medium FM value of 3.63, while ordinary river sand and corundum exhibited the lowest FM values of 3.21 and 3.20, respectively. It has been determined that all of the selected fine aggregates can be classified as coarse fine aggregate, as the majority of aggregates fall within the range of 2.4–4.0, with a few fine aggregates exhibiting slightly higher values.

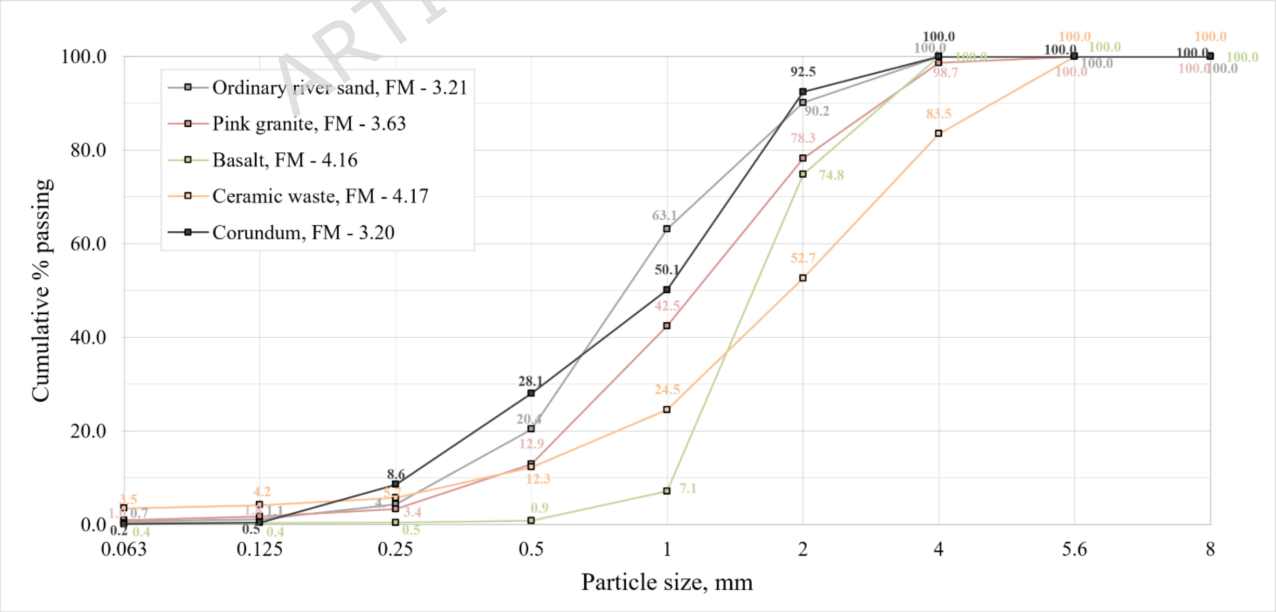


Fig. 4. Fine aggregate particle size distribution.

2.3 Mixture Formulation and Specimen Preparation

The compositions of the geopolymers were precisely designed with varying proportion ratios, using two aluminosilicate precursor materials (ceramic brick waste & waste metakaolin), two alkaline activators (NaOH & Na_2SiO_3), and five types of fine aggregates (ordinary river sand, pink granite, basalt, ceramic waste & corundum). Consequently, a total of 25 geopolymer mortar mix compositions were formulated, incorporating varying proportions of the identified constituents, as outlined in Table 3. The precursor materials CBW and MKW are blended at contents (F1 ÷ F5) of 100/0, 75/25, 50/50, 25/75 and 0/100 wt%. This study used two separate alkaline solutions (sodium silicate and sodium hydroxide) combined at a fixed mass ratio of 1.5:1.0 (Na_2SiO_3:NaOH) as part of the mix design. A constant morality of 8M was used for the sodium hydroxide solution through all of the designed compositions. Also, as the preparation process of sodium hydroxide solution generates exothermic reaction, the solution was cooled to room temperature before use. Our recent studies [36] have shown that increasing the sodium hydroxide dosage is essential for improving geopolymerization efficiency, particularly when larger quantities of metakaolin residue are introduced into the system. Metakaolin waste reacts faster than ceramic brick waste due to its higher Al_2O_3 content and greater amorphous phase, which promotes faster reactions during early curing. Consequently, the composition of the alkaline solution varies among mixtures to ensure the efficient geopolymerization process. In addition to variations in activator dosage, the total Na_2O content of the system is further influenced by the metakaolin waste precursor, which contains 7.4% Na_2O. Given the variation in the proportion of metakaolin across the F1–F5 mixtures, its contribution to the overall alkali content is not constant. Therefore, the overall SiO_2/Na_2O ratio was integrated to more accurately reflect the effective chemical environment of each mixture. This parameter furnishes a more comprehensive description of the system chemistry, accounting for both the alkaline activator and the intrinsic Na_2O contribution from the precursors, and is directly relevant to geopolymerization behaviour, gel formation, and resulting material properties.

Regarding fine aggregates, the objective was to utilize a constant fine aggregate to precursor (FA/P) ratio of 1.5:1.0 across all the designed geopolymer mortar compositions. However, this was not feasible because ceramic waste fine aggregates exhibited a significantly lower bulk density compared to other fine aggregates. Specifically, the lower mass of the particles resulted in a higher volume percentage within the mixture. Consequently, the FA/P ratio when utilizing ceramic waste fine aggregate exhibited a rotational range [0.57, 0.67, 0.80]:1.0.

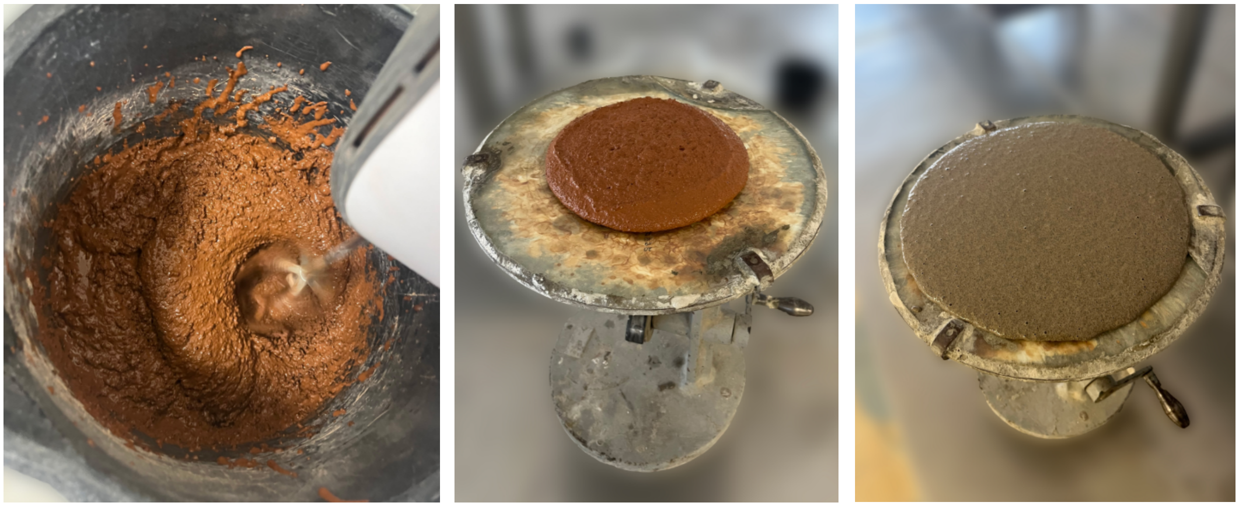
Table 3. Geopolymer mortar compositions (wt.%)

Mix ID	Precursor materials		Fine aggregate					Alkaline solutions		Mix design parameters				Spread diameter (flow table), mm
	CBW	MKW	Ordinary river sand	Pink granite	Basalt	Ceramic waste	Corundum	NaOH	Na_2SiO_3	AS/P[1]	FA/P[2]	W/S[3]	SiO_2/Na_2O	
F1-OSA	35.09	0.00	52.63					4.91	7.37	0.35	1.50	0.21	14.42	155
F1-GRA	35.09	0.00		52.63				4.91	7.37		1.50			145

F1-BAS	35.09	0.00			52.63			4.91	7.37		1.50			140
F1-CBW	52.05	0.00				29.73		7.29	10.93		0.57			130
F1-COR	35.09	0.00					52.63	4.91	7.37		1.50			150
F2-OSA	25.42	8.47	50.85					6.10	9.15	0.45	1.50	0.27	9.10	180
F2-GRA	25.42	8.47		50.85				6.10	9.15		1.50			180
F2-BAS	25.42	8.47			50.85			6.10	9.15		1.50			160
F2-CBW	35.43	11.81				31.50		8.50	12.76		0.67			155
F2-COR	25.42	8.47					50.85	6.10	9.15		1.50			170
F3-OSA	16.39	16.39	49.18					7.21	10.82	0.55	1.50	0.32	6.54	205
F3-GRA	16.39	16.39		49.18				7.21	10.82		1.50			195
F3-BAS	16.39	16.39			49.18			7.21	10.82		1.50			170
F3-CBW	22.56	22.56				30.07		9.92	14.89		0.67			160
F3-COR	16.39	16.39					49.18	7.21	10.82		1.50			205
F4-OSA	7.81	23.44	46.88					8.75	13.13	0.70	1.50	0.39	4.88	215
F4-GRA	7.81	23.44		46.88				8.75	13.13		1.50			210
F4-BAS	7.81	23.44			46.88			8.75	13.13		1.50			190
F4-CBW	10.00	30.00				32.00		11.20	16.80		0.80			175
F4-COR	7.81	23.44					46.88	8.75	13.13		1.50			220
F5-OSA	0.00	30.30	45.45					9.70	14.55	0.80	1.50	0.43	3.95	240
F5-GRA	0.00	30.30		45.45				9.70	14.55		1.50			230
F5-BAS	0.00	30.30			45.45			9.70	14.55		1.50			220
F5-CBW	0.00	38.46				30.77		12.31	18.46		0.80			210
F5-COR	0.00	30.30					45.45	9.70	14.55		1.50			235

AS/P[1] – alkali solution to precursor (powder) ratio; FA/P[2] – fine aggregate to precursor (powder) ratio; W/S[3] – water to solids ratio.

The specimen preparation for geopolymer mortar is comprised of three distinct stages, namely dry/wet mixing, casting, and curing. The dry mixing procedure entails the blending of dry precursor materials (CBW and MKW) for a duration of one minute. Concurrently, the alkaline solutions (NaOH and Na_2SiO_3) are mixed together for an equivalent period of time. Subsequently, the wet mixing procedure is initiated. In this procedure (see Fig. 5a), dry precursors are meticulously mixed with unified alkaline solutions using an electric mixer for a duration of three minutes. Thereafter, fine aggregates are gradually incorporated into the geopolymer paste, and the mixture is continuously blended for an additional two minutes until a homogeneous geopolymer mortar is achieved. The total duration of the mixing process was precisely six minutes.



(a) (b) (c)

Fig. 5. Geopolymer mortar mixture: (a) – wet mixing procedure; (b) – flow of mix F2 COR; (c) – flow of mix F5 COR.

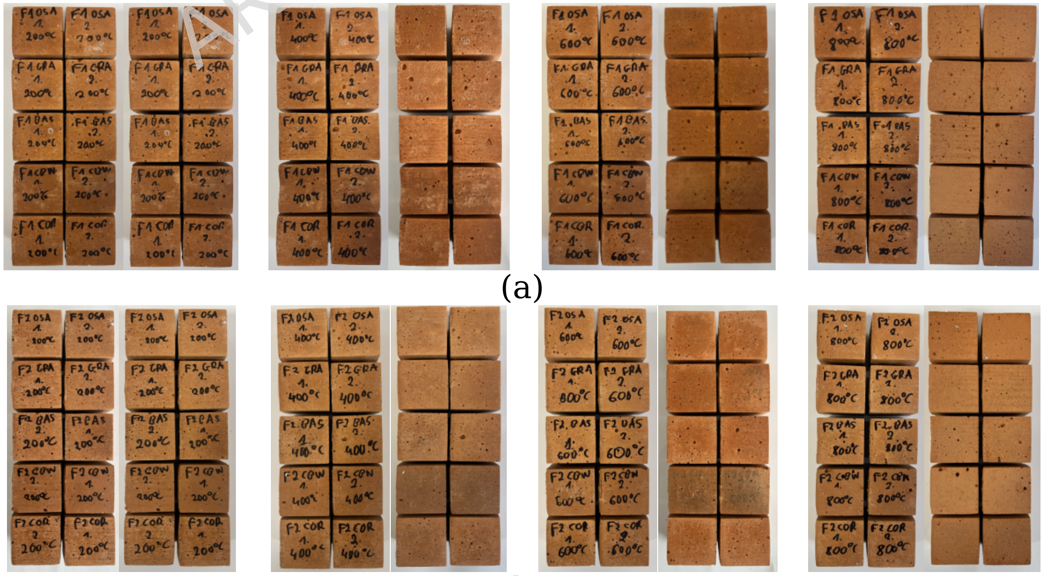
In regard to the mixture's workability, it was determined that the flow table test would be employed to evaluate the workability of all the designed geopolymer mortar compositions (see Fig. 5b & Fig. 5c). The experiment was carried out as follows: first, the standardized mold was filled with fresh geopolymer mortar; then, the cone was lifted and 15 jolts of the flow table were applied; finally, the resulting flow diameter was measured. The results of these tests are summarized in Table 3. The fundamental objective of the flow table method was to examine the impact of fine aggregate type on the fresh-state consistency of geopolymer mortars. Given the substantial number of designed geopolymer mortar compositions, the evaluation of flowability was restricted to mixtures containing the same precursor materials. Consequently, comparative analyses of flowability between geopolymers with disparate binding phases (i.e., varying precursor systems) were not conducted. For instance, the flowability of F1 mixtures was not compared with that of F2, F3, F4, or F5. This approach was adopted because an increase in metakaolin waste content requires a higher dosage of alkaline solution, resulting in an increased alkaline solution to precursor (AS/P) ratio and a concurrent increase in the water to solids (W/S) ratio. The rise in free water content that accompanies this process enhances flowability in mixtures with higher metakaolin waste precursor content. Consequently, direct comparisons between different precursor systems are rendered inappropriate. A tendency has been observed during the processing of the spread diameter results. Specifically, ceramic waste and basalt fine aggregate have been shown to have the lowest spread diameters. This observation has been made regardless of the selected precursor ratios (F1, F2, F3, F4, or F5). The coarser nature of ceramic waste and basalt fine aggregates, which contain the majority of their particle content on a 1mm sieve, is the underlying cause of the observed phenomenon. The absence of optimal particle distribution in these materials results in inter-particle friction and particle interlocking, consequently leading to mixes that are more rigid and less workable, accompanied by reduced flows. For the purpose of comparison, compositions containing ordinary river sand, pink granite, and corundum exhibited better particle distribution, which resulted in a significantly more consistent flowability. Furthermore, it should be noted that no additional water was utilized to enhance the workability of the fresh-state mixture, as all of the liquid phase is derived from alkaline solutions. Furthermore, it was observed that, as the number of jolts increased, some mixtures continued to flow.

The geopolymer mortar mixture was blended and cast into cubic silicone molds (40 mm × 40 mm × 40 mm) and compacted on a vibrating table to avoid the formation of unnecessary air bubbles. In order to mitigate the loss of moisture during the early stages of the curing process, polypropylene film was used to coat the molds. The geopolymer binder specimens were subsequently exposed to an environmental setting for a duration of 24 hours, characterized by an ambient temperature of 20 ± 2 ^{0}C and a relative humidity of 40%, allowing them to attain a state of repose. Subsequent to a 24-hour rest period, a furnace-curing regime was implemented, involving a temperature of 60 ^{0}C for a duration of 24 hours. The subsequent curing period was maintained under the aforementioned ambient temperature conditions until a duration of 28 days had elapsed. At that juncture, a compression strength test was conducted prior to and following exposure to elevated temperatures.

3. Results and Discussion

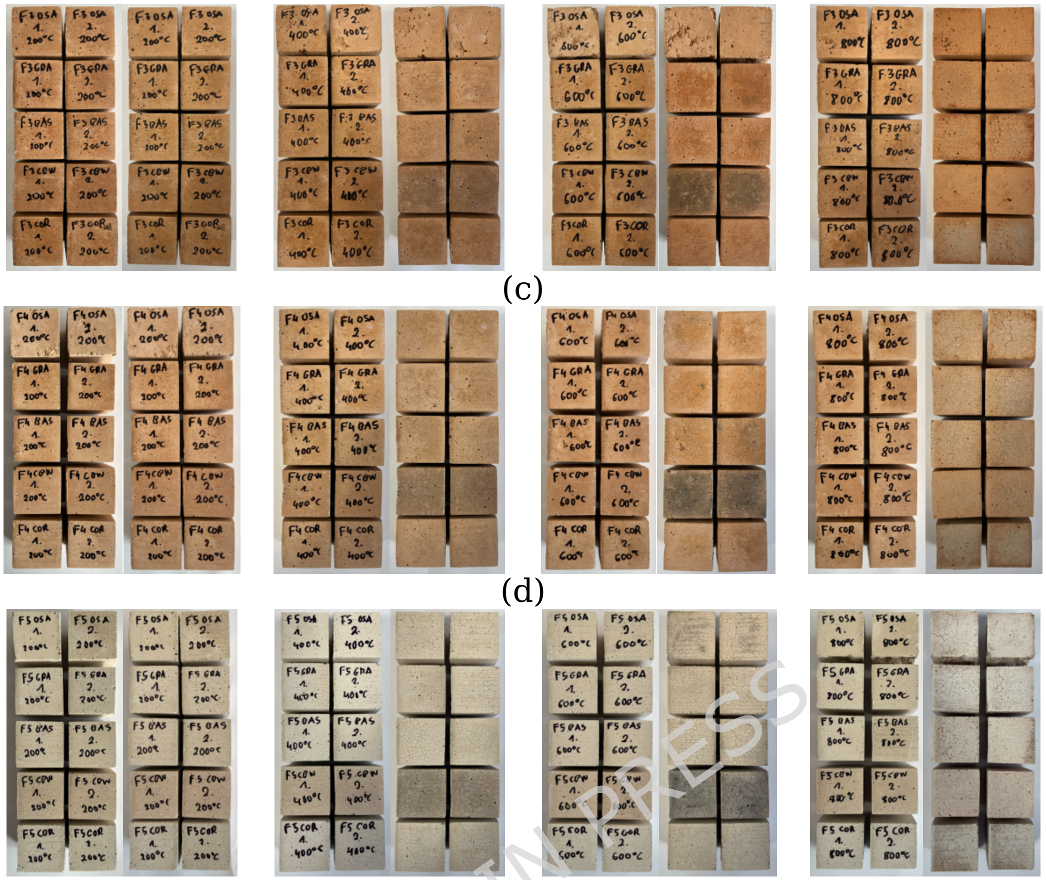
3.1. Visual Evaluation and Physical Properties Before and After Thermal Exposure

Subsequent to the thermal exposure at temperatures ranging from 200 to 800°C, a series of macroscopic evaluations were performed on the geopolymer mortar specimens to assess the occurrence of any visible alterations (see Fig. 6).



(a)

(b)



(c)

(d)

(e)

Fig. 6. Surface cracking of geopolymer mortars before and after high-temperature exposure at 200, 400, 600 and 800 ⁰C. (a) – F1 OSA, F1 GRA, F1 BAS, F1 CBW, F1 COR; (b) – F2 OSA, F2 GRA, F2 BAS, F2 CBW, F2 COR; (c) – F3 OSA, F3 GRA, F3 BAS, F3 CBW, F3 COR; (d) – F4 OSA, F4 GRA, F4 BAS, F4 CBW, F4 COR; (e) – F5 OSA, F5 GRA, F5 BAS, F5 CBW, F5 COR.

It has been observed that the pigmentation of geopolymer mortar undergoes a gradual fading process as the temperature increases. At temperatures of 800°C, the pale hue becomes predominant, particularly in compositions that contain higher proportions of ceramic brick waste precursor. The red tone exhibits a discernible loss at this temperature. The phenomenon of color degradation at elevated temperatures can be attributed to the oxidation of iron compounds at such high temperatures. The presence of iron is predominantly observed in compositions dominated by CBW precursors, exhibiting oxides with a percentage of Fe_2O_3 of 4.6%. This observation is consistent with the findings reported in the study by Nazari et al. [37]. Furthermore, it is evident that some compositions exhibit a transition in color from white/red to black (burnt-like) due to incomplete oxidation. In contrast, the specimens examined at 800°C exhibited no black (burnt-like) color, suggesting complete oxidation, phase transformation, and densification.

The severity of surface cracking was assessed qualitatively via a visual inspection method employing four descriptors (none, slight, moderate, severe). The results for the designed geopolymer mortar compositions and exposure temperatures are summarized in Table 4.

Table 4. Visual evaluation of surface cracking severity of geopolymer mortars exposed to high temperature environments.

Mix ID		Temperature			
Precursor content	Aggregate type	200 ^{0}C	400 ^{0}C	600 ^{0}C	800 ^{0}C
F1	OSA	none[1]	none	minor[2]	moderate[3]
	GRA	none	none	minor	moderate
	BAS	none	none	minor	moderate
	CBW	none	none	minor	none
	COR	none	none	none	none
F2	OSA	none	none	minor	minor
	GRA	none	none	minor	minor
	BAS	none	none	minor	moderate
	CBW	none	none	minor	none
	COR	none	none	none	none
F3	OSA	none	minor	moderate	moderate
	GRA	none	minor	moderate	moderate
	BAS	none	minor	moderate	severe[4]
	CBW	none	minor	minor	minor
	COR	none	none	minor	minor
F4	OSA	none	minor	moderate	severe
	GRA	none	minor	moderate	severe
	BAS	minor	moderate	severe	severe
	CBW	minor	moderate	severe	minor
	COR	none	minor	minor	minor
F5	OSA	minor	moderate	severe	severe
	GRA	minor	moderate	severe	severe
	BAS	moderate	severe	severe	severe
	CBW	moderate	severe	severe	severe
	COR	none	minor	moderate	severe

none[1] – no surface cracks; minor[2] – few fine surface cracks; moderate[3] – visible, partially connected surface cracks; severe[4] – dense, interconnected surface cracks.

Geopolymer mortar compositions with higher volumes of CBW precursor generally exhibited none to moderate cracking between 200 and 800°C. In contract, compositions containing a higher proportion of MKW precursor demonstrated moderate to severe cracking at the same high temperature conditions. This phenomenon is strongly associated with the excessive water utilized in the geopolymer system. Metakaolin waste precursor tends to necessitate a greater quantity of alkaline activator solution to achieve adequate geopolymerization reaction. However, as the alkaline solution to precursor (AS/P) ratio increases, an excess of water enters the system (higher W/S ratios). Consequently, at elevated temperatures, MKW-based geopolymers exhibit a propensity for more rapid evaporation and greater intensity of cracking, a phenomenon attributable to substantial mass loss. This mass loss exhibited a marked increase in proportion with rising temperature, suggesting enhanced moisture evaporation and thermal degradation of the geopolymer matrix. The corresponding mass loss and density variations are presented in Figure 7.

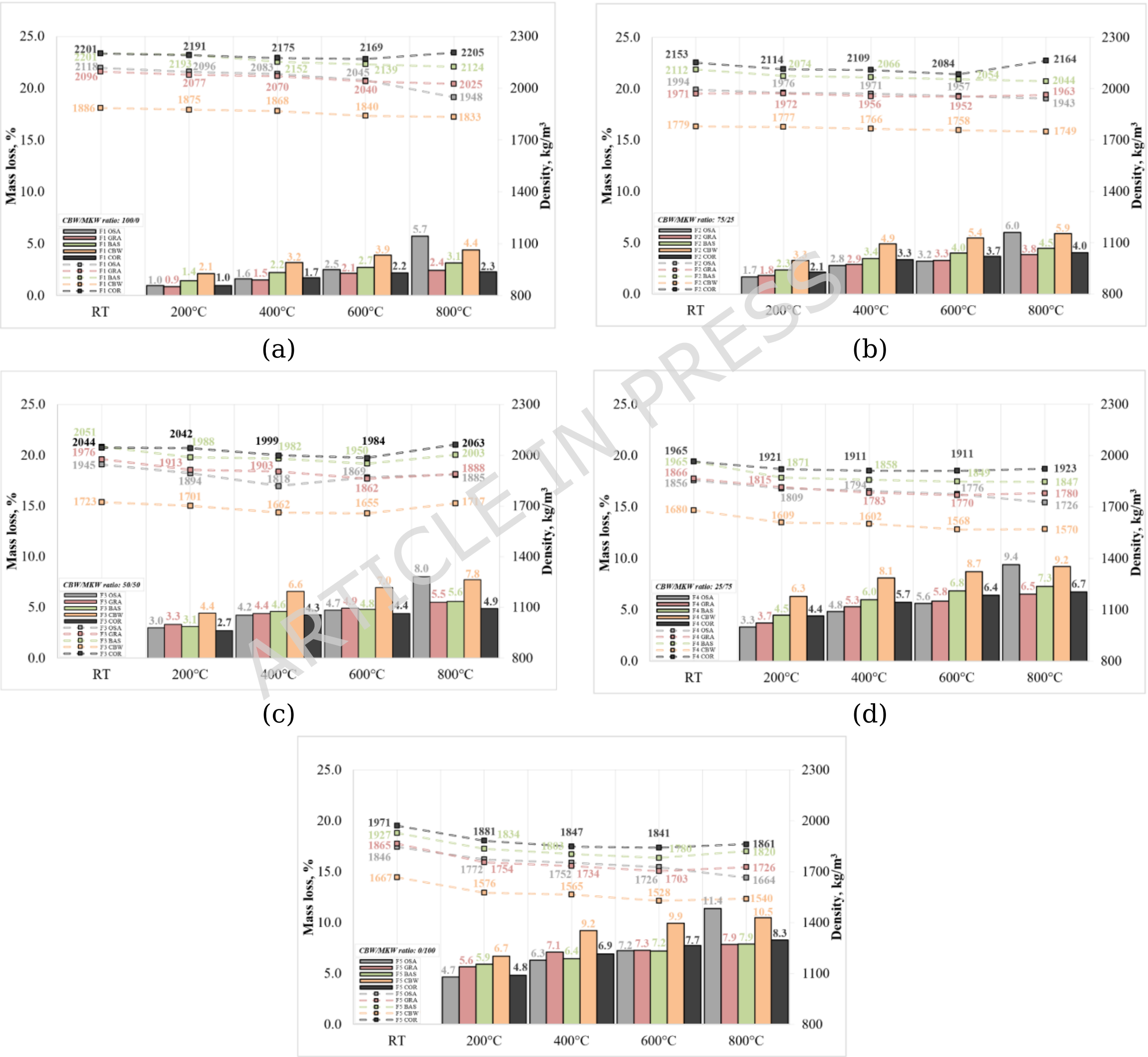
The occurrence of surface cracking was found to be contingent upon three constituent factors: precursor type, aggregate type, and high-temperature exposure. In regard to the impact of fine aggregate on surface cracking under high-temperature conditions, corundum and ceramic waste aggregates have been shown to yield the most favorable outcomes at temperatures up to 800°C. Regardless of the utilized binder phase (F1-F5), the presence of cracks is observed to be minimized. The phenomenon can be attributed to the high refractoriness of corundum aggregate, which has been observed to reach temperatures of up to 1580°C [38]. In comparison, ceramic waste fine aggregate has been shown to maintain stability at temperatures up to 1050°C [39], a capability that can be attributed to the sintering effect.

Mass loss and hardened density are strongly associated with the water-to-solids (W/S) ratio. Recent studies [40] have confirmed that the incorporation of an increasing quantity of ceramic brick waste precursors into the binder phase has been observed to result in a lower mass loss of geopolymer mortar and higher hardened geopolymer density at high temperatures compared to metakaolin waste precursors. CBW-based geopolymers necessitate a reduced quantity of free water for homogeneous mixing, consequently yielding lower W/S ratios. The elevated density of CBW particles contributes to the formation of denser hardened composites [41]. Consequently, even under high temperatures and with various fine aggregates, compositions with higher MKW content have been shown to exhibit a higher propensity for mass loss and a lower density.

However, in the context of examining the influence of fine aggregate on the mass loss and density of geopolymer composites at elevated temperatures, corundum fine aggregate demonstrated the lowest mass loss and highest density when subjected to exposures up to 800°C. The composition (F1 COR), which contains 100% ceramic brick waste precursor and corundum, exhibited optimal outcomes, with a minimal mass loss of 2.3% and a maximum density of 2205 kg/m^3 at an elevated temperature of 800°C. The composition with the same binder containing basalt (F1 BAS) and pink granite (F1 GRA) fine aggregates have a density of 2124 kg/m^3 and 2025 kg/m^3, respectively. These compositions exhibited a considerably higher mass loss of 3.1% and 2.4%, respectively. In contrast, the compositions containing ordinary river sand (F1 OSA) and ceramic waste (F1 CBW) fine aggregates tend to have the lowest densities of 1948 kg/m^3 and 1833 kg/m^3, respectively, along with the highest 5.7% and 4.4% mass losses.

It is evident that the designed compositions, irrespective of the binder type utilized, in terms of hardened geopolymer mass loss and density, are predominantly influenced by the origin of fine aggregate and their initial parameters, such as particle density, bulk density, and water absorption. In terms of particles and bulk densities, ordinary river sand, basalt, and granite exhibit comparable values; however, ordinary river sand demonstrates significantly higher water absorption. Consequently, geopolymers incorporating this fine aggregate are prone to greater mass loss under elevated temperature exposure. Ceramic waste aggregate, on the other hand, exhibits the lowest particle and bulk densities due to its non-natural origin and prior thermal treatment, which results in high porosity. This increased porosity corresponds to elevated water absorption, resulting in geopolymer composites with reduced density and high mass loss at elevated temperatures when ceramic waste fine aggregate is used. In contrast, corundum fine aggregate possesses the highest particle and bulk densities and minimal water absorption, promoting geopolymers with increased hardened density and reduced mass loss associated with free water evaporation at high temperatures.

In summary, the compositions that exhibited the highest hardened geopolymer densities, lowest mass loss, and least surface cracking at 800°C elevated temperature were those containing higher concentrations of CBW precursor and corundum fine aggregate (F1 COR, F2 COR, F3 COR).



(a) (b)

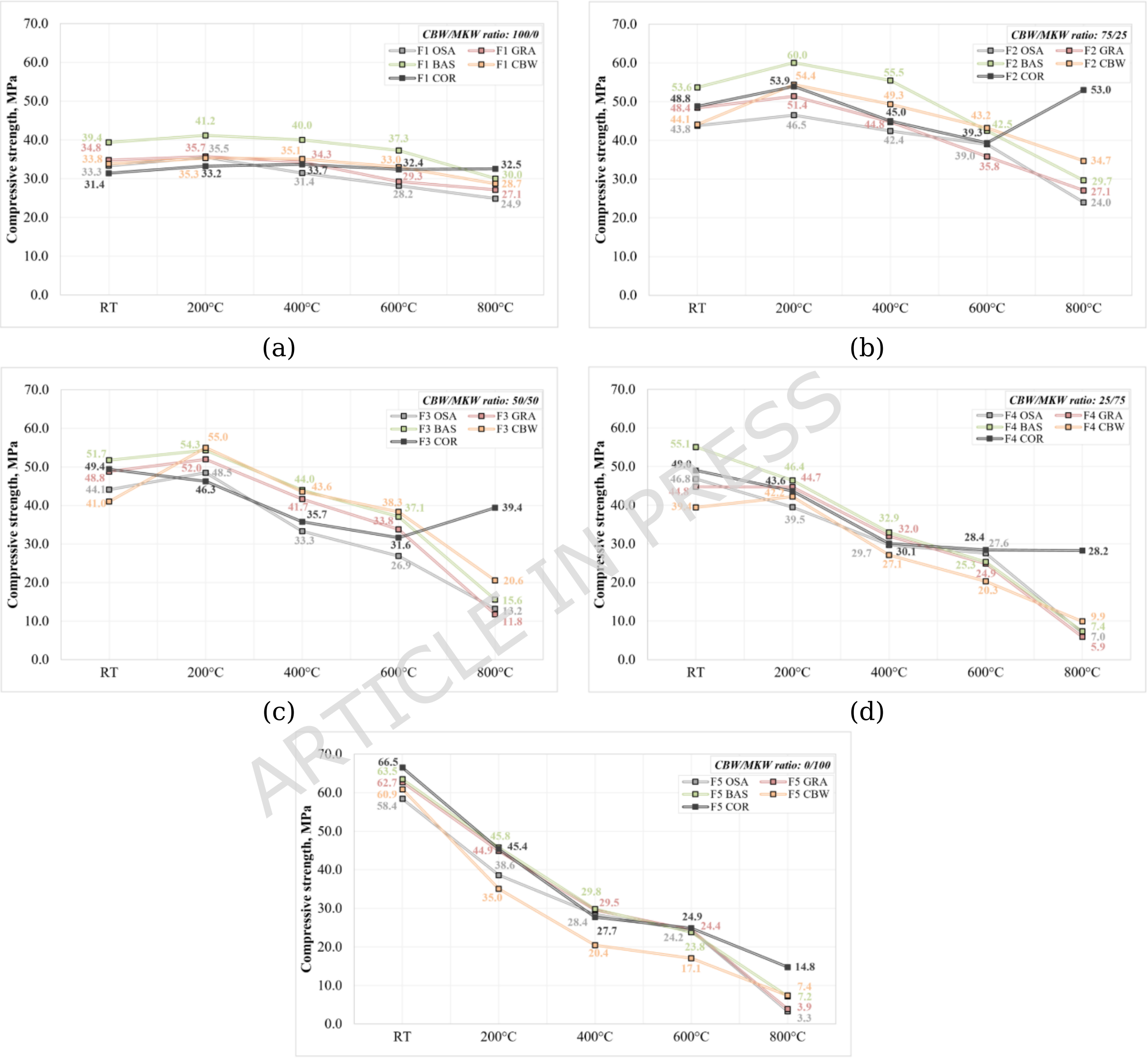
(c) (d)

(e)

Fig. 7. A relationship between geopolymer mortar density and mass loss before and after high-temperature exposures (200, 400, 600 and 800 ⁰C) for different CBW/MKW ratios: (a) – 100/0; (b) – 75/25; (c) – 50/50; (d) – 25/75; (e) – 0/100.

3.2. Compressive Strength Before and After Thermal Exposure

Compressive strength and residual strength are critical parameters for geopolymer mortar, as they represent its load-bearing capacity under ambient conditions and its ability to maintain mechanical performance after exposure to elevated temperatures. The initial compressive strength after 28 days at room temperature (RT) and the post-heating (residual) compressive strength after 200, 400, 600, and 800°C elevated temperature exposures are illustrated in a line chart (see Fig. 8).



(a) (b)

(c) (d)

(e)

Fig. 8. Geopolymer mortar compressive strength before and after high-temperature exposures (200, 400, 600 and 800 ⁰C) for different CBW/MKW ratios: (a) – 100/0; (b) – 75/25; (c) – 50/50; (d) – 25/75; (e) – 0/100.

Compressive strength and residual strength are critical parameters for geopolymer mortar, as they represent its load-bearing capacity under ambient conditions and its ability to maintain mechanical performance after exposure to elevated temperatures. The initial compressive strength after 28 days at room temperature (RT) and the post-heating (residual) compressive

strength after 200, 400, 600, and 800°C elevated temperature exposures are illustrated in a line chart (see Fig. 8).

Independent of fine aggregate type, the 28-day compressive strength at ambient temperature shows a clear increasing trend with higher metakaolin waste content in the binder. Compositions dominated by metakaolin waste have been shown to exhibit superior mechanical performance. In particular, the mixture containing 100% metakaolin waste binder (F5), irrespective of the fine aggregate type, has been demonstrated to achieve the highest compressive strength values, ranging from 58.4 to 66.5 MPa. This trend is consistent with our previous study [42] and is attributed to the higher reactivity of metakaolin waste compared to ceramic brick waste, arising from its higher Al_2O_3 content and greater amorphousness. These characteristics promote a more efficient geopolymerization process, resulting in the formation of a dense geopolymer matrix enriched with N-A-S-H gel and goosecreekite phases.

The 28-day compressive strength results demonstrate that basalt and corundum fine aggregate mixtures generally exhibited higher strength than mixtures containing ordinary river sand, granite, or ceramic waste, irrespective of binder composition. The highest compressive strengths for varying binder types were as follows: F1-BAS (39.4 MPa), F2-BAS (53.6 MPa), F3-BAS (51.7 MPa), F4-BAS (55.1 MPa), and F5-COR (66.5 MPa). Although the disparities among the fine aggregates were relatively negligible, basalt and corundum consistently exhibited marginally enhanced mechanical performance under ambient conditions. This enhancement can be attributed to the high strength and stiffness of basalt and corundum, in conjunction with their dense, low-porosity microstructure and favorable particle morphology [43, 44]. These characteristics enhance stress transfer and mechanical interlocking with the geopolymer binder. Their chemical stability in alkaline environments further stimulates the development of a dense and well-bonded interfacial transition zone, thereby contributing to the observed increase in compressive strength at 28 days.

A clear trend is observed regarding the performance of the different binder types (F1–F5) under elevated temperatures: geopolymer mortars with increasing CBW precursor content exhibit significantly higher thermal stability at 200, 400, 600, and 800 °C compared to MKW-based mortars. This tendency is especially noticeable at 800 °C, where the most robust MKW-based formulations (F5) only maintain a small portion of their initial compressive strength. Specifically, F5-COR retains 22.3% of its original strength, F5-BAS retains 11.3%, F5-GRA retains 6.2%, F5-CBW retains 12.2%, and F5-OSA retains 5.6%. The significant decline in compressive strength observed in MKW-based geopolymers at elevated temperatures is predominantly ascribed to their unfavorable thermal response, which is intimately linked to their high water-to-solid (W/S) ratio and the presence of excess physically bound water. The process of heating results in the rapid evaporation of both free water and water that is weakly bound to the substance. This leads to an increase in the internal vapor pressure. The increase in vapor pressure causes microcracking and pore coarsening to occur within the geopolymer matrix. Additionally, the ensuing thermal shrinkage and the mismatch between the binder and aggregate phases contribute to accelerated crack propagation. The combined effects of these factors result in a significant disruption of the integrity of the aluminosilicate network, leading to a substantial reduction in load-bearing capacity at elevated temperatures. In contrast to the general degradation in strength observed at extremely high temperatures of 800°C, there was an increase in compressive strength recorded at 200°C for compositions with higher contents of CBW precursor (F1, F2, and F3). This behaviour is consistent with the post-curing effects induced by thermal processes in the field of geopolymer composites involves the enhancement of compressive strength within a moderate temperature range, typically spanning 200–400°C. This phenomenon is commonly attributed to the intensification of geopolymerization processes. In this particular regime, an increase in ionic mobility, in conjunction with the ongoing condensation of aluminosilicate species, has been demonstrated to promote further gel formation, matrix rearrangement, and overall densification, thereby enhancing the material's mechanical performance [45]. Similar findings have been reported by Zhang et al. [46] where a low-calcium fly ash-based geopolymer exhibited an increase in compressive strength to 167% of its original value after heating to 200 °C, which was explained by continued geopolymer reaction and progressive densification of the binder structure under moderate thermal exposure.

In terms of the type of fine aggregate, exposure to high temperatures had the least harmful effect on geopolymer mortar containing corundum (COR) and ceramic waste (CBW) fine aggregates. Conversely, geopolymer composites with other fine aggregates exhibited the lowest residual strength at all tested temperatures. Among these aggregates, basalt performed comparatively better, while pink granite and ordinary river sand performed worse under elevated temperature exposure. Among the various geopolymer binder compositions that were examined, the mixture containing 75% CBW and 25% MKW exhibited the highest overall high-temperature resistance, as demonstrated by its superior residual strength across all fine aggregate types. Consequently, the ensuing sections will direct their focus toward an examination of the performance of this specific binder composition in conjunction with the various fine aggregates. The residual strength results at 800°C provide a clear distinction in performance between fine aggregates. Hardened geopolymer mortars, incorporating OSA, GRA, and BAS, demonstrated comparable residual strengths, retaining around 55% of their initial 28-day strength. Conversely, CBW and COR exhibited considerably higher residual strengths (78.7% and 108.6%, respectively), signifying their superior resilience to high-temperature exposure.

The residual compressive strength of geopolymer mortars following high-temperature exposure is found to be significantly influenced by the physicochemical nature of the fine aggregate. Ordinary river sand (OSA) fine aggregate demonstrated substandard performance at elevated temperatures, attributable to its inert, quartz-rich composition and the thermal expansion of quartz near 573 °C, which leads to thermal incompatibility with the geopolymer matrix, a property that facilitates microcracking [20, 47]. The diminished residual strength of pink granite (GRA) geopolymer mortars is ascribed to mineralogical heterogeneity and thermal expansion mismatch. These factors result in increased internal stresses at temperatures in excess of 600°C [48]. Basalt (BAS) fine aggregate demonstrated moderate performance, attributable to its relatively high thermal stability [49]. However, its chemical inertness and coarser particle size impose limitations on interfacial bonding with the geopolymer matrix. It was demonstrated that ceramic waste (CBW) fine aggregates exhibited a notable retention of residual strength at elevated temperatures. The observed behavior can be ascribed to their partial reactivity in alkaline environments, which fosters geopolymerization and matrix densification. Additionally, their inherent thermal stability as a pre-calcined material contributes to their properties. However, their comparatively coarser particle size may result in a slight reduction in interfacial contact. Corundum fine aggregate exhibited the highest residual strength, a consequence of its exceptional thermal stability, elevated melting point, and minimal thermal expansion. These properties contribute to the mitigation of microstructural damage and the preservation of mechanical integrity under conditions of extreme temperature exposure [26].

A comparison of the data presented in Figures 7 and 8 reveals a clear correlation between density, mass loss, and compressive strength. The findings indicate that the density evolution in response to temperature is significantly influenced by both the CBW/MKW ratio of the binder and the type of fine aggregate employed. In particular, an increase in metakaolin waste (MKW) content in the binder has been shown to lead to a systematic increase in mass loss and a corresponding reduction in density at elevated temperatures, regardless of the aggregate used. This behaviour is attributed to the higher alkaline solution demand of MKW-based systems, resulting in increased water-to-solids (W/S) ratios. Upon heating, the evaporation of excess free and physically bound water promotes pore formation and microcracking, ultimately leading to a reduction in density and structural integrity. This discrepancy is evident when representative compositions are juxtaposed. Mixtures incorporating corundum fine aggregate and a CBW-dominated binder (F2 COR) maintain high density even at elevated temperatures, with a density of approximately 2164 kg/m³ being attained at 800 °C and a minimal mass loss of 4.0% being recorded. In contrast, mixtures that contain 100% MKW binder and any type of fine aggregate (F5) exhibit significant degradation at 800 °C, with density values typically decreasing from 1667 – 1971 kg/m^3 to 1540 – 1861 kg/m^3, accompanied by elevated mass loss ranging from 7.9 to 11.4% and significantly reduced compressive strength, often falling below 5 – 15 MPa depending on the aggregate type. Despite variations associated with aggregate type, mixtures that maintain higher density after thermal exposure consistently exhibit superior compressive strength. Conversely, those with increased mass loss and reduced density show more severe mechanical degradation. These observations suggest that thermally induced mass loss and the associated increase in porosity are key factors in the deterioration of compressive strength in geopolymer mortars. Despite the variability of these effects in relation to binder composition and aggregate characteristics, a consistent correlation between density, mass loss, and mechanical performance is observed across all investigated systems. Similar relationships between density, weight loss, and strength degradation in fly ash based geopolymers have been observed in the studies by Kaya [50–52].

In summary, corundum (COR) fine aggregate demonstrated the highest residual strength, density and lowest mass loss when utilized with a ceramic brick waste-based geopolymer binder, underscoring its exceptional thermal stability and matrix compatibility. The performance of ceramic waste (CBW) fine aggregate was satisfactory, attributable to its partial reactivity and inherent thermal stability. Subsequent studies should examine the impact of varying fine aggregate content, given the potential to optimize the fine aggregate-to-precursor (FA/P) ratio, with the objective of enhancing workability, fresh mixture rheology, and overall mechanical performance. Additionally, research should assess the high-temperature resistance of the mixture over multiple heating cycles.

3.3. Two-Factor ANOVA Evaluation of Compressive Strength

To assess the statistical significance of the factors influencing compressive strength, a two-factor ANOVA (analysis of variance) was employed. The present method is employed to assess the impact of two independent variables, specifically binder type and exposure temperature, on the dependent variable, which is the compressive strength of the specimens. The analysis was performed separately for each type of fine aggregate, including ordinary river sand (OSA), pink granite (GRA), basalt (BAS), ceramic brick waste (CBW), and corundum (COR). As illustrated in Tables 4, 5, 6, 7, and 8, the results of the one-way analysis of variance (ANOVA) are summarized for specimens prepared with five types of aggregates. The dataset encompasses five binder types (F1–F5) and five exposure temperatures (RT, 200°C, 400°C, 600°C, and 800°C), with each temperature evaluated in five replicates.

The statistical significance of the effects of ordinary river sand (OSA) find aggregate and exposure temperature on the compressive strength of geopolymer mortars was subsequently evaluated, as illustrated in Table 5. Binder type groups demonstrate an average compressive strength ranging from 30.1 MPa (in binder F4) to 39.1 MPa (in binder F2), with variances increasing for certain binders. These variances indicate a sensitivity to thermal exposure. As the temperature increases, there is a substantial decline in compressive strength among temperature-exposed groups. At room temperature (RT), the average compressive strength of 45.3 MPa diminishes to 14.5 MPa at 800°C, thereby substantiating thermal degradation.

Table 5. Two-factor ANOVA results for the effects of binder type and exposure temperature on compressive strength of geopolymer mortars with ordinary river sand (OSA) fine aggregate.

Source of variation	Sum of squares (SS)	Degree of freedom (df)	Mean square (MS)	F-statistic (F)	Probability (P-value)	Critical F (Fcrit)

Binder type	285.36	4	71.34	1.44	0.267	3.01
Exposure temperature	2927.10	4	731.77	14.76	3.11×10^{-5}	3.01
Error	793.17	16	49.57	-	-	-
Total	4005.62	24	-	-	-	-

The outcomes of the two-factor (ANOVA) analysis indicate that the type of binder does not significantly impact the compressive strength of ordinary river sand (OSA) fine aggregate. The calculated F-statistic value of 1.44 is found to be less than the F-critical value of 3.01. The binder type exhibits no substantial impact under these conditions, indicating that thermal resistance is predominantly influenced by temperature exposure rather than binder composition for ordinary river sand (OSA) fine aggregate. Concurrently, exposure temperature exerts a profoundly significant influence on compressive strength. The calculated F-statistic value of 14.76 is more than four times higher than the F-critical value of 3.01. The analysis indicates that exposure temperature is the predominant factor influencing compressive strength, with strength losses accelerating beyond 400°C. The study by Wang et al. [53] supports the hypothesis that for ordinary river sand fine aggregates, accelerated strength loss beyond ~400-600°C is linked to the onset of the α–β quartz phase transformation. This transformation promotes internal cracking and decreases compressive capacity.

The findings of the two-factor (ANOVA) analysis for pink granite (GRA) fine aggregate indicate that exposure temperature exerts a significantly substantial effect on compressive strength, as evidenced by the calculation result ($F = 14.76$, $p \approx 3.11 \times 10^{-5} < 0.05$), as shown in Table 6. This outcome serves as confirmation that thermal exposure is the predominant factor influencing strength reduction in these systems. In contrast, the effect of binder type was not found to be statistically significant ($F = 1.69$, $p = 0.20 > 0.05$), indicating that variations among binders did not result in significant changes to compressive strength when evaluated across all temperature ranges.

Table 6. Two-factor ANOVA results for the effects of binder type and exposure temperature on compressive strength of geopolymer mortars with pink granite (GRA) fine aggregate.

Source of variation	Sum of squares (SS)	Degree of freedom (df)	Mean square (MS)	F-statistic (F)	Probability (P-value)	Critical F (Fcrit)
Binder type	404.41	4	101.10	1.69	0.20	3.01
Exposure temperature	3532.84	4	883.21	14.76	3.11×10^{-5}	3.01
Error	957.53	16	59.85	-	-	-
Total	4894.79	24	-	-	-	-

Conversely, average compressive strength undergoes a progressive decrease with increasing temperature, ranging from 47.9 MPa at room temperature to 45.7 MPa at 200°C, 36.4 MPa at 400°C, 29.6 MPa at 600°C, and 15.2 MPa at 800°C. The precipitous decline observed beyond 400°C can be attributed to the dehydration of hydration products and thermal cracking. As the temperature rises, a phenomenon referred to as "variance among binders" becomes more pronounced (e.g., F5 variance = 490.6). This observation suggests that thermal stress has the capacity to amplify performance discrepancies within specific binder–fine aggregate combinations.

From a practical standpoint, the pink granite (GRA) fine aggregate exhibit moderate thermal stability up to 400°C, yet undergo substantial strength reduction at 600–800°C. It is imperative to recognize that binder optimization, in isolation, will not suffice to counterbalance this decline. Consequently, the implementation of aggregate selection and thermal protection strategies is paramount for maintaining structural integrity under conditions of elevated temperature.

The findings of the two-factor ANOVA for basalt fine aggregate reveal that exposure temperature exerts a significant influence on compressive strength ($F = 15.64$, $p = 2.18 \times 10^{-5} < 0.05$). This outcome confirms thermal exposure as the predominant factor governing strength reduction, as illustrated in Table 7. In contrast, the binder type exhibited a borderline but non-significant effect ($F = 2.94$, $p \approx 0.053 > 0.05$). While the F-statistic approaches a significant threshold (Fcrit = 3.01), the $\alpha = 0.05$ criterion remains unmet. This suggests that binder variations may have a comparatively minor effect in relation to that of temperature.

Table 7. Two-factor ANOVA results for the effects of binder type and exposure temperature on compressive strength of geopolymer mortars with basalt (BAS) fine aggregate.

Source of variation	Sum of squares (SS)	Degree of freedom (df)	Mean square (MS)	F-statistic (F)	Probability (P-value)	Critical F (Fcrit)
Binder type	729.88	4	182.47	2.94	0.053	3.01
Exposure temperature	3880.34	4	970.09	15.64	2.18×10^{-5}	3.01
Error	992.45	16	62.03	-	-	-
Total	5602.68	24	-	-	-	-

Conversely, average compressive strength experiences a marked decrease with increasing temperature: from 52.7 MPa at room temperature, it declines to 49.6 MPa at 200°C, 40.5 MPa at 400 °C, 33.2 MPa at 600 °C, and 18.0 MPa at 800 °C. The pronounced reduction beyond 400°C is indicative of dehydration of hydration products and thermal cracking, a phenomenon consistent with other fine aggregate systems. A notable increase in variance among binders was observed at elevated temperatures (e.g., F5 variance = 462.4), suggesting that thermal stress significantly amplifies performance variations within binder-fine aggregate combinations.

From an engineering perspective, basalt fine aggregates demonstrate relatively good thermal stability up to 400°C and moderate performance at 600°C; however, strength loss at 800°C exceeds 65%, severely compromising load-bearing capacity. Preceding investigations have substantiated the thermal stability of basalt fine aggregates. Wang et al. [54] reported that triaxial compression tests on basalt rock demonstrated that the material's mechanical strength remained relatively stable up to 600°C. However, they also noted that after this temperature threshold was reached, there was a significant acceleration in the structural deterioration of the rock. This study indicates that, in the absence of appropriate fine aggregate selection and thermal protection strategies, binder optimization alone is insufficient to counteract this trend and maintain structural integrity in high-temperature conditions.

The findings of the two-factor ANOVA analysis on geopolymer mortars with ceramic waste (CBW) fine aggregate indicate that both exposure temperature and binder type have statistically significant effects on compressive strength, as outlined in Table 8. The exposure temperature exhibited a significant influence ($F = 6.27$, $p \approx 0.003 < 0.05$), thereby substantiating the notion that thermal exposure constitutes a primary factor in the deterioration of strength. In contrast to the findings observed in other aggregates, the impact of binder type is also noteworthy ($F = 3.51$, $p \approx 0.031 < 0.05$), indicating that binder composition plays a pivotal role in performance when CBW is utilized as a fine aggregate.

Table 8. Two-factor ANOVA results for the effects of binder type and exposure temperature on compressive strength of geopolymer mortars with ceramic waste (CBW) fine aggregate.

Source of variation	Sum of squares (SS)	Degree of freedom (df)	Mean square (MS)	F-statistic (F)	Probability (P-value)	Critical F (Fcrit)
Binder type	1134.04	4	283.51	3.51	0.031	3.01
Exposure temperature	2025.77	4	506.44	6.27	0.003	3.01
Error	1293.23	16	80.83	-	-	-
Total	4453.04	24	-	-	-	-

In addition, average compressive strength decreases progressively with temperature, from 43.8 MPa at room temperature to 44.4 MPa at 200°C, 35.1 MPa at 400°C, 30.4 MPa at 600°C, and 20.3 MPa at 800°C. The precipitous decline observed beyond 400°C can be attributed to the dehydration of hydration products and the onset of thermal cracking. It has been demonstrated that variance among binders increases significantly at higher temperatures (e.g., F5 variance = 433.5), indicating that thermal stress amplifies differences between binder–aggregate combinations.

In practice, ceramic waste (CBW) fine aggregates exhibit adequate thermal stability up to 400°C and reasonable performance at 600°C; however, substantial strength loss occurs at 800°C. Given the substantial impact of binder type on performance, meticulous optimization of binder composition is crucial when employing ceramic waste fine aggregates in high-temperature applications. This finding underscores the promise of CBW fine aggregates for sustainable construction, contingent upon the strategic selection of binders tailored to thermal exposure conditions.

The two-factor ANOVA indicates that exposure temperature exerts a statistically significant effect on compressive strength for systems containing corundum fine aggregate ($F = 3.93$, $p \approx 0.021 < 0.05$). However, the binder effect was found to be non-significant ($F = 2.35$, $p = 0.098 > 0.05$), as demonstrated in Table 9.

Table 9. Two-factor ANOVA results for the effects of binder type and exposure temperature on compressive strength of geopolymer mortars with corundum (COR) fine aggregate.

Source of variation	Sum of squares (SS)	Degree of freedom (df)	Mean square (MS)	F-statistic (F)	Probability (P-value)	Critical F (Fcrit)
Binder type	712.47	4	178.12	2.35	0.098	3.01
Exposure temperature	1193.33	4	298.33	3.93	0.021	3.01
Error	1213.47	16	75.84	-	-	-
Total	3119.28	24	-	-	-	-

The average compressive strength of the binder types exhibited a temperature-dependent variation, with a mean strength of 49.0 MPa at room temperature, decreasing to 44.5 MPa at 200°C, 34.4 MPa at 400°C, 31.3 MPa at 600°C, and 33.6 MPa at 800°C. It is noteworthy that, in contrast to other aggregates, the mean increases from 600°C to 800°C, a phenomenon attributable to elevated retention in specific binder–corundum fine aggregate pairs. It is noteworthy that composition F2 COR attains 53.0 MPa at 800°C (retention 108.6% vs. RT), while F1 COR achieves 32.5 MPa at 800°C (retention 103.5%).

However, the dispersion across binders at 800°C is substantial (14.8–53.0 MPa), suggesting that while the binder main effect is not statistically significant overall, binder selection still influences practical outcomes in this fine aggregate system. Given the absence of replication in the design (interaction absorbed in error), these results underscore the remarkable high-temperature resilience of corundum fine aggregate. Specifically, the F1 COR and F2 COR geopolymer mortar compositions demonstrate notable promise for service above 600°C, while the F5 COR exhibits substantial degradation at 800°C. A comparable high-temperature effect on geopolymer incorporating corundum aggregates was reported by Li et al. [26], where they underscore the material's excellent thermal compatibility and exceptional high-temperature performance above 600°C.

The two-factor ANOVA analysis showed which factors have the greatest influence on the changes in the compressive strength of geopolymer mortars at high temperatures. The most important factor is temperature – it significantly reduces compressive strength in all fine-aggregate systems ($p < 0.05$). This means that strength loss is mainly caused by thermal degradation processes, such as dehydration and microcrack formation, rather than by the binder composition or the type of fine aggregate.

The influence of the binder type was more complex and depended strongly on the fine aggregate used. In the OSA and GRA systems, differences between binders did not have a significant effect, indicating that their high-temperature behavior is mainly controlled by the properties of the aggregate itself. The BAS system showed only a weak, borderline binder effect ($p \approx 0.053$), suggesting that binder chemistry may play a role, but not a decisive one. In contrast, the CBW system showed a clearly significant binder effect ($p = 0.031$). This means that when ceramic waste is used as fine aggregate, binder selection becomes particularly important, as CBW is more sensitive to the composition of the geopolymer matrix – likely due to its heterogeneous mineralogy and variable thermal expansion.

The corundum (COR) fine aggregate performed exceptionally well. Mortars made with COR maintained very high strength even at 800 °C, and some binder and COR combinations, such as F2 COR, even exceeded their original strength, retaining more than 100%. This indicates that COR is not only highly resistant to elevated temperatures but may also promote microstructural densification or sintering effects.

Across all systems, compressive strength decreased as temperature increased, with the most pronounced drop occurring above 400 °C. This aligns with known mechanisms such as dehydration of geopolymer gels, formation of microcracks due to thermal incompatibility, and structural changes in the aluminosilicate network. The ANOVA results also show that the influence of the binder becomes more important at higher temperatures, suggesting that thermal stress amplifies the role of binder chemistry in maintaining structural stability.

From a practical perspective, the study clearly identifies which aggregates are most suitable for high-temperature applications. COR is the most resistant, maintaining performance up to 800 °C. BAS and GRA show moderate resistance and are suitable up to around 400 °C. CBW has promising potential, but it requires careful binder selection to reduce its sensitivity to matrix composition. Overall, the results highlight that ensuring the high-temperature reliability of geopolymer mortars depends on achieving good compatibility between the aggregate's mineralogy and the binder's properties.

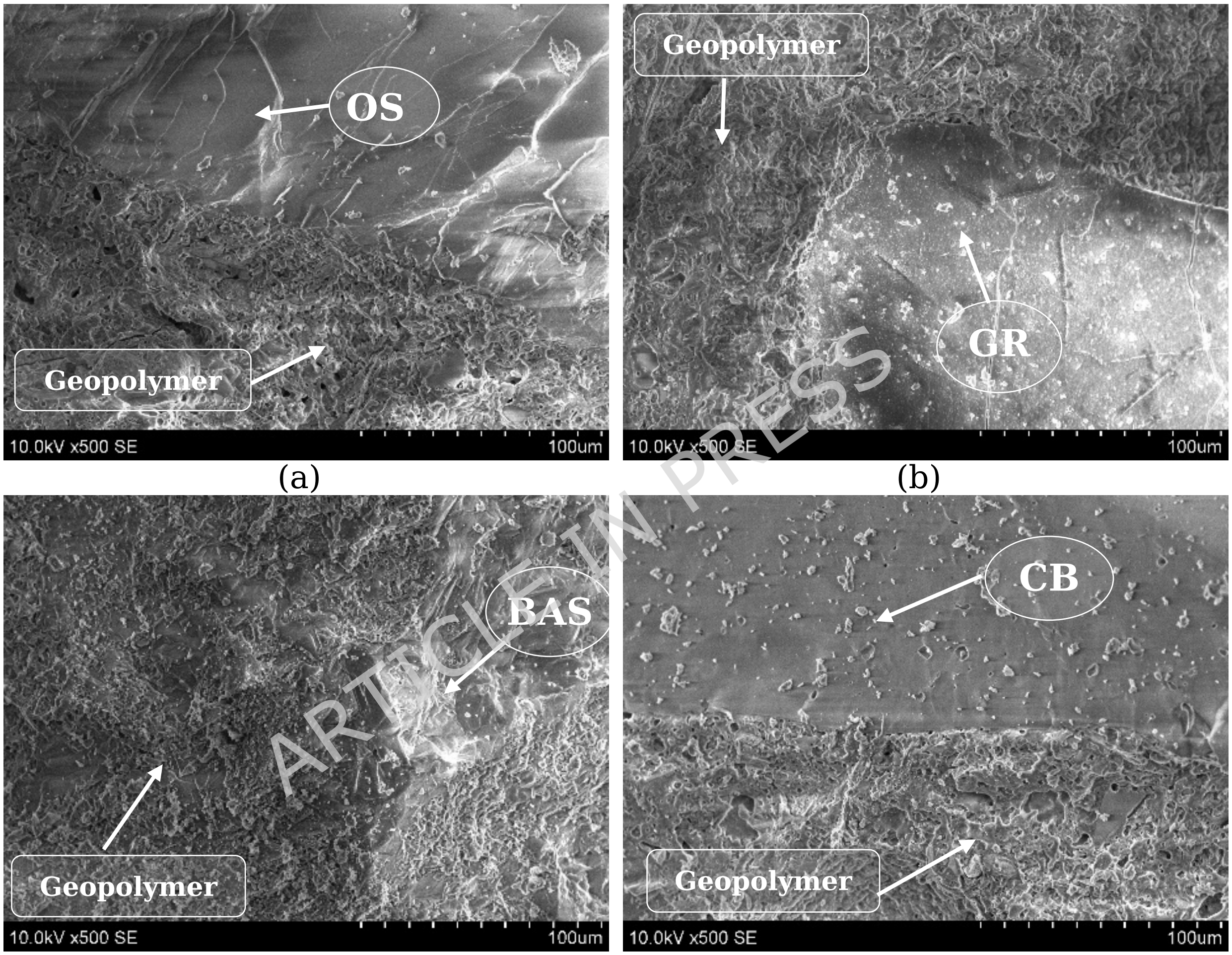
3.4. SEM analysis before high-temperature exposure

The microstructural analysis based on scanning electron microscopy (SEM) was used to evaluate the geopolymer mortar (F2 binder composition) incorporating five different fine aggregates – ordinary river sand (OSA), granite (GRA), basalt (BAS), ceramic waste (CBW), and corundum (COR). The microstructural images at 100 µm magnification are shown in Figure 9. The F2 binder composition was selected because it exhibited the highest mechanical performance according to our previous study [36]. This analysis enabled evaluation of the interaction between the fine aggregates and the geopolymer matrix, as well as identification of key microstructural features that may influence the mortar's resistance to high-temperature exposure.

For the ordinary river sand (OSA) fine aggregate (Fig. 9a), a heterogeneous matrix with pronounced micro-porosity and limited aggregate–matrix bonding is observed. The sand particles have smooth and chemically inert surfaces, which leads to the formation of weak interfacial zones. The presence of initial microcracks indicates that this aggregate may be sensitive to thermal deformation, explaining the lower residual strength after high-temperature exposure.

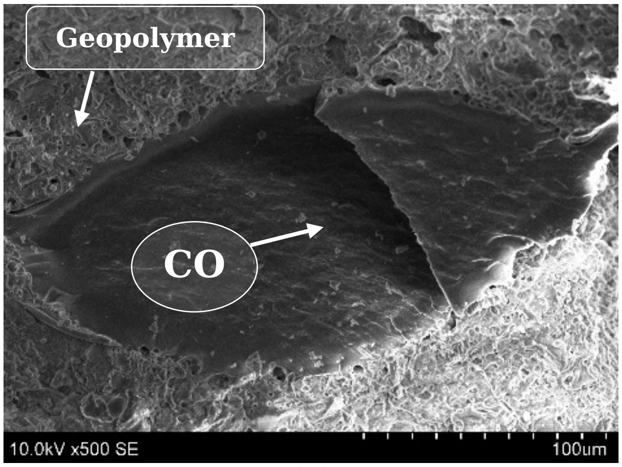
The pink granite (GRA) fine aggregate (Fig. 9b) exhibits a rough, angular surface that provides better mechanical interlocking with the matrix. However, microcrack clusters are visible around quartz-containing grains, arising from the heterogeneous mineral composition of granite. These defects may develop into propagating cracks under thermal shock, meaning that despite good initial bonding, granite is not an optimal aggregate for high-temperature applications.

The basalt (BAS) fine aggregate (Fig. 9c) forms a homogeneous microstructure with strong mechanical bonding. The matrix surrounding the basalt grains is dense, and porosity is limited. This indicates that basalt integrates effectively into the geopolymer system. Nevertheless, due to the presence of iron and magnesium silicates in basalt, phase transformations may occur at elevated temperatures, which later reduce thermal stability, even though the initial microstructure is favorable.



(a) (b)

(c) (d)



(e)

Fig. 9. The microstructure of geopolymer mortar made of F2 binder before high-temperature exposure: (a) – OSA; (b) – GRA; (c) – BAS; (d) – CBW; (e) – COR.

The ceramic waste (CBW) fine aggregate (Fig. 9d) exhibits a porous and reactive surface, which forms an exceptionally strong interface with the geopolymer matrix. The matrix surrounding the CBW particles is dense and free of pronounced interfacial cracks, while the surface texture enables additional silicate reactions to occur. This results in a stable, chemically compatible composite that shows no structural defects prior to heating. Such microstructural synergy explains the high residual strength observed after exposure to 800 °C.

The corundum (COR) fine aggregate (Fig. 9e) forms the densest and most stable microstructure. COR particles are extremely hard, thermally stable, and nearly non-porous, which ensures that the interfacial zone remains continuous and free of microcracks. The matrix around the corundum grains is homogeneous, and the bonding is stable. Because corundum has a very low thermal expansion coefficient, it does not induce stresses within the matrix, making it an optimal aggregate for high-temperature applications. This is supported by experimental results showing that COR-containing mortars retain or even exceed their initial strength after exposure to 800 °C.

The microstructure of geopolymer mortars, as well as their resistance to high temperatures, is closely related to the mineralogical composition, surface morphology, and thermal behavior of the fine aggregates used. Numerous studies show that the type of aggregate determines the quality of the interfacial transition zone (ITZ), the formation of microcracks, and the final strength of the composite, both before and after thermal exposure. Many authors agree that a denser microstructure, lower porosity, and stronger ITZ directly correlate with higher residual strength after exposure to high temperatures [55, 56]. CBW and COR fine aggregates consistently exhibit these characteristics, therefore, their use in geopolymer mortars is particularly promising for fire-resistant construction.

4. Conclusions

This study investigated the compressive strength and thermal resistance of geopolymer mortars exposed to temperatures up to 800 °C, focusing on the combined effects of precursor composition and fine aggregate type. The main conclusions are summarized as follows:

- All geopolymer mortars produced with 100% metakaolin waste (MKW) exhibited high initial (28-day) compressive strength, regardless of the fine aggregate type. The highest strengths were achieved with corundum (COR) and basalt (BAS) aggregates, reaching 66.5 MPa (F5 COR) and 63.5 MPa (F5–BAS), confirming their positive contribution to early-age mechanical performance.
- Mortars containing >50% ceramic brick waste (CBW) as precursor exhibited minimal surface cracking across all aggregate types, indicating superior thermal stability. In contrast, MKW-based mortars showed cracking initiation at 200–400 °C, consistent with their higher mass loss caused by elevated free-water content and accelerated evaporation. CBW-based mortars benefited from lower moisture content and partial reactivity at elevated temperatures, promoting matrix sintering and densification. Among the fine aggregates, COR and CBW aggregates demonstrated the least cracking at 800 °C due to their low thermal expansion and high thermal stability.
- Mortars incorporating 75% CBW and 25% MKW as precursors achieved the highest residual compressive strengths after thermal exposure. Mixtures with OSA, GRA, and BAS aggregates retained only about 55% of their initial strength, whereas those with CBW aggregate retained 34.7 MPa (78.7%) and those with COR aggregate reached 53.0 MPa (108.6%). These results clearly demonstrate that CBW and COR aggregates provide superior strength retention, attributable to their thermal stability and, in the case of CBW, beneficial partial reactivity at high temperatures.
- SEM analysis confirmed that aggregate type controls microstructural integrity even prior to thermal exposure. OSA and GRA aggregates formed weaker ITZs with early microcracking, BAS exhibited moderate structural coherence, while CBW and COR aggregates produced dense, homogeneous and thermally stable microstructures. These microstructural characteristics directly correlate with the observed residual strength trends after heating.
- Two-way ANOVA analysis identified temperature as the dominant factor affecting compressive strength for all aggregate types ($p < 0.05$), in agreement with the sharp strength deterioration observed above 400 °C. The binder effect was insignificant for OSA, GRA, and COR mortars, borderline significant for BAS ($p \approx 0.053$), and statistically significant for CBW ($p = 0.031$), indicating that CBW-based systems are particularly sensitive to binder composition.
- Overall, corundum (COR) was identified as the most thermally robust fine aggregate, maintaining excellent structural performance up to 800 °C. Ceramic brick waste (CBW) also showed outstanding performance,

especially when used both as precursor and fine aggregate. Basalt (BAS) and granite (GRA) provided moderate thermal resistance up to approximately 400 °C, while ordinary sand (OSA) was the least suitable for high-temperature applications.

In summary, the results demonstrate that high-temperature durability of geopolymer mortars requires careful alignment of aggregate mineralogy, precursor reactivity, and binder composition, with temperature remaining the primary governing parameter. CBW and COR based systems emerge as particularly promising solutions for fire-resistant geopolymer construction materials.

Data availability

All data generated or analyzed during this study are included in this published article.

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