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GEOPOLYMER COMPOSITE MADE FOR HIGH TEMPERATURE APPLICATIONS

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Abstract. Geopolymer composite for high temperature applications was made using masonry rubble waste resulting from building demolition (clay brick scraps and concrete scraps). It was known that the geopolymer has an initial fire resistance due to the inorganic network constituting its structure, but the concern for testing and improving the mechanical strength of this new material type exposed to high temperatures was not a research objective until recently.

Geopolymer manufacturing included traditional by-products such as granulated metallurgical slag (predominantly) and fly ash as alumina-silicate materials with cementitious properties as well as the alkaline activator, in which NaOH was replaced with KOH due to its superior thermal resistance. Results showed that compressive strength recorded acceptable losses up to 800°C and higher at 1000°C, while flexural strength increased at 600 and 800°C compared to reference value and then decreased after heating to 1000°C.

Keywords: geopolymer composite, granulated slag, fly ash, clay brick, demolition waste.

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1. Introduction

The end of the last century brought to the fore a new perception of the notion of economical and environmentally friendly material. It is about a new type of material with excellent cementitious and pozzolanic properties suitable for use in construction, based on waste and industrial by-products rich in alumina and silica, activated in the aqueous solution of some alkaline activators which facilitate the initiation and development of the geopolymerization reaction turning waste into so-called geopolymers (Matsimbe *et al.*, 2022).

The geopolymerization involves chemical reactions between different alumina-silicate oxides and silicates under high alkaline conditions, forming polymeric Si-O-Al-O bonds (Xu and van Deventer, 2000).

Geopolymer as a new type of material was patented in the last decade of the 20th century by the French researcher J. Davidovits (Davidovits *et al.*, 1994; Davidovits *et al.*, 1998) and had an exceptional contribution to changing the carbon footprint of traditional construction materials based on Portland cement achieved in the last two hundred years (Evolution, 2022). The geopolymer concept opened a new and important stage in the design and production of construction materials, allowing the intensive use of recycled alumina-silicate waste transformed by alkaline activation into products with newly created value.

A wide range of alumina-silicate material sources can be used for producing geopolymers such as natural clay, metakaolin, industrial by-products (fly ash, metallurgical slag, red mud, etc.), residual glass, waste of demolition and rehabilitation of construction, and other wastes (Giannopoulou *et al.*, 2023).

Until 10-15 years ago, research on the use of geopolymers as construction materials was limited to improving their mechanical performance at ambient temperature, without any interest regarding the evolution of these features after exposure to high temperature.

However, according to more recent research, geopolymers have demonstrated an important potential for use in applications that require resistance to high temperatures. Due to their inorganic structure, geopolymers have shown high thermal stability, far superior to traditional cement-based materials. These features allow geopolymers and geopolymer composites to be used in furnace masonry, thermal insulation or wall panels (Giannopoulou *et al.*, 2023; van Riessen *et al.*, 2009).

The main factors that modify the thermal stability and mechanical characteristics of geopolymers after exposure to high temperatures are the type of geopolymeric precursors, the aggregate nature, SiO₂/Al₂O₃ molar ratio, the composition of alkaline activator, and the heating rate of geopolymer (Lahoti *et al.*, 2019; Duxson *et al.*, 2007a; Kovalchuk and Krivenko, 2009).

Recent works (Barbosa and Mackenzie, 2003a; Barbosa and Mackenzie, 2003b; Duxson *et al.*, 2007b) showed that an excellent thermal stability of geopolymers based on metakaolin, using as alkaline activator solutions of

hydroxide and silicate of sodium or potassium, was obtained under the conditions where the curing process kept the amorphous structure until at the melting temperature of geopolymers or when $\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratios facilitated the formation of crystalline phases with high melting points. According to (Duxson *et al.*, 2007b), the proportion of crystalline phases generated at high temperature decreased with the increase of $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio, the unfinished crystallization of the amorphous phase contributing to decreasing the geopolymer mechanical strength. On the other hand, it was found that geopolymers activated with potassium hydroxide and silicate registered a low thermal deformation at high temperature. Another observation concerns the compressive strength of geopolymers based on fly ash or metakaolin as well as the combination of the two materials, which was affected by increasing the exposure temperature. Similarly, the geopolymers based on granulated blast furnace slag suffered a significant decrease of compressive strength after exposure to 1200°C for 1 hour. Instead, their flexure strength slightly increased by exposure to 1000°C and very much by exposure to 1200°C (Rovnanik *et al.*, 2013).

Due to their chemical properties and microstructure, geopolymers have an initial fire resistance, which recommends their use in applications involving high temperatures. According to (Lahoti *et al.*, 2019), the inorganic network that supports the structure of geopolymers contributes to the high fire resistance of these materials. Also, they contain significant amounts of physically and chemically bound water, which at high temperature evaporates, the material becoming endothermic.

The paper (Giannopoulou *et al.*, 2023) aimed at the experimental research of the possibilities of increasing the fire resistance of geopolymers prepared from brick waste and respectively, ceramic tile waste recycled from building demolition. The geopolymers made in this way were tested in terms of compression strength, apparent density, and mass loss, after their exposure to temperatures up to 1050°C. The thermal stability of geopolymers made with brick waste and respectively, with ceramic tile waste, tested at high temperatures of maximum 1050°C was satisfactory, without tendency for thermal deformation. The compressive strength of the brick waste-based geopolymer reached 19 MPa after the curing process for 7 days and kept values within the limits of 15-21 MPa after exposure to 600-1050°C. At the maximum thermal level, the geopolymer lost 8% from the density value and 10% of the value of its initial mass. The compressive strength of ceramic tile waste-based geopolymer reached 33 MPa after 7 curing days, decreased to 17 MPa after exposure to 600°C and 13 MPa after exposure to 800°C, but reached a value even slightly higher than the initial one at 1050°C. At the maximum temperature, the apparent density decreased by 9% compared to the initial value, while the product mass decreased by 7%.

The authors' team of the current paper previously performed several experiments aiming at the use of alumina-silicate waste and industrial by-products in order to manufacture geopolymer materials suitable for use in the

construction sector (Paunescu *et al.*, 2023a, b, c; Paunescu and Paunescu, 2023d; Paunescu *et al.*, 2023e). The determined physical and mechanical characteristics were limited only to normal temperature conditions, not to the requirement of these materials at high temperatures. The objective of this work is to extend characteristics measurement of geopolymers also after exposure to high temperatures.

2. Materials and Methods

The geopolymer prepared for the experiment described in the paper was composed of two industrial by-products with pozzolanic and cementitious properties (granulated blast furnace slag and coal fly ash) and two waste recycled from the demolition of buildings (clay brick waste and concrete waste). River sand as a fine aggregate (grain size below 3.5 mm) completed the list of solid component materials.

Granulated blast furnace slag purchased from ArcelorMittal Galati (Romania) more than 10 years ago constitutes a by-product of the industrial manufacture of pig iron. The slag granulation technique is usually applied in metallurgy by pouring the melt into a cold-water pool, the slag granule dimension being between 2-6 mm. Grinding the slag in a ball mill allowed the grain size to drop below 100 μm . The material chemical composition according to (Paunescu *et al.*, 2022) includes: 36.44% SiO_2 , 11.60% Al_2O_3 , 41.81% CaO , 5.80% MgO , 0.55% MnO , 0.78% Fe_2O_3 , 0.35% Na_2O , and 0.43% K_2O .

Coal fly ash, the most well-known and frequently used alumina-silicate industrial by-product in geopolymer manufacturing processes, was supplied by Paroseni-Thermal Power Plant (Romania) about 8 years ago, under the conditions that the plant used anthracite as fuel, having the effect of generating a fly ash containing low amounts of CaO . The initial grain size of the ash was below 250 μm and its mechanical processing led to obtaining a fine powder below 60 μm . The chemical composition of the fly ash was as follows: 54.4% SiO_2 , 26.5% Al_2O_3 , 3.5% CaO , 2.5% MgO , 1.5% TiO_2 , 4.8% Fe_2O_3 , 0.4% Na_2O , 0.6% K_2O , and 1.7% SO_3 as determined on the AXIOS type X-ray fluorescence spectrometer in the Metallurgical Research Institute Bucharest.

Theoretically, clay brick being a porous material influences the firing temperature (in the range of 800-1100°C), whose increase affects the mechanical and physical properties of the material (Karaman *et al.*, 2006), the low durability, and high quantity of water-absorbing. The chemical composition of clay brick includes: 48.7% SiO_2 , 13.7% Al_2O_3 , 10.0% CaO , 5.7% Fe_2O_3 , 3.7% MgO , and 2.5% K_2O (Šveda *et al.*, 2017). Recycled old clay bricks were crushed and ground in a ball mill, the resulted particles being sieved and selected the grain size under 90 μm .

The old concrete recycled from building demolition was isolated from masonry rubble to be used in the manufacture of geopolymer. The scraps were

crushed and ground in the ball mill in order to obtain particle sizes below 90 μm . The chemical composition of old concrete particles was determined according to (Jorat *et al.*, 2018), resulting in the following values: 71.9% SiO_2 , 4.0% Al_2O_3 , 12.1% CaO , 0.7% MgO , 0.2% TiO_2 , 1.5% Fe_2O_3 , 0.6% Na_2O , 0.7% K_2O and 0.4% SO_3 .

River sand, very rich in silica (over 96%) (Bala and Khan, 2013), was added in the mixture as fine aggregate. Its particle size was limited to maximum 3.5 mm and the particle selection was made by sieving.

The alkaline activator was composed of KOH pellets dissolved in distilled water (10 M molarity) and Na_2SiO_3 aqueous solution (8% Na_2O , 27% SiO_2 , and 65% water). KOH was preferred over NaOH (the other option recommended by the inventor J. Davidovits) due to the higher fire resistance of potassium-based geopolymers, resistant to temperatures above 1200°C (Sakkas *et al.*, 2015).

Three mixture versions for preparing geopolymer specimens in order to test the changes of their features after exposure to high temperatures were designed (Table 1).

Table 1
Experimental versions of the geopolymer preparation

Composition	Version 1 ($\text{kg}\cdot\text{m}^{-3}$)	Version 2 ($\text{kg}\cdot\text{m}^{-3}$)	Version 3 ($\text{kg}\cdot\text{m}^{-3}$)
Granulated blast furnace slag	260	240	220
Coal fly ash	50	70	90
Recycled residual clay brick	480	490	500
Recycled residual concrete	460	450	440
River sand	1250	1250	1250
10 M KOH	85	85	85
Na_2SiO_3	225	225	225

According to the data in Table 1, in this experiment the granulated blast furnace slag constituted the predominant alumina-silicate material related to coal fly ash in a weight ratio between 2.44:1 and 4:1. The waste recovered from the building demolition represented 960 $\text{kg}\cdot\text{m}^{-3}$ in each of the three experimental versions, but the clay/concrete weight ratio increased from 1.043 (version 1) to 1.136 (version 3). River sand as fine aggregate was used in weight of 1250 $\text{kg}\cdot\text{m}^{-3}$, representing 50% from the total amount of solids.

The manufacturing method of the geopolymer is generally an established method, the main difference between different particular applied methods referring to the curing period of the fresh material (mainly temperature and duration). The preparation of the mixture of solid materials as well as that of the components of the liquid alkaline activator were carried out in separate vessels. After the homogenization of the two mixtures, the alkaline activator was slowly

poured over the solid mixture, after which their mixing was carried out in an electrically operated mixer at 500 rpm for 5-7 min until the formation and homogenization of the paste. The paste was then poured into open metal moulds of cubic and rectangular shape, respectively. The paste curing took place according to the own method previously applied and tested by heating at 80°C for 24 hours in an electric oven. After the curing period, the specimens were removed from the mould and stored for hardening at room temperature under low humidity conditions for 7 and 28 days before determining the characteristics of samples and the influence of exposure to high temperature.

Testing the change of specimen characteristics through exposure to high temperatures (600, 800 and 1000°C) involves subjecting them to heating thermal regime starting from room temperature with an average rate between 8-10°C/min, maintaining for 2 hours at the maximum temperature and then relatively slow cooling at a rate of 4-5°C/min until the initial temperature.

Usual methods were applied to identify the physical and mechanical characteristics of specimens. Archimedes' principle was used to measure the apparent density of geopolymer samples (ASTM B962-23). 1000 kN electro-hydraulic mechanical testing machine was used for measuring the compressive strength (ASTM C 109). Three-point bending flexural test was utilized for determining the flexural strength with ASTM C293 standard. Measuring the water-absorbing of specimens for 24 hours was performed with ASTM D570 standard. The mass loss of geopolymeric products was identified by weighing the specimens before and after exposure to high temperature. The microstructural appearance of samples could be examined with the Biological Microscope MT5000 model, 1000 x magnification.

3. Results and Discussion

The results of determining the physical and mechanical features of geopolymer composite specimens at room temperature are presented in Table 2.

Table 2
Physical and mechanical features at room temperature of specimens

Feature	Version		
	1	2	3
Apparent density (kg·m ⁻³)	2498	2470	2448
Compressive strength (MPa)			
- after 7 days	24.6	21.1	18.3
- after 28 days	49.7	44.0	39.2
Flexural strength (MPa)			
- after 7 days	4.0	3.9	3.7
- after 28 days	8.2	7.9	7.8
Water-absorbing (vol. %)	6.3	6.5	6.8

The physical appearance of the three geopolymer composite specimens is shown in Fig. 1 and their microstructural appearance is revealed in Fig. 2.

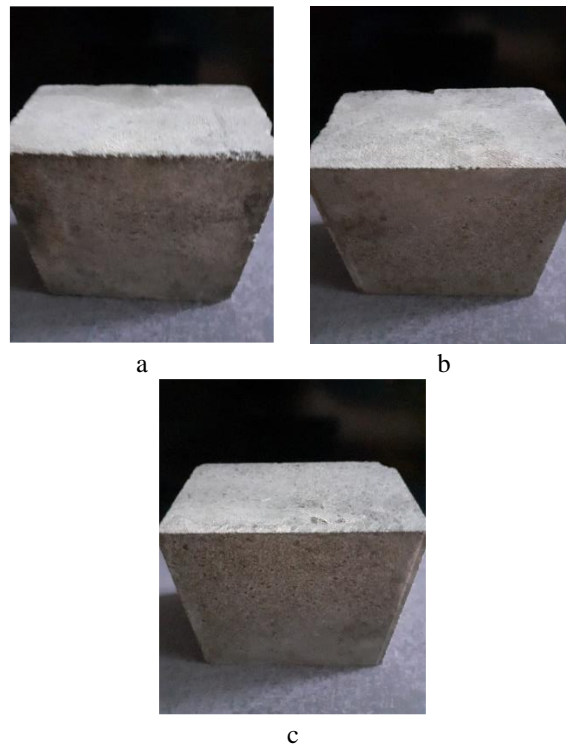
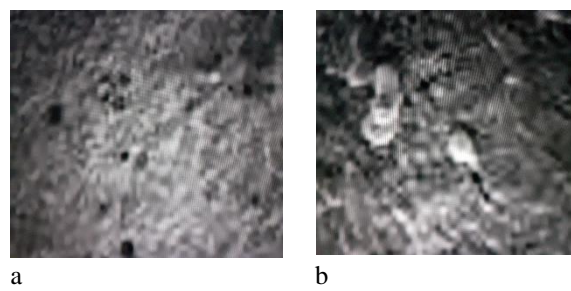


Fig. 1 – Physical appearance of geopolymer composite specimens
a – version 1; b – version 2; c – version 3.



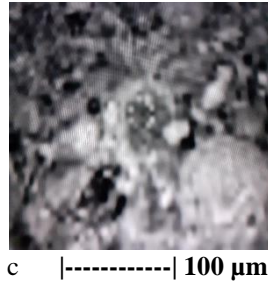


Fig. 2 – Microstructural appearance of geopolymer composite specimens
a – version 1; b – version 2; c – version 3.

The second stage of the measurement process carried out for highlighting the influence of exposing the samples to high temperatures (600-1000°C) was further achieved by their heating, maintaining, and cooling, successively reaching the limits of 600, 800, and 1000°C, respectively. The results obtained by applying the methods for determining the characteristics of the three specimens after the heat treatment at high temperatures are shown in Table 3. The measurements were performed after cooling the specimens to ambient temperature.

Table 3

Characteristics of geopolymer specimens after exposure to high temperatures

Characteristic	Specimen 1	Specimen 2	Specimen 3
Apparent density (kg·m ⁻³)			
- after heating at 600°C	2375	2349	2326
- after hearing at 800°C	2248	2223	2201
- after heating at 1000°C	2188	2110	2032
Mass loss (%)			
- after heating at 600°C	5.1	4.9	5.2
- after hearing at 800°C	10.0	10.0	10.2
- after heating at 1000°C	12.4	14.6	17.0
Compressive strength (MPa)			
- after heating at 600°C	48.0	43.1	37.9
- after hearing at 800°C	45.7	40.3	34.2
- after heating at 1000°C	40.2	32.5	28.4
Flexural strength (MPa)			
- after heating at 600°C	8.3	7.9	7.8
- after hearing at 800°C	8.5	8.2	8.0
- after heating at 1000°C	7.2	7.0	6.9
Water-absorbing (vol. %)			
- after heating at 600°C	6.4	6.5	6.9
- after hearing at 800°C	6.5	6.5	6.8
- after heating at 1000°C	6.0	6.1	6.1

According to the data in Table 3, the exposure of geopolymer composite specimens to high temperatures (between 600-1000°C) influences their physical and mechanical characteristics identified in normal temperature conditions, but to a small extent and especially at 1000°C.

The apparent density of specimens with values between 2448-2498 kg·m⁻³ without heat exposure, decreased after exposure to 600°C to values between 2326-2375 kg·m⁻³, reaching the lowest level after exposure to 1000°C (2032-2188 kg·m⁻³), due to the loss mass experimentally determined at 4.9-17.0% compared to the initial mass of samples.

The compressive strength, having initial values between 39.2-49.7 MPa, depending on the analyzed sample (according to Table 2), recorded reduction of these values due to the heat exposure, but as in the case of density, within small limits. Thus, specimen 1 with the highest strength value decreased from 49.7 MPa (reference value) to 40.2 MPa corresponding to heating at 1000°C.

Regarding the flexural strength, its reference values (7.8-8.2 MPa) corresponding to the data in Table 2, slightly increased by exposure to 600°C (7.8-8.3 MPa) and respectively, 800°C (8.0-8.5 MPa), decreasing to 6.9-7.2 MPa after exposure to 1000°C.

Water-absorbing measured in the range of 6.3-6.8 vol. % in the reference situation (Table 2) was influenced to a small extent by exposure to 600 and 800°C. Only the exposure to the temperature of 1000°C led to a slight reduction of its values reaching 6.0-6.1 vol. %.

Therefore, the technique adopted in this work for producing a geopolymer composite usable in constructions, resistant to sufficiently high temperatures avoiding the effects caused by a possible fire, proved viable following the measurements. The material mixture was based on recycled waste from building demolition (clay brick and concrete as old materials). Ground granulated blast furnace slag in predominant proportion and coal fly ash, as alumina-silicate industrial by-products were used due to their pozzolanic and cementitious properties. The alkaline activator required for the activation of alumina-silicate materials and developing the geopolymerization reaction was used by combining KOH pellets dissolved in distilled water with Na₂SiO₃ aqueous solution. The authors' team of the current paper used for the first time KOH replacing NaOH due to the superior resistance to thermal stress of the potassium-based geopolymer, according to the information taken on from the literature (Sakkas *et al.*, 2015).

The concern for preserving the mechanical strength properties of geopolymers usable in construction was not highlighted in the literature until recently. Giannopoulou *et al.*, 2023 is a singular example referring to making and testing geopolymers based on building demolition waste (residual bricks and ceramic tiles) exposed to temperatures up to 1050°C.

Considering the different composition of the material mixture as well as the different parameters of the curing process of the fresh material (temperature and duration), the current work originality is proven.

4. Conclusions

The objective of the work was the preparation of a geopolymer composite whose exposure to high temperatures (up to 1000°C) does not significantly change its mechanical properties. It was known that in general, the geopolymer has an initial fire resistance due to the use of some waste or industrial by-products rich in alumina and silica. However, until recently, no scientific work was recorded dedicated to testing the change in the mechanical characteristics of geopolymers exposed to high temperatures. The experiment described in this paper used granulated blast furnace slag and fly ash as alumina-silicate materials in a net ratio favourable to slag. Also, important quantities of waste from building demolition (clay brick scraps and concrete scraps) in a balanced weight ratio, were used in the starting mixture. Unlike the numerous experiments performed by the work authors aiming at the manufacture of different geopolymer types, for the first time the composition of the alkaline activator was changed, KOH being adopted as a substitute for NaOH in the mixture also including Na_2SiO_3 . The modification was justified by the superior thermal resistance of the potassium-based geopolymer. The test results showed that the apparent density of the geopolymeric product, the mass loss, and the compressive strength were negatively influenced, but to a small extent, by the exposure of the hot cured material and stored for 28 days at temperatures of 600 and 800°C. Influence of the exposure at 1000°C was felt to a greater extent, the density more decreasing, the mass loss increasing, and the compressive strength decreasing its value to the greatest extent. However, flexural strength had a different behaviour, slightly increasing after exposure to 600 and 800°C, and registering a more important decrease after exposure to 1000°C. The most important conclusion of the work was that the characteristics of the made geopolymer were acceptable at least after exposure to temperatures up to 800°C, with the mention that the flexural strength value even increased for 600 and 800°C exposure compared to measuring results without hot exposure.

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COMPOZIT GEOPOLIMERIC REALIZAT PENTRU APLICAȚII LA TEMPERATURĂ ÎNALTĂ

(Rezumat)

Compozitul geopolimeric pentru aplicații la temperaturi înalte a fost realizat folosind deșeuri din molozul zidăriei rezultate din demolarea clădirilor (spărturi de cărămidă de argilă și spărturi de beton). Se știa că geopolimerul are o rezistență inițială la foc datorită rețelei anorganice care constituie structura sa, dar preocuparea pentru testarea și îmbunătățirea rezistenței mecanice a acestui nou tip de material expus la temperaturi înalte nu a fost un obiectiv al cercetării până de curând. Fabricarea geopolimerului a inclus produse secundare tradiționale cum sunt zgura metalurgică granulată (în proporție predominantă) și cenușa zburătoare sub formă de materiale alumino-silicatică cu proprietăți de cimentare, precum și activatorul alcalin, în care NaOH a fost înlocuit cu KOH datorită rezistenței sale termice superioare. Rezultatele au arătat că rezistența la

compresiune a înregistrat pierderi acceptabile până la 800°C și mai mari la 1000°C, în timp ce rezistența la încovoiere a crescut la 600 și 800°C comparativ cu valoarea de referință și apoi a scăzut după încălzirea la 1000°C.