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SYNTHESIS AND MECHANICAL PROPERTIES OF ECO-FRIENDLY GEOPOLYMERS OBTAINED FROM WASTE MATERIALS

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Abstract

This paper investigates the synthesis and mechanical properties of geopolymers obtained from industrial waste - fly ash and granulated blast furnace slag - with the aim of developing environmentally friendly and sustainable construction materials. The raw materials were activated with alkaline solutions of water glass and 12M NaOH in a 1:2 ratio. The ratio of alkali to precursor (fly ash, slag and quartz sand) was 1:1. The dependence of the compressive strength on the addition of slag was tested for geopolymer pastes (C), and geopolymer mortars (P) based on fly ash and quartz sand, both with and without granulated blast furnace slag. The results show that adding up to 20% slag does not significantly affect the compressive strength of the geopolymer. The compressive strengths of geo ratio polymer pastes are 30-50% higher than those of geopolymer mortars. However, the compressive strengths of both geopolymer pastes and mortars after 2 and 7 days are twice as high as the standard values of compressive strengths of cement mortars after 2 and 7 days according to EN 197-1. Chemical analysis (XRF) confirmed the presence of high content of SiO_2, Al_2O_3 and CaO in the starting materials, which contributes to the formation of a strong geopolymer structure. The obtained results indicate the potential of geopolymers as a sustainable and environmentally friendly alternative to conventional binders in construction.

Key words: *geopolymers, fly ash, granulated blast furnace slag, compressive strength, XRF analysis, environmental protection.*

1. Introduction

Growing concerns about CO_2 emissions and pollution caused by the production of Portland cement have led to the intensive development of alternative, environmentally friendly construction materials. Geopolymers, inorganic polymers based on aluminosilicates, are among the most promising alternatives. Their synthesis involves the activation of raw materials rich in silicon and aluminum with alkaline solutions, resulting in amorphous or semi-crystalline structures with high mechanical and chemical properties. The advantages of geopolymers include compressive strength, refractoriness, and corrosion resistance [1,2,5-10]. Namely, the compressive strength of geopolymers after 7 days is significantly higher than the compressive strength of cement, and it is assumed that with additional research, faster construction of concrete structures based on geopolymers can be achieved.

The use of waste materials such as fly ash and granulated blast furnace slag offers a dual benefit: it reduces the amount of waste that would otherwise be landfilled, and at the same time enables the production of materials with a low ecological footprint. Fly ash, as a by-product of coal combustion, is rich in SiO_2 and Al_2O_3, while granulated blast furnace slag, a product of the metallurgical industry, contains significant amounts of CaO, MgO and Fe_2O_3, which strengthen the mechanical properties of geopolymers [3-5,15,16].

In environmental terms, the use of fly ash and granulated blast furnace slag as raw materials directly contributes to the reduction of CO_2 emissions, by avoiding energy-intensive production of Portland cement. It is estimated that the use of geopolymers can reduce CO_2 emissions by up to 80%, which makes this material particularly attractive for sustainable construction.

The aim of this paper is to produce geopolymers in BiH using fly ash from thermal power plants. There are

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four thermal power plants in BiH, each using different types of coal and therefore generating different types of fly ash as waste material. This paper presents only a small part of the research, using fly ash from the Stanari Thermal Power Plant near Doboj and granulated blast furnace slag from ArcelorMittal Steel Zenica. Since there are no standards for testing geopolymers, cement standards are used.

Alkali activation enables the production of innovative construction materials by reacting silicon- and aluminium-rich precursors with a highly alkaline solution. This solution, commonly an alkali hydroxide or silicate, acts as the chemical activator. By raising the pH of the mixture, the activator promotes dissolution of the raw materials and drives the formation of a new three-dimensional, concrete-like network structure [16]. The specific choice of activator (e.g., sodium hydroxide or sodium silicate) affects the pore architecture and binding phases of the resulting material, typically enhancing the formation of amorphous aluminosilicate gels known to improve chloride resistance. Appropriate curing conditions are also essential, as they support the development of a dense microstructure that limits chloride ingress [16].

The characteristics of the alkaline activator are key determinants of the reaction mechanisms and final performance of alkali-activated materials (AAMs). Increased alkalinity generally intensifies the activation reactions, producing denser and mechanically stronger matrices, while higher activator concentrations can accelerate reaction kinetics, leading to reduced setting times and increased early-age strength [16,19]. The most frequently used alkaline reagents in alkali-activation systems include hydroxides (e.g., sodium hydroxide and potassium hydroxide) and silicates (e.g., sodium silicate). Sodium hydroxide (NaOH) is a highly soluble, strongly alkaline compound that supplies an abundant concentration of hydroxide ions (OH^-) necessary for precursor dissolution. Potassium hydroxide (KOH) exhibits similar chemical behaviour and also contributes hydroxide ions to sustain the activation process. Sodium silicate (Na_2SiO_4), produced from the reaction of sodium carbonate with silica sand, provides both alkalinity and a reactive silica source, making it a versatile and widely used activator in AAM systems [12,13,16,18].

2. Experimental

2.1. Material and methods

The chemical analysis of the initial raw materials, as well as the prepared samples, was carried out using a Bruker S8 Tiger XRF spectrometer, which is routinely used for raw material analysis at the Kakanj cement plant.

X-ray diffractometry (XRD) was used to identify the crystalline phases present and to determine the crystallographic structure of the material. Powdered samples were pressed into compact pellets and analyzed using a Bruker D8 Endeavor XRD instrument. During the measurements, Cu Kα (λ = 1.5418 Å) radiation was employed, generated in the X-ray tube at 25 mA and 40 kV.

Differential thermal analysis (DTA) was employed to determine the temperatures at which phase transformations occur in the samples. The measurements were carried out using a Netzsch DTA 409 CD instrument. For the analysis of differential thermal curves, the method of comparison with appropriate standards from DTA curve catalogs was used.

AJEOL JSM-6460LV scanning electron microscope was used to examine and capture high-resolution images of the fracture surfaces of the samples. Imaging was performed at the University Center for Electron Microscopy, University of Novi Sad (Serbia).

The granulometric composition was analyzed using a Malvern Mastersizer 2000 laser particle size analyzer.

To measure flexural and compressive strength, the Kakanj cement factory used a Toni Technik press. Standard prisms were prepared according to EN 196-1 for flexural and compressive strength testing. Compressive and flexural strength were measured after 2, 7, and 28 days. Each prism was placed in the testing apparatus with its side surface on the support rollers and its longitudinal axis vertical to the supports. The load was applied vertically via the loading roller on the opposite face of the prism and increased uniformly at a rate of 50 ± 10 N/s until failure occurred. Upon fracture, the press automatically stopped, and the value was read directly from the press in MPa.

The raw materials used in this study included fly ash from the Stanari Thermal Power Plant, Doboj; granulated blast furnace slag from Arcelor Mittal Zenica; standard quartz sand in accordance with DIN EN 196-1; commercial water glass; and a 12 M NaOH solution prepared in the Chemistry Laboratory of the Faculty of Engineering and Natural Sciences, University of Zenica.

2.2. Synthesis of geopolymers

Geopolymers were synthesized in two ways, divided into two series. The first series consisted of pastes prepared by mixing fly ash and alkali with and without the addition of 20% granulated blast furnace slag. The second series was synthesized as mortar, following the procedure used for preparing cement samples for compressive and flexural strength testing with standard sand). The ratio of alkali Na_2SiO_3/NaOH = 2:1, while the ratio of alkali to the solid mixture (fly ash, granulated blast furnace slag and quartz sand) was 1:1. 12 M NaOH was used. The mixtures were homogenized and poured into 50 x 50 mm roller molds for the first series (GC), and 40 x 40 x 160 mm prism molds for the second series (GSP). The samples were hermetically sealed, due to the exothermic reaction, and after 24 hours they were removed from the molds. They were then placed in a drying oven at a temperature of 70 °C for 24 h in order to perform geopolymerization. After geopolymerization, the samples were left at room temperature until the moment of testing. It should be noted that the samples from series 2 were prepared according to the EN 196-1 standard for the preparation of cement samples. The designations of samples and mixtures are shown in Table 1.

Table 1 Labels of samples of prepared mixtures

Series	Sample labels	Alkali/raw material ratio	Na_2SiO_3/NaOH ratio	Fly ash [%]	Granulated blast furnace slag [%]	Quartz sand [g]
Series I	0GC	1	2	100	0	0
	20GC	1	2	80	20	0
Series II	OGSP	1	2	100	0	1350
	20GSP	1	2	80	20	1350

Figure 1 Preparation of paste samples (I series - GC)

Figure 2 Preparation of mortar samples (2nd series - GSP)

After the samples were prepared, the compressive strengths were tested after 2, 7 and 28 days.

3. Results and discussion

Table 2 shows the chemical composition of fly ash from the Stanari thermal power plant, and Table 3 shows the chemical composition of granulated blast furnace slag determined by the XRF method.

Chemical analysis (XRF) confirmed the presence of high SiO_2, Al_2O_3 and CaO in the starting materials, which contributes to the formation of a solid geopolymer structure.

Table 2 Chemical analysis of fly ash from the thermal power plant Stanari [20]

Oxides	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	MnO	TiO_2
Content [%]	44,21	19,06	8,56	20,98	2,33	1,78	1,55	0,058	0,70

Table 3 Chemical composition of granulated blast furnace slag "ArcelorMittal" Zenica [20]

Oxides	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	TiO_2	LOI
Content [%]	42,39	8,37	1,83	37,08	5,36	0,46	<0,01

Figures 3 and 4 show DTA/TG curves of fly ash and granulated blast furnace slag, which were performed at the Faculty of Engineering and Natural Sciences, University of Zenica. Fly ash and slag were heated to a temperature of 1000 °C at a rate of 20 °C/min.

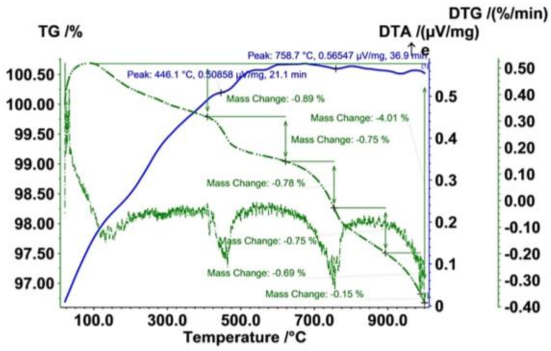

Figure 3 DTA/TG analysis of the fly ash of the TPP Stanari

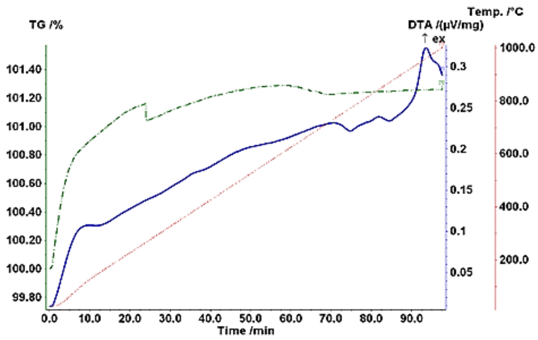

Figure 4 DTA/TG analysis of granulated blast furnace slag

Figure 9 provides a detailed representation of the changes that fly ash undergoes when heated in a controlled atmosphere. The initial minor change in mass is primarily due to the loss of physically adsorbed moisture or hygroscopic water. Fly ash exhibits good thermal stability within the range of 200 °C to ~ 600 °C, as shown by the relatively flat green TG (thermogravimetric) curve. The mass remains stable, which is a key characteristic of quality fly ash used in building materials. Above 600 °C there is a significant mass loss, which coincides with the sharp upward peaks in the blue DTA curve. Rising DTA peaks indicate exothermic reactions (release of heat). It is assumed that this mass loss is primarily attributed to the combustion or oxidation of remaining unburned carbon (LOI - loss on ignition) and the decomposition of compounds such as carbonates (e.g., calcite decomposes to calcium oxide and carbon dioxide) present in the ash. A slight mass gain can occur even above 600 °C due to the oxidation of ferrous compounds in the presence of an oxidizing atmosphere (air). The diagram in Figure 3 confirms that fly ash behaves as expected, especially in its reactions at high temperatures associated with carbon content and mineral decomposition.

Figure 4 shows that based on the thermogravimetric analysis (TGA) and differential thermal analysis (DTA) data, granulated blast furnace slag (GBFS) undergoes significant thermal events and decomposition reactions at high temperatures. The green TG curve is relatively flat and stable below approximately 600 °C (about 25 minutes). This indicates that the as-received GBFS sample is mostly dry and has a low loss on ignition (LOI) up to this temperature range, which is a desirable property for its use as a geopolymer additive. The most prominent features are the sharp upward peaks in the blue DTA curve at around 80-90 minutes (corresponding to temperatures above 900 °C). These are strong exothermic peaks, meaning that the material is releasing a significant amount of heat. The DTA peaks coincide with the steep decline in the green TG curve. This synchronized behavior indicates that the heat release is likely related to a chemical reaction that causes mass loss, such as decomposition or oxidation of the components within the granulated blast furnace slag (GBFS) at very high temperatures (typically above 800 °C). Given that the structure of granulated blast furnace slag is amorphous, it is assumed that this exothermic peak at a temperature of 990 °C corresponds to the recrystallization of the amorphous phase into a crystalline one, i.e., in melilit.

Analysis indicates that granular blast furnace slag (GBFS) is thermally stable up to a temperature of approximately 600 °C. Above this temperature, the granular blast furnace slag (GBFS) undergoes a significant, multi-stage decomposition process that is exothermic in nature, as evidenced by the weight loss on the TG curve and prominent upward peaks on the DTA curve.

Figures 5 and 6 show the XRD analysis of fly ash and slag, which was performed at the Kakanj Cement Factory.

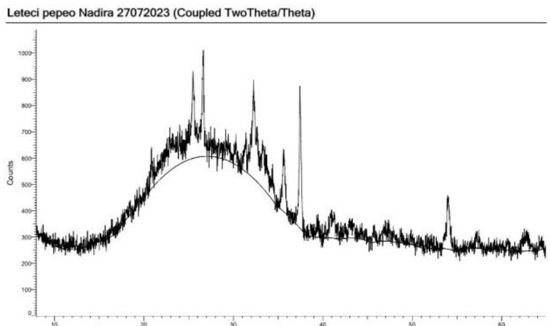

Figure 5 XRD analysis of fly ash [20]

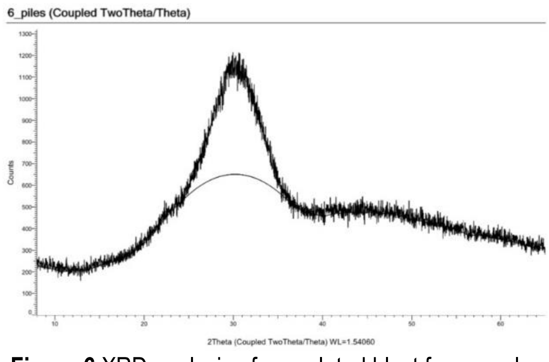

Figure 6 XRD analysis of granulated blast furnace slag [20]

Based on the XRD analysis of fly ash, shown in Figure 5, it can be seen that the fly ash is largely amorphous, but also contains a small percentage of crystalline structure, as indicated by several sharp peaks of quartz, mullite, hematite, and magnetite. The proportion of the amorphous phase is 74.5%. A large, broad amorphous peak in the sample, centered approximately between 20° and 30° (2θ angle), indicates a significant amount of non-crystalline (glassy) aluminosilicate material. This amorphous phase is typical of fly ash and contributes to its pozzolanic properties. Several sharp crystalline peaks are superimposed on the broad peak. The relative intensity and sharpness of these peaks, together with the prominence of the amorphous curve, suggest that this is probably an F-class ash, which typically has a low calcium oxide content and a high silica and alumina content.

Based on the XRD analysis of the slag shown in Figure 6, it can be noted that there are no sharp or sudden peaks, meaning np peaks that deviate from the others. A key feature of this sample of granulated blast furnace slag is the presence of a very broad, diffuse "peak" around an angle of approximately 30° rather than sharp, distinct peaks. The non-crystalline nature of the granulated blast furnace slag is clearly indicated by this X-ray.

Figures 7 and 8 show the granulometric composition of fly ash and slag.

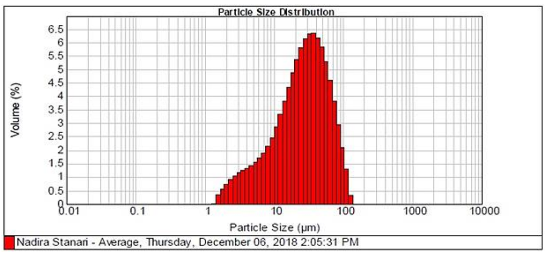

Figure 7 Granulometric composition of the fly ash of the TPP Stanari

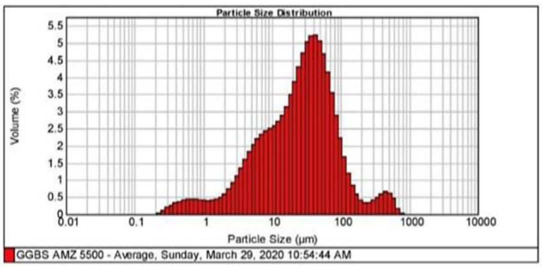

Figure 8 Granulometric composition of granulated blast furnace slag [20]

From Figure 7 it is evident that the particle size of the Stanari fly ash is relatively uniform. The highest peak in the distribution occurs around 40 µm to 50 µm particle size in the sample by volume. The distribution is skewed to the left (finer particles), but the majority of the material is concentrated in the range of 10 µm to 100 µm.

It can be seen from Figure 8 that the particle size of granulated blast furnace slag ranges from 0.3 to 1000 µm, with most of the material falling in the middle range. The highest peak (mode) in the distribution occurs between approximately 40 µm and 60 µm, indicating that this is the most common size range by volume in the sample. There is a minor peak around 400 to 600 µm, indicating a bimodal distribution (two primary size groups). An adequate particle size distribution and fineness improve particle packing and the flowability (workability) of concrete mixes.

SEM images of fly ash Stanari samples are shown in Figure 9.

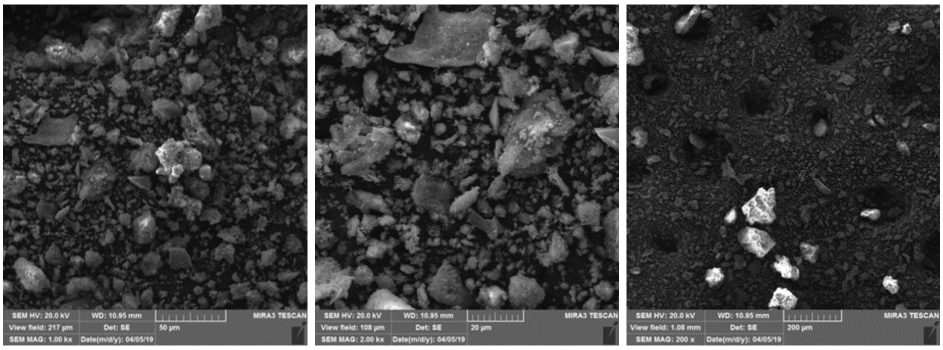

Figure 9 SEM images of fly ash samples Stanari

From the SEM image, it can be seen that the fly ash grains are not spherical but have an irregular fluffy shape. This appearance of fly ash grains is a consequence of the low temperature of coal combustion, which ranges from 850-900 °C. At such a low coal combustion temperature, the ash particles did not transition into a glassy state, but the non-combustible components found in the coal transitioned into other crystalline phases.

Figure 10 shows the compressive strength values for geopolymer samples prepared at the ratio A/LP = 1.0 and at the ratio VS/NaOH = 2.0, which were treated at a temperature of 70°C, and whose compressive strength was tested after 2, 7 and 28 days.

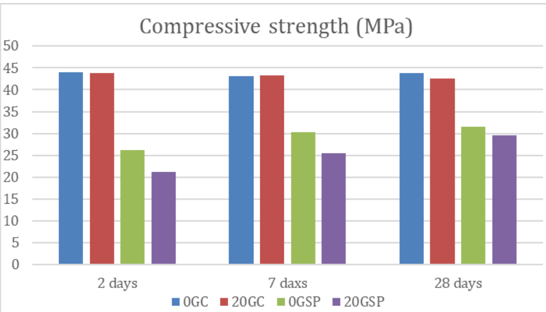

Figure 10 Dependence of the compressive strength of geopolymer pastes and mortars with and without the addition of slag

From Figure 10, it can be concluded that the compressive strengths of geopolymer pastes with (20GC) and without (0GS) the addition of 20% granulated blast furnace slag are approximately the same after 2 and 7 days. The 0GC and 20GC mixes consistently achieve the highest compressive strengths across all time points, both reaching approximately 44-45 MPa at 28 days. The 0GC and 20GC mixes start with high early strength (around 44 MPa), while the 0GSP and 20GSP mixes start much lower (around 26 MPa and 21 MPa, respectively). The compressive strengths of geopolymer pastes are 30-50% higher than those of geopolymer mortars (0GSP and 20 GSP). The diagram also shows that the compressive strengths of geopolymer mortars without (0GSP) the addition of granulated blast furnace slag are higher than that of geopolymer mortars with the addition of 20% slag (20GSP). However, the compressive strengths of both geopolymer pastes and mortars after 2 and 7 days are twice as high as the standard prescribed values for the compressive strengths of cement mortars after 2 and 7 days.

4. Conclusion

Research has shown that it is possible to produce geopolymers with high compressive strength using industrial waste - fly ash from the Stanari thermal power plant and granulated blast furnace slag from ArcelorMittal Zenica. The the compressive strengths of geopolymer pastes with (20GC) and without (0GS) the addition of 20% granulated blast furnace slag are approximately the same after 2 and 7 days. The 0GC and 20GC mixes consistently achieve the highest compressive strength across all time points, both reaching approximately 44-45 MPa at 28 days. The 0GC and 20GC mixes start with high early strength (around 44 MPa), while the 0GSP and 20GSP mixes start much lower (around 26 MPa and 21 MPa, respectively). The compressive strengths of geopolymer pastes are 30-50% higher than those of geopolymer mortars (0GSP and 20 GSP). Also, the compressive strengths of geopolymer mortars without (0GSP) the addition of granulated blast furnace slag are higher than those of geopolymer mortars with the addition of 20% slag (20GSP). However, the compressive strengths of both geopolymer pastes and mortars after 2 and 7 days are twice as high as the standard values of compressive strengths of cement mortars after 2 and 7 days, according to EN 197-1. This leads to the conclusion that geopolymer-based materials could speed up the construction of structures compared to construction with cement concrete. However, it is necessary to invest more into further research. With a significant reduction in ecological footprint, geopolymers based on fly ash and granulated blast furnace slag represent a sustainable and environmentally friendly alternative to cementitious materials.

Note

A part of this study was presented at the XVI International Mineral Processing and Recycling Conference, organized by the University of Belgrade, Technical Faculty in Bor, from 28–30 May 2025, Belgrade, Serbia.

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SINTEZA I MEHANIČKA SVOJSTVA EKOLOŠKI PRIHVATLJIVIH GEOPOLIMERA DOBIJENIH IZ OTPADNIH MATERIJALA

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Izvod

U ovom radu ispitivana je sinteza i mehanička svojstva geopolimera dobijenih iz industrijskog otpada - letećeg pepela i granulisane visokopećne troske - s ciljem razvoja ekološki prihvatljivih i održivih građevinskih materijala. Sirovine su aktivirane alkalnim rastvorima vodenog stakla i 12M NaOH u odnosu 1:2. Odnos alkalija i prekursora (leteći pepeo, granulisana visokopećna troska i kvarcni pijesak) bio je 1:1. Ispitana je zavisnost pritisne čvrstoće uzoraka sa i bez dodatka granulisane visokopećne troske geopolimernih pasti (C), kao i geopolimernih maltera (P). Rezultati pokazuju da dodatak granulisane visokopećne troske u količini do 20% ne utiče značajno na pritisnu čvrstoću geopolimera. Pritisne čvrstoće geopolimernih pasti su 30-50% veće od pritisnih čvrstoća geopolimernih maltera. Međutim, pritisne čvrstoće i geopolimernih pasti i maltera nakon 2 i 7 dana su dvostruko veće od standardnih vrijednosti pritisnih čvrstoća cementnih maltera nakon 2 i 7 dana prema EN 197-1. Hemijska analiza (XRF) potvrdila je prisustvo visokog sadržaja SiO_2, Al_2O_3 i CaO u sirovinskim materijalima, što doprinosi formiranju jake geopolimerne strukture. Dobijeni rezultati ukazuju na potencijal geopolimera kao održive i ekološki prihvatljive alternative konvencionalnim vezivima u građevinarstvu.

Ključne reči: *geopolimeri, leteći pepeo, granulisana visokopećna troska, pritisna čvrstoća, XRF analiza, zaštita okoline.*