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HIGH MECHANICAL STRENGTH-GEOPOLYMER CONCRETE BASED ON COAL FLY ASH AND GROUND RECYCLED RESIDUAL GLASS ADDED IN THE FINE AGGREGATE

BY

LUCIAN PĂUNESCU[1,*], BOGDAN-VALENTIN PĂUNESCU[2] and ENIKÖ VOLCEANOV[3,4]

[1]Cosfel Actual SRL Bucharest, Romania
[2]Consitrans SA Bucharest, Romania
[3]Faculty of Science and Materials Engineering, University “Politehnica” of Bucharest, Romania
[4]Metallurgical Research Institute SA Bucharest, Romania

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Abstract. Fly ash-geopolymer concrete using ground glass waste as a partial substitute for the fine aggregate was experimentally made in order to increase the mechanical strength of geopolymer. Using the method recently patented by the French Davidovits for activating the geopolymerization reaction of alumino-silicate materials in a highly alkaline liquid medium, this material type was produced in a gel state, poured into the mold and subjected to the steam curing process at 80ºC, followed by keeping at room temperature for 28 and 90 days respectively, before determining its characteristics. Additionally and simultaneously, constituting the work originality, the fine aggregate of the mixture was partially replaced up to 15% with ground glass waste. At the end of the curing process, the investigation of specimen characteristics highlighted the increase of compressive strength up to 62.7 MPa, i.e. 8.05% higher compared to the reference sample made without replacing the fine aggregate with glass.

Keywords: geopolymer concrete, fly ash, recycled glass waste, geopolymerization, alkaline activator.

*Corresponding author; *e-mail*: lucianpaunescu16@gmail.com

1. Introduction

Recently, the geopolymer concrete was proposed as a new environmentally friendly construction material (Abdul Aleem and Arumairaj, 2021) as a result of excluding the cement from the composition of concrete, the manufacture of which involving very high emissions of greenhouse gases (mainly carbon dioxide) and also excessively high consumption of fossil fuels (Moya *et al.*, 2010). The binder role of concrete in this case belongs to natural alumino-silicate materials (kaolin, metakaolin, rice husk ash, volcanic rock powder, etc.) or residual similar materials originating as industrial by-products (coal fly ash, granulated blast furnace slag, red mud, mining tailing, etc.). This type of cementitious materials with high pozzolanic properties used in the mixture for manufacturing the concrete as substitutes for cement contributes to the improvement of its mechanical properties and durability as well as the workability of the fresh material (Zhang and Malhotra, 1995). In terms of quality, the transformation of alumino-silicate materials into geopolymer with remarkable properties takes place following the geopolymerization reaction favoured by a highly alkaline liquid medium consisting of sodium hydroxide ((NaOH) and sodium silicate (Na_2SiO_3) (Davidovits, 1991). The geopolymerization process is a fast, very complex process. Silicon and aluminium minerals result in "three-dimensional polymeric chain and ring structure consisting of Si-O-Al-O bonds" (Davidovits, 1994). The complete understanding of this process is still incomplete and additional research is needed (Zhang and Malhotra, 1995; Rangan, 2008a).

Several geopolymer concretes were experimentally made using fly ash as residual alumino-silicate material. According to (Assi *et al.*, 2017), fly ash-geopolymer concrete is an excellent solution for valorizing residual materials and at the same time significantly reduces CO_2 emissions during the concrete manufacturing. The properties of geopolymer concrete compared to those of ordinary concrete include the following peculiarities: higher resistance to heat, resistance to all inorganic solvents, higher resistance to compression, water resistance, high working life before stiffening, non-toxic, stable at room temperature. Based on the experimental results of the characterization of geopolymer concrete, the compressive strength of geopolymer is very high by comparing with ordinary concrete made with Portland cement (of about 1.5 times). Also, the early strength of the geopolymer concrete is very high. Determining the workability of geopolymer concrete indicated values comparable to those of Portland cement concrete (Abdul Aleem and Arumairaj, 2021).

According to the paper (Guo *et al.*, 2010), geopolymer concrete was produced with fly ash and alkaline activator composed of NaOH and Na_2SiO_3 with the SiO_2/Na_2O molar ratio of 1.5. The weight proportion of Na_2O in fly ash composition was 10%. The compressive strength of the optimal geopolymer

specimen reached 63.4 MPa after the curing process at 75ºC for 8 hours followed by curing at room temperature for 28 days.

In another reference (Lloyd and Rangan, 2010), a fly ash-based geopolymer concrete activated in sodium silicate and sodium hydroxide solution (Na_2SiO_3/NaOH ratio of 2.5) is presented. The SiO_2/Na_2O ratio was 2. Fine sand (24.6 wt. %) and coarse aggregate (57.4 wt. %) completed the solid composition of the mixture. A naphthalene-based superplasticizer was added. The curing process of geopolymer concrete was carried out at 60ºC for 7 days. The compressive strength reached high values of 60 MPa determined after 1 day and 82 MPa after 4 days.

Results of studies on the experimental manufacture of fly ash-geopolymer concrete are presented in (Rangan, 2008b). Low-calcium fly ash (calcium below 5 wt. %) was preferred as alumino-silicate raw material, because the high proportion of calcium can alter the geopolymer microstructure and disturb the geopolymerization reaction. The particle size of fly ash was 80% below 50 μm. The amount of fly ash was 408 $kg \cdot m^{-3}$. Fine sand and coarse aggregate that characterize the composition of ordinary concrete were also used in the manufacturing process of geopolymer concrete. The quantities of fine and coarse aggregate were: 554 and 1294 $kg \cdot m^{-3}$, respectively. The alkaline activator was a combination of Na_2SiO_3 and NaOH 8M solution. The quantities of the activator components were 103 and 41 $kg \cdot m^{-3}$, respectively. A very low amount (6 $kg \cdot m^{-3}$) of superplasticizer was added. In the variant of using NaOH 14M, an addition of water (22.5 $kg \cdot m^{-3}$) was necessary. The fresh material produced by mixing the components of mixture (for 2.5-17 min) poured into molds was subjected to the curing process with steam at 60ºC for 24 hours followed by curing at room temperature for 21 days leading to obtaining compressive strengths between 38-53 MPa (depending on the mixing duration).

Another concrete fly ash-geopolymer was prepared by the well-known method of the French inventor Davidovits, consisting in the activation of the geopolymerization reaction of fly ash in aqueous solution of NaOH (8M and 14M concentration) and Na_2SiO_3 (Hardjito *et al.*, 2004). The adopted Na_2SiO_3/NaOH ratio was 0.4 and 2.5, respectively. The curing process at 60ºC for 24 hours continued at room temperature for up to 7 days led to obtaining compressive strength values of maximum 67.6 MPa.

Another residual material whose influence on the properties of construction concrete has been tested and shown in the literature (Afshinnia, 2019) is recycled glass waste. In the ground state, the glass represents a natural pozzolan contributing to improving the characteristics of fresh and cured concrete: workability, mechanical strength, resistance to the freeze-thaw cycle, resistance to sulfate. The pozzolanic quality of glass powder allows its use as a partial substitute for cement, but in this paper, cement is completely removed from the concrete composition, being replaced with geopolymeric materials.

Research on the use of residual glass as an aggregate in the concrete composition has been carried out in the last decades. Malik *et al.*, 2013 partially replaced (up to 30%) the fine aggregate (sand) in the concrete composition with waste glass with a particle size between 0.1-1.18 mm. As a result, the concrete samples had higher compressive strength after 28 days of curing, were cheaper, and environmentally friendly.

Tests to study the effect of replacing up to 30% of the fine sand aggregate with waste glass were carried out and are presented in (Ramana and Samdani, 2013). Experimental results indicated that compressive strength, tensile strength, and flexural strength increased corresponding to the replacement of fine aggregate with residual glass up to 15%, but decreased for further increasing replacement rates up to 30%.

According to the paper (Dabiri *et al.*, 2018), authors carried out research on the influence of replacing traditional fine aggregate with residual glass powder on the compressive strength and the concrete weight. Microsilica was added in low proportions to prevent the unwanted reaction between alkali and silica due to the high reactivity of aggregates containing glass (Afshinnia, 2019). The test results showed that by replacing the aggregate with glass particles, the compressive strength increased by over 30%. Instead, the concrete weight did not practically change. The optimal replacement rate of aggregate with waste glass was determined experimentally at 50%.

Other research (Kavyateja *et al.*, 2016) was carried out on the use of waste glass (particle size between 150 μm - 1 mm) as a partial substitute for sand in the fine aggregate of concrete manufactured with a water/cement ratio of 0.5. The replacement rates of fine aggregate with glass were varied between 0 - 40%. The curing process of fresh concrete poured in cube mold was initiated by keeping it in an oven for 24 hours, followed by removing the sample from the mold and performing curing by immersing it in the curing water tank. Determining the characteristics of the cured concrete was made at different time intervals between 3 - 90 days. According to the results, the compressive strength increased up to the replacement rate of 20% (reaching 46 MPa after 90 days), then suddenly decreased (to 35 - 37 MPa after 90 days) corresponding to the replacement rates of 30% and 40%, respectively. Experimentally, the tensile strength value decreased with increasing the glass content.

Glass waste has also been tested as a partial substitute for concrete coarse aggregate up to 50% (Ibrahim, 2020). The investigation of the concrete characteristics revealed that the density, water absorption, and consistency of fresh concrete decreased by up to 3.85 %, from 1.13 to 0.5 vol. %, and from 10 to 7.1 respectively, in the mentioned order. However, tensile and compressive strength increased with increasing the substitution rate up to 25%. The values of the two types of mechanical strength gradually increased up to about 15% compared to the reference concrete, after which they gradually decreased.

It should be mentioned that the use of waste glass as a partial substitute for aggregate presented above as information based on the literature refers to Portland cement concrete. The literature does not contain data on the substitution of glass waste for the aggregate of geopolymer concrete.

The objective of the current paper is to test the experimental manufacture of a geopolymer concrete based on low-calcium fly ash (class F), which also includes the partial replacement of sand from the fine aggregate with ground recycled glass waste. The aim of the research was to increase the mechanical strength of material obtainable by this method. The work originality consists in improving the production technique of fly ash-geopolymer concrete by the partial addition of glass waste in the composition of geopolymer fine aggregate.

2. Materials and Methods

Fly ash as an industrial by-product of the energy industry resulting from burning the coal in the boilers of thermal power plants and captured in electrofilters by purifying the emitted waste gases is an alumino-silicate material suitable for its use as a substitute for Portland cement in concrete manufacturing after its activation in a high alkaline medium. According to (Davidovits, 1994), the type of fly ash suitable for the geopolymerization process is class F, i.e. low-calcium fly ash according to the ASTM C 618-12 standard. This fly ash type is produced by burning anthracite (hard coal mines) or bituminous coal. Reserves of class F fly ash provided to the Romanian companies Daily Sourcing & Research SRL and Cosfel Actual SRL by Paroseni-thermal power plant 6-7 years ago, the period when this plant was operating with anthracite, were used by the authors in the current experiment.

The chemical composition of class F fly ash includes the following components:

Table 1

Chemical composition of class F fly ash (wt. %)

SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	TiO_2	Na_2O	K_2O	SO_3	LOI
54.4	26.5	4.8	3.5	2.5	1.5	0.4	0.6	1.7	2.3

The particle size of fly ash was below 35 μm after a supplementary grinding of the material provided by the plant.

The highly alkaline liquid medium required for the initiation and development of the geopolymerization reaction was created by mixing NaOH 10M dissolved in water and Na_2SiO_3 aqueous solution composed of 29.4% SiO_2, 14.7% Na_2O, and 55.9% water (Rangan, 2008b). NaOH was commercially purchased in the form of pellets and Na_2SiO_3 was purchased as an aqueous solution 38% concentration.

The fine aggregate was represented in the reference variant of quartz sand available on the market with grain size below 0.8 mm. The other experimental variants partially replaced the sand with recycled soda-lime glass waste in weight proportions of 5, 10, and 15%. Post-consumer colourless container glass was selected, washed, dried, broken, ground in a ball mill, and sieved, the grain size below 100 μm being chosen to be used in the experiment. Processing operations of recycled glass waste have been carried out in the Romanian company Bilmetal Industries SRL Popesti-Leordeni, Ilfov.

Coarse aggregate constituted of gravel had the majority granulometric fraction below 5 mm representing 63.3 wt. %, while the granulometric fractions between 5-10 mm and between 10-14 mm represented 24.2 wt. % and 12.5 wt. %, respectively.

The chemical composition of quartz sand, recycled colourless glass waste (Dragoescu *et al*., 2018), and gravel is shown in Table 2.

Table 2

Chemical composition of sand, colourless glass, and gravel (wt. %)

Material	SiO_2	Al_2O_3	CaO	MgO	Fe_2O_3	Na_2O	K_2O	SO_3
Quartz sand	98.8	0.77	0.01		0.05	0.22		-
Colourless glass	71.7	1.9	12.0	1.0	-	13.3	-	-
Gravel	87.5	6.1	0.28	0.03	1.62	2.08	-	0.06

The manufacture of geopolymer concrete involves some preparation rules. Thus, the alkaline activator consisting of NaOH dissolved in water and the aqueous solution of Na_2SiO_3 mixed for 4-5 min should be made in a separate container at least 24 hours before the preparation of the solid mixture. The optimal ratio between the two components was previously determined experimentally and has the value of 2.5. The mixture between fly ash as an alumino-silicate material with pozzolanic properties as well as fine and coarse aggregates was carried out by stirring for 5 min. After making the powder mixture, the liquid alkaline activator was slowly poured over the solid materials and the mixing continued for another 5 min until the gel was formed. The geopolymerization reaction had thus the conditions for transforming the alumino-silicate material into a geopolymer. Pouring the gel into a stainless steel cube mold and protecting it with thin plastic film were operations that preceded the initiation of the curing process. The first part of the process took place in an oven where steam blowing at 80ºC was carried out for 24 hours. The second stage meant the curing at room temperature (23 - 25ºC) for 48 hours in a thermally insulated enclosure under the conditions in which the concrete specimen was removed from the mold. After these three days of curing, the sample was free kept at room temperature until the moment of determining the characteristics of the specimen, i.e. maximum 90 days.

Four experimental variants were chosen based on the own experience (Paunescu *et al.*, 2023) combined with the information from the literature. The composition of these variants is shown in Table 3.

Table 3
Composition of geopolymer concrete variants

Composition ($kg \cdot m^{-3}$)	Variant 1	Variant 2	Variant 3	Variant 4
Fly ash	400	400	400	400
Quartz sand	550	522.5	495	467.5
Glass waste	-	27.5	55	82.5
Total fine aggregate	550	550	550	550
Coarse aggregate	1280	1280	1280	1280
NaOH 10M solution	40	40	40	40
Na_2SiO_3 solution	100	100	100	100
Water addition	20	20	20	20

As mentioned above, variant 1 (reference variant) was performed using a relatively common recipe for the manufacture of fly ash-geopolymer concrete without the addition of waste glass in the fine aggregate. In variants 2 - 4, the quartz sand was partially replaced with ground glass waste in weight proportions of 5, 10, and 15%, the total amount of fine aggregate being kept constant at 550 $kg \cdot m^{-3}$. Dosing the other solid materials (fly ash - 400 $kg \cdot m^{-3}$, and coarse aggregate - 1280 $kg \cdot m^{-3}$) as well as dosing the liquid alkaline activator composed of sodium hydroxide, sodium silicate, and water, i.e. 40:100:20 ($kg \cdot m^{-3}$) were kept constant in all tested variants.

The investigation methods adopted for measuring physical, mechanical, and microstructural characteristics of the four geopolymer concrete specimens are presented below. The density was determined taking into account the regular geometric shape of the product by weighing it with an electronic balance and relating this value to that of the sample volume (Metrology, 2015). Apparent porosity was calculated according to the ASTM C642-97 standard based on the results of dry, wet, and suspended specimen weights and reporting the difference between wet and dry weight to the difference between wet and suspended weight (Hui-Teng *et al.*, 2020). The method of immersing the concrete specimen under water for 24 hours (ASTM D570) was used to determine the level of water absorbed in the material mass and the 100 kN-compression fixture Wyoming Test Fixture allowed the identification of compressive strength of the tested concrete (Practical guide, 2018). The flexural strength measuring was performed according to SR EN ISO 1412:2000 (Curtu and Stanciu, 2011). Biological Microscope MT5000 model (1000 x magnification) was used for visualizing the microstructural aspect of geopolymer concrete samples.

3. Results and Discussion

Applying the investigation methods of geopolymer concrete specimens mentioned above led to determining their main physical and mechanical characteristics. Table 4 includes the experimental results after 28 and 90 days, respectively, of room temperature free curing process before measuring the sample characteristics.

Table 4

Main characteristics of geopolymer concrete specimens after the curing process

Variant	Density after 28/90 days ($kg \cdot m^{-3}$)	Apparent porosity after 28/90 days (%)	Compressive strength after 28/90 days (MPa)	Flexural strength after 28/90 days (MPa)	Water absorption after 28/90 days (vol. %)
1	2370/2389	22.7/22.5	45.0/58.4	12.3/12.9	12.1/12.1
2	2362/2379	23.0/22.8	46.4/61.3	12.5/13.1	10.8/10.8
3	2355/2370	23.2/23.1	47.0/62.2	12.6/13.1	7.9/8.0
4	2350/2363	23.3/23.2	47.4/62.7	12.7/13.2	4.0/4.1

The analysis of the data in Table 4 shows that the partial replacement of sand in the fine aggregate of geopolymer with waste glass (between 5 - 15 wt. %) leads to increasing the compressive strength after 90 days to 61.3 - 62.7 MPa compared to 58.4 MPa determined in the case of a fly ash-geopolymer concrete prepared without the addition of residual glass (variant 1). Extending the storage time of the specimen at room temperature from 28 to 90 days led to significant improvement of the compressive strength both in the reference variant (variant 1) from 45.0 to 58.4 MPa and for the beneficiary variants of replacing the sand with glass (variants 2 - 4) from 46.4 - 47.4 MPa to 61.3 - 62.7 MPa. This improvement of the mechanical strength by increasing the storage time is already well known, what is of interest in this research being the comparison between the strength values obtained by the addition of waste glass compared to those that characterize the reference variant. The result is increasing the compressive strength of variant 4 with 5.3% after 28 days and 7.4% after 90 days compared to variant 1.

The flexure strength registered much lower values between 12.3 - 12.7 MPa including variant 1 after 28 days and between 12.9 - 13.2 MPa after 90 days.

The geopolymer density decreased by the addition of glass from 2389 $kg \cdot m^{-3}$ (reference variant) to 2363 - 2379 $kg \cdot m^{-3}$ in variants 2 - 4 (in reverse order), while apparent porosity increased from 22.7% (reference variant) to 23.0 - 23.3% in variants 2 - 4 after 28 days and from 22.5% to 22.8 - 23.2% after 90 days. These changes in density and apparent porosity largely explain the tendency to improving the mechanical strength.

The influence of the partial replacement of sand with waste glass is evident in the case of water absorption. Its value decreased significantly from 12.1 vol. % (variant 1) to 4.0 - 10.8 vol. % (variants 2 - 4).

Appearance images of the four geopolymer concrete specimens are presented in Fig. 1 and the microstructural aspect of specimens is shown in Fig. 2.

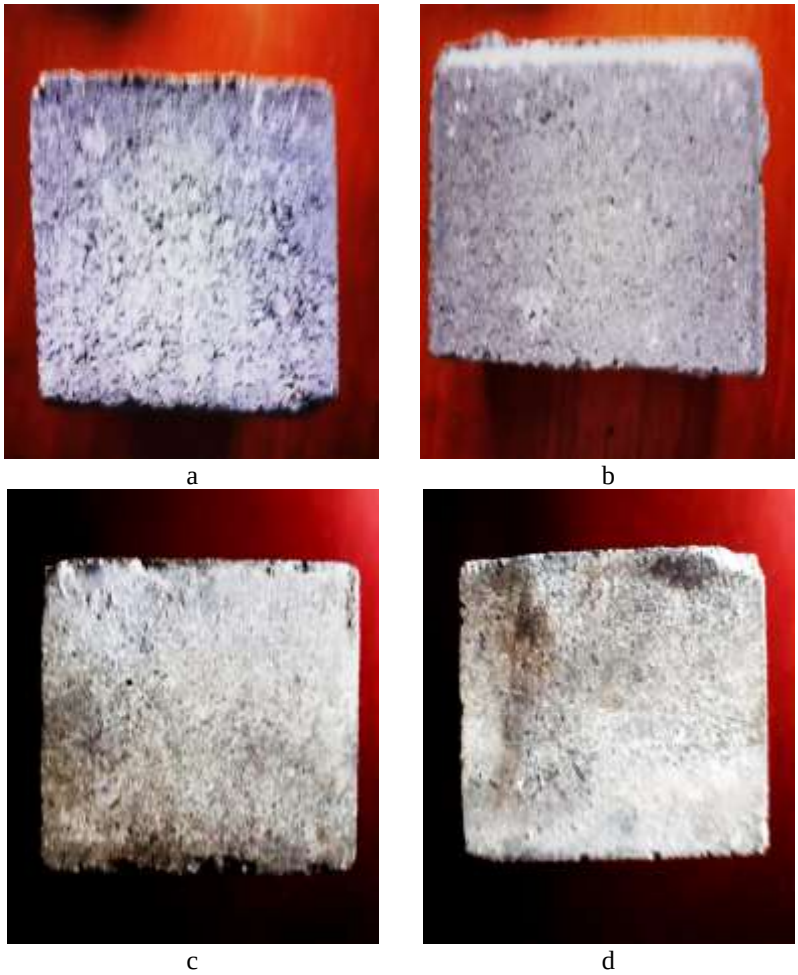

Fig. 1 – Appearance images of geopolymer concrete specimens
a – variant 1; b – variant 2; c – variant 3; d – variant 4.

A denser geopolymer concrete microstructure was observed especially in Fig. 2 (c and d) in the case of mixtures with the replacement of sand from the fine aggregate with ground glass waste (10 and 15%). A similar finding was also noted in the paper (Abdallah and Fan, 2014).

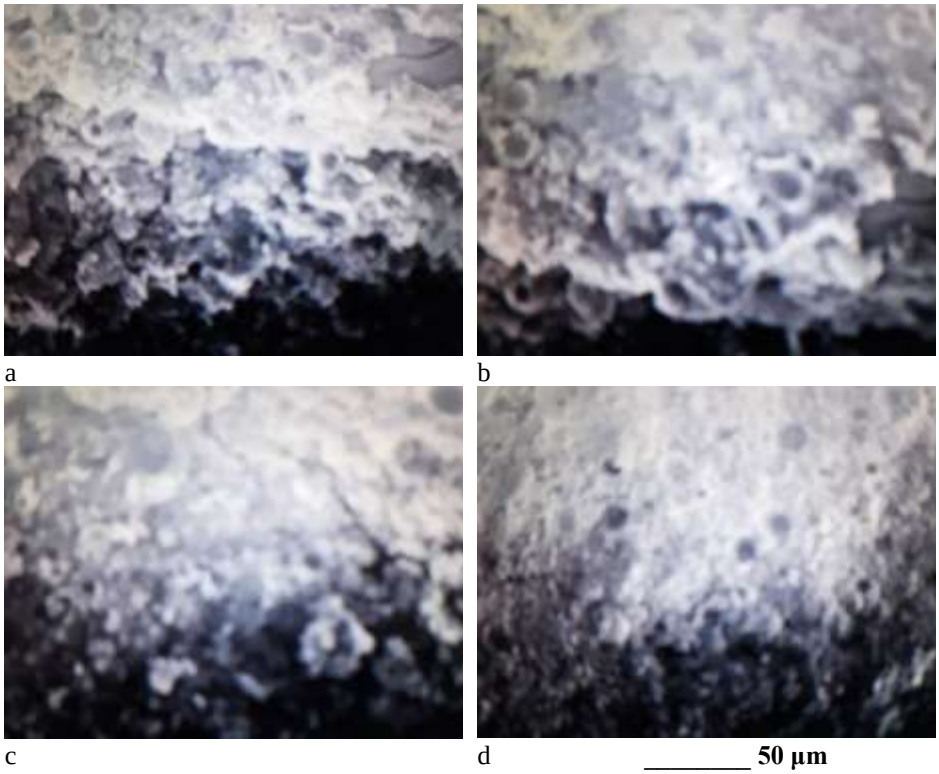

Fig. 2 – Microstructural aspect of geopolymer concrete specimens
a – variant 1; b – variant 2; c – variant 3; d – variant 4.

Considering the characteristics of fly ash-geopolymer concrete specimens with the partial replacement of sand from the fine aggregate with glass waste (variants 2 - 4), variant 4 was adopted as the optimal variant. The highest values of compressive strength (62.7 MPa) and flexural strength (13.2 MPa) were obtained after 90 days of curing in the case of replacing fine sand with 15% recycled colourless glass waste. The geopolymer density had the lowest value (2363 $kg \cdot m^{-3}$), apparent porosity had the highest value (23.2%), and water absorption was clearly the lowest of only 4.1 vol. %. In this experiment, the reference sample was performed with a similar manufacturing recipe except the change of fine aggregate with glass waste addition (variant 1) and the results were the comparison basis of its characteristics with those of the specimens manufactured with glass waste.

According to the literature (Malik *et al.*, 2013; Ramana and Samdani, 2013; Dabiri *et al*., 2018; Kavyateja *et al*., 2016), the partial replacement of the fine aggregate with waste glass is appropriate in variable maximum weight proportions between 15 - 30%, after which at higher proportions the effect on the

mechanical strength of the geopolymer concrete significantly decreases. For this reason, the current experiment tested the fly ash-geopolymer concrete manufacturing method combined with the partial replacement of the fine aggregate with waste glass, adopting the maximum limit of the replacement rate at 15%. Regarding the maximum level of mechanical strength value of the geopolymer, it is also variable depending to a large extent on parameters of the geopolymer concrete curing process. In general, the compressive strength value can exceed 60 MPa. From this point of view, the compressive strength value reached in this work (62.7 MPa) is at the level of performances reported in the literature.

4. Conclusions

The work aimed to improve the mechanical strength of fly ash-geopolymer concrete by partially replacing the fine aggregate (quartz sand) with ground recycled glass waste. The manufacturing technique used the method recently patented by the French researcher Davidovits for turning alumino-silicate materials (such as fly ash) into geopolymer by activating the geopolymerization reaction in a highly alkaline liquid medium consisting of NaOH and Na_2SiO_3 solutions. Simultaneously and additionally, constituting the work originality, the fine aggregate of the starting mixture was partially replaced up to 15 % with ground glass waste below 100 μm. The curing process with steam at 80 ºC of the solid and liquid materials mixture as a gel poured into the mold, continued with curing at room temperature for 28 and 90 days led to strengthening the geopolymer, whose compressive strength reached 62.7 MPa, being 8.05% higher compared to the reference specimen made without replacing the fine aggregate with waste glass. The compressive strength value reached in this work is at the level of performances reported in the literature. Except for the compressive strength, it was found that the density of geopolymer concrete decreased with increasing the glass content in the fine aggregate. Also, water absorption showed an obvious decrease with the increase of glass proportion and the microstructure of geopolymer became denser with the glass addition in aggregate.

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BETON GEOPOLIMERIC CU REZISTENȚĂ MECANICĂ ÎNALTĂ PE BAZĂ DE CENUȘĂ ZBURĂTOARE DE CĂRBUNE ȘI STICLĂ REZIDUALĂ RECICLATĂ MĂCINATĂ ADĂUGATĂ ÎN AGREGATUL FIN

(Rezumat)

Un beton geopolimeric pe bază de cenușă zburătoare utilizând deșeu de sticlă măcinată ca înlocuitor parțial al agregatului fin a fost fabricat experimental în scopul creșterii rezistenței mecanice a geopolimerului. Utilizând metoda recent brevetată a francezului Davidovits pentru activarea reacției de geopolimerizare a materialelor aluminosilicatice într-un mediu lichid înalt alcalin, acest tip de material a fost produs in stare de gel, turnat într-o matriță și supus procesului de întărire cu abur la 80ºC, urmat de păstrarea la temperatura camerei pentru 28 și respectiv, 90 zile înaintea determinării caracteristicilor sale. Suplimentar și simultan, constituind originalitatea lucrării, agregatul fin al amestecului a fost parțial înlocuit până la 15% cu deșeu de sticlă măcinată. La finalul procesului de întărire, investigarea caracteristicilor probei a evidențiat creșterea rezistenței la compresiune până la 62,7 MPa, adică cu 8,05% mai mare față de proba de referință fabricată fără înlocuirea agregatului fin cu sticlă.