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REDUCING THE CARBON FOOTPRINT BY MAKING GEOPOLYMER MATERIALS FOR CEMENT-FREE CONSTRUCTION BASED ON WASTE AND INDUSTRIAL BY-PRODUCTS

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ABSTRACT: High-strength cement-free geopolymer concrete with 62.9 MPa (after 28 curing days) was experimentally produced from blast furnace slag, recycled concrete, and metakaolin as alumina-silicate binders as well as silica fume as silica-rich ultra-fine powder. Combining slag with metakaolin and silica fume allowed to increase its proportion up to 58.1 wt. %, without affecting shrinkage and hardness. Residual concrete from demolition was also used for making the geopolymer concrete as coarse aggregate as well as alumina-silicate binder together with fly ash, but not with granulated slag, metakaolin, and silica fume, this constituting the paper originality.

KEYWORDS: geopolymer concrete, alumina-silicate binder, granulated slag, recycled concrete, metakaolin.

1. INTRODUCTION

The term "geopolymer" decisively imposed itself in the international scientific world in the first 20 years of the 21th century. According to Matsimbe et al. [1], the geopolymer is the result of the polycondensation reaction that generates a three-dimensional matrix of alumina-silicates. Complex structures of hydrated gel and polymerized networks are contained in binary geopolymer systems and favourably influence the flowability, strength, and durableness of these materials. Industrial by-products with high calcium content such as granulated slag, coal ash, and phosphogypsum introduced in mixtures for manufacturing geopolymers provide gels of the C-S-H and C-A-S-H types characterized by dense polymerized grids which improve resistance and setting durations.

According to [2], the geopolymer concept belongs to the French researcher J. Davidovits from 1991 [3] being used for alkali alumina-silicate binders. Large quantities of alumina-silicate materials (rich in alumina and silica) suitable for the manufacture of geopolymers are available in the world (such as: fly ash, metallurgical slag, metakaolin, clay, red mud, volcanic stone, etc.). Their preparing requires low energy consumption and the result of these operations means very low CO_2 emissions (169 kg $CO_2 \cdot m^{-3}$) compared to the industrial manufacture of common Portland cement that emits over 300 kg $CO_2 \cdot m^{-3}$ into the atmosphere [2]. In principle, the production of geopolymer involves mixing finely ground alumina-silicate materials with a liquid alkaline agent composed of Na_2SiO_3 or K_2SiO_3 and NaOH or KOH. Hardening the geopolymer at an early age is achieved very quickly, so that in about 4 hours it is possible to reach 70 % of the final value of the compressive strength [2].

Geopolymer concrete manufactured using alumino-silicate materials has higher durability compared to traditional concrete under similar curing conditions. Also, fly ash in the geopolymer concrete composition leads to superior chemical stability compared to the reference concrete [4].

The paper [5] considered that the new type of concrete brings several technical, environmental, and economic advantages, maintaining high mechanical strength, high durability, better workability, low permeability, higher resistance to acid, low shrinkage cracking, faster production process, lower curing duration, and cost reduction.

Li et al. [6] presented a manufacturing solution for geopolymer concrete using a binder composed of granulated slag, gypsum, and EAF (electric arc oven) slag resulting very good compression resistance after 28 days (more than 50 MPa).

The integral replacing of cement with fly ash in the mixture composition for manufacturing the geopolymer concrete was presented in the paper [7]. The solution composed of Na_2SiO_3 and NaOH (in a ratio of 1.5-2.5) contributed to the activation of the geopolymerization reaction. The hardening procedure carried out at 60-75 °C for half or one day facilitated the increase of the geopolymer compressive strength to over 50 MPa.

Using SiO_2/Na_2O report of 1.5 of the alkaline agent and 10 % for the report between Na_2O/fly ash, Guo et al. [8] obtained high values of compressive strength in the manufacture of a geopolymer based on coal ash. Curing the fresh concrete at 75 °C for 8 hours and storing it for 28 days allowed reaching 63.4 MPa.

The work [9] presented a review on the preparation of the geopolymer based on coal ash without the use of ordinary Portland cement. The composition of the alkaline activator included Na_2SiO_3 and NaOH with a molarity between 7-10 M. The ratio of activator components varied between 0.67-1. It was observed that by the increase of temperature, the geopolymerization reaction was more quickly and the geopolymer reached about 70 % of its final resistance in only 3-4 hours of curing. Thus, by hardening between 60-90 °C for 1-3 days, compression strength of up to 50 MPa could be obtained.

Other authors [10] presented effects of slag addition (below 20 %) into the mix preponderantly containing coal ash. The alkaline agent represented 35-40 % from the binder quantity and the ratio of activator components Na_2SiO_3/NaOH was within the limits of 1.5-2.5. The results of slag supplementation had favourable effects on flowability and mechanical resistance of the geopolymer concrete in case of performing the curing process at ambient temperature. Higher slag ratio and lower ratios between activator components led to increasing the strength, but to the slight reduction in workability.

Ramani and Chinnaraj [11] presented a manufacturing variant of the geopolymer utilizing the combination between slag and rice husk ash by completely replacing the cement. The rice husk ash ratio was experimentally enlarged to 30 %. The mixture contained geopolymer binders (390 $kg \cdot m^{-3}$), fine and coarse aggregates (total 1850 $kg \cdot m^{-3}$), alkaline agent (160 $kg \cdot m^{-3}$), superplasticizer (1.5 %), and water (60 $L \cdot m^{-3}$). The Na_2SiO_3/NaOH proportion was maintained constant during the experiment at 2.5. The highest value of compression resistance reached 62 MPa after 3 curing days, 68 MPa after 7 days, and 71 MPa after 28 days in the optimal version including 10 % rice husk. Over this value of rice husk ash, the compression strength decreased sharply.

Another combination between alumina-silicate wastes with the role of suitable replacement cementitious materials for Portland cement (coal ash and residual construction concrete recycled from demolitions) was used by authors of the current work for making the geopolymer concrete [12]. The waste activation by facilitating the geopolymerization reaction was made using the alkaline agent solution composed of Na_2SiO_3 and NaOH in 2.41 ratio. Fine and coarse aggregates were used as well as silica fume as a superplasticizer to help increase strength and keeping adequate workability. Experimental results showed obtaining high values of compressive strength reaching in the optimal version (with 383 $kg \cdot m^{-3}$ fly ash, 230 $kg \cdot m^{-3}$ concrete waste, and 38 $kg \cdot m^{-3}$ silica fume) 58.9 MPa after 28 days and 35.8 MPa after 7 days. Also, durableness resistance testing to acid, sulphate, and chloride showed excellent results.

The recent using the slag in the manufacture of geopolymer has shown that it exhibits good compression resistance at an early age [13]. Some disadvantages of using the slag have been identified: high shrinkage and low hardness. They can be eliminated by the combined use slag-metakaolin or metakaolin-fly ash-silica fume.

It was experimentally recovered that the addition of micro-silica in the initial mixture together with slag has the ability to develop the reactivity of blast furnace slag through the filling effect, favouring the increase in compressive strength [14].

Also, silica fume reduces bleeding and segregating the concrete (although this requires adequate curing), improves its resistance in chemically aggressive environments, and inhibits alkali-silica reactions that can cause severe damage to the concrete structure [15].

The current work aimed at the geopolymer concrete production using a by-product of the metallurgical industry (granulated slag), a recycled waste from construction demolition (residual concrete), alumina-silicate material (metakaolin) coming from the mineral kaolin, a secondary product of the manufacture of metallic silicon or silicon alloys in EAF metallurgical oven (silica fume), natural fine and coarse aggregates (river sand and natural granite gravel). Also, water-reducing additive (polycarboxylate ether) was chosen for improving the concrete durableness by lowering the water/cementitious proportion, reducing permeability, and improving flowability [16]. The alkaline activating agent adopted for the activation of alumina-silicate materials were Na_2SiO_3 and NaOH in aqueous solution.

2. MATERIALS AND METHODS

2.1 Materials

The solids used in this experiment were nominated above: ground granulated slag, residual concrete recycled from construction demolition, metakaolin, silica fume, sand and natural granite gravel as aggregates as well as polycarboxylate ether as water-reducing additive.

Granulated slag procured from ArcelorMittal Galati (Romania) is a secondary product of manufacturing the metallurgical pig iron, granulated through casting the molten product in a cold water-pool. The particle dimensions of slag is between 2-6 mm. For utilizing in the current experiment, the slag was ground for reducing its size under 100 µm. The oxide composition of the slag was the following: 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 [17].

The main components of the concrete composition recycled from demolition were 49.8 % SiO_2, 16.9 % CaO. 9.8 % Al_2O_3, and 7.6 % Fe_2O_3. The recovered concrete pieces were crushed and ground and the grain size selected after sieving was below 100 µm.

Metakaolin considered the most effective pozzolanic material usable in the manufacture of concrete, produced from mineral kaolin, is available on the market as a porous powder with a mean dimension under 20 µm, originating from China. Its chemical composition consists of 50-60 % SiO_2 and 30-40 % Al_2O_3 [18].

Silica fume as a by-product of metallurgy industry (Silica fume, 2023), in the form of an ultrafine amorphous silica-rich powder (particle size around 0.3 µm) was commercially procured originating from China. The chemical composition of silica fume contains 93.6 % SiO_2, 1.8 % CaO, 1.1 % MgO, 0.8 % Al_2O_3, 0.5 % Fe_2O_3, and 0.1 % Na_2O [19].

River sand with the grain size under 1.3 mm as fine aggregate as well as natural granite gravel with particle dimensions under 17 mm as coarse aggregate were also utilized in the mix for making the geopolymer concrete.

Polycarboxylate ether in form of powder commercially available (originally from India) was used for improving workability by reducing water/cementitious ratio [16].

Except for the solid components of the mixture, the liquid solution of the alkaline agent required for initiating the polymerization reaction was also included in the list of materials. Na_2SiO_3 (or water glass) was used together with NaOH dissolved in water as alkaline agent for the geopolymer production. Na_2SiO_3 is commercially available as a liquid solution with 38 % concentration. NaOH (or caustic soda) is available on the market in pellet form suitable for water-dissolving. The molarity of 12 M was adopted.

The materials were weighed, dosed, and mixed forming six experimental variants presented in Table 1.

Table 1. Components of experimental variants

Component	Version 1 ($kg·m^{-3}$)	Version 2 ($kg·m^{-3}$)	Version 3 ($kg·m^{-3}$)	Version 4 ($kg·m^{-3}$)	Version 5 ($kg·m^{-3}$)	Version 6 ($kg·m^{-3}$)
Ground granulated blast furnace slag	145	167	200	230	268	305
Residual concrete recycled from demolition	295	263	220	180	132	85
Metakaolin	50	60	70	80	90	100
Silica fume	35	35	35	35	35	35
Fine aggregate (under 1.3 mm)	560	560	560	560	560	560
Coarse aggregate (under 17 mm)	810	810	810	810	810	810
Na_2SiO_3	193	193	193	193	193	193
12 M NaOH	80	80	80	80	80	80
Polycarboxylate ether	3.5	3.5	3.5	3.4	3.4	3.4
Working water	220	220	220	220	220	220
Water/binder ratio	0.419	0.419	0.419	0.419	0.419	0.419

According to the data in Table 1, the main variables in the composition of prepared mixtures are the amounts of ground slag and residual concrete recycled from building decommissioning, and metakaolin. The total consumption of the three materials was 490 $kg·m^{-3}$, but each of them had values that varied within large limits between 145-

305 kg·m^{-3} (in the case of slag), between 85-295 kg·m^{-3} (in the case of concrete residual), and between 50-100 kg·m^{-3} (in the case of metakaolin). Silica fume, which completed the binder mixture, had a constant value (35 kg·m^{-3}) for all versions tested.

2.2 Methods

Geopolymer concrete manufacturing method consists of the activation of alumina-silicate raw materials through contact with the liquid solution of the alkaline agent. This innovative manufacturing method is the basis of the recent invention of the French researcher J. Davidovits. The alkaline solution facilitates starting and developing the polymerization chemical process, which turns alumina-silicate waste into a geopolymer with remarkable mechanical and physical properties, having the ability to replace cement-based concrete that has become ecologically and economically inappropriate [20]. According to [21], geopolymerization is a very complex chemical process which still requires the involvement of several scientific specialties for the complete understanding all stages of the conversion of alumina-silicates into geopolymers.

The small-scale making of the geopolymer concrete samples did not require specialized equipment of the type used in construction for dosing and mixing the mix components. These operations were performed in laboratory conditions without negatively influencing the quality of works. The fresh concrete casted into metallic moulds was inserted into a small sealed chamber to be subjected to the first hardening operation at 80 °C (with hot air) for one day, provided by an electric preheater. Then, the hardened specimens were released from the moulds and stored freely for 7 and respectively, 28 days before their characterization.

2.3 Characterization methods

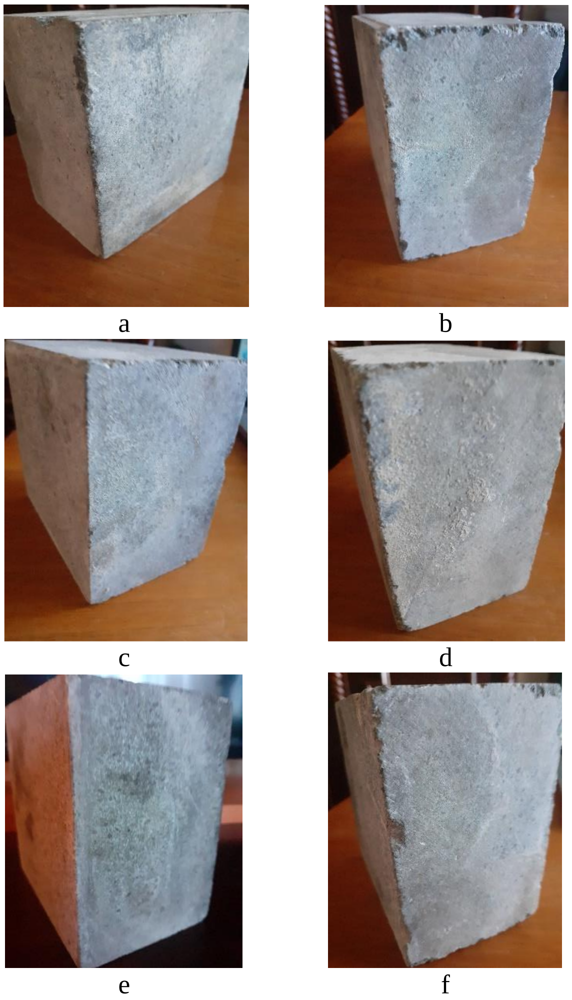
The main feature of the green geopolymer, workability, was measured utilizing the slump test equipment (ASTM C143-10). The gravimetric measuring technique [22] was used for determining the density value after the hardening period of 28 days. Water absorption was measured through the immersing method of specimens under water (ASTM D570) [23]. The compression resistance was identified with 100 kN-hydraulic axial press equipment (EN 826-2013), and the flexural strength was determined applying the third-point load test (ASTM C78). The resistance to acid, sulphate, and chloride was tested by immersing the samples in 5 % H_2SO_4 solution for 28 days, and 10 % NaCl solution for 28 days (ASTM C1898-20, SIA 262/1:2008, and CSN EN 12390-11:2015). Features at the microstructural scale of specimens were analyzed with the MT5000-Biological Microscope, 1000 x magnification.

3. RESULTS AND DISCUSSION

3.1 Results

The workability of the fresh geopolymer concrete tested with Abram's cone method, under the conditions of using the Na_2SiO_3/NaOH ratio of 2.41 and the NaOH molarity of 12 M, reached the slump value of 149 mm. According to [24], workability is negatively influenced by the increase of molarity in the 8 M-18 M range. Adopting the value of 12 M proved to be an appropriate choice for at least satisfactory workability of the fresh geopolymer.

Appearance of the hardened geopolymer concrete samples after 28 days is shown in Figure 1.



a b

c d

e f

Figure 1. Aspect of hardened geopolymer concrete specimens
a – version 1; b – version 2; c – version 3; d – version 4;
e – version 5; f – version 6.

Generally, the appearance of specimens is of compact materials, with high strength and durableness. The clear differentiation of the six experimental versions that led to obtaining the specimens in Figure 1 is difficult to achieve.

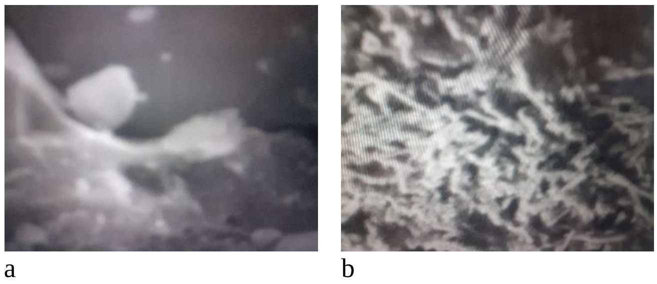
Investigating the mechanical, physical, and resistance to chemical attack features of cured geopolymer concrete specimens led to the data presented in Table 2.

Table 2. Characteristics of cured samples

Characteristic	**Variant**					
	1	2	3	4	5	6
Density (kg·m^{-3})	2336	2343	2349	2355	2360	2368
Water absorption (vol. %)	1.7	1.7	2.0	1.9	2.2	2.4
Compression resistance (MPa)						
- after 7 days	35.0	35.5	35.9	37.2	38.6	40.1
- after 28 days	41.5	46.0	50.2	54.3	58.5	62.9
Flexural resistance (MPa)						
- after 7 days	3.3	3.8	4.2	4.7	5.2	5.8
- after 28 days	7.1	8.0	8.9	9.7	10.5	11.4
Durableness resistance to:						
- acid	93.0	92.7	92.8	92.0	91.0	92.4
- sulphate	93.7	94.0	94.3	95.0	95.5	95.3
- chloride	90.3	90.7	91.2	90.4	90.8	90.0

According to the data presented in Table 2, density of geopolymer concrete had the tendency to increase its values with the increase of the slag/residual concrete ratio, reaching 2368 kg·m^{-3} in version 6 (for a ratio between the two wastes of 4.76). Water absorption was kept within reduced limits (in the range of 1.7-2.4 vol. %) and at a normal level compared to other geopolymer concretes. Compression strength reached high limits of its values, especially after storing the specimens for 28 days (between 41.5-62.9 MPa), the influence of the blast furnace slag content being important for reaching the maximum value. The same conclusion is also valid in the case of storing the specimens for 7 days, the data of compression resistance being within the limits of 35.0-40.1 MPa. Also, flexural resistance reached high levels of 11.4 MPa (for storage of 28 days) and 5.8 MPa (for storage of 7 days). Durableness resistance in the acid, sulphate, and chloride environment highlighted the ability of geopolymer concrete on its resistance to chemical attack.

Microstructural aspect of samples is shown in Figure 2.



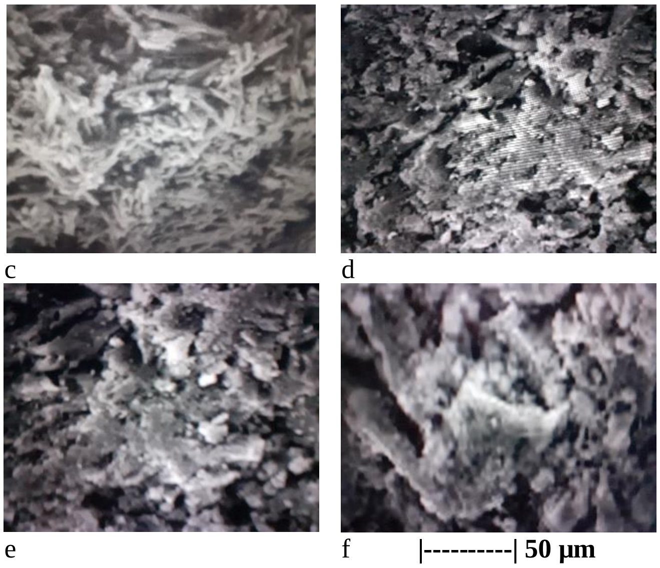


Figure 2. Microstructural aspect of cured geopolymer concrete specimens
a – variant 1; b – variant 2; c – variant 3; d – variant 4;
e – variant 5; f – variant 6.

The influence of increasing the slag ratio in the mix of experimental variants 1-6 becomes more and more distinct in images of Figure 2. The more loaded matrix of the concrete specimens due to the presence of slag in increasing amounts has the role of improving their mechanical properties. This mechanical improvement was also noted by measuring the compressive strength, whose values are indicated in Table 2.

3.2 Discussion

Metallurgical slag used in the mix for making the geopolymer concrete experimentally proved that it can contribute to the increase of compression resistance, but simultaneously favours growing the shrinkage and the decrease of hardness. Metakaolin and silica fume added together the slag showed ability in eliminating these disadvantages. Thus, the group of materials with the role of binder composed of slag, residual concrete coming from demolition, metakaolin, and silica fume, in different ratios, was adopted in this experiment with the aim of obtaining high compressive strength, maintaining suitable properties for the workability of geopolymer concrete. Polycarboxylate ether as a known water-reducing additive was also adopted to increase the geopolymer durability by reducing the water/binder ratio (0.419), reducing permeability as well as improving the workability (149 mm by the slump test).

Among the six versions tested, the most appropriate for the experiment objectives was version 6, in which the four used binders had the following weight proportions: 58.1 wt. % blast furnace slag, 16.2 wt. % residual concrete, 19.0 % metakaolin, and 6.7 wt. % silica fume.

The work originality was the simultaneous use of slag, residual concrete, and metakaolin as alumino-silicate binders with cementitious properties as well as silica fume as a silica-rich nanomaterial. The weight proportion of blast furnace slag could thus be increased up to 58.1 % due to its combination with metakaolin and silica fume, without affecting the shrinkage and hardness of the geopolymer concrete. The residual concrete coming from building demolition was previously used as coarse aggregate for preparing concrete (Rahal, 2007; Etxeberria *et al.*, 2007), but not as an alumino-silicate binder.

4. CONCLUSIONS

The use of granulated slag together with recycled concrete from building dismantling as alumina-silicate binders for making geopolymer concrete without coal fly ash contribution was the objective of the experiment presented in the current paper. The starting mixture also included metakaolin and silica fume for possible increasing the slag proportion beyond the limits at which shrinkage and hardness are affected, reaching the highest value of 58.1 wt. %. The recycled concrete was not used as a coarse aggregate as in other previous works, but properly processed as a cementitious material with the role of binder and this constituted the main element of the paper originality. The workability improvement was achieved by using a water-reducing additive (polycarboxylate ether). The fresh material poured into the mould was hardened with warm air at 80 ℃ for 1 day and after releasing from the mould it was stored freely for 7 and 28 days. The investigation data of the cured geopolymer features showed that by growing the slag proportion, compression resistance increased up to 62.9 MPa (after 28 days), while the flexural resistance increased up to 11.4 MPa (after 28 days). The resistance of geopolymer concrete to chemical products attack highlighted excellent durableness values.

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