HIGH STRENGTH-GEOPOLYMER BUILDING MATERIAL

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Abstract: New geopolymer concrete with high mechanical strength - 58.9 MPa after 28 days of curing - was experimentally made under environmental friendly and economic conditions. The high-strength-geopolymer is baed on coal fly ash and building concrete waste as geopolymer materials suitable for completely substituting the cement in concrete structure. The alumino-silicate geopolymer materials with binder role were activated in liquid alkaline medium (sodium silicate and sodium hydroxide) for facilitating the polymerization reaction that turns the alumino-silicate wastes into geopolymer concrete. The use for the first time in this experiment of recycled building concrete waste from demolition is the work originality.

Keywords: geopolymer, fly ash, recycled concrete waste, alkaline activator, polymerization reaction, compressive strength.

1. INTRODUCTION

Alumino-silicate materials from industrial by-products and wastes represent a viable alternative to ordinary Portland cement as a concrete binder. Silica and alumina-rich, the geopolymers inserted in the concrete composition as substitutes for cement bring numerous advantages to this important construction material, improving workability, durability, mechanical strength, acid resistance, considerably reducing greenhouse gas emissions during the manufacturing process and lowering the price due to significant decreasing the energy consumption compared to cement-based concrete [1, 2].

The interest of researchers for the use of coal fly ash, blast furnace slag, rice husk, red mud, etc. in geopolymer concrete manufacturing processes appeared relatively recently. In the last decade of the last millennium, Davidovits, J. discovered the method of activating these by-products and wastes through the polymerization reaction, obtaining high strength concretes [3, 4]. Numerous works published in the last decade in the literature confirm this concern.

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Generally, coal fly ash and blast furnace slag are the main by-products used as cement substitutes in proportions of 15-20 and 30-40 wt. % respectively, of the cementitious material mixture. According to Kumar Metha et al. [5], over 50 % fly ash can be used, keeping cement consumption of maximum 200 $kg \cdot m^{-3}$.

Manufacturing recipe in which cement is completely replaced with fly ash and its activation for polymerization reaction is achieved with aqueous solution of sodium silicate and sodium hydroxide (in 1.5-2.5 weight ratio) is presented in paper of Sofi et al. [6]. The curing process at 60-75 ºC for 12-24 hours led to obtaining high compressive strength of over 50 MPa.

According to Malhotra [7], using typical range of component materials, different levels of high-performance concrete strength were reached. In the experiments carried out by the author, cement was used in low proportions (from 120-130 $kg \cdot m^{-3}$ to 100-120 $kg \cdot m^{-3}$) being partially substituted with fly ash. Constant amounts of aggregate (coarse and fine) being used in all cases and with decreasing water/cement ratio from 0.40-0.45 to 0.30-0.32, led to increasing the mechanical strength of concrete after 28 days of curing from 20 MPa up to 40 MPa. High-strength concretes needed the addition of a superplasticizer in order to obtain low water/cement ratio.

High compressive strength was obtained by making a coal fly ash-geopolymer concrete using an alkaline activator of sodium silicate solution and sodium hydroxide. It has been found that 1.5 molar ratio SiO_2/Na_2O of activator favours the increase of compressive strength and also, the 10 % mass ratio Na_2O/coal fly ash was suitable for the high-strength of the concrete. The highest compressive strength value of 63.4 MPa was reached by curing the fresh concrete at 75 ºC for 8 hours followed by curing at room temperature for 28 days [8].

Recent research on the influence of nanoparticle addition in the geopolymer composition has shown that compressive strength, bending strength and abrasion resistance as well as the workability can be improved by using silica and alumina nanoparticles [9]. The amount of nanoparticles related to the amount of cement is effective for properties of geopolymer concrete between 0.2-1.2 wt. % (for silica) and 0.5-4.0 wt. % (for alumina).

The experiment described by Adak et al. [10] was focused on the use of nano-silica in the fly ash-geopolymer mortar composition. Three molar concentrations (8, 10 and 12M, respectively) of the alkaline activator together with various proportions of nano-silica addition (up to 10 % of fly ash amount) were tested. The geopolymer mortar with the addition of 6 % nano-silica showed the highest improvement of compressive strength, flexural strength, and tensile strength under the conditions of curing at room temperature for 28 days. The cause of this improvement in mechanical properties was experimentally identified to be due to turning the amorphous compound into a crystalline compound.

The work of Mohd Mustafa Al Bakri et al. [11] presents information from the literature on the manufacturing techniques of geopolymer concretes without the use of cement. The binder role of cement in ordinary concrete is taken over by fly ash as the main geopolymer used in these processes. Fly ash has pozzolanic properties like the cement and is silica and alumina-rich. The alkaline solution that includes sodium silicate and sodium hydroxide is recommended to have a molarity of 7-10M. Sodium silicate is considered the most suitable alkaline activator because it contains silicon (dissolved and partially polymerized), which reacts easily, is incorporated into the reaction products and contributes to improving the characteristics of the mortar. According to the working method, the previously prepared alkaline solution is mixed with the binder and then with the aggregate (fine and coarse) in order to form fresh concrete. The components ratio of the alkaline solution Na_2SiO_3/NaOH falls within the range of 0.67-1. The molar concentration of NaOH (between 10-20 M) has a minor effect on the strength of geopolymer concrete. During the fresh concrete curing, the polymerization process takes place [4]. Increasing the process temperature, the polymerization becomes faster and the concrete gains about 70 % of its strength in 3-4 hours of curing. The compressive strength increases by curing at 60-90 ºC for 24-72 hours and can reach 40-50 MPa. Geopolymer concrete manufacturing with completely removed of cement leads to obtaining a material resistant to aggressive environments: acid and marine environments, high temperature and fire.

Experimental manufacture of geopolymer concrete without cement, but with a combination of coal fly ash with other geopolymer materials is also shown in the literature. Preparation of material mixture in which fly ash was replaced in proportions up to 30 wt. % with ground granulated blast furnace slag and the alkaline activator was composed of a solution of sodium silicate and sodium hydroxide mixed in the ratio 1.5 led to the manufacture of geopolymer concrete by curing in ambient conditions [12]. The compressive strength of the material reached

43.61 MPa and the bending strength 5.45 MPa. The curing process in ambient conditions showed the possibility of manufacturing the geopolymer concrete for in-situ applications.

Deb et al. [13] present results of granulated blast-furnace slag including in proportions up to 20 wt. % into the binder mixture predominantly represented by fly ash. The alkaline activator varied between 35-40 % of the amount of binder and the ratio Na_2SiO_3/NaOH had values within the limits 1.5-2.5. It was observed that the addition of blast furnace slag over fly ash had a significant favorable effect on the workability and on the strength of geopolymer under conditions of curing at ambient temperature. However, at higher proportions of slag and the lower ratio between Na_2SiO_3 and NaOH, with the strength increasing the workability is slightly reduced.

The effects of the partial substitution of fly ash with high MgO-blast furnace slag and the change of the SiO_2/Na_2O mass ratio of the alkaline activator (between 0.25-1.0) were analyzed in [14]. The results showed that increasing the fly ash content increased the initial setting time, but the effect on the final setting time was very low, although early age compressive strength was decreased. The fly ash increasing content had no effect on drying shrinkage, but lowered the autogeneous shrinkage.

Another combination of geopolymer materials (ground granulated blast furnace slag and rice husk ash) was used for the manufacture of geopolymer concrete [15] completely replacing the usual Portland cement. The proportion of rice husk ash in the mixture with the predominant blast furnace slag was gradually increased up to 30 wt. %. The mixture composition included the combination of blast furnace slag and rice hush ash geopolymers (394 $kg \cdot m^{-3}$), coarse and fine aggregate (1848 $kg \cdot m^{-3}$), alkaline activator (Na_2SiO_3 and NaOH) of 158 $kg \cdot m^{-3}$, superplasticiser (1.5 %) and water (59 L/m^3). The Na_2SiO_3/NaOH ratio was kept constant at 2.5. The compressive strength of geopolymer concrete reached the highest values (62.3 MPa at 3 days of curing, 67.6 MPa at 7 days, and 70.7 MPa at 28 days) in the variant with 10 wt. % rice husk ash. Above this limit, the compressive strength values strongly decreased.

The current work aimed at the experimental manufacture of a high-strength geopolymer concrete based on coal fly ash and building concrete waste recovered from building demolition as geopolymer alumino-silicate materials using also nano-silica addition for increasing the compressive strength. Improving the strength was significantly accentuated by the curing process with hot air at 80 ºC for 6 hours followed by curing at room temperature for 28 days. The work originality consists in the choice of building concrete waste as a geopolymer material used in mixture with coal fly ash activated with alkaline solution of Na_2SiO_3 and NaOH.

2. METHODS AND MATERIALS

2.1. Methods

The method adopted in this experiment is based on the complete replacement of the usual binder of traditional concrete (Portland cement) with industrial by-products and wastes rich in silica and alumina. These materials available in the world can play the role of binder in the structure of new concrete geopolymer, so that the quality of the building material is not affected. Obviously, the energy consumption for processing the raw material is incomparably lower compared to the consumption required for the industrial manufacture of cement. Also, the large amounts of greenhouse gases emitted into the atmosphere during the manufacture of cement are practically removed.

The method of turning alumino-silicate wastes into geopolymer concrete is based on the polymerization reaction of these wastes through alkaline activation with Na_2SiO_3 and NaOH solution. According to the literature [4], the polymerization reaction in an alkaline medium of minerals based on silica and alumina has as a result a three dimensional polymeric chain and ring structure consisting of Si-O-Al-O bonds. It should be mentioned that until now a clear mechanism regarding the setting and curing of the geopolymer concrete has not yet been accepted by researchers. However, in principle, the reaction mechanism could contain the following stages: the dissolution of silicon and aluminum atoms of raw material under the action of hydroxide ions, transport / orientation / condensation of precursor ions into monomers, and setting or polycondensation / polymerization of monomers into polymeric structures. It is not clear whether these stages can take place separately or simultaneously, overlapping each other.

The preparation of fresh geopolymer concrete was carried out in several stages. The alkaline activator composed of the aqueous solution of Na_2SiO_3 and NaOH dissolved in it was made 24 hours in advance. The liquid solution was then poured over the homogeneous mixture of fly ash, concrete waste, coarse and fine aggregate, and nano-silica. The mixture was stirred with VEVOR electric overhead stirrer mixer corrosion resistance laboratory (0-3000 rpm) at 1000 rpm for 3 min until slurry was formed. It was poured into a mold with removable walls and was inserted into a thermally insulated enclosure for hot air curing at 80 ºC for 6 hours. Then the second stage of the curing process was carried out by keeping the specimen removed from the mold at room temperature (18-25 ºC) for 28 days.

2.2. Materials

The binder used for the geopolymer manufacturing was composed by coal fly ash and recycled construction concrete waste from building demolition. The use of construction waste constitutes the work's originality. The two raw materials are rich in silica and alumina according to the data in Table 1. Coal fly ash as an industrial by-product was provided by the Paroseni-thermal power station (Romania) having the grain size below 200 μm. It was ground in a ball mill and sieved, the grain size below 40 μm being selected. Building concrete waste was recovered from the rubble of building demolition, broken, and ground in the ball mill to grain size below 100 μm.

Obtaining the geopolymer concrete composition is conditioned by the activation of alumino-silicate materials with an alkaline activator represented by an aqueous solution of sodium or potassium silicate and sodium or potassium hydroxide (solid in form of pellets) dissolved in the silicate solution. In this experiment, sodium silicate and hydroxide were used allowing to obtain a microstructure characterized by closed pores, unlike the geopolymers prepared by potassium-activation [16]. The ratio between the two components of the alkaline activator, recommended in the literature within limits of 1.5-2.5, was chosen at 2.41. Na_2SiO_3 CAS: 10213-79-3 Sigma-Aldrich was commercially purchased. NaOH in the form of pellets, easily soluble in water, was used from the material reserves of Cosfel Actual SRL.

Table 1. Oxide composition of solid materials.

Oxide composition	Coal fly ash (wt.%)	Concrete waste (wt.%)	Sand (wt.%)	Nano-silica (wt.%)
SiO_2	46.5	68.9	97.9	92.3
Al_2O_3	23.7	18.7	1.1	1.3
CaO	7.9	1.6	-	1.6
MgO	3.2	0.5	-	0.9
Fe_2O_3	8.6	6.5	0.5	1.0
Na_2O	6.0	0.2	-	0.25
K_2O	4.1	1.5	0.3	0.79
TiO_2	-	1.3	-	-
SO_3	-	-	-	0.11

The composition of starting materials also included the aggregate represented by river sand (coarse aggregate below 5 mm and fine aggregate below 1 mm). The oxide composition of sand is shown in Table 1.

Nano-silica also known as silica fume is a by-product of metal silicon or ferrosilicon alloy production in the iron and steel industry. Nano-silica is a very reactive pozzolanic material contributes to increasing the mechanical strength of geopolymer concrete [10]. This product is available on the market, with the maximum particle size being 100 nm. Its oxide composition can be also seen in Table 1.

2.3. Methods of characterization the geopolymer concrete specimens

Characterizing the geopolymer concrete specimens aimed at the following determinations: density, water absorption, thermal conductivity, compressive strength, durability test (acid, chloride, and sulphate resistance), microstructural configuration, and X-ray diffraction test (XRD). The density was measured using the gravimetric method [17] and the test of water absorption (BS 1881-122: 2011 and A1: 2020) was made by the specimen immersion in distilled water for 1 hour. Room temperature-thermal conductivity was determined with HFM 446 Lambda apparatus based on the heat-flow method (SR EN 1946-3:2004). Measuring the compressive strength

according to EN 826:2013 was performed with 10 kN-hydraulic axial press machine. The acid resistance test was carried out by immersing the specimens in 5 % solution of sulfuric and hydrochloric acid for 4 weeks (ASTM C1898-20), the sulphate resistance test was made by immersion in 10 % sodium sulphate for 4 weeks (SIA 262/1:2008, appendix D), and chloride resistance test was carried out by immersion in 10 % sodium chloride solution for 4 weeks (CSN EN 12390-11:2015). These tests consisted in comparing the new compressive strength values with the previous. The microstructural configuration of specimens was analyzed with the Biological Microscope MT5000 model with the captured image, 1000 x magnification. The XRD investigation (according to EN 13925-2: 2003) was made with an X-ray diffractometer Bruker-AXS D8 Advance with CuKα radiation.

3. RESULTS AND DISCUSSION

3.1. Results

Four geopolymer concrete compositions were designed to test their manufacture. According to Table 2, 881 $kg \cdot m^{-3}$ aggregate was mixed with the binder composed of coal fly ash (between 383-389 $kg \cdot m^{-3}$) and concrete waste (between 224-230 $kg \cdot m^{-3}$) which adds up to a constant amount of 613 $kg \cdot m^{-3}$ as well as the variable addition of nano-silica between 15-38 $kg \cdot m^{-3}$. Nano-silica had percentage values related to the amount of binder between 2.45-6.20 %. The total amount of solid mixture had values between 1509-1532 $kg \cdot m^{-3}$, decreasing from variant 1 to variant 4. The alkaline activator amount formed from the aqueous solution of Na_2SiO_3 and NaOH (12 M), their ratio being 2.41, had the constant value of 750 $kg \cdot m^{-3}$ representing 49.0-49.7 % of the total solid amount.

Table 2. Composition of the experimental variants.

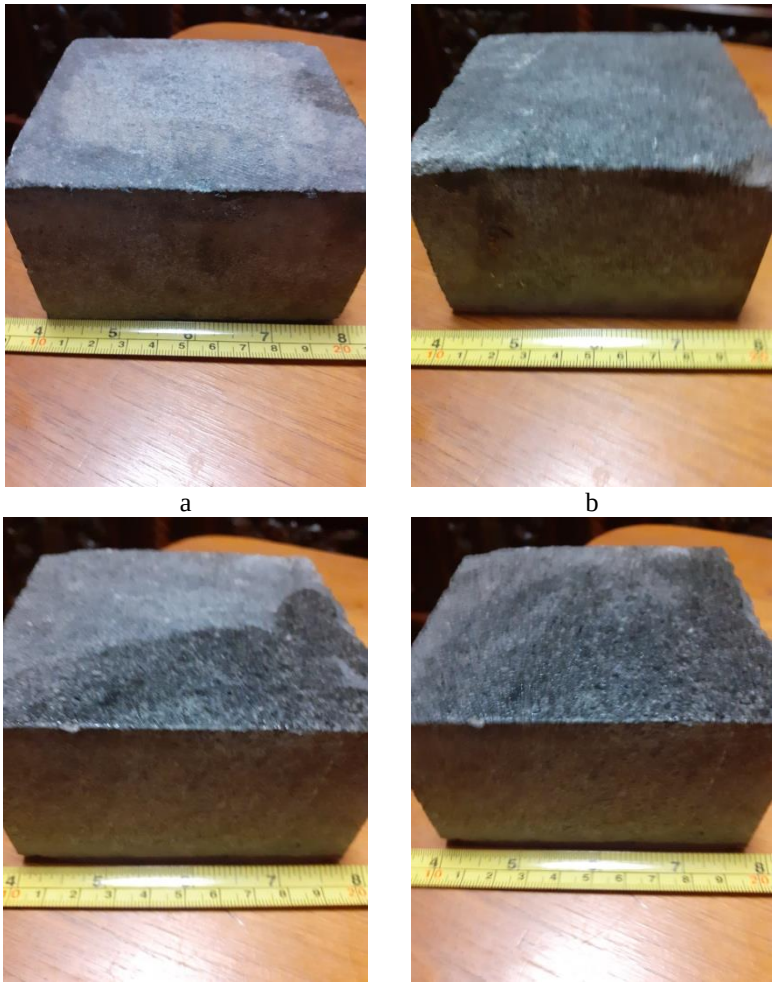
Material composition	Variant ($kg \cdot m^3$)			
	1	2	3	4
Aggregate				
- coarse (< 5 mm)	498	498	498	498
- fine(< 1 mm)	383	383	383	383
Coal fly ash (< 40 μm)	383	385	387	389
Concrete waste (< 100 μm)	230	228	226	224
Nano-silica (< 100 nm)	38	30	22	15
Total solid	1532	1524	1516	1509
Sodium silicate (38 % solution)	530	530	530	530
Sodium hydroxide	220	220	220	220
Total liquid	750	750	750	750
Total concrete	2282	2274	2266	2259

The fresh geopolymer concrete was poured into cubic molds with the size 100 x 100 x 100 mm for the curing process mentioned above. The appearance of cured specimens after 28 days is shown in Figure 1.

According to the characterization methods of the geopolymer specimens mentioned above, the determination of their peculiarities was carried out after finishing the curing process of the fresh concrete (28 days). In the case of compressive strength, measurements were also made after 7 days of curing. The results are presented in Table 3.

Analyzing the data presented in Table 3, the very high values of the compressive strength after 28 days of curing (between 41.7-58.9 MPa) can be noticed. Also, the intermediate measurements of the strength after 7 days indicate high values (between 27.3-35.8 MPa). Unlike other types of geopolymer concrete (geopolymer foams) recently made by Chithambar Ganesh et al. [18] which aimed to obtain porous structure by using a foaming agent (usually hydrogen peroxide) having a density within the limits of 400-500 $kg \cdot m^{-3}$, thermal conductivity between 0.080-0130 $W \cdot m^{-1}K^{-1}$, but compressive strength below 6 MPa, the geopolymers produced by the technique presented in this paper have a relatively high density (2260-2283 $kg \cdot m^{-3}$) and high thermal conductivity (1.35-1.93 $W \cdot m^{-1}K^{-1}$) which does not confers heat-insulating properties. Instead, water absorption is much lowered (below 1.2 vol. %) compared to geopolymer foams. The resistance tests of geopolymers against acid, sulphate, and chloride attack showed very good durability. The lowest loss of mechanical strength was

recorded at the attack with sulphate (93.5-95.2 %) and the highest at the attack with chloride (90.2-91.3 %), but generally, the losses are very low.



a b c d

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

Table 3. Characteristics of geopolymer concrete specimens.

Variant	Density ($kg \cdot m^{-3}$)	Water absorption (vol. %)	Thermal conductivity ($W \cdot m^{-1}K^{-1}$)	Compressive strength (MPa)		Durability resistance test (%)		
				After 7 days	After 28 days	Acid	Sulphate	Chloride
1	2283	1.2	1.93	35.8	58.9	92.7	93.5	90.6
2	2275	0.9	1.74	32.1	53.0	91.8	94.3	91.3
3	2267	0.7	1.47	29.8	47.3	92.0	95.0	90.7
4	2260	0.4	1.35	27.3	41.7	92.8	95.2	90.2

Images of the microstructural configuration of the analyzed specimens are shown in Figure 2.

According to the pictures in Figure 2, the most compact microstructure is that corresponding to variant 1, in which the highest proportion of nano-silica was used (6.20 % of the total geopolymer binder), whose role in increasing the mechanical strength of concrete is known. The other proportions of nano-silica used in variants 2, 3 and 4 were 4.89, 3.59, and 2.45 respectively, gradually decreasing the mechanical properties of the specimens.

On the other hand, the contribution and effectiveness of the curing process must be taken into account, contributing to the significant increase of the material strength as a result of the polymerization reaction. In the case of the experiment described in this paper, the parameters of the curing process were kept constant in all tested variants, so that the main difference between variants is explained by the variable proportion of the nano-silica addition.

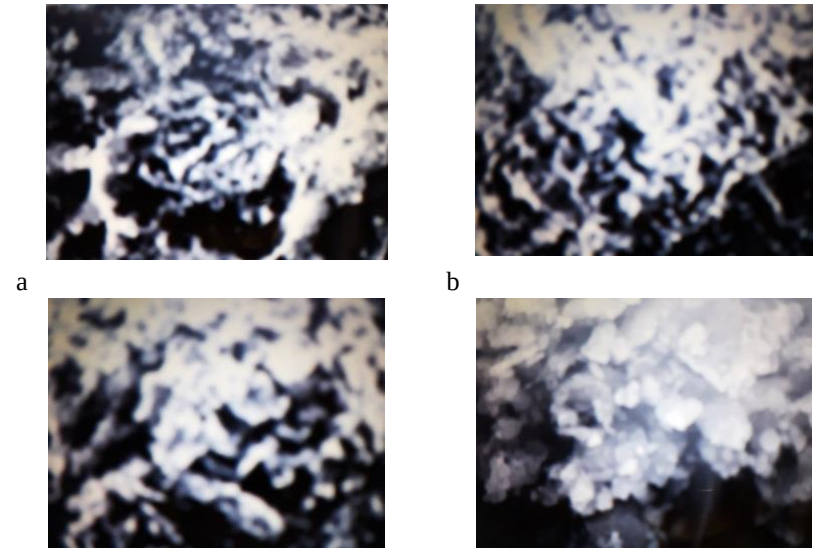


Fig. 2. Microstructural configuration of the geopolymer concrete specimens: a – variant 1; b – variant 2; c – variant 3; d – variant 4.

The used alumino-silicate materials are amorphous materials. During the geopolymerization process, a new phase also amorphous of geopolymer concrete is formed. Under these conditions, the crystalline phases in the geopolymer concrete specimens are insignificant. The only trace of the crystalline phase identified (hydroxy-sodalite, $Al_3HNa_4O_{13}S_3$) is due to the formation of crystalline zeolitic phases. Hydroxy-sodalite is a microporous crystalline alumino-silicate hydrophilic zeolite with small pore size. Its presence in the structure of analyzed materials is not significant.

Analyzing all the data related to the results of determining the characteristics of geopolymer concrete specimens, variant 1 was chosen as optimal variant.

3.2. Discussion

In the last 10 years, numerous geopolymer concrete variants were experimentally made including both geopolymer foams and high-strength geopolymers depending on the manufacturing method adopted.

The objective of the current work was the experimental production of geopolymer concrete with high compressive strength made in environmentally friendly and economic conditions. According to the literature, other works have used coal fly ash as a geopolymer material as a complete or partial substitute for cement. Coal fly ash available as an industrial by-product of the energy industry has often been associated with granulated blast furnace slag also an industrial by-product of the iron and steel industry. Cement, as a traditional binder of ordinary concrete, is considered guilty for excessive emissions of greenhouse gases into the atmosphere as well as for very high energy consumption (fossil fuel) under the current world conditions of hydrocarbon and ecological crisis (climatic overheating of the planet).

By their composition, alumino-silicate geopolymer materials have the requested qualities to be excellent concrete binders and adequate substitutes for cement. Except for coal fly ash and blast furnace slag, the list of possible geopolymer materials that can be used as binders in the manufacture of geopolymer concretes is longer, being also including rice husk ash, red mud, ash of incineration the municipal solid waste, etc. The present paper included recycled building concrete waste of construction demolition to be used together with coal fly ash.

The test results showed the feasibility of this association of geopolymer materials, with the proportion of recycled concrete waste in the mixture with fly ash being within the limits of 36.5-37.5 wt. %. The compressive strength of the concrete after 28 days of curing reached the maximum value of 58.9 MPa, being approximately similar to the values obtained in other experiments.

4. CONCLUSIONS

The present research aimed to manufacture a geopolymer concrete in which the traditional role of cement was taken by coal fly ash as a by-product of the energy industry and recycled concrete waste from the demolition of buildings. The activation of the two geopolymer materials was carried out with the usual alkaline activator containing Na_2SiO_3 solution and NaOH dissolved in aqueous solution. Additionally, low amounts of nano-silica were used with a recognized role in increasing the mechanical strength of geopolymer concrete. The fresh slurry resulting from stirring together the solid and liquid mixtures were subjected to the curing process into a mold in a thermally insulated enclosure under the influence of hot air at 80 ºC for 6 hours followed by curing at room temperature for 28 days. The manufacturing process allowed obtaining a maximum compressive strength of 58.9 MPa after 28 days and a maximum of 35.8 MPa after 7 days, performances corresponding to the use of the highest ratio of nano-silica of 6.20 % of the total geopolymer binder. The results obtained in this experiment were excellent both in terms of compressive strength and the durability of the material to acid, sulphate, and chloride attack as well as the low water absorption (below 1.2 vol. %). The originality of the work consisted in the use of recycled building concrete waste from demolition.

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