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Geopolymer concrete based on fly ash and slag using recycled aggregate from building demolition

Beton geopolimeric pe bază de cenușă zburătoare și zgură utilizând agregat reciclat din demolarea clădirilor

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Abstract. *A new version of the geopolymer concrete composition including recycled aggregate from the demolition of buildings as fine aggregate was tested in this experiment. Different weight proportions were tried together with the natural aggregate (river sand) and determinations of mechanical and physical characteristics of the geopolymer were carried out. Given that the main effect of the recycled aggregate addition was increasing compressive and flexural strengths, while the other characteristics decreased, the solution adopted was corresponding correlation of characteristic values to obtain a product with optimal properties (version 3): 2105 kg·m^{-3}-density, 4.3 %-water-absorbing, 54.9 MPa-compressive strength, 9.2 MPa-flexural strength, and 22.9 GPa-elasticity modulus.*

Key words: *geopolymer concrete, recycled aggregate, building demolition, granulated blast furnace slag, compressive strength.*

Rezumat. *O nouă variantă a compoziției betonului geopolimeric incluzând agregat reciclat din demolarea clădirilor a fost testată în acest experiment. Au fost încercate diferite proporții masice împreună cu agregatul natural (nisip de râu) și au fost efectuate determinări ale caracteristicilor mecanice și fizice ale geopolimerului. În condițiile în*

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care principalul efect al adăugării agregatului reciclat a fost creșterea rezistențelor la compresiune și încovoiere, în timp ce celelalte caracteristici au scăzut, soluția adoptată a fost corelația corespunzătoare a valorilor caracteristicilor pentru a obține un produs cu proprietăți optime (varianta 3): 2105 kg·m^{-3}-densitatea, 4,3 %-absorbția apei, 54,9 MPa-rezistența la compresiune, 9,2 MPa-rezistența la încovoiere, și 22,9 GPa-modulul de elasticitate.

Cuvinte cheie: beton geopolimeric, agregat reciclat, demolarea clădirii, zgură granulată de furnal, rezistența la compresiune.

1. Introduction

The new concept regarding the economical and environmentally friendly material type patented and developed by the French inventor J. Davidovits in the last decade of the 20th century has aroused the interest of manufacturers and users in the field of construction materials]1]. The new material called geopolymer is an amorphous semi-crystalline material formed by the geopolymerization reaction between alumino-silicate materials and an alkaline activator. The geopolymer precursors require to be rich in alumina and silica, playing a major role in the hardening process of the geopolymer as well as in formation of the N-A-S-H gel together with other elements ensuring the strength feature of the material [2]. The geopolymer formed in this way has the ability to replace the traditionally binder used in the manufacture of concrete (ordinary Portland cement) due to its pozzolanic properties as well as mechanical and durability ones. The processing of materials that make up the geopolymer requires low energy consumption and almost negligible emissions of greenhouse gases (carbon dioxide) [3].

According to the literature [4], coal fly ash is the main geopolymeric material, as a by-product of the energy industry, used as a substitute binder for cement in the construction concrete manufacturing process. Silicon and aluminum oxides from fly ash composition with low calcium content have the ability to react with the alkaline activator solution forming the geopolymer paste that binds together the fine and coarse aggregate as well as other un-reacted materials constituting the geopolymer concrete. Recent experiments using ground granulated blast furnace slag [5] have shown that geopolymers made with metallurgical slag exhibit good early-age mechanical resistance properties, especially compressive strength. However, some disadvantages of using the slag have been identified such as high shrinkage and low hardness. These disadvantages were overcome by the combined use of slag together with metakaolin or fly ash as well as the use of metakaolin together with fly ash and silica fume. Kim et al. [6] observed that adding silica fume as a precursor together with slag could improve the reactivity of blast furnace slag through the filling effect, leading to increasing the compressive strength.

An economic solution was recently tested in the process of manufacturing geopolymer concrete using concrete waste recycled from the demolition of buildings in the form of coarse aggregates [7, 8] due to their higher weight proportion in the concrete composition. The European Commission considers that construction and demolition waste represent 25-30 % of the total waste [9]. According to the data provided by Tam et al. [10], currently EU countries produce around 200 million tons of recycled aggregates, of which about 80 % are used for building construction and about 20 % for road construction. The decrease of mechanical features as a result of recycled aggregates application was improved by the addition of superplasticizers. The paper [11] aimed at the application of these recycled wastes in the form of fine aggregates replacing natural sand. The similar effect of decreasing the mechanical strength of concrete was counteracted by the use of superplasticizers.

Geng and Sun [12] have identified the particle surface of fine recycled concrete aggregate as being rough, with many pores, and microcracks, that differentiate this aggregate type from fine natural aggregate. According to [13], average values of recycled aggregate density are between 2.18-2.56 $g \cdot cm^{-3}$ and water-absorbing for 24 hours has average values of 9.9 vol. %. Water-absorbing ability is increasing in the case of lower sizes of the aggregate. Thus, the concrete made with fine recycled aggregate requires more mixing water compared to usual concrete, increasing the water/cement ratio. As a result, mechanical strength and durability decrease and shrinkage increases.

According to Pereira et al. [14], lower water/cement ratio improves compactness and mechanical features of concrete and an effective method is the use of superplasticizers. However, few works on concrete durability made with fine recycled concrete aggregate and superplasticizer are known. The concrete durability is characterized by carbonation and penetration of chloride ions.

The replacement in proportions of 30-100 % of fine natural aggregate with fine recycled aggregate in a reference concrete of 60 MPa as well as the use of a carboxylate-based superplasticizer and a water/cement ratio between 0.41-0.48 led to decreasing the compressive strength after 28 days by 3.7-7.6 % corresponding to the replacement proportions mentioned above and the increase of water-absorbing by immersion by 16.8-46 %. The carbonation depth and the chloride penetration coefficient increased by 40-110 % with the increase of the proportion of aggregate replacement [15, 16].

The experiment carried out by Cartuxo et al. [11] by replacing fine natural aggregate with fine recycled aggregate reached acceptable results by using a high-performance superplasticizer (SikaPlast 898) based on the combination of modified polycarboxylates in aqueous solution that works by electrostatic and respectively, by steric repulsions. The use of superplasticizer SikaPlast 898 decreased the following characteristics of cement-based concrete: water/cement ratio (up to 25 %), water absorption by immersion (up to 43 %), capillary water absorption (up to 66 %), carbonation depth at 91 days (up to 80 %), and the chloride migration coefficient (up to 46 %).

The work [17] is a review recently carried out by Thomas et al. in the field of using aggregates recycled from the construction demolition in the composition of geopolymer concrete. According to the conclusions of this study, the workability of geopolymer concrete is improved by increasing the quantity of recycled aggregates.

The compressive strength has higher values compared to cement-based concrete and is improved by increasing the addition of blast furnace slag. The curing process of the geopolymer concrete with embedded recycled aggregate having slag in the binder composition is advantageous if the process temperature is ambient, while in the case of the binder containing fly ash a higher temperature (below 100 °C) is appropriate, leading to increasing mechanical properties. The tendency to decrease the compressive strength was observed in the case of geopolymer concrete with coarse recycled aggregate due to the higher water absorption compared to coarse natural aggregate, but also due to the inferior properties of the recycled aggregate. The microstructural characteristics are superior to those of the reference concrete. The abrasion resistance of the geopolymer made by mixing fly ash with 5-15 % Portland cement and metakaolin is higher. Water absorption and sorptivity of geopolymer concrete are increased by increasing the proportion of recycled aggregate. The resistance of concrete to the attack of sulfuric acid is very high. Regarding the penetration of chlorides through the geopolymer concrete containing recycled aggregates, the resistance level is acceptable within certain limits. From the point of view of authors, the replacing problem of traditional natural aggregates with aggregates recovered from the building demolition is at a stage where scientific research should continue to improve knowledge in order to industrially apply the optimal technical solution.

Tan et al. [18] presented in their work an effective method of increasing the mechanical strength of geopolymer concrete by introducing into its composition the ground mixture of recycled aggregate from demolition and metallurgical slag. Both the compressive strength and the flexural strength of the geopolymer concrete have increased. According to the reported results, the compressive strength could reach 70 MPa after 7 days.

The authors' team of the current paper has recently made several experimental versions of geopolymer concrete [19-22] using different alumino-silicate wastes as a substitute for Portland cement, but until now they have not tried use recycled residual concrete from building demolitions as a substitute for fine natural aggregate. In the work presented below, fly ash and ground granulated blast furnace slag were chosen as alumino-silicate industrial by-products for the complete replacement of ordinary Portland cement. Recycled construction concrete from the building demolition was recovered to be used as fine aggregate of the geopolymer concrete after its mechanical processing in order to obtain by crushing-grinding the particle size range below 3 mm. Under the conditions of dwindling sand reserves worldwide [23], the approach to other sources, such as the case of residual concrete from demolitions, is justified. To compensate for the increased water-absorbing capacity of the recycled aggregate compared to the natural one, a suitable additive (polycarboxylate ether) was used.

2. Methods and materials

The adopted method is based on the complete replacement of the traditional concrete binder (Portland cement) with alumino-silicate industrial by-products: coal fly ash and granulated furnace slag having adequate cementitious properties to replace cement as a binder. The purpose of adopting these materials rich in alumina and silica, existing in significant quantities in the world industry, is the significant reduction of fossil fuel consumption and implicitly, of greenhouse gas emissions into the atmosphere compared to the cement manufacturing process.

The method of transforming alumino-silicate materials into geopolymers is based on the geopolymerization reaction, which is the essence of Davidovits' invention. The reaction, activated in an alkaline environment, leads to the formation of three-dimensional structure of the polymer chain and the ring formed by Si-O-Al-O bonds. Although the reaction mechanism has not yet been accepted by all specialists, in principle it could include the following stages: the dissolution of silicon and aluminum atoms under the action of hydroxide ions from the alkaline solution, the transport and condensation of precursor ions into monomers, and the polymerization of monomers into polymer structures [24].

The fresh geopolymer concrete preparation involves several stages. First, the alkaline activator containing sodium silicate (Na_2SiO_3) solution and sodium hydroxide (NaOH) in the form of solid pellets dissolved in water is separately prepared. The mixture of the liquid solution is then poured over the mixture of solid materials (fly ash and ground granulated blast furnace slag), the two components being mixed for 3 min until the paste is formed. The fine aggregate obtained by grinding the recycled concrete from the demolition, coarse aggregate consisting of natural gravel with dimensions below 18 mm, the additive (polycarboxylate ether) as a fine powder, and supplementary water were added over the paste. After a new mixing for about 5 min, the fresh geopolymer concrete was poured into metal molds and introduced into a sealed room for the curing treatment with hot air at 80 °C for 24 hours. After the completion of the mentioned treatment, the hardened specimens were removed from molds and stored for 7 and 28 days before testing their physical-mechanical features.

The materials used in this experiment were: coal fly ash and granulated blast furnace slag as binder completely replacing Portland cement, solution of Na_2SiO_3 and NaOH in the form of pellets soluble in water composing the alkaline activator, ground recycled concrete from demolition and natural sand as fine aggregate (below 2.7 mm), natural gravel (below 18 mm) as coarse aggregate, and polycarboxylate ether as a water-reducing additive.

Class F-fly ash was the type of ash purchased over 7 years ago and stored at Daily Sourcing & Research Bucharest, from Paroseni-Thermal power plant (Romania). The ash captured in electro filters of the plant after the fine purification of waste gases resulting from anthracite burning had grain size below 200 μm and required additional mechanical processing (grinding) to reduce the size below 40 μm. The chemical composition of fly ash with low CaO content (3.5 %) suitable for the production of geopolymer is shown in Table 1.

Granulated blast furnace slag was purchased from ArcelorMittal Galati (Romania) about 10 years ago and stored at the Metallurgical Research Institute Bucharest. The slag was taken in the form of granules between 2-6 mm and was ground to sizes below 80 μm. Its chemical composition is presented in Table 1.

Table 1

Chemical composition of fly ash and slag (wt. %)

Material	SiO_2	Al_2O_3	TiO_2	Fe_2O_3	CaO	MgO	Na_2O	K_2O	SO_3
Class F fly ash	54.4	26.5	1.5	4.8	3.5	2.5	0.4	0.6	1.7
Granulated blast furnace slag	36.4	11.6	-	0.8	41.8	5.8	0.3	0.4	-

Alkaline activator solution was composed of an aqueous solution of Na_2SiO_3 (38 % concentration) and NaOH pellets dissolved in water (12 M molarity). Alkaline activator components were commercially purchased.

Fine aggregate of geopolymer concrete was represented by natural sand (river sand) and recycled residual building concrete from demolition. River sand had dimensions in the range of 1.2-2.7 mm and his chemical composition mainly contained SiO_2 (97.5 %). Waste building concrete was arbitrarily selected from a demolition site. The chemical composition determining of the selected specimen was carried out in Metallurgical Research Institute on the X-ray fluorescence spectrometer. The main components of the concrete were 49.8 % SiO_2, 16.9 % CaO, 9.8 % Al_2O_3, and 7.6 % Fe_2O_3. The sample was crushed and ground, the particle size selected for the experiment being below 3 mm.

The coarse aggregate of geopolymer concrete was represented by natural gravel with grain size under 18 mm. Its oxide composition included 55.9 % SiO_2, 18.0 % CaO, 4.2 % Al_2O_3, and 3.0 % Fe_2O_3.

Polycarboxylate ether (in form of fine powder) originally from India was chosen as a water-reducing additive.

Four experimental variants were adopted in this experiment for making geopolymer concrete using fine recycled aggregate from building demolition, being presented in Table 2.

Table 2

Composition of geopolymer concrete variants

Composition	Version ($kg \cdot m^{-3}$)			
	No. 1	No. 2	No. 3	No. 4
Class F-fly ash	380	330	290	225
Ground granulated blast furnace slag	70	120	160	225
Na_2SiO_3	214	214	214	214
12 M NaOH	89	89	89	89
Fine aggregate - recycled concrete aggregate (below 3 mm) - natural sand (1.2-2.7 mm)	 - 930	 140 790	 350 580	 550 380

Composition	Version ($kg \cdot m^{-3}$)			
	No. 1	No. 2	No. 3	No. 4
Coarse aggregate (granite gravel) (below 18 mm)	920	920	920	920
Polycarboxylate ether	3.2	3.3	3.5	3.5
Water	132	132	132	132

Table 2 includes different ratios between the binder components of mixture (fly ash and slag). Thus, the ash/slag ratio has values within the limits of 1.00-5.43, the weight proportion of ground granulated blast furnace slag increasing from 15.6 % (version 1) to 50 % (version 4).

The main interest element of this experiment was changing the ratio between the components of the fine aggregate of geopolymer: recycled concrete aggregate and natural sand. Version 1 contained only natural sand (930 $kg \cdot m^{-3}$), while version 4 changed this ratio in favour of recycled aggregate (550 $kg \cdot m^{-3}$) compared to natural sand (380 $kg \cdot m^{-3}$). The coarse aggregate (granite gravel) did not influence this experiment having adopted a constant value of 920 $kg \cdot m^{-3}$. As mentioned above, polycarboxylate ether was added into the geopolymer composition having the role to reduce the water content required by recycled concrete aggregate used as a fine aggregate. The components of the alkaline activator (Na_2SiO_3 and NaOH) were adopted in the ratio 2.40 and the Na_2SiO_3/binder ratio was chosen at 0.48 [25].

The investigation of geopolymer concrete characteristics was carried out in two directions: one regarding the workability parameters of fresh concrete and another regarding the physical, mechanical, and microstructural features of hardened concrete. The workability of fresh concrete was determined using Abram's cone (EN 12350-2: 2006). The density was measured by the Archimedes' method using the water-intrusion method (ASTM D792-20). The water absorption was determined by the method of immersing the sample under water (ASTM D570). Compressive strength was examined using the method contained in EN 12390-3: 2001 and flexural strength was measured applying the three-point bend test on the specimen (SR EN ISO 14125: 2000). Modulus of elasticity was determined in conformity with ASTM C469-02e1. The microstructural peculiarities of geopolymer concrete specimens were analyzed with Biological Microscope model MT5000 with image captured, 1000 x magnification.

3. Results and discussion

Abram's cone test for slump flow indicated a good level of workability of the fresh geopolymer concrete, the slump flow values falling within the limits of 180-215 mm, according to the data in Table 3.

Table 3

Values of Abram's cone test for slump flow

Version	No. 1	No. 2	No. 3	No. 4
Slump flow (mm)	215	206	193	180

Generally, fresh mixes of geopolymer concrete have good viscosity and cohesion properties. By increasing the amount of blast furnace slag in the binder composition, the tendency to reduce these properties values dwindles. The explanation of Thomas et al. [17] for this evolution is the high amount of liquefied calcium ions of the slag and its fast reaction with the alkaline activator forming calcium silicate hydrate, that precipitates. Recycled concrete aggregate, having an inherent mortar content on its surface, absorbs water in larger quantities compared to natural aggregate during mixing, thus reducing the workability of geopolymer concrete.

The following stage of determining the specimen features referred to the mechanical, physical, and microstructural characteristics of geopolymer concrete made after the curing process of fresh concrete poured into molds, followed by the storage of specimens removed from the molds for 7 and 28 days. The density, water absorption ability, compressive strength, flexural strength, modulus of elasticity, and microstructural peculiarities of the four geopolymer concrete specimens have been investigated. Results are presented in Table 4.

Table 4

Mechanical and physical characteristics of geopolymer concrete specimens

Characteristic	Version			
	No. 1	No. 2	No. 3	No. 4
Density ($kg \cdot m^{-3}$)	2320	2216	2105	2030
Water absorption (vol. %)	3.8	4.0	4.3	4.5
Compressive strength (MPa) - after 7 days - after 28 days	 31.0 44.5	 34.7 49.8	 38.9 54.9	 43.1 63.2
Flexural strength (MPa) - after 7 days - after 28 days	 3.5 7.2	 3.9 8.5	 4.3 9.2	 5.9 11.2
Modulus of elasticity (GPa)	34.8	28.3	22.9	16.8

Analyzing the data in Table 4, it is found that measuring density results of geopolymer concrete samples incorporating recycled concrete waste from demolitions as a fine aggregate indicate significant decreases with increasing this waste proportion in the fine aggregate composition up to 59.1 % (2030 $kg \cdot m^{-3}$) compared to 2320 $kg \cdot m^{-3}$ obtained in the version using only natural fine aggregate.

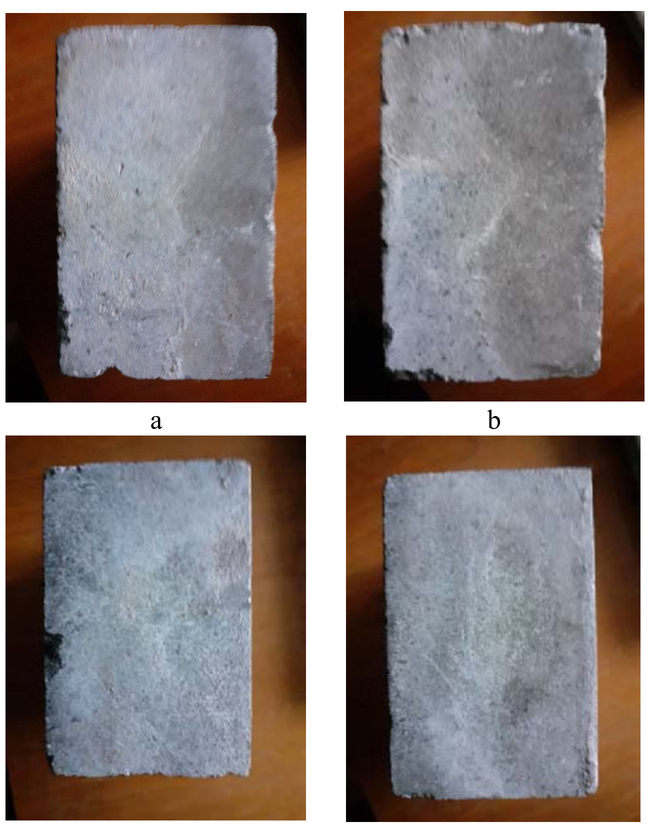
It was experimentally found that the use of recycled aggregate from demolitions has higher water absorption due to the cracks created during the recycling process, thus affecting the workability of the fresh geopolymer. On the other hand, the partial replacement of fly ash with blast furnace slag has the opposite effect, reducing the water absorption. Table 4 practically shows the combined result of the two opposite effects, observing a slight increase in absorbing water in versions with higher inclusion of recycled aggregate. Thus, water absorption increased from 3.8 vol. % (version 1) to 4.5 vol. % (version 4).

Compressive strength of geopolymer concrete with embedded recycled aggregate as well as using fly ash and especially, blast furnace slag as complete substitute for cement as a binder proved to increase significantly, reaching very high values after 28 days (up to 63.2 MPa) and also after 7 days (up to 43.1 MPa).

In principle, flexural strength is negatively influenced by embedding recycled aggregate in fine aggregate composition. However, the addition of more than 30 % blast furnace slag in the binder mixture contributed to the significant improvement of the strength under the conditions of high proportions of recycled aggregate of at least 50 %. According to Table 4, the maximum flexural strength values reached 11.2 MPa after 28 days and respectively, 5.9 MPa after 7 days in version 4 (with 59.1 % recycled aggregate and 50 % blast furnace slag).

Determining the modulus of elasticity of geopolymer concrete with embedded recycled aggregate highlighted a sharp decrease of its value with the increase of recycled aggregate weight proportion up to 59.1 %. Thus, the modulus of elasticity decreased from 34.8 GPa (version 1) up to 16.8 GPa (version 4).

Images of geopolymer concrete specimens with embedded recycled aggregate made in this experiment are shown in Fig. 1.



a b

c d

Fig. 1. Geopolymer concrete specimens with embedded recycled aggregate
a – version 1; b – version 2; c – version 3; d – version 4.

All four specimens shown in Fig. 1 have compact and homogeneous surfaces, typical for construction materials with high mechanical strength.

Pictures representing microstructural peculiarities of geopolymer concrete specimens with embedded recycled aggregate are shown in Fig. 2.

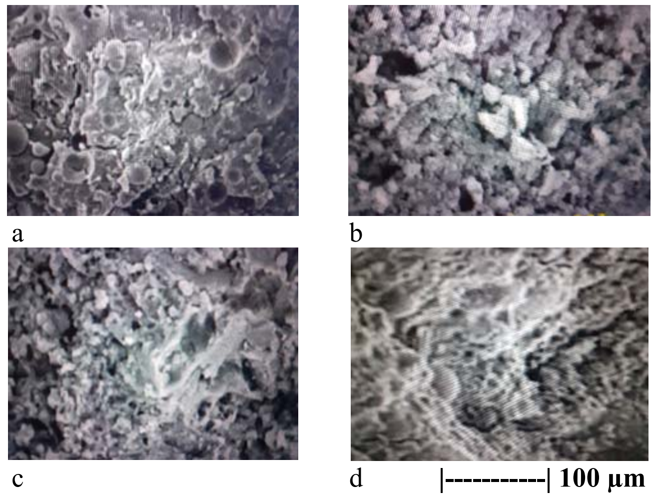


Fig. 2. Microstructural peculiarities of geopolymer concrete specimens with embedded recycled aggregate
a – version 1; b – version 2; c – version 3; d – version 4.

The spherical shape of the fly ash granules is visible in the first two pictures (a and b) of Fig. 2, then it fades in (c), so that picture (d) shows a predominantly typical structure containing metallurgical slag [26].

The comparative analysis of the geopolymer concrete specimens incorporating recycled aggregate made in this experiment requires the investigation of the limits of their characteristics influenced by the proportions of the recycled aggregate compared to the natural one in the geopolymer concrete composition. The testing of the mechanical and physical properties of specimens incorporating between 0-59.1 % recycled aggregate in the fine aggregate composition identified the decrease in the geopolymer density, the slight increase in water absorption, the significant increase in the compressive strength, the increase in flexural strength, especially at proportions above 50 %, and the sharp decrease of modulus of elasticity. Also, the slump flow test showed a slight tendency to decrease the geopolymer workability.

Choosing the optimal experimental variant by correlating the appropriate characteristics as a whole led to the determination of version 3 as the best of the four made versions. Features of this version include the density of 2105 kg·m^{-3}, water absorption of 4.3 vol. %, compressive strength of 38.9 MPa after 7 days and 54.9 MPa after 28 days, flexural strength of 4.3 MPa after 7 days and 9.2 MPa after 28 days as well as modulus of elasticity of 22.9 GPa.

4. Conclusions

The paper aimed at the manufacture of geopolymer concrete based on fly ash and ground granulated blast furnace slag using different additions of recycled concrete waste from the demolition of buildings in the form of aggregate together with natural fine aggregate (sand). The interest of researchers for the valorization of waste resulting from the demolition of buildings as recycled aggregate under the conditions of its application in the geopolymer concrete is relatively recent, as long as the geopolymer itself is a recent invention. Until now, the world research in this field has not reached optimal results that are unanimously recognized. The research team of the current work has recently carried out several researches on making geopolymer concretes having original contributions on the binder mixture from different alumino-silicate wastes, but the combination of binder solutions with those of geopolymer fine aggregate is made for the first time. The originality of the work consists in the correlation of the excellent features obtained for compressive strength (maximum 63.2 MPa) and flexural strength (maximum 11.2 MPa) with other decreasing properties such as density, water-absorbing, and modulus of elasticity, so that the overall performance of geopolymer to be valuable.

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