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NONCONVENTIONAL PROCEDURE OF MORE INTENSE VALORIZATION OF ALUMINA-SILICATE WASTE FOR PREPARING NEW GEOPOLYMERIC MATERIALS SUITABLE IN CONSTRUCTION

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ABSTRACT: A new and original dosing of alumina-silicate wastes in preparing mixture of the geopolymeric material is presented in this paper. Unlike the usual preponderant utilization of fly ash and ground blast furnace slag, the article adopted a major proportion of concrete waste from building demolition compared to the other two mentioned components, aiming at obtaining high strength geopolymers. The result of applying this new dosing in the material mixture activated with alkaline solution was increasing the compression and flexural resistance of the geopolymer, especially at earl age of curing (7 days). The experiment showed that by mixing 310-470 $kg \cdot m^{-3}$ of concrete waste from demolition, 230-254 $kg \cdot m^{-3}$ of fly ash, 102-120 $kg \cdot m^{-3}$ of blast furnace slag, and 4 $kg \cdot m^{-3}$ of silica fume, the best performing mechanical characteristics of the geopolymer can be obtained. The optimal values of denseness were between 1.64-2.04 $g \cdot cm^{-3}$ and those of heat conductance were between 0.218-0.306 $W \cdot m^{-1} \cdot K^{-1}$.

KEYWORDS: alumina-silicate, geopolymer, concrete waste, alkaline activator, compression resistance, earl age.

1. INTRODUCTION

The method of activating residual materials with alkali has been found since the middle of the 20th century and was considered as a promising solution for replacing or reducing Portland cement in order to reduce CO_2 emissions in the atmosphere during the industrial manufacturing process. Through this original technique, the principal phase comes from the silicate or alumina-silicate components of waste as a result of the chemical reaction with alkali metals. According to [1], the reaction leads to the formation of a Na_2O-Al_2O_3-SiO_2-H_2O type system. Except for the consistent reduction of the amount of CO_2 emissions, the tendency to recycle solid alumina-silicate waste (intensively exhibited in the last decades) can reduce the environmental problems caused by landfills, which contaminate the atmospheric air and underground water. It has been experimentally observed that CO_2 emissions can thus be reduced by at least 43 % compared to the situation of using Portland cement [2]. Also, the resistance to early age of materials manufactured by alkali activation was improved and their temperature resistance increased [1].

By using alkali-activation technology, alumina-silicate-based solid wastes, such as granulated blast furnace slag, fly ash, red mud, and different bio-ashes, can be transformed into alternative cementitious materials to Portland cement for reducing the carbon footprint of the building and keeping activities of concrete structures [1].

Other alternative inorganic industrial wastes, except those mentioned above, have also been tested for the manufacture of alkali-activated binders. In the work [3], metallurgical, energy, mining, construction-demolition, chemical, ceramic, or agro-industry wastes corresponded to the intended purpose.

The activators used in activating process of alumina-silicate materials are grouped into two main categories: caustic alkaline products (NaOH and KOH) [4, 5] and respectively, alkaline-earth products [$Ca(OH)_2$ and $Mg(OH)_2$] [6, 7]. Also, other weak acid salts (such as Na_2CO_3, K_2CO_3, Na_3PO_4, K_3PO_4, Na-COOH, K-COOH, etc.) can be applied as activators. Water glass (Na_2SiO_3) is well known as a very effective activator [8].

According to the literature, several associations of alumina-silicate materials with different activators have been tested. Thus, metakaolin and rice husk ash were activated with NaOH or KOH [4, 5], ground blast furnace slag was associated with NaOH and Na_2SiO_3 [7], acetylene sludge and fly ash were activated with $Ca(OH)_2$ [9], and blast furnace slag was chemically treated with Na_2SiO_3, NaOH, and Na_2CO_3 [8].

In the last decades, the exceptional scientific contribution of the French scientist J. Davidovits had a decisive role in the transformation of alumina-silicate wastes into special polymers called geopolymers. They are characterized by better physio-mechanical and chemical peculiarities compared to similar commercially existing construction materials [10]. A wide variety of new materials have been made due to the geopolymerization reaction of inert materials rich in silica and alumina activated in an alkaline environment [11]. The mentioned geopolymerization reaction carried out in an alkaline liquid environment is, according to the experts' opinion, a complex reaction developed in several stages, not necessarily successive ones. The generation of geopolymer involves dissolution and hydrolysis processes, completed with condensation in the system shown above, the level of silicon and sodium concentrations being essential in the condensation process.

Several factors influence the reaction between alkali and alumina-silicate materials (such as: chemical composition of basic materials, temperature and curing method, type of activators [12-14]. According to the literature, in a system with high content of calcium (or magnesium), the product resulted by hydration, including calcium (magnesium) alumina-silicate hydrates, with a layered structure, is known as C-A-S-H system. On the other hand, a system with low calcium (magnesium) content forms a tetrahedral structure in the form of a three-dimensional gel. These structures are influenced by the silicon/aluminum ratio and the reaction medium [15].

According to [16], the manufacture of fly ash-based geopolymer concrete using the geopolymerization reaction in an alkaline environment was experimentally proven to be favoured by the reduced content of calcium oxide (CaO) of less than 3 wt. %, the total content of SiO_2 and Al_2O_3 of about 80 wt. % with Si/Al ratio of about 2. As an alkaline activator, the combination of Na_2SiO_3 solution and NaOH dissolved in deionized water was recommended.

The paper [17] analyzed the influence of use high temperatures (up to 95 °C) in the curing process on the phase composition, microstructure, and strength of geopolymeric materials based on Class F fly ash under conditions of applying the alkaline activator composed of sodium silicate and sodium hydroxide solution. Adopting relative high temperatures in the range 75-95 °C of the material thermal treatment during the curing process determined significant increasing its compression strength even reaching 60 MPa.

Recently, the Romanian team that authored the current work carried out several experiments on the production of geopolymeric materials with heat-insulating properties by methods including the use of aqueous alkaline environment. Geopolymer foam with heat-insulating features was produced by expanding alumina-silicate mix including fly ash and clay brick waste in alkaline activator environment composed of water glass and NaOH in aqueous solution. Other components of the mix were expanded perlite, quartz sand, and olive oil (as a surfactant). The principal features of the geopolymer foam were low density, low heat conductivity, and relatively high compression resistance [18].

Porous material based on residual bottle was non-conventionally made with anthracite as a pore-feeder agent utilizing the oxidizing atmosphere of the oven. Water glass as a protective agent for carbon particles and manganese dioxide as a pore-forming agent had the main role in obtaining cellular glass with remarkable physio-mechanical, thermal, and microstructural properties. The optimal product was made by heating to 790 °C, using 1 % anthracite, 11.5 % water glass, 1.8 % MnO_2, and 5 % water. The apparent density of the product was low (0.21 $g \cdot cm^{-3}$) and also heat conductivity had a low value (0.039 $W \cdot m^{-1} \cdot K^{-1}$) [19].

Applying very efficient high-rate microwave heating (nearly 20 $°C \cdot min^{-1}$), cellular glass was manufactured from residual flat glass (90 %), using NaOH (4 %) in deionized water as a foaming agent, and borax (6 %) as a flow agent. The product density was 0.32 $g \cdot cm^{-3}$, heat conductivity of 0.06 $W \cdot m^{-1} \cdot K^{-1}$, and compression strength of 3 MPa [20].

Metakaolin-based geopolymer composite with addition of refractory filler wastes (super-aluminous concrete and silica fire bricks) recycled from demolition of a warming oven. Applying the alkaline activation technique, heating to 90 °C, and storing the samples for 28 days allowed preserving the mechanical stability of the geopolymer composite. The loss of compressive and flexural strength after exposure to 900 °C compared to the reference specimen was considered acceptable for use under conditions where high temperature stability is required [21].

A new method of manufacturing strength and porous cellular glass with load-bearing properties from recycled flat glass utilizing at the same time, two expanding agents: glycerol (liquid) and eggshell (solid), applying the non-conventional method of microwave heating. The results of this experiment showed that a cellular glass with low bulk density (0.16-0.19 $g \cdot cm^{-3}$) and high compression strength (up to 7.6 MPa) was prepared [22].

Excellent strength cement-free geopolymer concrete (62.9 MPa) after 28 days of curing was obtained from ground blast furnace slag, recycled concrete from building demolition, and metakaolin (as alumina-silicate binders). Silica fume as silica-rich fine powder was also added. Mixing these ingredients and the application of the mentioned preparation method (temperature, storage time) allowed obtaining the geopolymer concrete [23].

Using a higher than usual weight proportion of recycled concrete from building demolition, together with blast furnace slag as a by-product of the metallurgical industry, coal fly ash as a by-product of the energy industry as well as low proportion of silica fume, an ultrafine siliceous powder, constituted the principle recipe experimentally tried in the current work, aiming at the production of an environmentally friendly material with excellent mechanical resistance as an alternative to the traditional construction materials available on the market.

2. METHODS AND MATERIALS

2.1 Methods

The method adopted in this article is based on the principle of transforming the alumina-silicate materials available in the world, most often in the form of industrial by-products and waste, activated under the influence of alkaline solutions with an activating role, which favour the development of the geopolymerization reaction and the formation of geopolymers. The method of generating them as materials with remarkable physio-mechanical and chemical characteristics belongs to the French researcher J. Davidovits through his exceptional patents and governs almost the entire material transformation process of alumina-silicate waste.

Manufacturing the geopolymers has become in the last decades a process recognized by all researchers in the field and is considered a process of the future due to its contribution to the planet greening by eliminating or significantly reducing polluting technologies that consume large amounts of fossil fuel.

Basically, there are two types of materials in this experiment as a physical state. Ground blast furnace slag, coal fly ash, recycled concrete waste from demolition, and silica fume are solid materials. Their preparation (grinding, sieving, and mixing) was done in a metal container. The components of alkaline activator included an aqueous solution (38 % concentration) of sodium silicate (water glass) as well as a liquid solution composed of NaOH in the form of solid pellets dissolved in distilled water. The two mentioned types of aqueous solution were mixed by stirring in a glass vessel. Then, the liquid mixture was poured over the solid mixture and further, the two different mixture types were homogenized by mechanical stirring for about 3 min, observing the formation of a relatively viscous suspension. The next operation was pouring the suspension into cylindrical moulds and placing them in an electric laboratory oven, the temperature chosen for the hardening treatment being 85 °C for 8 hours. After the thermal hardening of specimens, they were removed from the moulds and stored in an isolated room with low humidity for a maximum of 28 days. The determination of mechanical specimen features was performed both after 28 days and after 7 days of storage.

2.2 Materials

As mentioned above, recycled industrial by-products as blast furnace slag and coal fly ash, and recycled concrete waste from demolition, large available in the world, so relatively cheap, were used in this experiment. Silica fume (or microsilica) as an ultrafine powder (0.10-0.15 µm) resulting from the silicon-based ferroalloy manufacturing process is a more expensive product, but the amount utilized in the starting mixture is very low.

Granulated blast furnace slag with sizes between 2-6 mm was purchased and stored 10-12 years ago from ArcelorMittal Galati (Romania). The chemical composition of the blast furnace slag is shown in Table 1. Coal fly ash (below 250 µm) resulted during the coal burning process in Paroseni-Thermal power station (Romania), also procured and stored 8-10 years ago. The ash grain size was reduced under 80 µm). The fly ash composition is also presented in Table 1. Building concrete waste was recovered from demolition process. This waste was broken and ground at dimensions within the limits of 10-80 µm. The chemical composition of the concrete waste [24] is included in the data of Table 1.

Silica fume (99.9 % purity) was commercially purchased, originating from China.

As mentioned above, the alkaline activator was composed of an aqueous solution (38 %) of Na_2SiO_3 (also known as water glass) and an aqueous solution of NaOH as solid pellets dissolved in distilled water. Both the water glass solution and the NaOH pellets were purchased from the market.

Table 2 presents the versions adopted for preparing the geopolymeric materials and Figure 1 shows images of the cross section of the geopolymer specimens.

2.3 Methods utilized for determining the specimen characteristics

The density was measured by the Archimedes' procedure using the water-intrusion method (ASTM D792-20). The water absorption was identified through the method of immersing the specimen under water (ASTM D570). Compression strength was determined utilizing the method provided by ACI 318M-08 (cylindrical shape specimen Ø 50 x 80 mm) and flexural strength was measured applying the three-point bend ASTM C78-02 (beam-shaped specimen 160 x 20 x 10 mm). Heat conductivity measuring method was based on the standard ASTM C177. The water uptake was determined by immersing method of the sample under water (ASTM D570). The microstructural particularities of the geopolymer specimens were analyzed with Biological Microscope model MT5000, 1000 x magnification.

Table 1. Chemical composition of alumina-silicate materials (wt. %)

Material	**SiO_2**	**Al_2O_3**	**CaO**	**MgO**	**Fe_2O_3**	**Na_2O**	**K_2O**	**TiO_2**	**SO_3**
Ground blast furnace slag	35.9	11.8	42.0	5.5	0.7	0.2	0.5	-	-
Coal fly ash (class F)	54.9	26.3	3.0	2.4	4.9	0.5	0.5	1.2	1.6
Recycled concrete waste	68.9	18.7	1.6	0.5	6.5	0.2	1.5	1.3	-

Table 2. Adopted versions for preparing geopolymeric materials

Composition	**Version (kg·m^{-3})**					
	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6
Ground blast furnace slag	120	114	108	102	96	90
Coal fly ash	230	238	245	254	262	270
Recycled concrete waste from demolition	310	360	410	470	525	570
Silica fume	4	4	4	4	4	4
10M NaOH	96	95	97	96	96	97
Na_2SiO_3	220	220	222	221	222	223
Water addition	17	19	20	22	23	24

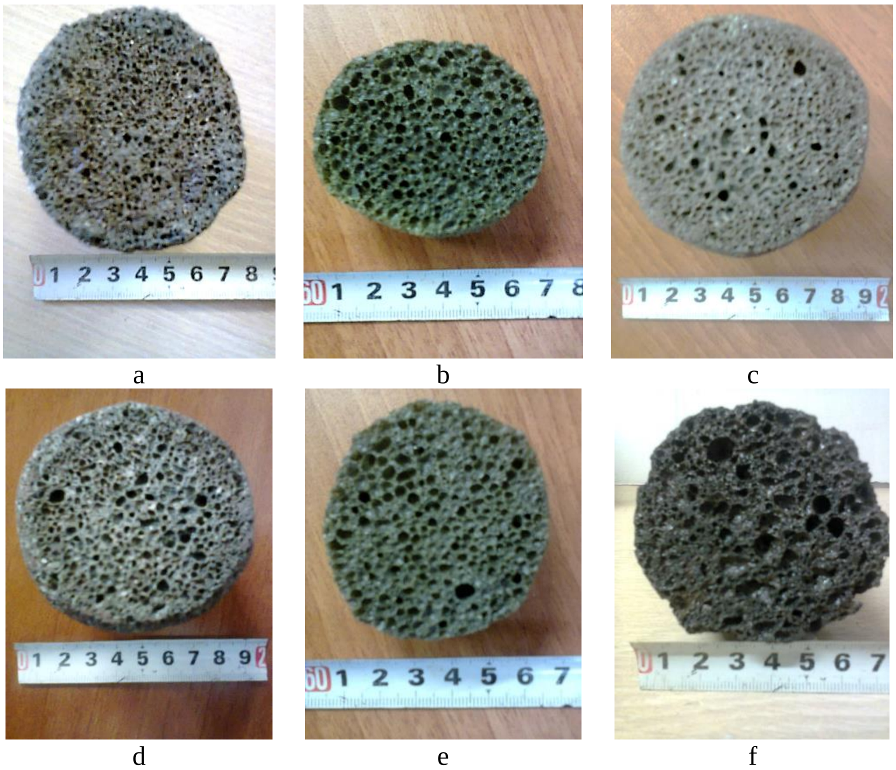

Figure 1. Images of the cross section-geopolymer specimen appearance
a – version 1; b – version 2; c – version 3; d – version 4; e – version 5; f – version 6.

According to the data in Table 2, the activator/alumina-silicate waste ratio evolved following a downward curve between versions 1-6, falling from 0.479 to 0.344. The main cause was the significant increase in the proportion of concrete waste from demolitions, whose value increased from 310 to 570 kg·m^{-3}. In principle, the coal fly ash/blast furnace slag ratio between 1.92-3.00 was adopted, increasing between 1-6 versions.

3. RESULTS AND DISCUSSION

3.1 Results

According to Figure 1, the pictures show the physical change of porous aspect of specimens starting from version 1 to version 6 due, primarily, to the continuous significant increase in the weight proportion of the recycled concrete waste from demolition.

In microstructural terms, images presented in Figure 2 show increasing the pore size of the six specimens from under 1 mm (corresponding to version 1) to almost 3 mm (corresponding to version 6). If the first four versions have quite uniform porous structures, instead the structures that characterize versions 5 and 6 are relatively coarse.

The measurements to determine the mechanical features of specimens were carried out after 7 days of curing to identify their resistance level at an early age as well as after 28 days to see the level of final resistance of the geopolymeric products.

The physio-mechanical and heat features of the six specimens of geopolymeric materials are presented in Table 3.

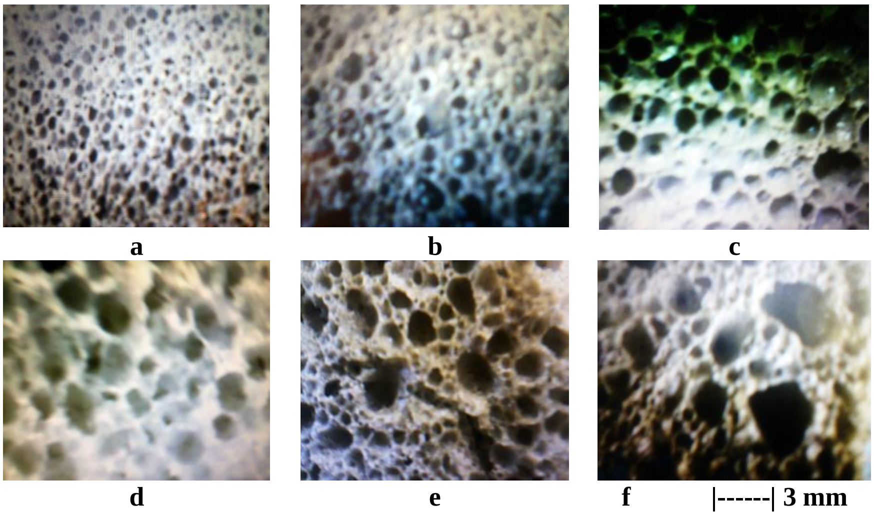

Figure 2. Microstructural appearance of geopolymeric specimens

a – version 1; b – version 2; c – version 3; d – version 4; e – version 5; f – version 6.

Table 3. Physio-mechanical and heat features of geopolymeric materials

Feature	Version 1	Version 2	Version 3	Version 4	Version 5	Version 6
Denseness (g·cm^{-3})	2.04	1.91	1.80	1.64	1.30	1.12
Porousness (%)	28.5	29.8	30.3	32.8	34.0	36.1
Heat conductance (W·m^{-1}·K^{-1})	0.306	0.273	0.233	0.218	0.197	0.171
Compression resistance (MPa)						
- after 7 days	55.0	50.3	46.1	40.5	34.2	28.3
- after 28 days	54.5	51.0	47.2	42.3	37.0	29.4
Flexural resistance (MPa)						
- after 7 days	4.7	4.5	4.0	3.7	3.3	3.0
- after 28 days	5.8	5.6	5.2	4.6	3.9	3.6
Water uptake (%)	5.2	5.4	5.4	5.6	5.5	5.5

The characterization results analysis of geopolymeric material specimens in Table 3 highlighted the high level of their mechanical resistance, especially at the early age of curing. Thus, compression resistance values after 7 days of curing had high values between 23.3-55.0 MPa after a decreasing curve between versions 1-6. The values of the same resistance type at the end of the 28 days were within the limits of 29.4-54.5 MPa, also following a decreasing curve.

Flexural resistance values had also a relatively high level both at early age (3.0-4.7 MPa) and at 28 days of curing (3.6-5.8 MPa).

The denseness and the heat conductance had relatively high values (between 1.12-2.04 g·cm^{-3} and respectively, 0.171-0.306 W·m^{-1}·K^{-1}).

Water uptake values measured through the sample immersion under water showed their constancy at a relatively normal level (between 5.2-5.6 %) for this type of material.

3.2 Discussion

The tests carried out in this experiment have experimentally demonstrated that alumina-silicate wastes are adequate, economical, and environmentally friendly for production new material types called geopolymers, especially applicable in construction. These findings are not a novelty in the last decades, but the use in high proportion of the recycled concrete waste from the building demolition. In this paper, results obtained under conditions of utilizing in the range of 310-570 kg·m^{-3} in the starting mixture for preparing the geopolymer are presented.

In terms of the strength of the new material, the high values of compression and flexural resistance are remarkable, especially at an early age (after 7 days of curing). The compressive strength values reached over 40 MPa in the first four versions, under conditions of use between 310-470 kg·m^{-3} of concrete waste. Flexural resistance values reached over 4.0 MPa in the first three versions. The continuation of the curing process up to 28 days contributed to increasing the values of the two mechanical resistance types, but the growth rate was diminished. Thus, the compressive strength increased within the limits of 42.3-54.5 MPa in the first four versions and the flexural strength reached 5.2-5.8 MPa in the first three versions. It was thus concluded that by using 310-470 kg·m^{-3} of recycled concrete waste, 230-254 kg·m^{-3} of coal fly ash, and 102-120 kg·m^{-3} of ground granulated blast furnace slag as well as 4 kg·m^{-3} of silica fume, the conditions are created for obtaining the most performance results of the geopolymeric material.

4. CONCLUSION

The work objective was to design and test a geopolymer type based on the use of significantly higher weight ratio of concrete waste recovered from building demolition compared to the usual proportions of alumina-silicate wastes (mainly fly ash and metallurgical slag). Results were excellent especially regarding the compression and flexural resistance at earl age (42.3-54.5 MPa and 5.2-5.8 MPa, respectively).

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