RESIDUAL ALUMINO-SILICATE MATERIALS (FLY ASH, SLAG, CLAY, AND GLASSWARE) FOR A NONCONVENTIONAL ECOLOGICAL POLYMER OF THE FUTURE

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ABSTRACT: Alumino-silicate wastes frequently used in the manufacturing process of geopolymer composites (fly ash and blast furnace slag) were mixed with alumino-silicate wastes non-used previously in this process (clay brick waste and glassware waste). The mixture activation with an alkaline activator based on NaOH 8M and Na_2SiO_3 aqueous solution that facilitates the geopolymerization reaction developing contributed to the conversion of waste into geopolymer composite, a new type of cheap and ecological materials with mechanical and physical properties suitable for the building sector. The optimal characteristics of specimens were: density of 2042 $kg \cdot m^{-3}$, apparent porosity of 19.6 %, compression strength of 53.7 MPa, and water absorption of 6.9 vol. %.

KEYWORDS: alumino-silicate waste, geopolymer composite, fly ash, slag, alkaline activator, compression strength.

1. INTRODUCTION

In the first two decades of the 3rd millennium, an accented trend to replace some usual raw materials, whose manufacturing process is unfriendly to the environment and great primary energy consumer exhibited worldwide. Especially, the construction material industry was strongly affected by stopping the Portland cement production, the traditional binder of construction concrete and the requirement of its replacing by other material types with close characteristics existing in natural state (metakaolin, clay, volcanic ash, etc.) or in form of waste or industrial by-products (coal fly ash, granulated blast furnace slag, red mud, incinerator ash of municipal solid waste, waste paper sludge, rice husk ash, wheat straw ash, etc.). The optimal basis method was proposed and patented by the French researcher J. Davidovits [1], according to which the activation in alkaline concentrated solution of alumino-silicate materials facilitates the geopolymerization reaction developing forming a new product named geopolymer [2, 3] with excellent mechanical, physical, and structural features, even superior to the cement-based concrete. Numerous research works have been achieved in the world on experimental manufacture of geopolymer area testing different alumino-silicate resources and using several alkaline solution concentration, whose results were recently published in the literature [4-10].

According to the literature, the most suitable alumino-silicate residual material usable for the production of geopolymers is coal fly ash, an industrial by-product captured in electrofilters following the coal burning in the boilers of thermal power plants in energy industry. In the world, large coal fly ash quantities result from the mentioned industrial process (over 300 million tons annually [11]), containing SiO_2 (35-56 %), Al_2O_3 (16-28 %), CaO (3-9 %), Fe_2O_3 (5-9 %), and others. This by-product is a residual material, which would create serious problems of storage in landfills with major ecological implications, if there were not already several industrial fields interested in its use as a raw material (in construction as glass foams [12], glass-ceramics [13], and aggregates [14], in ecology for treatment of wastewater as adsorbents [15], in metallurgy for high-entropy alloy preparation [16], etc.).

The best-known geopolymer manufacturing techniques are based on coal fly ash or the fly ash-granulated blast furnace slag mixture. In both variants, the alumino-silicate material is finely ground to grain sizes below 25-35 μm. The technique by which the geopolymerization reaction is initiated is the pouring over the solid powder mixture of an aqueous solution of NaOH and Na_2SiO_3 between 8 and 14M having the role of alkaline activator and mixing the two phases until slurry is formed. The slurry is poured into metal molds and subjected to the final curing process, which can be carried out at room temperature or at about 70-80 ºC, in a neutral atmosphere or in air. The duration of the curing process is variable, generally between 8-16 hours. Usually, determining the mechanical, physical, thermal, and microstructural characteristics of geopolymer specimens is performed after 7 or 28 days of free curing at room temperature.

According to data presented in the work [17], fly ash-geopolymer experimentally manufacturing under conditions in which the alkaline activator was characterized by SiO_2/Na_2O molar ratio in value of 1.5 and Na_2O/fly ash weight ratio represent 10 % allowed to reach the highest value of the compression strength of 63.4 MPa after the curing process at 75 ºC for 8 hours followed by the curing at room temperature for 28 days. The composition of Na_2SiO_3 was the following: 9.1 % Na_2O, 29.2 % SiO_2, and 61.7 % H_2O.

The combined use of fly ash (60-90 wt. %) and granulated blast furnace slag (10-40 wt. %) as raw materials for preparing the geopolymer was tested by the authors of the paper [18]. The alkaline activator was composed of NaOH 6-14M, used to dissolute the alumino-silicate material mixture and Na_2SiO_3 with the role of a binder. The Na_2SiO_3/NaOH weight ratio was adopted at 2.5, which allowed obtaining high compression strength (over 47 MPa). The preparation of NaOH-Na_2SiO_3 mixture involved its keeping at room temperature for 24 hours. After the formation of slurry resulting from mixing the solid powder materials and the liquid alkaline activator, it was poured into cube molds (50x50x50 mm). After pre-curing at room temperature for 24 hours, the curing process followed at 60 ºC into an oven for 24 hours. Next, the geopolymer specimens were taken out of the oven and kept at room temperature for 28 days. The density of fly ash-slag-geopolymer had values within the limits of 2055-2100 $kg \cdot m^{-3}$, higher by comparing with fly ash-geopolymer density (between 1570-2050 $kg \cdot m^{-3}$). According to the measurement results, porosity (about 17 %) and water absorption (about 7 vol. %) characteristic of the fly ash-slag-geopolymer were lower than those of the fly ash-geopolymer. Porosity and water absorption values decreased by increasing the blast furnace slag content from 10 to 20 %. The conclusion of the work was that the high value of density and low values of porosity and water absorption contributed to the improvement of compression strength of fly ash-slag-geopolymer compared to the strength of fly ash-geopolymer.

Except the use of coal fly ash and granulated blast furnace slag in the manufacturing process of geopolymer priority analyzed in the literature, other residual alumino-silicate materials have been recently tested. Thus, volcanic ash containing 44.9 % SiO_2, 13.3 % Al_2O_3, 6.7 % Fe_2O_3, 5.1 % CaO, 3.6 % MgO, 1.5 % K_2O was experimentally used together with fly ash, pozzolan, metakaolin, and mining tailing for making geopolymer composite [6]. NaOH and Na_2SiO_3 in aqueous solution were added as alkali activator in order to activate the geopolymerization reaction. The compression strength reached 35.1 MPa under the conditions of a volcanic ash-pozzolan mixture and a value of SiO_2/Al_2O_3 ratio of 7.09.

The glass waste as a possible alkaline activator was experimentally investigated in the making process of fly ash-geopolymer. The paper [19] comparatively analysed the properties of cement-geopolymer performed with NaOH 8M, NaOH 10M and 15 % water glass (Na_2SiO_3) as well as NaOH 10M and 15 g residual glass. Mixing the raw material based on alumino-silicates and alkaline activator in the mentioned variants led to obtaining a hydrated alkaline alumino-silicate gel favouring the geopolymerization reaction developing. Experimental results confirmed the viability of substituting water glass, usually used in geopolymer preparing by activating residual alumino-silicate materials.

The paper [20] showed that the silicon contained in the residual glass dissolved in the NaOH-Na_2SiO_3 aqueous solution could lead to effects similar to those of the silicon in water glass. Thus, it was concluded that glass waste could take over the role of activator in alkaline activated systems. A treatment with NaOH/Na_2CO_3 with pH = 13.6 facilitated the partial dissolution of silicon contained in glass waste in its most reactive monomeric form. Experimentally, the aqueous solutions resulting from the treatment of residual glass behaved as an alkaline activator dissolving the furnace slag and forming compounds similar to those resulting from the preparation with water glass. The experiment indicated that the composition of slurry prepared with water glass and activators containing residual glass were similar.

The current paper is the result of authors' research in the area of making geopolymer composites, in which residual materials with high silica and alumina content (clay brick waste from building demolition and laboratory glassware waste) have been tested in mixture with coal fly ash and granulated blast furnace slag aiming at the valorization of these resources with low greenhouse gas emissions in the atmosphere and low energy consumption.

2. METHODS AND MATERIALS

2.1 Methods

The method applied for making the geopolymer composite is generally a method recognized by all researchers who approach this field. Thus, the raw material consisting of alumino-silicate materials is processed by breaking, grinding, sieving and selecting by the grain size. The geopolymer manufacturing process involves a very low grain size range (below 50-60 μm). Separately, the alkaline activator which usually includes NaOH 8M (in the form of pellets soluble in distilled water) and Na_2SiO_3 aqueous solution is prepared by mixing its alkaline components with the mixer. The alkaline liquid formed is kept for 24 hours at room temperature. Then, the liquid activator is slowly poured over the solid powder mixture and their mixing is continuing at a high rate (over 1000 rpm) for 15-20 min, until the slurry is formed. Next, the slurry is poured into metal molds, which are then covered with a thin plastic sheet to avoid the water loss. A pre-curing process at room temperature for 24 hours is carried out before starting the curing process at 75 ºC for 24 hours in an electric oven. During this time, the wet material is protected with a thin plastic sheet to avoid the humidity loss. At the end of the process, geopolymer specimens are removed from the oven and stored at room temperature for 28 days in an isolated enclosure, after which the determination of their characteristics can be carried out. The making process of composite geopolymer took place in the experimental base of Cosfel Actual SRL Bucharest.

The geopolymerization reaction constitutes the base of the raw material transformation process based on alumino-silicate materials into a covalently bonded three-dimensional network. The bonds are of the type $[\text{-Si-O-Al-O-}]_n$, according to [21]. Geopolymerization is considered an extremely complex process [22] characterized by its unfolding in three steps, which can overlap influencing each other. Therefore, the detailed understanding of the conversion mechanism of alumino-silicate precursors into geopolymers is difficult and requires a large number of experimental techniques.

2.2 Materials

The materials used as raw materials in this experiment were: coal fly ash provided by Paroseni thermal power station (Romania), granulated blast furnace slag provided by ArcelorMittal Galati (Romania), clay brick waste recycled from building demolition, and alumino-silicate glass waste recovered from residual laboratory glassware.

The chemical composition of materials mentioned above was determined on the X-ray fluorescence spectrometer type AXIOS-sequential from Metallurgical Research Institute Bucharest (Romania), the results being shown in Table 1.

Table 1. Chemical composition of materials

Com-posi-tion	**Coal fly ash (%)**	**Blast furnace slag (%)**	**Clay brick waste [23] (%)**	**Alumino-silicate glass waste (%)**
SiO_2	46.5	37.4	56.4	47.0
Al_2O_3	23.7	6.4	27.4	15.5
CaO	7.9	39.9	1.2	24.3
Fe_2O_3	8.6	6.9	7.2	0.3
MgO	3.2	3.5	1.4	9.7
Na_2O	6.0	0.1	1.0	-
K_2O	4.1	0.2	4.4	0.7
MnO	-	2.3	-	-

Granulated blast furnace slag initially had the grain size below 4 mm and coal fly ash provided by the thermal power station had the grain size below 250 μm. The two types of material were subjected to grinding in a ball mill and selected by sieving to sizes below 40 μm. Clay brick waste recycled from building demolition and residual laboratory glassware were broken into pieces below 10 mm and repeatedly ground in the ball mill until the grain size of the two material types was reduced below 30 μm. The processing operations took place in the specialized workshops of University "Politehnica" of Bucharest and Metallurgical Research Institute.

Alkaline activator was composed of NaOH pellets and 38 % Na_2SiO_3 aqueous solution, both alkaline products being commercially purchased.

2.3 Methods of characterizing the geopolymer specimens

Density was determined by weighing the specimen and reporting its mass to the volume easily calculated due to its regular geometrical shape [24]. Apparent porosity was calculated according to ASTM C642-97 as the report of difference between wet weight and dry weight and the difference between wet weight and suspended weight of the specimen [18]. Heat conductivity was measured using a heat-flow-meter HFM 448 Lambda (according to SR EN 1946-3: 2004) at the ambient temperature. Water absorption was identified by water immersion method for 24 hours (according to ASTM D570). For determining the compression strength, 100 kN-compression fixture Wyoming Test

Fixture [25} was used and the microstructural aspect of specimens was investigated with Biological Microscope TM5000 model with captured image, 1000 x magnification.

3. **RESULTS AND DISCUSSION**

3.1 Results

As mentioned above, the powder mixture of residual alumina-silicate materials mainly contained coal fly ash (within the limits of 74-84 wt. %) decreasingly distributed in the four experimental variants according to Table 2. Granulated blast furnace slag was adopted in the range of 6-13 wt. % allocated in increasing proportions compared to fly ash. Except the two residual materials frequently used in experiments presented in the literature in the experimental manufacturing process of geopolymer composites, the current experiment included two other alumino-silicate wastes extremely rarely used or not used in this type of manufacturing process (clay brick waste from building demolition and residual laboratory glassware). According to the data in Table 2, the weight ratio of clay waste was kept constant at 5 wt. % and that of glassware increased in the range of 5-8 wt. %.

Table 2. Experimental variants

Composition (wt. %)	Variant			
	1	2	3	4
Coal fly ash	84	81	78	74
Blast furnace slag	6	8	10	13
Clay brick waste	5	5	5	5
Alumino-silicate glass waste	5	6	7	8

The alkaline activator parameters adopted in this experiment based on own previous results and information from the literature were: NaOH concentration of 8M, solid/liquid weight ratio of 3.2, and Na_2SiO_3/NaOH molar ratio of 1.45. These parameters were kept constant in all experimental variants.

The use of determining techniques of physical, thermal, mechanical, and microstructural features mentioned above allowed their identification. Results including density, apparent porosity, heat conductivity, compression strength, and water absorption corresponding to the four experimental variants of making the geopolymers are shown in Table 3.

Table 3. Characteristics of geopolymer composites

Characteristic	**Variant**			
	1	2	3	4
Density ($kg \cdot m^{-3}$)	1973	1995	2025	2042
Apparent porosity (%)	26.3	24.5	22.0	19.6
Heat conductivity ($W \cdot m^{-1} \cdot K^{-1}$)	0.359	0.390	0.429	0.461
Compression strength (MPa)	39.8	44.0	50.2	53.7
Water absorption (vol. %)	8.6	8.0	7.5	6.9

Examining the results on physical, thermal, and mechanical characteristics of geopolymer composite specimens it can be seen that the decrease of fly ash/blast furnace slag weight ratio from 84/6 to 74/13 allowed the increase of compression strength from 39.8 to 53.7 MPa. Under the conditions in which the proportion of clay waste was constant in the mixture of starting materials, it is obvious that the increasing addition of glassware waste up to 8 wt. % also influenced the improvement of the geopolymer mechanical properties. The decrease of fly ash ratio and the increase of slag and glassware ratios had the effect of increasing the density value (from 1973 to 2042 $kg \cdot m^{-3}$) and heat conductivity (from 0.359 to 0.461 $W \cdot m^{-1} \cdot K^{-1}$) simultaneously with reducing the apparent porosity. These modifications of physical and thermal properties significantly influenced mechanical characteristics of specimens, the compression strength reaching the highest value (53.7 MPa) in case of variant 4, that was adopted as the optimal experimental variant. The high level of compression strength is comparable to that obtained in the case of fly ash-slag-geopolymer made and presented in the literature [18].

Appearance images of geopolymer composite specimens manufactured in this experiment are presented in Figure 1. The images are suggestive for the compact and very dense character of specimens.

The investigation of geopolymers at the microstructural level (Figure 2) shows major structural differences between the specimen corresponding to variant 1 (with the highest concentration of fly ash) and the specimen corresponding to variant 4 (with the highest weight participation of blast furnace slag in the geopolymer composition).

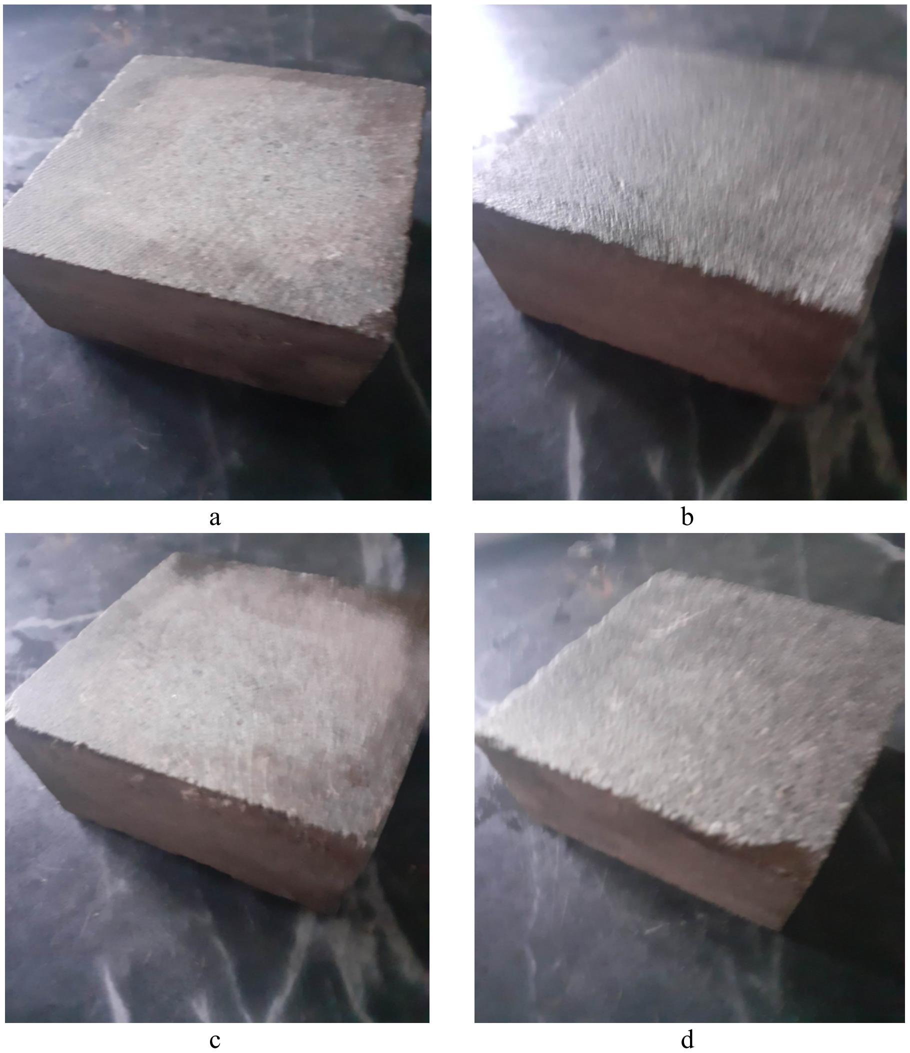

Figure 1. Appearance images of geopolymer composite specimens
a – variant 1; b – variant 2; c – variant 3; d – variant 4.

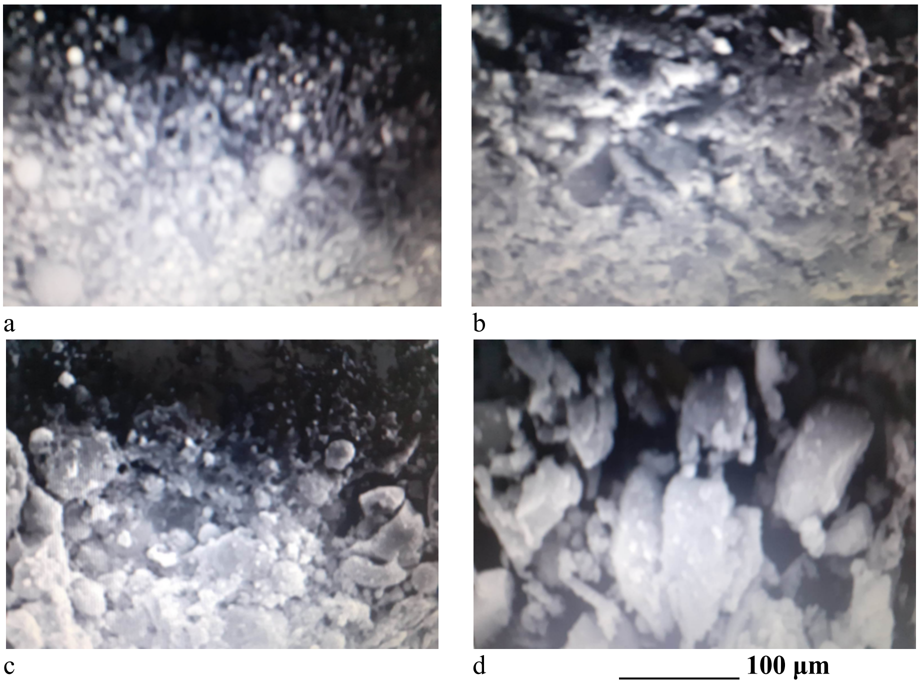

Figure 2. Microstructural aspect of geopolymer composite specimens
a – variant 1; b – variant 2; c – variant 3; d – variant 4.

3.2 Discussion

The production of geopolymer composites as materials whose process is ecological and consumes very low energy is of exceptional importance for the future of our planet. It is largely based on recycling alumino-silicate wastes as raw material.

To facilitate this operation, the selective waste collection technique is required. Some industrial wastes are the result of specific technological processes, such as coal fly ash in the energy industry and granulated blast furnace slag in the iron and steel industry. These are collected in the activity sector that produces them and thus they are easy to recover for recycling.

Other alumino-silicate wastes, such as those resulting from building demolition (clay brick waste) or laboratory glassware waste, are not practically collected selectively and their recovery from a landfill that includes mixed municipal waste would be extremely difficult.

In the case of the experimental manufacturing process of geopolymer composite presented in this paper, clay brick waste as well as laboratory glassware waste were collected by the authors, the low waste amounts being easy to procure.

4. CONCLUSION

The paper aimed at testing the making process of geopolymer composite using a mixture of alumino-silicate waste based on two types of industrial waste frequently tested previously (fly ash and blast furnace slag) and two types practically unused in previous experiments (clay brick waste from building demolition and laboratory glassware waste from activities specific to the chemical industry, pharmaceutical industry, medicine, food industry, etc.).

Alumino-silicate waste containing high proportions of silica and alumina can be converted into geopolymer composites, new ecological and cheap materials with mechanical and physical properties suitable for the building sector. The use of an alkaline activator (NaOH 8M and Na_2SiO_3 aqueous solution) allows the development of the geopolymerization reaction, the basis of this type of process.

The optimal geopolymer specimen was made from coal fly ash (74 %), blast furnace slag (13 %), clay brick waste (5 %), and glassware waste (8 %). The alkaline activator had the NaOH concentration of 8M, solid/liquid weight ratio of 3.2, and Na_2SiO_3/NaOH molar ratio of 1.45. The geopolymer density was 2042 $kg \cdot m^{-3}$, apparent porosity was 19.6 %, compression strength reached 53.7 MPa, and water absorption had the value of 6.9 vol. %.

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