NONCONVENTIONAL ECOLOGICAL AND LOW-ENERGY CONSUMPTION TECHNIQUE TO PRODUCE HIGH-STRENGTH GEOPOLYMER COMPOSITE BASED ON RESIDUAL MATERIALS AS A NEW TYPE OF CONSTRUCTION MATERIAL

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ABSTRACT: High-strength geopolymer composite, a new type of construction material, based on granulated blast furnace slag and coal fly ash reinforced with PET fibres obtained by recycling and processing PET bottles was experimentally made. According to Davidovits' invention, an aqueous solution of sodium hydroxide and sodium silicate representing the alkaline activator was the basic element of the process favouring the development of the geopolymerization reaction, which involves the residual alumino-silicate materials (blast furnace slag and fly ash). The final curing process of the composite in the state of slurry poured into molds constituted the originality of the work by alternating the curing at room temperature for 20 hours with curing by maintaining the material at 75 ºC for 10 hours and then continuing the curing process at room temperature for 7 and 28 days respectively. The composite geopolymer had remarkable mechanical properties (compression strength of 60.1 MPa and flexural strength of 11.5 MPa) corresponding to the maximum proportion of PET fibres of 2.3 wt. % and final curing process time of 28 days. The composite made from residual materials is suitable for its use in ecological production conditions and with extremely low energy consumption.

KEYWORDS: geopolymer composite, alumino-silicate, PET fibre, fly ash, slag, alkaline activator.

1. INTRODUCTION

The serious consequences of changing the terrestrial climate caused by the continuous destruction of the protective ozone layer due to the huge emissions of greenhouse gases (billions tons of carbon dioxide) has suddenly become a major problem of humanity highlighted since the first decade of the new millennium. According to World Meteorological Organization in September 2019, the average global temperature of the planet has increased by at least 1 ºC compared to pre-industrial levels and there is the risk that if the current rate of CO_2 emissions is maintained, the climate will overheat by 3 ºC in the year 2100.

This alarming situation as well as the global oil crisis that started after 1975 imposed a new orientation towards recycling waste and industrial by-products and their re-introduction in high energy-consuming technological processes, even being completely or partially replaced some basic materials from traditional industrial activity. Other recycled materials provided the raw material for the manufacture of new value-added products.

A very eloquent actual example is the Portland cement, considered for a long time as the only binder of concrete for construction. Its industrial manufacturing process involves an excessively high consumption of fossil fuel and implicitly, very high emissions of greenhouse gases into the atmosphere. Ever since the end of the 20th century, the French researcher J. Davidovits patented the geopolymer, an "inorganic polymeric new material" based on alumino-silicate residual materials coming from numerous industrial processes as by-products (coal fly ash, rice husk, granulated blast furnace slag, red mud, etc.) or existing in nature (clay, kaolin, metakaolin, rice husk ash, volcanic rock powders) [1-3]. According to Davidovits' invention, the geopolymer is synthesized due to the reaction between the lumina-silicate materials and a high-concentration aqueous solution of an alkaline activator. The geopolymerization reaction turns the lumina-silicate particles into a geopolymeric composite, in principle a good building material that completely eliminates the use of cement in the manufacturing process. Thus, geopolymer is an environmentally friendly material that contributes to the reduction of greenhouse gas emissions and also eliminates through recycling important quantities of industrial waste [4, 5]. The energy consumption allocated to the manufacture of fly ash-geopolymer is generally 60 % lower compared to the making process of Portland cement and CO_2 emissions are diminished by over 80 % [6].

Recently experiments whose results were published in the literature [7] have provided favourable information regarding the manufacture of geopolymer foams using lumina-silicate materials (coal fly ash), hydrogen peroxide as an expanding agent as well as mixture of sodium hydroxide (in the form of flakes) and aqueous solution of sodium silicate as an alkaline activator for the initiation and development of the geopolymerization reaction. Geopolymer foam had the characteristics of a thermal insulation material suitable for application in construction with density within the limits 0.2-0.8 $g\cdot cm^{-3}$, thermal conductivity over 0.037 $W\cdot m^{-1}\cdot K^{-1}$, compressive strength between 0.5-2 MPa, and pore size in the range of 0.5-3 mm.

The current work is focused on the manufacture of a geopolymer with high mechanical strength at the level of traditional compact concrete (produced with Portland cement). From this point of view, the geopolymer foam is not interesting and will not be analyzed in this paper.

Due to the durability, the availability of raw materials, and the relatively low price, ordinary construction concrete is a material of major importance. Therefore, the need to find materials with binding properties comparable to those of Portland cement is of great interest throughout the world. The geopolymer invented by Davidovits and qualitatively improved in the last years clearly represents the optimal solution for the complete replacement of cement. Increasing the compression strength and flexural strength, i.e. increasing the durability of geopolymer could be achieved by reinforcing it with fibres.

The technique of making a geopolymeric composite reinforced with carbon fiber felt by infiltrating geopolymer slurry into carbon fiber felt was presented in the paper [8]. The flexural strength of the reinforced product was significantly improved reaching the maximum value of 51.5 MPa.

An efficient method of controlling cracks caused by exposure to high temperature was obtained by Zhang et al. by adding carbon fibres into geopolymer [9].

Short steel fibres (2 wt. %) were used in the paper [10] for producing fly ash-geopolymer by alkaline activation. According to the provided experimental data, the behaviour of geopolymer at multiple cracks as well as the flexural strength were almost similar compared to materials manufactured with cement as a binder.

The efficiency of carbon and steel fibres used as geopolymer reinforcement additives has been experimentally proven, the mechanical properties of the polymer products being significantly improved. However, the high cost of these fibres has negatively influenced their use at an acceptable level [6].

Several types of fibre used for the reinforcement of geopolymer composites were comparatively analyzed based on the experimental results in the work [11]. Geopolymer composite based on fly ash reinforced with PET (polyethylene terephthalate) fibres cured at 65-70 ºC and composite based on mixture of fly ash and granulated blast furnace slag cured at room temperature were compared with composite based on cement reinforced with the same fibre proportions. Also, the comparison was made on similar composites reinforced with commercially available polypropylene fibre. The compressive strength of the geopolymeric composites, regardless of the type of reinforcing fibre, was higher compared to that of Portland cement-based composites. On the other hand, the geopolymer based on fly ash and blast furnace slag had slightly higher compressive strength compared to fly ash-geopolymer reinforced with PET fibres. The compressive strength of all the composites reinforced with polypropylene fibres was slightly higher than that of the composites reinforced with PET fibres. By increasing the volumetric proportion of PET fibre between 1-5 % the increase of the flexural strength of both geopolymeric composites was observed. The cement-composite reinforced with PET fibres did not register changes of the flexural strength. Reinforcing with polypropylene fibre shows opposite trends.

Another method of improving the mechanical properties of geopolymer composites was presented in [6], using polyvinyl alcohol in the form of fibres and powder, respectively. Polyvinyl alcohol fibres [12] are monofilament fibres that scattered in the concrete or geopolymer matrix form a network of multi-directional fibres contributing to the reinforcement process of material. The paper [6] referred to the reinforcement of geopolymer based on fly ash. Blast furnace slag was also used as a raw material to increase the reactivity of the geopolymer. The alkaline activator required for the geopolymerization reaction to take place was a mixture of sodium hydroxide (NaOH) and sodium silicate solution (Na_2SiO_3). The work studied the effect of adding polyvinyl alcohol in two forms (fibres and powder) on the mechanical properties, resistance to freeze-thaw cycles, and thermal decomposition properties of geopolymeric composites. Experimental results indicated that the fibre reinforcement was more effective compared to that with powder. The highest values of compressive strength (41.11 MPa) and flexural strength (8.43 MPa) were recorded using polyvinyl alcohol fibre.

The literature of recent years also contains other variants of geopolymer reinforcement. One of them is that presented in the work [13]. The geopolymer used fly ash as the basic raw material with the addition of 5-20 % alccofine (microfine material based on low calcium silicate slag) as a partial substitute for ash, and zeolite sand between 10-40 % as a partial addition to the fine aggregate. Rubber fibres (2 %) were added as a reinforcing agent. The alkaline activator was, as usual, composed of NaOH and Na_2SiO_3 solution. The curing process of the fresh material was performed in 7 and 28 days. Compressive strength was improved by up to 46.6 %, split tensile strength increased by up to 22.1 %, and flexural strength by up to 14.1 %. Workability decreased with increasing the zeolite content and water absorption decreased by 26 % compared to conventional concrete.

Fibres of vegetable origin were also used to reinforce the geopolymer. According to [14], pineapple fibres (between 0-0.5 % of the mortar mass) were added to the raw material (only fly ash) for the manufacture of geopolymer mortar composites. The concentration of the NaOH solution for activating the geopolymerization reaction was 14M and 16M, respectively. The length of the fibres was varied between 10-30 mm. The fibre length of 30 mm, the weight proportion of added fibres of 0.5 %, and the concentration of NaOH 16M were experimentally determined as optimal values to reach the highest level of compressive strength (41.5 MPa) and flexural strength (9.2 MPa).

In this work, the authors chose the technique of using polyethylene terephthalate (PET) fibres as a reinforcing agent for the experimental manufacture of high-strength geopolymer composite based on granulated blast furnace slag and coal fly ash. The fibres were prepared under laboratory conditions by chopping recycled PET bottles to sizes between 3-7 mm. The curing treatment of the fresh material in the slurry state was performed through an own technique, combining the curing process at room temperature for 20 hours, followed by curing at 75 ºC for 10 hours and keeping the cooled geopolymer at room temperature for 7 and 28 days respectively, when the characterization measurements of specimens were made.

2. METHODS AND MATERIALS

2.1 Methods

The principle of manufacturing a geopolymer using alumino-silicate materials (residual or natural) was stated in the invention of the French researcher Davidovits and is based on the geopolymerization reaction for whose development the presence of a high-concentration aqueous solution of alkaline activator is necessary. When the alkaline hydroxide (NaOH) comes into contact with the alumino-silicate material, the dissolution and hydrolysis of aluminum and silicon occurs according to reactions (1) and (2) [2, 15].

$$Al_2O_3 + 3H_2O + 2OH^- = 2[Al(OH)_4]^- \qquad (1)$$

$$SiO_2 + 2OH^- = [SiO_2(OH)_2]^{2-} \qquad (2)$$

After these reactions take place, a gel (slurry) is formed that changes its structural organization. Thus, the system is characterized by several gel phases. In the final stage, hardening takes place, in which the system is polymerized, becoming a solidified mass.

2.2 Materials

The raw material used in this experiment was composed of coal fly ash and granulated blast furnace slag. Polyethylene terephthalate fibres prepared by chopping recycled PET bottles at dimensions between 3-7 mm were used to reinforce the geopolymeric composite. In principle, the blast furnace slag was used for improving the reactivity of the geopolymer.

The alkaline activator was a mixture of NaOH flakes soluble in water and Na_2SiO_3 solution. The role of NaOH was to correct the SiO_2/Na_2O molar ratio of the activator, so that to be suitable for manufacturing the geopolymer composite. NaOH was commercially purchased in the form of flakes. The chemical composition of Na_2SiO_3 solution purchased from the market included 15.6 % Na_2O, 30.5 % SiO_2, and 53.9 % water.

Coal fly ash as an industrial by-product was provided by Paroseni thermal power station (Romania) whose oxide composition was also provided by the manufacturer and is shown in Table 1. Granulated blast furnace slag was provided by ArcelorMittal Galati (Romania) about 5 years ago, having the oxide composition indicated in the same table.

Table 1. Oxide composition of fly ash and slag

Composition	Coal fly ash (wt. %)	Blast furnace slag (wt. %)
SiO_2	53.1	37.7
Al_2O_3	23.7	9.4
CaO	7.9	45.4
MgO	3.2	4.9
Fe_2O_3	8.6	0.4
Na_2O	3.5	0.4
K_2O		1.7

Granulated blast furnace slag was ground in a ball mill. The slag granulation was selected by sieving under 60 μm. Coal fly ash with initial grain size below 200 μm was ground in the ball mill at a fine granulation under 32 μm.

Preparing the composite was carried out in several stages. First, the alkaline activator was prepared. NaOH and Na_2SiO_3 in the form of aqueous solution (14M) were mixed in an electric mixer with the molar ratio of 1.22 and then cooled to room temperature. Previous own tests have shown that this molar ratio gives the alkaline activator high reactivity and workability. PET fibres were added to the geopolymer slurry. The water/binder ratio was adopted at 0.45. The mixing of the raw material components (fly ash and slag) was done with the mixer at low rate for the beginning. Slowly, water and the aqueous solution of the alkaline activator were added to the mixture of solids, then the mixing was done at high rate of 1000 rpm for 4 min. The PET fibres were added to the wet mixture (slurry) until their dispersion was completely achieved. Then, the slurry was poured into a stainless steel mold with dimensions 110x35x35 mm and was vibrated to remove air bubbles using a concrete vibrating table (350x350 mm) made of stainless steel. The specimens were covered with a thin layer of plastic to avoid water loss and subjected to the curing process at room temperature for 20 hours, followed by the treatment at 75 ºC for 10 hours in an electric laboratory oven. The curing process was continued at room temperature in an enclosure for 7 and 28 days, respectively.

2.3 Characterization methods of specimens

Usual methods for characterizing ceramic samples were adopted to identify the physical, mechanical, thermal, and microstructural properties of the geopolymer composite specimens made in this experiment. Due to their regular shape, density was calculated by measuring the mass, determining the volume, and reporting the two values [16]. Thermal conductivity was measured with a heat-flow meter HFM 446 Lambda (SR EN 1946-3:2004) at room temperature. 100 kN-compression fixture Wyoming Test Fixture was used for determining the compression strength [17]. The flexural strength of the geopolymer was measured by the method of supporting the specimen as a lever and applying a flexural force with a constant rate until failure (SR EN ISO 14125: 2000) [18]. Water absorption was made by water immersion method for 24 hours (ASTM D570). Microstructural pictures of specimens were achieved with Biological Microscope MT5000 model with captured image, 1000 x magnification.

3. RESULTS AND DISCUSSION

3.1 Results

Four experimental variants were adopted according to Table 2.

Table 2. Experimental variants

Parameter	Variant			
	1	2	3	4
Activator/fly ash weight ratio	0.44	0.44	0.44	0.44
Slag/fly ash weight ratio	1.05	1.05	1.00	1.00
PET fibre (wt. %)	1.5	1.8	2.1	2.3
Water/binder weight ratio	0.45	0.45	0.45	0.45

The peculiarities of the four experimental variants varied in terms of the weight proportion of PET fibre within the limits of 1.5-2.3 wt. % and to a small extent regarding the slag/fly ash weight ratio having the value 1.05 in variants 1 and 2 as well as 1.00 in variants 3 and 4. The activator/fly ash weight ratio was kept constant at 0.44, and water/binder weight ratio constantly had the value of 0.45. The mixture of alumino-silicate materials (fly ash and granulated blast furnace slag) has the role of binder.

Recently, Shilar et al. experimentally found the influence of the alkaline activator concentration on some physical and mechanical properties of geopolymers [19]. Thus, density of these composite materials is increasing when the concentration evolves from 8M to 18M. The compression strength reaches the highest values in the case of the activator concentration of 15M, after which it decreases with the increase of the concentration up to the domain of 18-20M. Flexural strength has considerably lower increasing values corresponding to the concentration within the limits of 8-14M, after which it begins to slightly decrease for higher concentrations (18M). An almost similar evolution has water absorption, which first increases for concentrations between 8-

15M, decreasing for the further increase in concentration towards 18-20M.

Main physical, thermal, and mechanical features of geopolymer composite specimens determined through characterizing methods mentioned above are shown in Table 3.

Table 3. Features of geopolymer composite specimens

Feature	Variant			
	1	2	3	4
Density ($g \cdot cm^{-3}$)	1.65	1.69	1.74	1.81
Thermal conductivity ($W \cdot m^{-1} \cdot K^{-1}$)	0.348	0.359	0.368	0.382
Compression strength (MPa) - after 7 days curing - after 28 days curing	40.4 56.5	41.3 56.9	44.9 58.6	46.0 60.1
Flexural strength (MPa) - after 7 days curing - after 28 days curing	9.9 10.7	10.5 11.2	10.8 11.4	11.9 11.5
Water absorption (vol. %)	12.3	12.8	13.1	13.2

The analysis of the data in Table 3 shows that the mechanical properties of the geopolymer composite reinforced with PET fibres and cured for 7 and 28 days respectively, reached high values, especially the compressive strength after 28 days of curing (60.1 MPa). The PET fibre weight proportion was the highest (2.3 wt. %) corresponding to variant 4. The optimal blast furnace slag/fly ash weight ratio was 1.00 and the alkaline activator/fly ash weight ratio was 0.44. The 7 days-curing process allowed to reach a high value (46.0 MPa), but significantly lower compared to the 28 days-treatment. The flexural strength had considerably lower values even after 28 days curing (within the limits of 10.7-11.5 MPa), the influence of PET fibre-weight proportion on the mechanical properties of geopolymer composite not being clearly differentiated. In fact, the influence of other types of fibre used in experiments described in the literature on flexural strength was generally similar, the greater amount of fibres contributing in a small extent to the improvement of this mechanical characteristic. The density of the geopolymer composite had high values (between 1.65-1.81 $g \cdot cm^{-3}$) specific to a dense material, being slightly influenced by the increase in the proportion of PET fibre in the range of 1.5-2.3 wt. %. As a consequence of the rather high values of the density, the thermal conductivity of specimens recorded relatively high levels (in the range of 0.348-0.382 $W \cdot m^{-1} \cdot K^{-1}$), but in accordance with the normal values of this type of composite material. Also, the measurements to determine the absorption of water in the geopolymer mass showed a relatively normal level (between 12.3-13.2 vol. %), without observe a significant influence of the PET fibre proportion on water absorption.

Images of the physical appearance of the four geopolymer composite specimens in order of increasing PET fibre proportion according to Table 2 are presented in Figure 1.

Microstructural images of the four geopolymer composite specimens are shown in Figure 2.

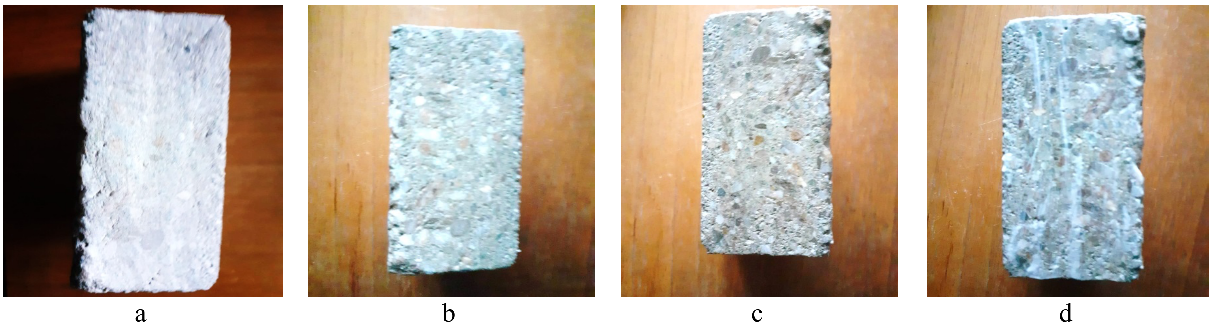

Figure 1. Images of the physical appearance of geopolymer composite specimens
a – variant 1; b – variant 2; c – variant 3; d – variant 4.

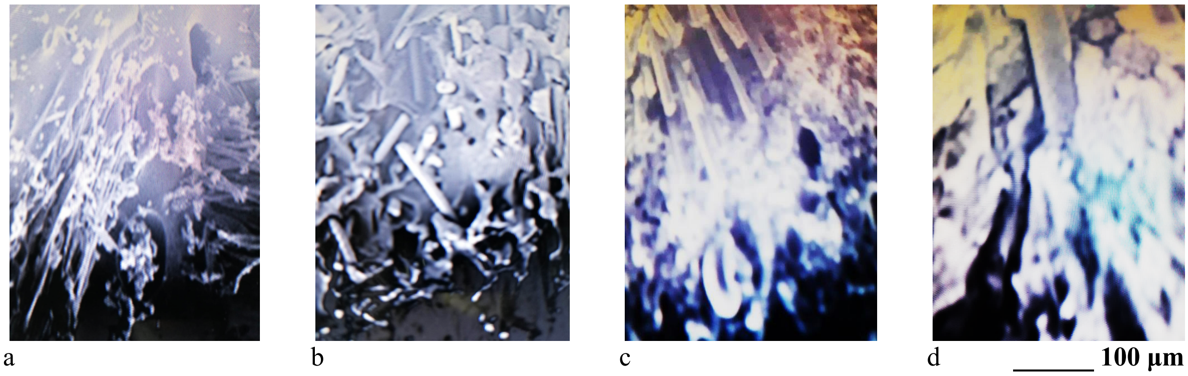

Figure 2. Microstructural images of geopolymer composite specimens
a – variant 1; b – variant 2; c – variant 3; d – variant 4.

The images in Figure 2 show the evolution of specimen microstructures characterized by the increase of fibre proportion in the composite mass. Also, the fibre size (between 3-7 mm) had an important role in the geopolymer reinforcement process. Image (d) corresponding to variant 4 represents an optimal microstructure by comparison with the other experimental variants due to the highest degree of occupation the inner space of the material with reinforcing elements.

3.2 Discussion

The manufacture of the new type of construction material based on the geopolymerization reaction of some residual alumino-silicate materials (blast furnace slag and fly ash) in the presence of the alkaline activator solution has become very interesting due to the ecological benefits (elimination of greenhouse gas emissions) and energy (extremely low consumption) during geopolymer processing. Currently, the global ecological and energy problems that affect the entire planet have imposed urgent solutions. The construction sector is seriously affected by the need to stagnate the production of cement, the main binder of construction concrete.

Research from all over the world has recently been involved in proposing construction materials based on both porous and dense geopolymers to meet the technical requirements of this activity field. The current work falls within these concerns and the geopolymer composite presented in the work is a suitable material for this purpose.

4. CONCLUSION

The objective of the work was the experimental manufacture of high-strength geopolymer composite based on granulated blast furnace slag and coal fly ash, alumino-silicate materials resulting as by-products of the iron and steel industry, and the energy industry, respectively. The method of activating this material type for the geopolymerization reaction to take place is contained in Davidovits's invention and is based on the contact of alumino-silicate materials with the alkaline activator (aqueous solution of NaOH and Na_2SiO_3). The reinforcement of the material mixture was made with variable weight ratio of PET fibres (1.5-2.3 wt. %) recycled and processed from post-consumer PET bottles. The geopolymer composite is attractive as a construction material considering that its manufacture is ecological (no greenhouse gas emissions) and the required energy consumption is extremely low. From this point of view, this composite type represents an excellent alternative solution to construction materials based on cement, considered undesirable in the current conditions of the global ecological and energy crisis. The work originality results from the method applied by the authors for the curing process consisting in the own alternation of curing at room temperature for 20 hours with that of maintaining at 75 ºC for 10 hours continued with curing at room temperature for 7 and respectively, 28 days in order to obtain high values of the mechanical strength of the geopolymer composite. The product had remarkable mechanical properties (compression strength of 60.1 MPa and flexural strength of 11.5 MPa) corresponding to the maximum proportion of PET fibres of 2.3 wt. % and final curing process time of 28 days.

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