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THE USE OF KERATIN PROTEIN AS ANIMAL FIBERS TO MAKE A GEOPOLYMER BASED ON ALUMINO-SILICATE INDUSTRIAL BY-PRODUCTS

BY

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Abstract. Keratin protein fiber existing in poultry feathers was incorporated (in the maximum volumetric proportion of 1.7%) in the starting mixture prepared for making a geopolymer concrete based on alumino-silicate industrial by-products (granulated blast furnace slag and fly ash). The aqueous solution of the alkaline activator composed of NaOH and Na_2SiO_3 facilitated the development of the geopolymerization reaction generating the geopolymer. After the curing process of fresh geopolymer, the strengthened specimens kept for additional 7 and 28 days were investigated to determine their mechanical and physical characteristics. The results of investigations showed an increase in compression strength up to 51.2 MPa and flexural strength up to 15.8 MPa (after 28 days). The geopolymer made from waste with very low energy consumption and in ecological conditions is suitable for its application in construction sector.

Keywords: keratin protein, animal fiber, geopolymer, blast furnace slag, flexural strength.

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1. Introduction

Under the current conditions of a deep global ecological crisis due to the climate disruption caused by the damage to the ozone protective layer of the planet as well as an older hydrocarbon crisis that started over 40 years ago, the industry in general and the construction materials industry in particular have undergone major changes. A new trend is becoming increasingly important regarding the relationship with waste and industrial by-products, having as objective their large-scale recycling (plastic, metal, glass, food waste, paper, textile, etc.) and the partial or total abandonment of certain raw materials whose manufacture requires high energy/fossil fuel consumption and which release in the atmosphere large amounts of greenhouse gases (CO_2) that destroy the ozone layer (e.g. cement).

Except for the most commonly used industrial by-products (coal fly ash and granulated blast furnace slag), numerous recycled materials have allowed the manufacture of new products, especially suitable for the construction materials industry: residual glass (Higuchi *et al*., 2021), construction materials from demolition (Wagih *et al.*, 2013), residual rubber (Siddika *et al.*, 2019). On the other hand, natural organic waste such as coir, palm, kenaf, jute, sisal, banana, sugarcane, bamboo, etc. (Suhail *et al.*, 2022; Shadheer Ahamed *et al*., 2020)) and even animal waste (Kurien *et al*., 2022) started to be used as an addition in composite materials, especially for their reinforcement, thus avoiding expensive materials such as fibers of steel (Chanh, 2023), carbon (Atiyeh and Aydin, 2020), glass (Devi *et al*., 2022), etc.

An exceptional invention belonging to the French researcher Davidovits *et al.*, (1994), which has already proven its usefulness since the end of the 20th century, radically changed the composition and manufacturing technique of the most well-known and strength construction material: cement-based concrete. Alumino-silicate materials in the form of waste and industrial by-products (mainly fly ash and granulated blast furnace slag), with suitable pozzolanic properties as a binder replacing cement, were activated in contact with high alkaline liquid solutions containing NaOH (or KOH) and Na_2SiO_3 or (K_2SiO_3) (Nodehi and Taghvaee, 2022) initiating and developing the complex reaction of geopolymerization that allows the transformation of silica and alumina-rich waste into geopolymers. These new materials have even better mechanical characteristics compared to ordinary concrete, require much lower energy consumption and pollutant emissions are also greatly lowed.

The current work was based on the design of the most used secondary products of the energy industry (fly ash) and the metallurgical industry (granulated blast furnace slag). Several previous papers presented in the literature of the last 10-15 years used the two types of residual materials either separately or together for making the geopolymer concrete.

The method of preparing a geopolymeric composite reinforced with carbon fiber (5 vol. %) was presented by Yan et al., 2016. The geopolymeric gel obtained by activating the calcined metakaolin powder (alumino-silicate material) with the alkaline activator solution was poured over the carbon fiber felt. The flexural strength of the reinforced composite reached a remarkable maximum value of 51.5 MPa.

Short steel fibers (2 wt. %) were incorporated into the mixture for making fly ash-geopolymer through alkaline activation with NaOH and Na_2SiO_3. The significant improvement of the strength of geopolymer at multiple cracks and the flexural strength were experimentally observed (Shaikh, 2013).

Although the effectiveness of the incorporation of steel and carbon fibers for geopolymer reinforcement has been practically proven by increasing the mechanical characteristics, the high cost of these fiber types is disadvantageous for their large-scale application (Cai *et al*., 2022).

The comparative analysis of several types of geopolymer-reinforcing fibers was carried out by Shaikh in 2021. Thus, the use of polyethylene terephthalate (PET) fibers in a fly ash-based geopolymer subjected to hot curing process at 65-70ºC as well as in a geopolymer based on fly ash and granulated blast furnace slag cured at room temperature were compared with cement-based composites reinforced with the same type and proportion of fibers. Also, the analysis included composites reinforced with polypropylene fibers. Compression strength of reinforced composites was significantly higher (by 10-13%) than cement-based composites. The geopolymer based on fly ash and granulated blast furnace slag reinforced with PET fibers had slightly higher compression strength compared to fly ash-based geopolymer reinforced with PET fibers. The compression strength of all the composites reinforced with polypropylene fibers was slightly higher compared to the composites reinforced with PET fibers. By growing the volumetric ratio of PET fibers with 1-1.5%, the increase of flexural strength of both composites was observed. The composite based on cement reinforced with PET fibers did not show modifications in flexural strength and reinforcing with polypropylene fibers indicated an inverse trend.

The use of polyvinyl alcohol fibers as an increasing method of mechanical strength of geopolymer composite was analysed by Cai *et al*., 2022. These are mono-filament fibers which, incorporated in concrete or geopolymer structure, generate a network of multi-directional fibers favouring the reinforcement process of material. The work presented the reinforcement with polyvinyl alcohol fibers and powder, respectively, of a geopolymer based on fly ash and granulated blast furnace slag activated with an alkaline solution consisting of NaOH and Na_2SiO_3. The results showed that the reinforcement with fibers was more effective than that with powder. Optimal values of compression strength and flexural strength were 41.11 MPa and 8.43 MPa, respectively.

Comparative results of the reinforcement of a cement-based concrete with polyvinyl alcohol fibers are shown in (Jabour *et al*., 2021). The concrete was made with Portland cement (450 kg·m^{-3}), fine aggregates (930 kg·m^{-3}), coarse aggregates (760 kg·m^{-3}), and polymer fibers (within the limits of 0.5-1 vol. %). After 28 curing days, the compression strength value reached 41.5 MPa.

Another modern manufacturing technique of fly ash-based geopolymer also including alccofine (between 5-20%), a microfine material based on low calcium silicate slag, and zeolite sand (between 10-40%) as raw materials was tested by Ganesh Babu *et al.*, 2021. Rubber fibers (2%) were added as reinforcing fibers. The activation of alumino-silicate materials was performed with the alkaline solution consisting of NaOH and Na_2SiO_3. After 7 and 28 curing days, compression strength and tensile strength increased by 46 and 14% respectively, compared to ordinary concrete. Instead, the workability decreased with the increase of zeolite ratio.

Fibers of vegetable origin (jute, flax, cotton, sisal, hemp, coir, palm, kenaf, sugarcane, bamboo, pineapple) were also used in both cement-based composites and geopolymer composites (Laverde *et al.,* 2022; Shadheer Ahamed *et al*., 2020; Zulfiati *et al*., 2019). To a lesser extent, some animal fibers (wool, hair, poultry feathers, mulberry silkworms) were incorporated into the composition of mixtures for different composites (Alyousef *et al*., 2020; Mendoza *et al*., 2019; Kurien *et al.,* 2022).

An animal fiber type containing keratin protein has been found in animal body. If α-keratin exists in all animals forming hooves, claws and horns, β-keratin is produced only in the body of birds, reptiles and amphibians (Keratin, 2023). This sulfur-rich protein has been tested in several applications and it has been found that it can be effectively used as reinforcing fiber in concretes (in volumetric ratios below 2%) influencing their mechanical and physical properties, especially the flexural strength (Hamoush and El-Hawary, 1994, Reddy and Yang, 2007).

As mentioned above, the current work has adopted the solution of using together the most important alumino-silicate industrial waste (granulated blast furnace slag and fly ash) as well as keratin protein fibers recovered from chicken feathers, i.e. animal fibers, for producing under ecological and economic conditions a high-strength geopolymer concrete reinforced with natural fibers.

2. Materials and Methods

According to investigations carried out by Tesfaye *et al.*, 2017, the chicken feather includes crude protein (82.4%), crude fibers (2.2%), crude lipid (0.8%), ash (1.5%), and water (12.3%). In terms of chemical composition, the feather contains carbon (64.5%), oxygen (22.3%), nitrogen (10.4%), and sulfur (2.6%).

The first operation of feather processing was washing and sterilizing them with deionized water mixed with NaOH solution and detergent, followed by free drying at ambient temperature, at a humidity of less than 65% (Reddy and Yang, 2007; Alonso *et al.*, 2013).

Among the component parts of the chicken feather, barbs represent the keratin-rich lateral threads that form from the main branch of the feather. To use the barbs in the experiment, they were cut to lengths of 8-15 mm. The bulk density of fibers was determined to be below 0.3 $g{\cdot}cm^{-3}$, much lower compared to other mineral or natural fibers (over 1.5 $g{\cdot}cm^{-3}$) used for reinforcing concretes or other composites.

Alumino-silicate industrial by-products used in experiment for making the geopolymer concrete completely substituting the Portland cement were granulated blast furnace slag provided by ArcelorMittal Galați (Romania) over 10 years ago and fly ash coal supplied by Paroseni-Thermal power plant (Romania) 7-8 years ago when this plant used anthracite as fuel and the CaO content in fly ash was low (under 5%) (Paunescu *et al.*, 2023b). Granulated slag with initial grain size between 2-6 mm was ground to below 80 μm and fly ash supplied below 200 μm was also ground to grain size below 60 μm. The chemical composition of wastes was determined on the X-ray fluorescence spectrometer type AXIOS-sequential from Metallurgical Research Institute Bucharest. The results are shown in Table 1.

Table 1

Chemical composition of wastes (wt. %)

Waste	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	TiO_2	MnO	Na_2O	K_2O	SO_3
Slag	37.4	6.4	6.9	39.9	3.5	-	2.3	0.1	0.2	-
Ash	54.4	26.5	4.8	3.5	2.5	1.5	-	0.4	0.6	1.7

According to Davidovits' invention (Davidovits *et al*., 1994), the recommended method for activating alumino-silicate waste and developing the geopolymerization reaction is the use of a highly alkaline solution composed of NaOH and Na_2SiO_3. The weight composition of Na_2SiO_3 includes 35% SiO_2, 16.8% Na_2O, and 46.4% water (Vora and Dave, 2013; Blaszczyński and Król, 2017). The method was also adopted in this experiment. 10M NaOH in the form of flakes soluble in water and Na_2SiO_3 aqueous solution (concentration 38%), both available in the market, were mixed to form the alkaline activator necessary for the production of geopolymer.

Quartz sand as fine aggregate containing 98.8% SiO_2 and having the grain size under 2 mm as well as gravel as coarse aggregate containing 87.5% SiO_2, 6.1% Al_2O_3, 2.1% Na_2O, and 1.6% Fe_2O_3 having the grain size within the limits of 5-12 mm (of which 80% represented the range between 5-8 mm) were the other solid components of mixture.

Several testing versions (presented in Table 2) were adopted for this experiment, in which the fly ash/blast furnace slag ratio varied between 2.46-3.50, the total amount of alumino-silicate waste being constant at 450 $kg{\cdot}m^{-3}$.

Volumetric proportion of animal fibers was within the limits of 0.7-1.7% (i.e. between 2-5 kg·m^{-3}), avoiding approaching the maximum limit of 2% experimentally determined (Alonso *et al.*, 2013) which affected the geopolymerization process and the increasing trend of the mechanical strength value (especially the flexural strength) of geopolymers. Fine and coarse aggregate amounts were maintained at constant values (880 and 970 kg·m^{-3}). Molarity of NaOH solution was chosen at 10M and the ratio of alkaline activator components Na_2SiO_3/NaOH was constant (2.00) in all testing versions. Alkaline activator/alumina-silicate binder had values in the range of 0.77-0.81.

Table 2
Composition of testing versions

Composition	Version 1 (kg·m^{-3})	Version 2 (kg·m^{-3})	Version 3 (kg·m^{-3})	Version 4 (kg·m^{-3})
Granulated blast furnace slag	100	110	120	130
Fly ash	350	340	330	320
Chicken feather fiber	2	3	4	5
Fine aggregate (sand < 2 mm)	880	880	880	880
Coarse aggregate (gravel between 5-12 mm)	970	970	970	970
10M NaOH solution	116	118	120	122
Na_2SiO_3 solution	232	236	240	244
Water addition	50	55	60	65

The first rule of preparing the mixture for the manufacture of geopolymer concrete was the separate preparation of the liquid and solid components, respectively. NaOH flakes dissolved in water and Na_2SiO_3 were stirred for 4 min at a rate of 300 rpm. Separately, ground slag and fly ash were mixed for 3 min with an electric stirrer and then animal fibers were inserted into this mix and the stirring process continued for another 3 min at a rate of 300 rpm. The next stage of preparing the raw material was the slow pouring of the liquid mixture over the homogenized solid, stirring this mixture until the formation of the gel and pouring the gel into metal molds for the curing process of the fresh material. The last stage of the geopolymer concrete manufacturing process was curing the material, first by blowing steam at 80ºC for 24 hours, followed by curing at room temperature for 48 hours. After removing from the molds, the geopolymer products were kept at room temperature for 7-28 days. The measurement of their mechanical characteristics was performed after 7 and 28 days, respectively.

The basic principle of turning alumino-silicate waste into a geopolymer is creating conditions for the development of the geopolymerization reaction, according to Davidovits' invention. This complex reaction initiated by alkaline activation with NaOH solution of silica and alumina-rich materials that generates dissolution and hydrolysis of silicon and aluminium occurs according to following reactions (Paunescu *et al.*, 2023a; Nawaz and Sivakumar, 2020; Davidovits, 1989).

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

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

As a result of these chemical equations (at room temperature), a gel is produced, whose structural features are changed and several gel phases are formed. The effect of these transformations was the polymerization of the fresh material, which by curing became a solidified product. 3D-structure of the type of polymer chain with Si-O-Al-O bonds characterizes the new material called "geopolymer" (Singh, 2018).

The investigation methods adopted for determining the geopolymer concrete features are presented below. The density was measured based on the regular geometric shape of the sample through its weighing and dividing this value to the calculated sample volume (Metrology, 2015). Apparent porosity was determined using the ISO 15901-2:2022 standard. The method of immersing the concrete specimen under water (ASTM D570) was used to identify the water volume absorbed by the material during 1 day. 100 kN-compression fixture Wyoming Test Fixture was used for measuring the compression strength value of geopolymer (A Practical Guide, 2018). The flexural strength measuring was carried out on the universal testing machine (SR EN ISO 14125:2000). Biological Microscope MT5000 model (1000 x magnification) was used for investigating the microstructural appearance of geopolymer concrete specimens.

3. Results and Discussion

The preparation of testing versions of geopolymer concrete reinforced with animal fibers was made at an experimental scale. The curing treatment of the fresh material, first with steam of 80ºC for 24 hours and then at room temperature for 48 hours was performed. After this treatment, specimens were kept at room temperature for 7-28 days before the determination of geopolymer characteristics, whose results are presented in Table 3.

Table 3

Main features of cured geopolymer concrete samples

Feature	Version 1	Version 2	Version 3	Version 4
Density (kg·m^{-3}) - after 7 days - after 28 days	 2428 2454	 2414 2440	 2400 2431	 2392 2417
Apparent porosity (%) - after 7 days - after 28 days	 23.3 22.7	 23.5 23.0	 23.6 23.1	 23.7 23.2
Compression strength (MPa) - after 7 days - after 28 days	45.2 50.0	45.4 50.6	45.5 50.9	45.6 51.2
Flexural strength (MPa) - after 7 days - after 28 days	 14.0 14.4	 14.6 15.0	 15.0 15.5	 15.2 15.8
Absorbed water (vol. %) after 28 curing days	4.0	3.6	3.0	2.8

The progressive increase in the weight proportion of chicken feather fiber from 2 to 5% led to structural changes in the geopolymer concrete, which influenced its physical and mechanical characteristics. According to the data in Table 3, the density of the specimens was reduced both in the case of determinations made after 7 days, and those made after 28 days.

Thus, the density value decreased from 2428 to 2392 kg·m^{-3} in the first case and from 2444 to 2397 kg·m^{-3} in the second. By increasing the storage time of the geopolymer from 7 to 28 days, in the conditions of obtaining a more compact material, a slight increase (by about 1.1%) of the density was observed. Thus, after 28 days, the density of the four versions decreased from 2454 to 2417 kg·m^{-3}.

The apparent porosity was inversely influenced by the evolution of density values. However, the increase in porosity was insignificant in terms of value after the two storing periods.

The mechanical strength (compression and flexural strength) was improved after storing the geopolymer for 7 and 28 days. Compression strength increased more with the addition of 2-3 kg·m^{-3} (versions 1-2) of animal fiber and less with the addition of 4-5 kg·m^{-3} (versions 3-4) after storage for 7 days. The strength reached the highest value (45.6 MPa) corresponding to version 4. The increase in the compression strength value was recorded in the entire range of animal fiber addition (2-5 kg·m^{-3}) reaching the maximum level of 51.2 MPa after 28 days corresponding to version 4 (with 5 kg·m^{-3} of fiber).

Flexural strength was favourably influenced to the highest degree by the incorporation of chicken feather fibers in volumetric ratios of maximum 1.7% (i.e. 5 kg·m^{-3}). According to the literature (Rangan, 2008), the maximum limit of chicken feather fiber incorporation of 2% affects the flexural strength of geopolymer and causes its value to begin to decrease. Therefore, the fiber maximum proportion tested in this experiment was below the mentioned limit. Flexural strength values were within the limits of 14.0-15.2 MPa after 7 days and 14.4-15.8 MPa after 28 days, being higher compared to usual values of geopolymer concrete without natural fiber addition.

Geopolymer-absorbed water determined by its immersion under water has shown that proportions between 2.8-4.0 vol. % characterizes this property of the material. In the case of the highest amount of animal fiber used in the experiment (5 kg·m^{-3}), the level of absorbed water reached the minimum value of 2.8 vol. %. Similar results were reported in the literature (Luhar and Khandelwal, 2015) in the case of making fly ash-based geopolymer concrete without reinforcing fiber addition.

Images of geopolymer concrete specimens reinforced with animal fibers are presented in Fig. 1.

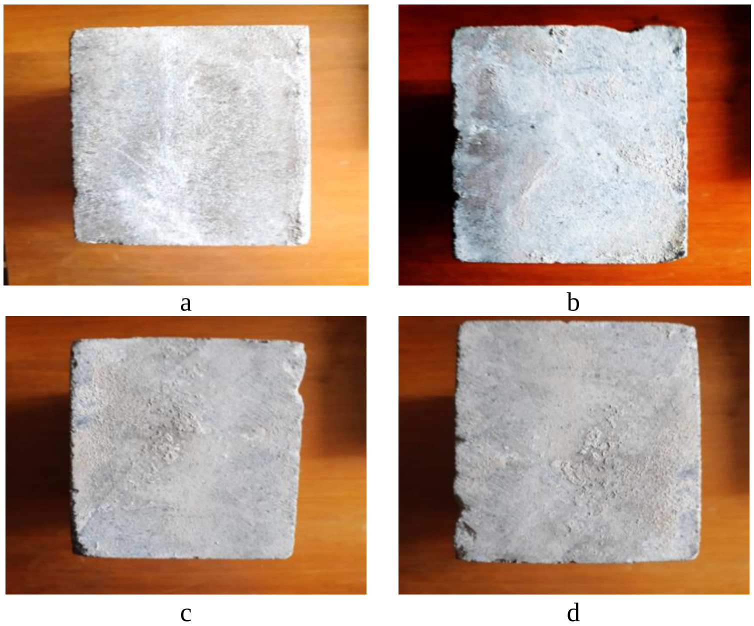

Fig. 1 − Images of geopolymer concrete specimens reinforced with animal fibers; a – version 1; b – version 2; c – version 3; d – version 4.

Microstructural appearance of geopolymer concrete specimens reinforced with animal fibers is shown in Fig. 2.

The geopolymer pictures reveal the robustness and compactness of the four testing versions. Practically, it is difficult to identify the specimens according to the macrostructural aspect provided by Fig. 1. According to Fig. 2, the distribution of chicken feather fibers is visible in microstructural images, the volume occupied by them increasing from version 1 to version 4.

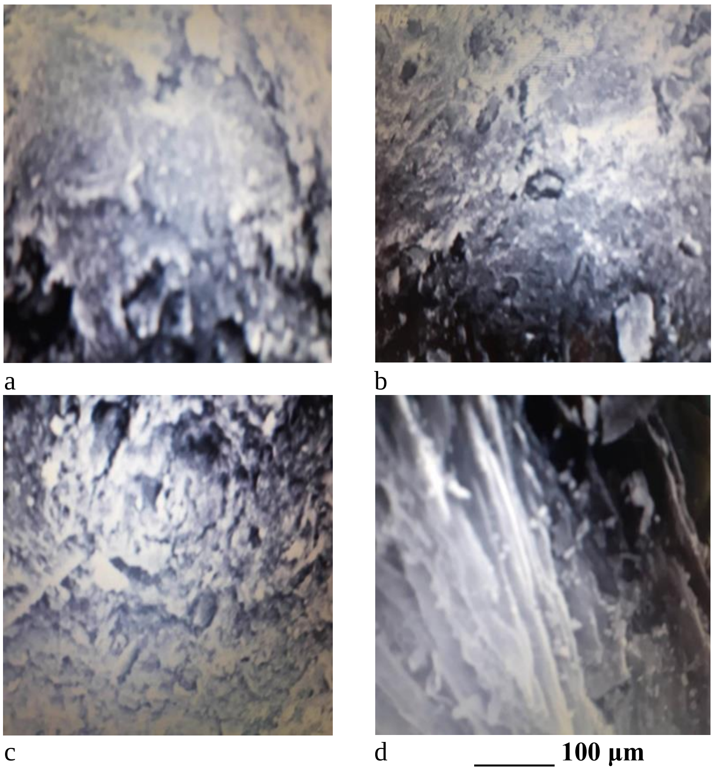

Fig. 2 − Microstructural appearance of geopolymer concrete specimens reinforced with animal fibers;
a – version 1; b – version 2; c – version 3; d – version 4.

In general, the predominant objective of making the geopolymer concrete as well as of cement-based concrete is to obtain high mechanical properties and especially, high-compression strength. A wide range of mineral and natural fibers were applied to strengthen concrete or geopolymer and the obtained results were conclusive. The highest performances were achieved using corrosion-resistant steel fibers, first applied in the case of cement-based concrete and recently, also extended in the case of geopolymer concretes (Liu *et al.*, 2020; Aisheh *et al.*, 2022). Even though the workability of fresh geopolymer depreciates with the increase in fiber content and the reduction of their diameter, the mechanical strength reaches very high values (up to 180 MPa). The use of granulated blast furnace slag in the mixture for producing geopolymer concrete reinforced with steel fibers is indicated for obtaining a very high level of compression strength (Gülsan *et al.*, 2019).

Geopolymer concrete was designed as an inexpensive and ecological silica and alumina-rich material using waste and industrial by-products. Unlike these peculiarities of raw material, stainless steel fibers are very expansive materials, which require high primary energy consumption. Therefore, the idea of using a natural fiber type, available in satisfactorily large quantities, with extremely low processing costs, was chosen by the authors for the reinforcement of geopolymer. An animal fiber type as a food industry waste represented by chicken feather fibers was adopted.

Except for the advantage of improving the mechanical characteristics of geopolymer concrete, the technical valorization of animal waste in geopolymer manufacturing process represents a solution to protect the environment by reducing the residual amount of feathers thrown to the landfill.

The important role of the curing process of fresh geopolymer should not be neglected, it greatly contributes to the improvement of the mechanical strength of material. The techniques used to carry out the curing process are relatively different, being adopted by each manufacturer. In this experiment, own treatment methods tested in several previous processes were used, which, although they are basically similar to traditional methods, have peculiarities that differentiate them.

4. Conclusions

The work aimed at making a geopolymer concrete based on granulated blast furnace slag and fly ash using chicken feather fibers as a food industry waste. The principle of alumino-silicate waste activation through contact with the alkaline activator solution consisting of NaOH and Na_2SiO_3 for the development of the geopolymerization reaction and the generation of geopolymer concrete, according to Davidovits' invention, was adopted in the experiment. Animal fibers were incorporated into the starting mixture in volumetric proportions between 0.7-1.7% (i.e. 2-5 $kg \cdot m^{-3}$) in four test versions. The effect of animal fibers was tested for the first time in the case of a geopolymer concrete, after several experiments on cement-based concrete were previously reported in the literature. After carrying out the curing process of fresh geopolymer with steam at 80ºC for 24 hours, followed by the treatment at room temperature for 48 days and then keeping the specimens for 7 and 28 days at room temperature, the mechanical and physical characteristics of final products were determined. The highest values of compression strength (51.2 MPa) and flexural strength (15.8 MPa) were obtained in the version using the maximum volumetric proportion of feather fibers (1.7%). In this version, the density had minimum values (2392 $kg \cdot m^{-3}$ after 7 days and 2417 $kg \cdot m^{-3}$ after 28 days) and adsorbed water was minimal (2.8 vol. % after 28 days).

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UTILIZAREA PROTEINEI CU CHERATINĂ CA FIBRE DE ORIGINE ANIMALĂ PENTRU A PRODUCE UN GEOPOLIMER PE BAZĂ DE PRODUSE SECUNDARE INDUSTRIALE ALUMINO-SILICATICE

(Rezumat)

Fibra proteică de cheratină existentă în penele păsărilor de curte a fost încorporată (în proporție volumetrică maximă de 1,7%) în amestecul de pornire pregătit pentru realizarea unui beton geopolimer pe bază de produse secundare industriale alumino-silicatice (zgură granulată de furnal și cenușă zburătoare). Soluția apoasă a activatorului alcalin compus din NaOH și Na_2SiO_3 a facilitat dezvoltarea reacției de geopolimerizare, care generează geopolimerul. La finalul procesului de întărire a geopolimerului proaspăt, probele întărite stocate pentru încă 7 si 28 zile au fost examinate pentru determinarea caracteristicilor lor mecanice și fizice. Rezultatele investigațiilor au arătat o creștere a rezistenței la compresiune până la 51,2 MPa și a rezistenței la încovoiere până la 15,8 MPa (după 28 zile). Geopolimerul realizat din deșeuri cu un consum foarte redus de energie și în condiții ecologice este adecvat pentru aplicarea sa în sectorul construcției.