Environmentally friendly technique for making high-strength geopolymer concrete reinforced with animal fibers as food industry waste

Tehnică ecologică de producere a unui beton geopolimeric cu rezistență înaltă armat cu fibre animale ca deșeuri ale industriei alimentare

Lucian Păunescu[1], Adrian Ioana[2], Bogdan Valentin Păunescu[3]

[1] Cosfel Actual SRL
95-97 Calea Grivitei street, M4 room, sector 1, Bucharest 010705, Romania
E-mail: lucianpaunescu16@gmail.com

[2] University „Politehnica" of Bucharest, Faculty of Science and Materials Engineering,
313 Independence Splai, sector 6, Bucharest 060042, Romania
E-mail: adyioana@yahoo.com

[3] Consitrans SA
56 Polona street, sector 1, Bucharest 010504, Romania
E-mail: pnscbogdan@yahoo.com

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Abstract. *Applying the method of reinforcing cement-concrete with chicken feather fibers was extended to fly ash-based geopolymer concrete. Four manufacturing recipes were used, in which the fiber amount was modified between 6-9 kg·m^{-3} and NaOH molarity in the alkaline activator varied from 8M to 10M. Curing process of the fresh geopolymer was performed with 80 ºC-steam for 24 hours and then at room temperature for 36 hours. The specimen mechano-physical characterization was made after 7 and 28 days and the results showed obtaining high values of compression strength of 45.1 and 47.8 MPa and flexural strength of 14.5 and 15.6 MPa.*

Key words: *geopolymer concrete, chicken feather fiber, fly ash, alkaline activator, mechanical strength.*

Rezumat. *Aplicarea metodei de armare a betonului din ciment cu fibre din pene de pui a fost extinsă la betonul geopolimeric pe bază de cenușă zburatoare. Au fost utilizate patru rețete de fabricare , în care cantitatea de fibre a fost modificată între 6-9 kg·m^{-3}, iar molaritatea NaOH în activatorul alcalin a variat de la 8M la 10M. Procesul de îmtărire a geopolimerului proaspăt a fost efectuată cu abur la 80 ºC timp de 24 ore și apoi la temperatura camerei timp de 36 ore. Caracterizarea mecano-fizică a probei a fost făcută după 7 și 28 zile, iar rezultatele au arătat obținerea unor valori mari ale rezistenței la compresiune de 45,1 și 47,8 MPa și rezistenței la încovoiere de 14,5 și 15,6 MPa.*

Cuvinte cheie: beton geopolimeric, fibră din pene de pui, cenușă zburătoare, activator alcalin, rezistență mecanică.

1. Introduction

Cement-based concrete has long been considered the most used construction material, the change in its hierarchical position being due to the ecological and energy crisis that our planet is currently facing. Portland cement is the concrete component that generated to a great extent this serious crisis and, starting from the end of the last century, it seems that geopolymer concrete [1] has become the replacement material.

Traditional concrete had some disadvantageous features including low values of tensile strength, ductility, and energy absorption. The low level of tensile strength was explained by low toughness as well as structural defects and microcracks of concrete. The solution considered optimal for increasing toughness was the incorporation of reinforcing fibers [2].

The first mention in the literature of the use of fibers in the composition of concrete in the modern age of technology was in 1963 [3]. The first fibers were wires in a metal reinforcement placed into the concrete mass, having the role of avoiding the internal cracks propagation. Since from the 1960s, the reinforcement method with steel fibers dispersed in cement-based concrete has seen a wide development in construction. The decrease of shrinkage and implicitly, shrinkage cracking of fresh concrete during the curing process is one of the advantages of using fibers in the concrete structure. Theoretically, this crack type appears in the unreinforced material when the evaporation rate of water on the surface of concrete is greater than the rate of water migration from the inside to the surface. The shrinkage gradient causes the appearance of surface cracks, which can be counteracted for example by a very low volume of added steel fibers [2].

Other mechanical properties of concrete such as wear resistance, durability, and fatigue resistance have been improved by applying the fiber reinforcement technique not only in the field of buildings, but also in bridges, highways, airport runways, etc. In load-bearing applications, the use of steel fiber reinforcement is a frequently encountered procedure [2].

Normally, the concrete reinforcing fibers should be easily embedded in the volume of the material mixture, to be resistant in the cementitious medium and to develop a good mechanical strength. The main disadvantage of the use of steel fiber reinforcement still remained the high cost of corrosion-resistant steel fibers [4]. Except steel, other materials such as glass, natural cellulose, carbon, nylon, polyethylene, etc. are already known in the form of concrete reinforcement fibers [5, 6].

Different solid wastes such as rubber, paper, plastic, textiles, etc. present special interest of researchers for their use as concrete reinforcing fibers. Thus, the role of fibers recovered from residual tires on the concrete shrinkage was researched in [2] and recycled fibers from paper waste coming from used journals were analyzed for cement reinforcement [7]. Steel shavings have been tested as fibers in concrete [8] and food packaging waste has provided usable fibers in concrete subjected to extreme climatic conditions of successive freeze-thaw [9].

Various natural fibers coming from sisal, jute, coir, bamboo, hemp, flax, and others were experimentally investigated for their using in concrete structure. The results showed that sisal and coir fibers have the most important contributions to improving the mechanical features of concrete [10].

Among the natural fibers tested in concrete reinforcement, the use of animal fibers is obviously less frequent. Previous research has revealed that the protein called "keratin" identified in wool [11], mulberry silkworms, and poultry feathers [12-14] plays a major role in concrete reinforcing. According to the mentioned literature, chicken feather fiber is an excellent substitute for synthetic fibers. Feather barbs have a unique peculiarity regarding its cross-section, which is no longer found in other natural fibers based on protein (wool and mulberry silkworm).

By adding animal fibers to the initial material mix, higher durability and mechanical resistance have been experimentally proven in recent decades. Poultry feathers are still largely considered food industry waste and are thrown into landfills, harming the environment health in the respective areas. The qualitative influence of some mechanical properties of concrete due to poultry feathers embedded in its structure was tested [15]. Mainly, the flexural strength is favoured, while the material density decreases by comparing with ordinary concrete based on Portland cement. It was found that the flexural strength was improved after 14-56 curing days in the case of 1% of chicken feathers addition, while this mechanical effect was observed only after 56 curing days in the case of concrete with 2% of feathers addition. Increasing the proportion of feathers above this limit contributed to the decrease in flexural strength.

The present work aimed at the application of a still untested solution for manufacturing the fly ash-based geopolymer concrete using residual chicken feathers as an addition of natural animal fibers for the geopolymer reinforcement. In this situation, the components of the mixture have completely ecological features. Adopting the recent remarkable invention of the French researcher J. Davidovits [1], the geopolymer is manufactured on the basis of waste or industrial by-products consisting of alumino-silicate materials with pozzolanic properties, which have the ability to completely replace ordinary Portland cement. The conditions for manufacturing cement using the actual technology are totally inadequate to current ecological and energetic requirements, while coal fly ash used as a raw material in the geopolymer concrete composition is a by-product of the energy industry. In the experiment presented in this paper, the technique of activating the alumino-silicate waste indicated by the inventor was adopted, i.e. its contact with very alkaline aqueous solution consisting of $NaOH$ and Na_2SiO_3 capable of developing the geopolymerization reaction. This complex reaction leads to the transformation of alumino-silicate waste into a geopolymer with excellent mechano-physical properties. The originality of the work is the use of natural animal fibers (particularly, chicken feather fibers) in the process of reinforcing the geopolymer concrete. Animal fibers have previously been applied in the case of cement-based concrete, but they have not been tested so far in the case of geopolymer concrete, practically the substitute of traditional cement-based concrete.

2. Materials and methods

So-called "barbs" of the chicken feather are the keratin-rich lateral parts, which start from the main branch of the feather. Several previous investigations presented in the literature on the chicken feather embedded in the concrete structure, showed the possibility of modifying mechanical and physical features of the traditional cement-based concrete. Barbs of the chicken feathers were cut with scissors at maximum lengths of 15 mm in preparing process for their use as reinforcing fibers in this experiment. Compared to other natural fibers (natural cellulose and wool), the density value of chicken feathers is significantly low (below 0.8 $g \cdot cm^{-3}$) [16]. According to the literature [17], chicken feather contains 82.36 % crude protein, 2.15 % crude fibers, 0.83 % crude lipid, 1.49 % ash, 12.33 % moisture. Its chemical composition includes 64.47 % carbon, 10.41 % nitrogen, 22.34 % oxygen, and 2.64 % sulphur. Processing feathers for their use in experiment included the following operations. First, the feathers were washed and sterilized with water deionised mixed with 6 % NaOH solution and detergent to remove dirt. Then, they were freely dried at room temperature (22-25 ºC) in conditions of relative humidity of 65 %, according to some recommendations taken over from [14, 16].

The basic recipes of making fly ash-based geopolymer concrete both in variants of making porous products and in variants of high-strength products are generally known from the literature.

According to the paper [18], in the manufacturing process of geopolymer concrete, it is indicated to use class F-fly ash characterized by the low content of CaO (below 5 %), because higher weight proportions can affect the geopolymerization reaction and the microstructure of the final product. Class F-fly ash represents the ash resulting from burning anthracite or bituminous coal. The ash used in the experiment was part of the batch of fly ash provided by Paroseni-Thermal Plant (Romania) in 2016, when the power plant used anthracite in the combustion process. The chemical composition of fly ash from Paroseni plant included 54.4 % SiO_2, 26.5 % Al_2O_3, 4.8 % Fe_2O_3, 3.5 % CaO, 2.5 % MgO, 1.5 % TiO_2, 0.4 % Na_2O, 0.6 % K_2O, 1.7 % SO_3, and 2.3 % LOI. The particle size of fly ash used during the experiment was below 60 µm obtained as a result of supplementary grinding, the initial size of ash being below 200 µm.

Alkaline activator adopted by authors was composed of 8M NaOH or 10M NaOH in form of solid flakes dissolved in deionized water and 38 %-Na_2SiO_3 aqueous solution. Na_2SiO_3/NaOH ratio had values in the range of 1.43-2.50.

The usual aggregates (fine and coarse) were also introduced into the composition of solid mixture. Silica-rich quartz sand was the fine aggregate (560 $kg \cdot m^{-3}$) having 98.9 % SiO_2. Its grain dimension was below 2 mm. Gravel (87.5 % SiO_2 and 6.1 % Al_2O_3) constituted the coarse aggregate, the grain size varying within the limits of 4-14 mm. Over 75 % of the amount of coarse aggregate (1290 kg·m^{-3}) had the grain size between 4-8 mm. The amounts of the two aggregate types were kept constant during the experiment.

The water addition amount was in the range of 5-15 kg·m^{-3}, being influenced by the NaOH molarity between 8M and 10M.

To make the geopolymer concrete reinforced with animal fibers, four testing versions were adopted according to the data in Table 1.

Table 1

Composition of the testing versions

Composition	Version 1 (kg·m^{-3})	Version 2 (kg·m^{-3})	Version 3 (kg·m^{-3})	Version 4 (kg·m^{-3})
Fly ash	410	410	410	410
Fine aggregate (sand)	560	560	560	560
Coarse aggregate (gravel)	1290	1290	1290	1290
Chicken feather fiber	6	7	8	9
8M NaOH solution	40	50	-	-
10M NaOH solution	-	-	60	70
Na_2SiO_3 solution	100	100	100	100
Water addition	5	5	15	15

The volumetric proportion of chicken feather fibers was tested in the range of 0.75-1.12 %, avoiding approaching the upper limit of the 2 % that affects the geopolymerization process [16]. Taking into account the fiber density value, their quantities were calculated at 6-9 kg·m^{-3}.

The first condition for processing geopolymer concrete is the separate preparation of the two types of mixtures (liquid and solid) in separate vessels. NaOH flakes (commercially available) dissolved in deionized water with molarities of 8M and 10M, respectively, were mixed with Na_2SiO_3 aqueous solution (also available on the market). Mixing was performed by stirring at a rate of 500 rpm for 5 min. Finely ground fly ash together with fine sand and coarse aggregate were mixed in another container for 3 min, then chicken feather fibers were added. Mixing was continued for another 3 min. The second step of the preparation process was pouring the liquid mixture containing the alkaline activator over the homogenized solid. Their mixing by stirring with a speed of 300 rpm for about 5 min took place until the formation of the gel. It was poured into several metal molds protected with thin plastic film. Introduced into a thermally insulated and sealed room, the fresh geopolymer was subjected to the curing process, first with steam at 80 ºC for 24 hours and then at room temperature for 36 hours. After completing this treatment, the hardened specimens removed from molds were also kept at room temperature until their characteristics were investigated, for variable periods of 7 and 28 days, respectively.

The investigation methods adopted for determining geopolymer concrete sample features are presented below. The density was measured based on the regular shape of the material by weighing it with an electronic balance and dividing this value to the calculated sample volume [19]. 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 evaluate the water volume absorbed by the material during 24 hours. 100 kN-compression fixture Wyoming Test Fixture was used for the identification of compression strength value of the geopolymer concrete [20]. The flexural strength measuring was carried out according to SR EN ISO 1412:2000. Biological Microscope MT5000 model (1000 x magnification) was used for analyzing the microstructural configuration of geopolymer concrete samples.

3. Results and discussion

Preparing the four testing versions of fly ash-based geopolymer concrete reinforced with chicken feather fibers was performed in laboratory conditions. After the curing treatment made in two stages, the first with 80 ºC-steam blowing for 24 hours and the second at room temperature for 36 hours, the cured products were investigated by usual mentioned methods for determining their mechanical and physical features after free storage at room temperature for 7 and 28 days, respectively.

Table 2

Main features of cured geopolymer concrete samples reinforced with chicken feather fibers

Feature	Version 1	Version 2	Version 3	Version 4
Density (kg·m^{-3})				
- after 7 days	2420	2406	2392	2384
- after 28 days	2435	2415	2398	2388
Apparent porosity (%)				
- after 7 days	23.1	23.3	23.4	23.5
- after 28 days	22.9	23.2	23.3	23.4
Compression strength (MPa)				
- after 7 days	44.8	45.0	45.1	45.1
- after 28 days	47.1	47.4	47.6	47.8
Flexural strength (MPa)				
- after 7 days	14.0	14.5	14.8	15.0
- after 28 days	14.5	14.9	15.3	15.6
Water uptake (vol. %) after 28 curing days	4.1	3.5	3.1	2.8

Changing the quantity of feather fibers from version 1 to version 4 between 6-9 kg·m^{-3} allowed to observe the structural modification of geopolymer influencing its density. It decreased from 2420 to 2384 kg·m^{-3} after 7 curing days as well as after 28 curing days from 2435 to 2388 kg·m^{-3}. By increasing the keeping time of geopolymer concrete specimens from 7 to 28 days, a normal slight increase of the material density was observed.

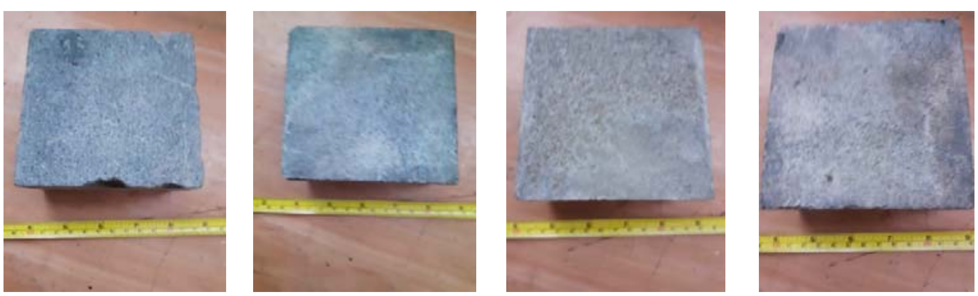
Normally, the apparent porosity is inversely influenced by the density value. In this experiment, the increase of porosity was insignificant both after storing the specimens for 7 days and after 28 days.

The use of animal fibers combined with the traditional technique of curing the fresh material led to reaching very high values of compression strength both after 7 days (44.8-45.1 MPa) and after 28 days (47.1-47.8 MPa). The increasing amounts of fibers in testing versions 1-4 influenced this strength, especially in the fiber amount range of 6-7 $kg \cdot m^{-3}$ and less in the range of 8-9 $kg \cdot m^{-3}$, in case of fresh products stored for 7 days and in the whole range in case of geopolymers stored for 28 days. Thus, the highest value of the compression strength (47.8 MPa) was reached by the specimen containing 9 $kg \cdot m^{-3}$ of chicken feather fibers (i.e. about 1.12 vol. %) stored for 28 days before measuring this mechanical strength type.

The influence of animal fiber addition on the flexural strength was beneficial keeping low enough volumetric proportion (below 2 %) not to cause the beginning decreasing trend of flexural strength experimentally found in [18]. According to the data in Table 2, the strength increased from 14.0 to 15.0 MPa after 7 curing days and from 14.5 to 15.6 MPa after 28 curing days. Flexural strength values were higher compared to other values of geopolymer concrete without natural fiber reinforcing.

Water uptake of geopolymer concrete specimens measured by immersion under water showed that volumetric proportions within the limits of 2.8-4.1 % were obtained. Increasing the amount of chicken feather fibers allowed lowering the level of absorbed water below 3 % in case of using 9 $kg \cdot m^{-3}$ of fibers. Almost similar results were obtained in case of manufacturing fly ash-based geopolymer concrete (without reinforcing fibers addition) presented in [21].

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



a b c d

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

Microstructural pictures of specimens made with fly ash as alumino-silicate waste and reinforced with chicken feather fibers as animal fibers are shown in Fig. 2.

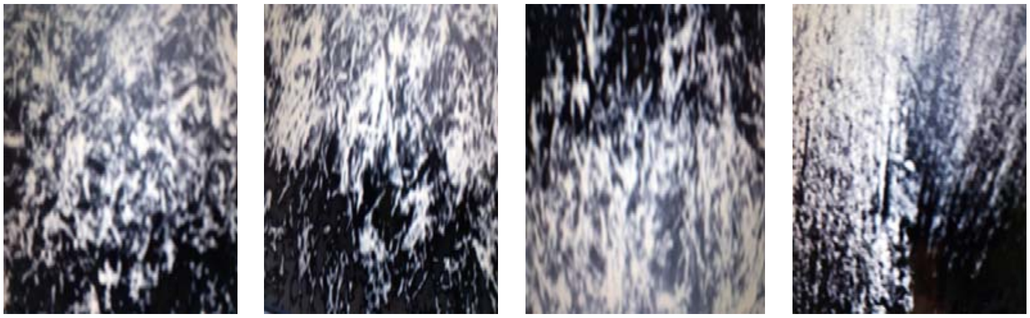


Fig. 2. Microstructural images of geopolymer concrete samples reinforced with animal fibers
a – testing version 1; b – testing version 2; c – testing version 3; d – testing version 4.

Images in Fig. 1 shows macrostructural compactness and robustness of all testing versions indicating very high mechanical strength of the made geopolymer samples. Fig. 2 indicates the presence of fibers in the microstructure of geopolymer samples composition in increasing volumetric proportion from version 1 to version 4.

The main concern of research in the field of manufacturing high-strength geopolymer concrete is to obtain high mechanical strength, especially compression strength. The addition of different fiber types applied to ordinary cement-concretes has proven to be effective. The best mechanical results were obtained with corrosion-resistant steel fibers. Recently, similar research on the use of steel fibers was also extended on geopolymer concretes [22, 23] and mechanical strength results were promising. Although the workability of the fresh geopolymer decreased with the increase in the content of fibers and the decrease in their diameter, the mechanical strength increased significantly, being reached extremely high values of compression strength (up to 180 MPa) in the particular case of slag-based geopolymer reinforced with steel fibers [24].

Geopolymer concrete is known as an inexpensive and environmentally friendly construction material using alumino-silicate waste and by-products. In the world, stainless steel fibers are very expensive products, their production involving high energy consumption. For this reason, in the current paper a type of animal fiber (poultry feathers) was chosen, which is practically a waste of the food industry in the field of poultry meat processing. The raw material is widely available, although the experiment presented above required only a very low amount of feathers supplied from a small private farm in Romania.

Except for the technical advantage of incorporating feather fibers in the manufacturing process of geopolymer concrete to increase its mechanical strength, the use of this animal waste contributes to regional environmental protection reducing the large amounts of feathers thrown into landfills.

It should be mentioned the important role of the curing process of fresh geopolymer (also applied in the case of ordinary cement concrete) on mechanical characteristics of the final product. Although, in principle, relatively similar steps are used, this process is adopted by the manufacturer having its own particularities. In the current experiment, authors applied their own curing mode, also used in other experiments with some changes. Considering the wide variety of mixture types used for the production of concrete, geopolymers or other similar construction materials the role of material type, amounts, processing degree before mixing, nature of admixtures, etc., on the one hand and the parameters of the curing process of fresh mixture, on the other hand, has not been precisely determined.

4. Conclusions

The main objective of the work was to test the reinforcement of a fly ash-based geopolymer concrete with chicken feather fibers. The volumetric proportion of these animal fibers was kept within a limited range between 0.75-1.12 % (i.e. 6-9 $kg \cdot m^{-3}$) in order not to affect the efficiency of the geopolymerization reaction of transforming the alumino-silicate waste (fly ash) into a geopolymer. The principle of fly ash activation was achieved through the method recommended by the remarkable Davidovits' invention, using aqueous solution of NaOH and Na_2SiO_3. Chicken feathers are residual materials of the food industry containing over 85 % keratin protein. The paper originality consists in the use of poultry feathers, non-tested in the world for reinforcing in the manufacturing process of geopolymer concrete. The own technique adopted for curing the flesh geopolymer concrete consisted of treatment by blowing steam at 80 ºC for 24 hours, followed by room temperature-curing for 36 hours. The investigation of physical and mechanical features of specimens was performed after their freely keeping at room temperature for 7 and 28 days, respectively. The feature values after 7/28 days corresponding to the optimal version (with 9 $kg \cdot m^{-3}$ of chicken feather) were: 2384/2388 $kg \cdot m^{-3}$ for density, 23.5/23.4 % for apparent porosity, 45.1/47.8 MPa for compression strength, 14.5/15.6 MPa for flexural strength, and 2.8 vol. % after 28 curing days for water uptake.

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