Agro-waste addition in the mixture for building geopolymer concrete manufacture

Adăugarea unui deșeu agricol în amestecul pentru fabricarea betonului geopolimeric pentru construcții

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Abstract. *Geopolymer concrete based on fly ash including an agro-waste (corn cob ash) was chosen as the optimal version as a result of the experiment in which granulated blast furnace slag was also used in the other versions. The alkaline activator solution for developing the geopolymerization reaction was kept. Also, usual curing technique was used to increase the mechanical strength. The geopolymer concrete specimen with corn cob ash addition made in the optimal version had the following features: density-2369 kg·m^{-3}, porosity-34.8 %, heat conductivity-0.509 W·m^{-1}·K^{-1}, compressive strength-25.9 MPa (after 7 days) and 39.8 MPa (after 28 days).*

Key words: *geopolymer concrete, agro-waste, corn cob ash, alumino-silicate waste, alkaline activator.*

Rezumat. *Betonul geopolimeric pe bază de cenușă zburătoare incluzând un deșeu agricol (cenușa de ştiulete de porumb) a fost ales ca variantă optimă ca urmare a experimentului în care zgura granulată de furnal a fost de asemenea utilizată în celelalte variante. Soluția de activator alcalin pentru dezvoltarea reacției de geopolimerizare a fost menținută. De asemenea, tehnica uzuală de întărire a fost aplicată pentru creșterea rezistenței mecanice. Proba de beton geopolimeric cu adaosul cenușii de știulete de* *porumb produs in varianta optimă a avut următoarele caracteristici: densitatea-2369 kg·m^{-3}, porozitatea-34,8 %, conductivitatea termică-0,509 W·m^{-1}·K^{-1}, rezistența la compresiune-25,9 MPa (după 7 zile) și 39,8 MPa (după 28 zile).*

Cuvinte cheie: beton geopolimeric, deșeu agricol, cenușă de știulete de porumb, deșeu aluminosilicatic, activator alcalin.

1. Introduction

Ever since the beginning (in the middle of the 19th century), the modern manufacturing process of conventional construction materials has represented a high carbon footprint. According to [1, 2], the manufacture of cement, that requires high consumption of fossil fuel to reach the process temperature of 1450 ºC, releases about 0.85 tons of CO_2 for each ton of cement, being one of the excessive sources of greenhouse gases emitted into the atmosphere worldwide. The need to develop ecological and sustainable construction materials and at the same time, cheap and accessible without affecting their quality, represents the main challenge of scientific research in this field.

Agricultural waste such as sugarcane bagasse, wheat straw, rice husk, etc. are not yet valorized properly. The usual strategies include throwing them in landfills and incinerating, which aggravates the problems of environmental protection. However, recent experimental results regarding the use of agro-waste for the manufacture of viable construction materials are known. Except recycled demolition waste of building and industrial waste, the incorporation of agro-waste has been tested for the production of concrete blocks [3]. Rice husk, sawdust, peanut shell, rice straw, and coconut shell have been tried. The results showed that the blocks with agricultural waste had acceptable mechanical strength, but the durability was satisfactory only in the case of using coconut shell and peanut shell.

According to Rahman et al. [4], the fly ash bricks were significantly improved by the addition of 10 % palm oil ash and the highest value of compressive strength was achieved by combining fly ash with palm oil ash in a 1:1 weight ratio.

According to [5], several agro-waste biomass types were analyzed for their possible use in the construction sector. The main type of cereal crop that generates straw is composed of wheat (65 %), rice (50 %) and to a lesser extent barley, sorghum, rye and oats. Coconut husk is one of the main types of agro-waste biomass as well as corn cob, oil palm, and maize husk. Most of the component parts of crops (leaves, stems, fruits, seeds, etc.) are suitable for the manufacture of new bio-based products. In the manufacture of building materials in concrete industry as well as in bricks production, the use of rice husk and sugarcane bagasse as biomaterials in the form of ash and to a lesser extent as fibers is known.

Several works note that silica (SiO_2) is the main component of the ash of agricultural waste. According to [6], the silica content in rice husk ash and sugarcane bagasse is much higher, reaching values within the limits of 60-95 %. Also, the pozzolanic properties of these ashes determine their effectiveness for increasing the level of physical, mechanical, and thermal characteristics (durability, strength, heat conductivity, porosity, workability, etc.) of bio-based products.

Fibers from cereal straws are used in the manufacture of bricks and as filler in polymer molds for structural reinforcement and thermal/ acoustic insulation [7, 8]. These fibers used in making bio-composites replace partially or totally the wood. Due to the high content of natural fibers, coconut husk is also used in this field. According to [9], coconut husk and rice husk are suitable for the manufacture of biopolymers used in construction.

Currently, one of solutions for the thermal insulation of buildings is the use of flax and hemp fibers, but the level of their application is quite limited. A growing interest in ecological and renewable materials has been noted lately. The use of natural fibers as a component of the insulation is directed especially towards the construction sector. Cellulosic insulations having a higher moisture regain compared to inorganic materials are recommended for old wooden buildings. Recently, several types of mattresses and loose-fill insulations have been developed. Despite their previous use, flax and hemp insulations are considered as new materials in the field of insulations [10].

According to [11], agriculture can become a major source of raw material for different sectors of the economy including the construction sector as a priority. The energy saving due to the efficient thermal insulation of the building is a basic factor of the rational use of energy, directly influencing the level of environmental pollution and greenhouse gas emissions in the atmosphere [12]. The paper [11] analyzes the role of agricultural waste in the concrete industry as aggregate substitutes (sawdust, rice husk, coconut kernel, and cork granules), and Portland cement substitutes (rice husk ash) as well as straw bales used in construction [13] and considers that the use of agricultural waste creates interesting perspectives in the field of ecological construction.

The research team of the authors of the current work has already made a concrete composite with coal fly ash and granulated blast furnace slag as main raw materials reinforced with hemp fibers as agricultural waste [14]. The made product was a concrete geopolymer using alumino-silicate materials representing industrial by-product activated with aqueous alkaline solution composed of sodium silicate (Na_2SiO_3) and sodium hydroxide (NaOH). The experimental results showed the following geopolymer features: density of 327 $kg \cdot m^{-3}$, heat conductivity of 0.094 $W \cdot m^{-1} \cdot K^{-1}$, compressive strength of 8.1 MPa (after 28 curing days), and water absorption of 2.6 vol. %.

The present work aimed at the use of an agricultural waste (corn cob ash) together with industrial alumino-silicate by-products (such as fly ash and granulated blast furnace slag) which by their pozzolanic properties allow the complete replacement of Portland cement for manufacturing a strength geopolymer concrete. Considering that all these raw materials are recycled agricultural or industrial waste, the deeply ecological particularity of the manufacturing process is obvious contributing to the valorization of these residual materials and the significant reduction of greenhouse gas emissions in the atmosphere. The originality of this paper is the addition in variable weight proportions of agro-waste to the already known combination of alumino-silicate waste, the effects on the geopolymer concrete features being analyzed.

2. Materials and methods

Corn cob is the hard part in the middle of the corn and base of the kernel. Generally, it is used as firewood in the household, but also for the production of numerous food and industrial products including corn oil, starch, sweeteners, beverage, industrial alcohols, etc. [15] and the resulting ash is thrown into landfills creating environmental problems. Corn cob ash can be considered an agricultural by-product, can be recycled and re-used in various ways. One of the possible utilization is as a pozzolanic material in cement and concrete. The ash is silica-rich [16]. According to [17], the chemical composition of corn cob ash contains: 79.3 % SiO_2, 1.0 % Al_2O_3, 0.6 % Fe_2O_3, 9.0 % CaO, 1.5 % MgO, 3.8 % K_2O, 10.8 % LOI.

In this experiment, corn cobs were dried outdoor in the sun heat for 1-2 days to remove the moisture, after which the calcination process took place in a ceramic crucible inserted in a muffle oven at 700 ºC for at least 5 hours, transforming the crystalline state of ash in an amorphous state. The grain size of corn cob ash was under 40 μm. Several calcination methods of corn cobs (relatively similar) were described in the literature [18-20].

Alumino-silicate industrial by-products used in experiment were coal fly ash recycled from energy industry and granulated blast furnace slag recycled from metallurgy industry. Fly ash was provided by Paroseni-Thermal Power Plant (Romania) about six years ago. The oxide composition of fly ash was the following: 54.8 % SiO_2, 24.2 % Al_2O_3, 8.6 % Fe_2O_3, 4.8 % CaO, 3.4 % MgO, 4.2 % (Na_2O + K_2O). The grain size of this material was under 200 μm and required an additional grinding to grain size under 80 μm. Granulated blast furnace slag was provided by ArcelorMittal Galati (Romania) 7-8 years ago and had the following chemical composition: 37.4 % SiO_2, 6.4 % Al_2O_3, 6.9 % Fe_2O_3, 39.9 % CaO, 3.5 % MgO, 2.3 % MnO, 0.1 % Na_2O, 0.2 % K_2O. The slag granulation was within the limits of 2-6 mm and its mechanical processing reduced the grain size under 100 μm.

Quartz sand with a content of about 98.8 % SiO_2 and a grain size below 2 mm was used in the starting mixture as fine aggregate, while gravel with a chemical composition of 87.5 % SiO_2, 6.1 % Al_2O_3, 2.1 % Na_2O, and 1.6 % Fe_2O_3 and grain size below 6 mm was chosen as coarse aggregate.

According to the invention of the French scientist Davidovits, J. [21] and the principle of alkaline activation of alumino-silicate materials [22], the mixture of sodium hydroxide (NaOH) soluble in water and aqueous solution with a concentration of 38 % of sodium silicate (Na_2SiO_3) was used. NaOH was commercially purchased in the form of flakes. Also, the Na_2SiO_3 solution was available on the market and could be diluted with distilled water to correct the solution viscosity.

The remarkable invention of the transformation of alumino-silicate waste into a superior geopolymeric material in terms of quality by activation in a highly alkaline medium, which facilitates the development of geopolymerization reaction, was the basis of the method adopted in this experiment. The effect of transforming alumino-silicate waste into a geopolymer is the possibility of significantly reducing or completely eliminating ordinary Portland cement from the composition of traditional concrete and obtaining a product with higher physico-mechanical properties under the conditions of its manufacture without greenhouse gas emissions (CO_2). The addition to the mixture of alumino-silicate materials of an agricultural waste with a very high availability (corn cob ash), silica-rich and having pozzolanic properties that give it the ability to be used in the manufacture of cement and construction concrete constitutes the completion of the mentioned adopted method.

The geopolymerization reaction is perceived by researchers as a complex reaction. The contact between the alkaline solution (NaOH) and the alumino-silicate materials generates the dissolution and hydrolysis of silicon and aluminum, which take place according to the reactions shown below [23-25].

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

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

As a result of these reactions (at room temperature), a gel is formed, that modifies its structural features and several gel phases appear. At the end, the system is polymerized, forming a solidified mass due to its hardening. A three-dimensional polymeric chain and ring structure based on Si-O-Al-O bonds is generated [26].

A pozzolanic material is, in terms of chemistry, a material rich in silica and alumina without cementing properties. Water added to such material gives it cementing properties by forming calcium silicate hydrate (C-S-H) gel. C-S-H gel is the main product of hydration of Portland cement and has the ability of increasing the strength of cement-based materials [27, 28]. Corn cob ash is a silica-rich precursor and it was experimentally found that it can partially substitute the Portland cement in a cement concrete due to its pozzolanic properties. In the experiment described below, the cement was completely eliminated from the mixture composition, the alumino-silicate waste playing the role of cementitious materials.

The method of preparing geopolymer concrete included the following steps. The dry alumino-silicate materials (fly ash and granulated blast furnace slag) together with corn cob ash, quartz sand, and coarse aggregate (gravel) in the dosages adopted for each experimental version were mixed in a metal container with an electrically operated stirrer for 5 min. The preparation of the alkaline activator took place separately in a glass vessel, being mixed NaOH flakes dissolved in distilled water, Na_2SiO_3 solution, and water addition by stirring for 5 min. After mixing, the alkaline activator was slowly poured over the solid mixture, after which the stirring process continued for another 5 min, generally until the gel was formed. The gel representing the fresh geopolymer was poured into a metal mold protected on the inside with thin plastic film and placed in a thermally insulated room for the curing treatment carried out by blowing steam at 80 ºC for 24 hours. The hot curing process was followed by room temperature curing for 48 hours. Keeping the specimen removed from the mold for free curing also at room temperature was carried out in an isolated room until the tests for characterizing the specimen were carried out after 7 and 28 days, respectively.

Composition of the four experimental versions is shown in Table 1.

Table 1

Composition of experimental versions

Composition	Experimental version ($kg \cdot m^{-3}$)			
	1	2	3	4
Fly ash	370	370	370	388
Granulated blast furnace slag	46	32	19	-
Corn cob ash	46	61	74	74
Quartz sand	670	670	670	670
Coarse aggregate (gravel)	850	850	850	850
NaOH 8M	120	120	120	120
Na_2SiO_3 solution	250	250	250	250
Water addition	60	60	60	60

According to [29], the calcium content could influence geopolymer gel products through the appearance of hydrated calcium alumino-silicate (C-A-S-H) along with hydrated calcium silicate (C-S-H) in alkali-activated geopolymer. The effect of Ca^{2+} on the geopolymerization process is still unclear. An improvement in compressive strength after 7 days for an addition of 3 % CaO in the material mixture was observed. Considering that the blast furnace slag provided by Arcelor Mittal Galati had a relatively high content of CaO (39.9 %) as well as the controversies in the literature regarding the effect of calcium, in this paper it was chosen to significantly reduce the weight proportion of blast furnace slag in the mixture, even up to zero, simultaneously with the increase in corn cob ash content.

Weight proportion of fly ash, blast furnace slag, and corn cob ash was varied according to the data in Table 1 as follows. In the first three versions, the proportion of fly ash was relatively constant around 80 %, while in version 4, the proportion of fly ash increased to 84 %. Blast furnace slag continuously decreased from 9.9 to 4.1 % between versions 1 and 3 and reached zero in version 4. The corn cob value increased from 9.9 % (version 1) to 16.0 % (version 3) and remained almost constant in version 4. The other components of the mixture had constant values in all four versions. The sand was 27.8 %, and coarse aggregate had the value of 35.2 %. Na_2SiO_3 and NaOH represented 10.4 and 5.0 %, respectively, their weight ratio being 2.08. The weight ratio between the total alumino-silicate materials (fly ash and slag) and the total alkaline activator decreased from 1.12 (in version 1) to 1.05 (in version 3), reaching 0.78 (version 4) in the case of complete elimination of the blast furnace slag. Water addition was kept constant at 2.5 %.

Characterizing methods of geopolymer specimens were the following. Density was determined by weighing the specimen mass relating to its volume [30]. Porosity was calculated identifying the proportion of porous material (apparent density) compared to the same material without pores obtained by melting and cooling (true density) [31]. The method utilized for measuring the heat conductivity was the heatflow method according to SR EN 1946-3:2004 standard. Compressive strength was measured with TA.XTplus Texture Analyzer and flexural strength was determined using SR EN ISO 1412:2000 [32]. The method of immersing under water the geopolymer specimen was applied for determining the water absorbed by this (ASTM D570). Investigation of the microstructural configuration was carried out with Biological Microscope TM5000 model with captured image, 1000 x magnification.

3. Results and discussion

The application of characterizing methods of geopolymer concrete specimens after 7 and 28 curing days led to the results shown in Table 2.

Table 2

Features of geopolymer concrete specimens

Feature	Experimental version			
	1	2	3	4
Density (kg·m^{-3})	2412	2400	2383	2369
Porosity (%)	29.8	31.7	33.7	34.8
Heat conductivity (W·m^{-1}·K^{-1})	0.518	0.514	0.510	0.509
Compressive strength (MPa)				
- after 7 days	22.5	20.8	20.4	25.9
- after 28 days	31.4	31.1	30.8	39.8
Flexural strength (MPa)				
- after 7 days	2.9	3.0	2.9	3.7
- after 28 days	3.6	3.7	3.7	4.1
Water uptake (vol. %)	2.1	2.3	2.2	2.4

The analysis of the data in Table 2 showed a slight decrease of the geopolymer density with the reduction of blast furnace slag proportion in the starting mixture, the density value starting from 2412 kg·m^{-3} (version 1) and going down to 2369 kg·m^{-3} (version 4). The porosity fell within low limits (29.8-34.8 %) following a slightly increasing slope from the value corresponding to the specimen of version 1 to that of the specimen made in version 4. Due to the high density and low porosity of the samples, heat conductivity had relatively high values (0.509-0.518 W·m^{-1}·K^{-1}). The compressive strength was influenced by changing the weight proportion of alumino-silicate waste (fly ash and slag) and agricultural waste. It is possible that the high content of CaO in the slag composition, at least in version 1 in which the blast furnace slag represented 1/8 of the total fly ash-slag amount, favorably influences the compressive strength value after 7 days of curing (22.5 MPa) according to the hypothesis presented in [29]. In versions 2 and 3 characterized by reducing the proportion of slag and increasing the proportion of agricultural waste, the compressive strength slightly decreased, both after 7 and after 28 days, to increase significantly in both curing variants in version 4. The value of compressive strength after 28 days reached the maximum level of 39.8 MPa. An almost similar situation was also registered in the case of specimen flexural strength. After remaining relatively constant in versions 1-3, the flexural strength reached its maximum value in the case of version 4 after 7 days (3.7 MPa) and also after 28 days (4.1 MPa), demonstrating that the best solution in terms of quality was the use of corn cob ash as an agricultural waste widely available in Romania mixed with coal fly ash, a by-product of energy industry, activated with the alkaline solution composed of NaOH and Na_2SiO_3. The maximum weight proportion of corn cob ash in the mixture with fly ash was 16 %.

The microstructural aspect of the four specimens corresponding to the tested experimental versions is presented in Fig. 1.

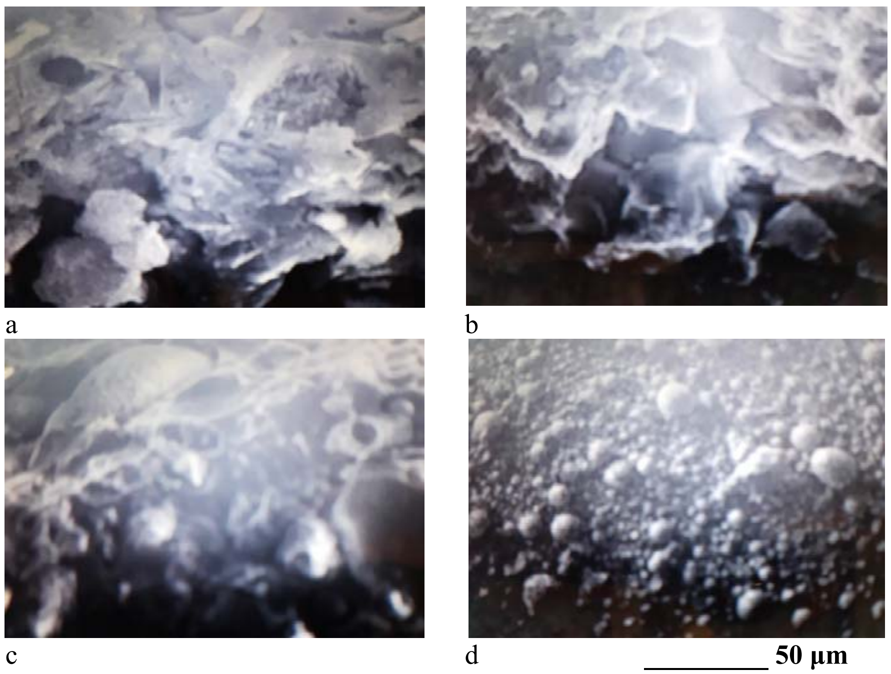

Fig. 1. Microstructural aspect of geopolymer concrete specimens
a – version 1; b – version 2; c –version 3; d – version 4.

Fig. 1 practically shows the transition from typical structures in which fly ash and granulated blast furnace slag are predominant (a) and (b), to structures in which the peculiarities of the fly ash structure become predominant, especially (d).

The results showed that the manufacture of geopolymer as designed by the French inventor Davidovits is an excellent modern technique of great importance for environmental protection based on residual alumino-silicate materials. The addition of other silicate wastes in the starting mixture, such as corn cob ash, is a very current trend that also contributes to the manufacture of products with suitable qualities for the construction sector. In this experiment, preparing the corn cob ash was made starting from corn cobs provided by an agricultural producer. Their transformation into ash by calcination was carried out in laboratory with electricity consumption. It should be noted that in reality, corn cob ash is practically an industrial by-product, which is thrown into the landfill. So, the energy consumption that generates ash as a secondary product is a useful consumption in several industrial activities, according to the previous specification in this work (chapter 2). Practically, version 4 of this paper after removing the granulated blast furnace slag used in versions 1-3 and keeping fly ash as an alumino-silicate industrial by-product as well as the alkaline solution for its activation became a typical fly ash-based geopolymer concrete also including the agricultural waste (16 %).

4. Conclusions

The work aimed at manufacturing geopolymer concrete based on fly ash and granulated blast furnace slag as alumino-silicate waste activated with liquid solution of NaOH and Na_2SiO_3 combined with the addition of a silica-rich agricultural waste (corn cob ash) to the starting mixture. Considering the controversial influence in the literature of the calcium content in the material mixture on the geopolymer features as well as the high proportion of CaO (39.9 %) in the composition of blast furnace slag provided by ArcelorMittal Galati, the solution of using a low slag content and its reducing in versions 1-3 up to the zero level in version 4, at the same time as the agricultural waste content increases up to a maximum of 16 wt. % in the same experimental version was adopted. The solid and liquid components of the mixture were processed separately and then together until a gel was formed, which was poured into a mold and subjected to the curing process. The geopolymer concrete specimen with corn cob ash addition made in the optimal version (version 4 without slag content) had the following features: density of 2369 $kg \cdot m^{-3}$, porosity of 34.8 %, heat conductivity of 0.509 $W \cdot m^{-1} \cdot K^{-1}$, compressive strength of 25.9 MPa (after 7 days) and 39.8 MPa (after 28 days), flexural strength of 3.7 MPa (after 7 days) and 4.1 MPa (after 28 days). Compared to other geopolymer concrete characteristics reported in the literature, the product had excellent compressive and flexural strength being suitable for using in the construction sector. The viability of adding the agro-waste in the form of corn cob ash for making the geopolymer was confirmed.

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