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Geopolymer brick composite made from building demolition materials

Compozit de cărămidă geopolimerică fabricată din materiale rezultate din demolarea clădirilor

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Abstract. *Unlike most manufacturing recipes of geopolymer materials containing fly ash, the current work adopted the association of clay brick and concrete resulting from building demolition together with metakaolin and blast furnace slag as materials with cementitious and pozzolanic properties suitable for substituting the extreme pollutant-Portland cement. Except for the original composition of the material mixture, the authors have adopted their own curing regime of fresh pasta at 80 ℃ for 24 hours, followed by traditional storage at room temperature for 7-28 days. Geopolymer brick properties were excellent through the appropriate correlation of mechanical and physical features.*

Key words: *building demolition, metakaolin, blast furnace slag, concrete waste, geopolymer brick.*

Rezumat. *Spre deosebire de majoritatea rețetelor de fabricare a materialelor geopolimerice conținând cenușă zburătoare, lucrarea curentă a adoptat asocierea cărămizii de argilă și beton rezultate din demolarea clădirilor împreună cu metacaolin și zgură de furnal, ca materiale cu proprietăți de cementare și pozzolanice adecvate pentru înlocuirea cimentului Portland extrem de poluant. Exceptând compoziția originală a amestecului material, autorii au adoptat propriul lor regim de întărire a pastei proaspete*

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la 80 °C pentru 24 ore, urmat de tradiționala stocare la temperatura ambiantă pentru 7-28 zile. Proprietățile cărămizii geopolimerice au fost excelente prin corelarea adecvată a caracteristicilor mecanice și fizice.

Cuvinte cheie: demolarea clădirii, metacaolin, zgură de furnal, deșeu de beton, cărămidă geopolimerică.

1. Introduction

According to the literature [1], clay bricks were among the first man-made synthetic materials for the construction of buildings. In principle, their realization is simple, consisting of mixing clay with water. Hardening techniques have evolved over time, from facile drying in the sun heat, to thermal treatment in industrial ovens.

Clay brick is a crystalline ceramic type. Its development as a suitable material for construction was achieved through the additional use of industrial and agricultural waste. The main role of these wastes in the brick making is as a pore-supplier inside the clay body [2].

Clay bricks are manufactured by firing the raw material of clay or shale, forming a sintered porous structure [3]. Some forms of clay or shale contain organic or mineral matter, which can release gases as a result of heating, creating the adequate conditions for expanding the pelletized particles. The lightweight aggregate of expanded clay, known as LECA [4], is manufactured through this technology, the process temperature around 1200 °C being reached in a rotary kiln.

During the thermal processes at high temperatures for the production of traditional construction materials, large amounts of greenhouse gases (CO_2) are released into the atmosphere, causing major ecological problems. In these conditions, except for the need to adopt technologies with lower consumption of fossil fuel, the recycling of industrial by-products as well as the collection for the effective use of waste from usual domestic consumption, have become extremely important concerns for humanity. In the last decades, thanks to the inventions of exceptional scientific value by the French researcher J. Davidovits regarding the turn of waste rich in alumina and silica, available in the world in very large quantities, into geopolymers with physical, mechanical, chemical properties, superior to existing construction materials on the market, the opportunity was created to manufacture and test a large number of materials with new value created using the geopolymerization reaction in an activated alkaline medium [5].

Geopolymerization reaction of alumina-silicate waste in presence of alkaline activator solution is considered by specialists as a complex reaction that occurs in several stage, which can overlap. In principle, the geopolymer formation includes dissolution and hydrolysis processes, followed by condensation, which occurs in the Na_2O-Al_2O_3-SiO_2-H_2O system, sodium and silicon concentrations being decisive for the condensation process [6].

According to the literature [7-10], but also from the authors' team own experience [11-13], fly ash, metakaolin, and granulated blast furnace slag were synthesized, the effect obtained being energy savings and the reduction of geopolymer manufacturing costs. Except for the mentioned alumina-silicate materials, recycled materials from the demolition of buildings were also used (residual concrete, residual bricks, and other components of the rubble from the demolished masonry) [14-16].

A peculiarity of preparing fly ash-based geopolymer and especially, the influence of curing at temperatures below 100 °C on its microstructure and strength was analyzed in the work [17]. It was found that the long-term precuring at ambient temperature of fresh geopolymer specimens before their introduction into the oven for hot curing for 24 hours is a beneficial process for increasing the material resistance. Class F-fly ash by-product was activated in the alkaline solution composed of Na_2SiO_3 and NaOH. J. Davidovits, the inventor of the geopolymerization process, has used except for the alumino-silicate waste, specially prepared highly reactive clay, the mixture being activated for developing the geopolymerization reaction through direct contact with the liquid alkaline activator. By comparison, the activation energy of fly ash is higher than that required in the case of granulated blast furnace slag.

Geopolymer bricks represent the latest technical innovation in the field of bricks [18] consisting of the introduction of additional cementitious materials (fly ash and granulated blast furnace slag) activated by an alkaline activator. The application of this innovative technology allowed the significant reduction of CO_2 emissions by removing the need for cement. It was experimentally found that geopolymer bricks made with fly ash require curing at a higher temperature. This curing process allows obtaining superior compression strength and lower moisture absorption. According to the paper of El-Naggar et al. [19], the manufacture of geopolymer bricks using alumina-silicate waste (clay brick waste, slaked lime waste, dealuminated kaolin, and caustic soda) offered the possibility of producing lightweight bricks (density of around 1000 $kg \cdot m^{-3}$), the compression strength still reaching 1.4 MPa.

According to [20], the use of ceramic dust waste can contribute to reducing the cost of geopolymer brick manufacturing. Durability of the same brick type produced from mine tailings significantly increased [21]. The introduction of alkali-activated industrial waste into the geopolymer brick composition can noticeably influence its physical, mechanical, and chemical properties [22].

A new original approach regarding the influence of the making process of geopolymer as well as the product itself on the environment was recently developed. According to the authors of the paper [23], the life cycle assessment of the new geopolymers based on waste from the demolition of constructions by the complete replacement of traditional Portland cement started from the adoption of precursors in the form of waste: red clay brick, hollow brick, roof tile, concrete, and glass as well as recycled concrete aggregates. The geopolymer as a result of chemical reactions between alumina-silicate oxides (alumina and silica) and the alkaline activator offers major advantages through reducing CO_2 emissions by 89 % and energy consumption by 60 % (according to authors' estimates).

Residual materials recycled from demolitions (brick waste) and by-products of the metallurgical industry (granulated blast furnace slag) were experimentally tested [24] to obtain a high-performance composite material as an alternative to concrete with similar characteristics. The results showed that the mixture of brick waste from demolitions (with low amorphous mass) with more reactive precursors (such as the slag) can lead to performing high strength composites (compressive strength of over 60 MPa).

Several works mentioned in [23], which used fly ash, metakaolin, granulated blast furnace slag as well as sodium silicate together with sodium hydroxide as an alkaline activator for the manufacture of geopolymer, revealed the environmental implications of some geopolymeric materials (fly ash considered un-adequate for the geopolymerization reaction [25], silicates such as sodium silicate in the activator composition that should be quantitatively reduced to make the geopolymer more environmentally friendly, etc.). Of course, the results mentioned above are controversial, especially given that the respective research was not considered completed.

According to [18], class F-fly ash (with low CaO content) from the thermal power plant was the main alumina-silicate industrial by-product used for the manufacture of geopolymer brick. Also, ground granulated blast furnace as a by-product of the metallurgical industry was added together with fly ash in the starting mixture. As a fine aggregate, a type of sand available in India was chosen having the specific gravity of 2.52 $g \cdot cm^{-3}$ and particle size after sorting by sieving between 0.15-4 mm. Sodium silicate/sodium hydroxide ratio was kept at 2.5. Testing the geopolymer brick specimens, where the fly ash/slag ratio varied between 100 and 3 %, and the fine aggregate reported to the binder (fly ash and slag) was kept relatively constant at 2.64-2.71 was performed after 7 and 28 curing days. Determining results showed that the highest values of compression strength were obtained using 25 % fly ash and 75 % slag (25.2 and 51.7 MPa, respectively), water-absorbing slightly varied between 6.0-7.1 %, the lowest value corresponding to the same proportions of the binder components. The acid resistance increased with the increase of the blast furnace slag ratio, the maximum weight loss being reached for the use of only fly ash.

The rapid rate of infrastructure development in the world has led to an excessive increase in waste from demolition and construction. Storing them in landfills is unacceptable in ecological terms, so their recycling and using as a precursor material for the manufacture of geopolymers is the best method of efficient capitalization. The attempt to optimize the design of geopolymer brick from construction waste using full factorial design methodology was recently carried out by Maase and Shrivastava [26]. The loss of water by the geopolymer during the geopolymerization reaction led to an increase in the workability of the fresh mix. The properties of the geopolymer in the fresh state were affected by the silicon/aluminum, sodium/aluminum, silicon/sodium as well as solid/liquid ratios. Different conditions of the curing process were tested (40-85 °C for 5-72 hours, followed by room temperature curing). Ground brick waste (under 90 μm) as a precursor, fine sand as well as Na_2SiO_3 and NaOH solution as an alkaline activator were the materials experimentally used. The molarity of 4 M and the Na_2SiO_3/NaOH ratio of 1.5 were the main optimal parameters.

Different variants of geopolymer mixtures including waste brick powder from demolitions were prepared and tested by Fořt et al. [27]. Various compositions of alkaline activator and curing conditions were applied. The results showed that the reaction rate at early age decreases with the increase of the sodium silicate modulus and with increasing the temperature of the curing process. Compared to metakaolin-based geopolymer, the reaction rate is slower due to the low content of the brick amorphous phase. By lowering the temperature of the curing process, the microstructural compactness gradually decreases and the specific volume of pores increases. With the increase of the sodium silicate modulus, regardless of the curing temperature value, the weight loss of the material decreases, while the dehydration of N-A-S-H and C-A-S-H gels occurs. For geopolymers cured at temperatures below 60 °C, it was found that most of the crystalline phases are similar to those of the precursor, highlighting only a partial geopolymerization, while in the case of geopolymers cured at 60-80 °C, the formation of zeolitic phases could be observed, confirming the completion of the geopolymerization process.

Recycled construction and demolition waste (CDW) was used according to Ducman et al. [28] for the manufacture of geopolymeric panels. The process involved the fine grinding of residual bricks, concrete, and mortar as well as wood chips as recycled waste. Fly ash, metakaolin, and granulated blast furnace slag had the binder role, activated by contact with the alkaline mixture consisting of potassium silicate (K_2SiO_3) and potassium hydroxide (KOH), another version of the alkaline activator predicted by J. Davidovits in his patents related to the geopolymerization of alumina-silicate waste. The results of measuring the characteristics of geopolymeric panels showed that they are suitable for cladding the building façades. Flexural strength corresponding to the panels made with brick waste, concrete, and mortar reached 5.5 MPa, while that of the panels using wood chips reached 4.3 MPa. The flexural modulus of elasticity of the two panel types had values of 2.02 and 1.38 GPa, respectively.

The work [29] proposed the synthesis of geopolymeric binders based in a very high proportion on waste provided by the building demolition. The ceramic waste constituted the alumina-silicate precursor, while the glass waste provided the silicate material necessary for the preparation of the alkaline activator. In this experiment, CDW-based geopolymers with a compression strength within the limits of 10–44 MPa were produced. The used materials contained 80–90 wt % CDW, depending on the preparation method of the activator. The paper proposed a procedure for increasing the capitalization rate of construction and demolition waste (CDW) in the geopolymer manufacturing process. Brick waste is an excellent alumina-silicate precursor. Also, window glass shards, recycled and properly processed, can be oriented towards greening the activator solution due to the high silica content.

Recently, several researchers in the field of constructions [22, 25, 29] have experimentally found that silicon and alkali contents of the alkaline activator solution seriously affects the geopolymer brick characteristics, causing the formation of additional crystalline phases such as zeolites and carbonates, which negatively influence the mechanical brick properties. Recycled glass waste from building demolition materials could effectively replace conventional activators (sodium or potassium silicate and sodium or potassium hydroxide) without affecting the level of compressive strength of the final product.

Taking into account the own previous experience of the authors' team of the current work as well as the paper conclusions mentioned above, especially in the case of the manufacture of geopolymer brick, in this work it was decided to use high proportion-recycled waste from the rubble resulting from the building demolition (clay brick scrap and concrete scrap) as well as metakaolin and granulated blast furnace slag, all having adequate cementitious and pozzolanic properties. Fine river sand was chosen as aggregate, while the chemical activation of alumina-silicate materials specified above was carried out by adopting the traditional type of alkaline activator composed of Na_2SiO_3 and NaOH proposed by the French inventor J. Davidovits.

2. Method and materials

The geopolymer brick manufacturing method included the independent preparation in separate vessels of the liquid alkaline activator and respectively, of the mixture composed of solid materials. The liquid solution of Na_2SiO_3 (38 % concentration) thus purchased from the market had SiO_2/Na_2O molar ratio of 1.8. Commercially available NaOH solid pellets, dissolved in distilled water (molarity 10 M), were slowly introduced into the vessel containing Na_2SiO_3 under the conditions of its continuous stirring, until the Na_2SiO_3/NaOH weight ratio reached 2.5. Stirring the liquid mixture continued for about 10 hours. The liquid solution of the activator should contain 23.5 % SiO_2, 18.5 % Na_2O and 58 % water. Separately, the dry solid mixture was prepared, including metakaolin, ground granulated blast furnace slag (below 100 µm), ground clay brick scrap and ground concrete scrap (under 90 µm) as well as fine river sand (under 3.5 mm) as fine aggregate. Na_2SiO_3, until a paste was formed. The homogenized paste corresponding to each specimen was poured into a metal rectangular mould with dimensions 250 x 120 x 50 mm. For the curing process, the specimen was introduced into a drying oven at 80 °C for 24 hours. At the end of this process, the sample was removed from the mould and stored at room temperature for 7 and 28 days before determining its characteristics.

Materials used in experiment were the following.

Metakaolin is a dehydroxylated form of the clay kaolinite [30]. It is commercially available as 1-2 µm-fine particles. Its chemical composition includes: 53.0 % SiO_2, 43.8 % Al_2O_3, 1.70 % TiO_2, 0.43 % Fe_2O_3 [31].

Granulated blast furnace slag procured from ArcelorMittal Galati (Romania) ten years ago is a by-product of metallurgical industry. The molten slag was granulated by pouring into cold water pool. The grain size of granulated slag is in the range of 2-6 mm. In this experiment, the particle size was reduced below 100 µm by grinding the slag in a ball mill. The chemical composition of the slag is the following: 36.44 % SiO_2, 11.60 % Al_2O_3, 41.81 % CaO, 5.80 % MgO, 0.55 % MnO, 0.78 % Fe_2O_3, 0.35 % Na_2O, and 0.43 % K_2O [32].

Clay bricks are porous materials, their porosity influencing the low durability, high amount of water-absorbing, and firing temperature. Compressive strength of old clay bricks exhibits a wide value range between 1.5-32 MPa, up to 50 MPa [33]. The chemical composition of clay brick contains 48.7 % SiO_2, 13.7 % Al_2O_3, 10.0 % CaO, 5.7 % Fe_2O_3, 3.7 % MgO, and 2.5 % K_2O [34].

According to Jorat et al. [35], the chemical composition of concrete scrap recycled from the building demolition contains: 71.9 % SiO_2, 4.0 % Al_2O_3, 12.1 % CaO, 0.7 % MgO, 0.2 % TiO_2, 1.5 % Fe_2O_3, 0.6 % Na_2O, 0.7 % K_2O and 0.4 % SO_3. As mentioned above, particle sizes of clay brick scrap and concrete scrap used in this experiment were reduced under 90 μm as a result of their mechanical processing.

The fine river sand (below 3.5 mm) obtained after selection by sieving was utilized as fine aggregate. This product has high silica content (over 96 %) [36].

The current experiment proposed testing four manufacturing recipes of geopolymer bricks, in which the less used combination including clay brick waste and concrete scraps recovered from the building demolition was tried, without involving the most commonly used material for the production of geopolymers (fly ash). Metakaolin and granulated blast furnace slag as materials with cementitious and pozzolanic properties were preferred to the ash. The sand as a fine aggregate and the alkaline activator solution completed the material list included in the making recipe. The four experimental versions containing the dosage of the mentioned materials are shown in Table 1.

Table 1

Experimental versions of making the geopolymer brick

Material composition	Version ($kg \cdot m^{-3}$)			
	No. 1	No. 2	No. 3	No. 4
Granulated blast furnace slag	60	90	120	150
Metakaolin	250	220	190	160
Clay brick waste	450	445	440	435
Concrete waste	450	455	460	465
Sand (below 3.5 mm)	1210	1210	1210	1210
10 M NaOH	90	90	90	90
Na_2SiO_3	220	220	220	220

The investigation methods utilized in this work are known methods usually used in research activities. Archimedes' principle was used to measure the apparent density of geopolymer brick specimens. Using ASTM C642-97 standard, the apparent porosity was determined by dividing the difference between wet and dry weight by the difference between wet weight and suspended weight of the sample. Thermal conductivity was measured at room temperature using the HFM448 Lambda heat-flow-meter (SR EN 1946-3:2004). 100 kN-compression fixture Wyoming Test Fixture was used to determine the compressive strength. The flexural strength was determined using the method recommended in SR EN ISO 1412:2000, i.e. the three-point bending test. Specimen immersion under water for 24 hours (ASTM D570) allowed the measurement of water-absorbing. The microstructural aspect of specimens could be analyzed with the Biological Microscope MT5000 model, 1000 x magnification.

3. Results and discussion

Table 2 centralizes the results of the measurements performed for the physical, mechanical, and thermal characterization of geopolymer brick specimens.

Table 2

Physical, mechanical, and thermal features of geopolymer brick specimens

Characteristic	Version			
	No.1	No.2	No.3	No.4
Apparent density ($kg \cdot m^{-3}$)	2372	2389	2396	2402
Apparent porosity (%)	39.7	35.4	30.0	26.7
Heat conductivity ($W \cdot m^{-1} \cdot K^{-1}$)	0.361	0.402	0.456	0.511
Compressive strength (MPa)				
- after 7 days	18.3	20.4	22.4	24.9
- after 28 days	36.8	43.5	50.1	59.7
Flexural strength (MPa)				
- after 7 days	2.4	3.0	3.6	3.9
- after 28 days	3.9	4.8	5.9	7.1
Water-absorbing (vol. %)	6.7	7.1	6.9	6.8

The presence in increasing proportions of granulated blast furnace slag in the mixture composition significantly influenced the compressive strength of the geopolymer, both after 7 curing days (maximum 24.8 MPa) and especially after 28 days (maximum 59.7 MPa). The same conclusion is not valid in the case of flexural strength, its values being more moderate (2.4-3.9 MPa after 7 days and 3.9-7.1 after 28 days). A possible explanation would be the contribution of the amorphous mass from the clay brick waste composition.

In physical terms, the apparent density of geopolymer brick composite registered a slight increase (from 2372 $kg \cdot m^{-3}$ in experimental version 1, to 2402 $kg \cdot m^{-3}$ in version 4) due to the increase in the proportion of blast furnace slag compared to metakaolin. Also, the rather low porosity of the geopolymeric product decreased under the conditions of preparating the recipe of version 4 compared to version 1 (from 39.7 to 26.7 %).

Referring to water-absorbing, it recorded values in the range of 6.7-7.1 vol.%, considered normal [18] due to the presence of clay in the brick waste composition.

Images of the geopolymer brick specimens are shown in Fig. 1.

a

b

c d

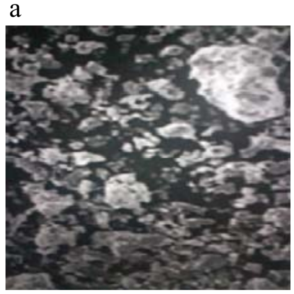
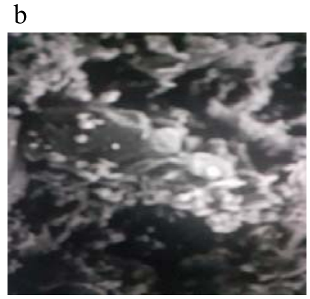
Fig. 1. Images of geopolymer brick specimens
a – specimen 1; b – specimen 2; c – specimen 3;
d – specimen 4.

The surface appearance of geopolymeric brick samples is slightly porous in the case of the first two versions (a and b) due to the physical peculiarities of clay brick waste, after which this appearance fades in the case of the last versions (c and d), that have lower proportions of clay brick in composition.

The microstructural images presented in Fig. 2 highlights the change of characteristics of the four specimens by decreasing the proportion of clay brick waste and increasing that of granulated blast furnace slag.

a b

c d **|-------| 5 µm**

Fig. 2. Microstructural pictures of geopolymer brick specimens
a – specimen 1; b – specimen 2; c – specimen 3;
d – specimen 4.

It can be observed the reduction of spots representing the amorphous mass areas typical for clay brick as well as obtaining a higher compactness (Fig. 2d) mainly due to the blast furnace slag.

The comparative analysis of the own experimental results with other results obtained in the field of geopolymer brick and published in the literature highlighted their similarity. Although the current work completely substituted one of the most frequently used by-products (fly ash), adopting the combination of metakaolin-granulated blast furnace slag, with cementitious and pozzolanic properties almost similar to those of ash, the performances of the final product were comparable. Also, the new method of including in the mixture for manufacturing geopolymers of the waste resulting from the building demolition (concrete and brick scraps) was adopted and applied in the recipe for the manufacture of geopolymer brick. An important role in obtaining a resistant and dense geopolymeric material is represented by the method adopted for curing the fresh material. In the conditions of a wide variety of known curing techniques, in the experiment presented in this work the authors adopted an own technique verified in several relatively similar experiments obtaining valuable results.

4. Conclusions

The objective of the current work was to test the manufacture of geopolymer brick composite without the contribution of the most frequently used material in similar processes (fly ash), replaced by metakaolin and granulated blast furnace slag as materials with cementitious and pozzolanic properties. This constituted the main element of the work originality. Building demolition waste (concrete and clay brick scraps) were also included in the mixture composition as alumina-silicate materials suitable for manufacturing the geopolymer. The curing process of the fresh material was carried out according to the authors' own method, being a secondary element of the work originality. High compressive strength values (up to 59.7 MPa after 28 days and up to 24.9 MPa after 7 days) were obtained, being almost similar to values reported in the literature. In physical terms, the geopolymer brick in the optimal version (with the highest slag proportion) had a dense and relatively compact structure, with apparent density of 2402 $kg \cdot m^{-3}$, porosity of 26.7 % and heat conductivity of 0.511 $W \cdot m^{-1} \cdot K^{-1}$. The experimentally made geopolymer brick is suitable for application in construction, being resistant, environmentally friendly, and little expensive.

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