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ALKALI-ACTIVATED FLY ASH BASED GEOPOLYMER PAVING BLOCKS: GREEN MATERIALS FOR FUTURE CONSERVATION OF RESOURCES

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Abstract

Both Circular Economy and Sustainable Development are concepts that focus on resource efficiency. This involves a complex waste management process with a high degree of recycling and recovery. Rapid population growth and urbanization in developing countries have led to large amounts of solid waste and, consequently, increased environmental degradation of the environment. Designing resilient, sustainable materials and buildings by using recycled materials or industrial by-products, which can replace some or all of the cement, leads to the development of a sustainable, low-carbon economy and furthermore to the conservation of natural resources. Industry is developing at an accelerated pace, consequently the development of modern construction materials in the economic world is more than necessary. The aim of this research is to present preliminary results regarding the design of the alkali-activated fly ash-based geopolymer paving blocks produced using Romanian local raw materials, to present several parameters that affect their mechanical properties and to assess their intended use in terms of legislation.

***Keywords**: Alkali-activation; Geopolymer paving blocks; Circular Economy; Sustainable Development*

Introduction

The increased utilization of Portland cement as main material in the production of concrete and the well-known disadvantages in terms of environmental pollution and green house gas emissions during its production which are not sustainable made policy makers worldwide realize the importance of facility for pedestrians and non-motorized vehicles in an urban infrastructure setup which could be more environmentally friendly. Worldwide, concrete is the most versatile, durable and reliable building material, requiring large quantities of Portland cement to produce. The environmental problems associated with the production of Portland cement are well known and are a closely monitored issue in terms of carbon dioxide emissions and other pollutants. Producing such a large volume of cement/concrete is directly associated with the environmental problems that arise - cement production is responsible for around 8-10% of total carbon dioxide emissions. Each tonne of produced cement requires 60, up to 130kg of fuel oil or equivalent, depending on the type of cement and the manufacturing process being used, and about 110kWh of electric generated power. A tonne of manufactured

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OPC releases between 0.8 and 1.1 tonnes of CO_2 into the atmosphere, as a consequence of burning fuel and calcining limestone [1, 2].

Rapid population growth and urbanization in developing countries have led to large amounts of solid waste and thus increased environmental degradation with 90-95% of all waste in the world is dumped on open land or in landfill sites. This creates considerable environmental problems. At the same time, there are sustained efforts worldwide to find alternative ways of using these types of wastes, given the limited natural reserves, for the development of modern construction materials.

Developed coutries are extensively focusing on the infrastructure development to improve mobility of its people and goods and realized the significance of better facilities for pedestrians and non-motorised vehicles. Urban areas that support and facilitate walking, could easily improve community health, vitality, and safety and could also improve the use of public transit, increase pedestrians by reducing car dependence, and promote cleaner air and green public space [3].

Both Circular Economy and Sustainable Development are concepts that focus on resource efficiency. This involves a complex waste management process with a high degree of recycling and recovery, therefore preventing the use of natural resources for the production of ordinary Portland cement and conservationg the natural environment. Research into the production and optimisation of geopolymeric materials is fundamentally driven by the need, identified both in the global environmental context and at national level, to implement the principles of sustainable development with sustainable resource consumption, to recover existing waste and to prevent new waste being generated [4-8]. The main focus on the development of geopolymer binders is based on available data and superior performance applications that could be achieved by these types of materials compared to existing construction materials [1-8]. The high mechanical properties (compressive strength some even above 80MPa), achieved at very early ages, when the heat treatment step is included in the manufacturing process, have demonstrated their applicability in obtaining prefabricated construction products, where the performance benefits and evaluated life cycle costs exceed the initial production costs, with specific uses in non-structural, pedestrian applications (footpaths, pedestrian areas), bicycle paths, parking lots, roads, highways, industrial premises, etc. The research also contributes to the timeliness and need for continuous and updated studies on the identification and valorisation of various materials resulting from technological processes in the energy industry, metallurgy etc. [9-13].

The main focus on the development of geopolymer binder mixtures is based on available data and superior performance applications that could be achieved by these types of materials compared to existing construction materials. For the production of geopolymer concrete, choosing the source materials largely depends on their availability and cost, the type of process to which they are subjected and specific requests from users [14]. There is a lot of research regarding the production of alkali-activated fly ash-based geopolymer materials, but their implementation requires special procedures, because there are not yet unitary regulations governing the manufacture of this type of material. They are carried out on the basis of special technical specifications, complementary to existing norms [15, 16].

Besides the mechanical properties of the alkali-activated geopolymer materials, there are also advantages in construction industry. Studies showed the potential of geopolymer binders to reduce climate change impacts due to cement production, which can be estimated to 44–64% improvement in greenhouse gas emissions. In case of precast concrete products and applications, geopolymer concrete has shown satisfactory and promising results [17-29].

Research on the development of alkaline activated geopolymer binder systems using local industrial waste is in line with the national and international research, development and innovation objectives by increasing the contribution to the advancement of frontier knowledge, through research carried out in niche areas, to create a comparative and emerging lead in the development of geopolymer materials and by internationalising research in Romania.

The aim of this research is to present preliminary results regarding the design of the alkali-activated fly ash-based geopolymer paving blocks produced using Romanian local raw materials, to present several parameters that affect their mechanical properties and to assess their intended use in terms of legislation. Based on previous results obtained for alkali-activated geopolymer concrete [30, 31], research directions on the material development and technological tranfer were identified, highlighting the approach of a current research theme which includes "Sustainable Development" and "Circular Economy" concepts.

Experimental part

Materials

In order to produce the prefabricated paving blocks, preliminary alkali-activated fly ash-based geopolymer concrete mixtures were selected, based both on studying the literature and starting from a rigorous selection of source materials that will be used to produce this type of material. The evolution of the geopolymer concrete mix design was developed based on the observations collected in the experimental research and initial practical assessments, the fresh state appearance and the performance of the materials at a certain time [30, 31].

Fly ash

Low calcium-fly ash from a power plant in Romania was chosen as main raw material for the production of the alkali-activated fly ash-based geopolymer paving blocks. Its chemical composition was established through X-ray fluorescence methods. As seen in Table 1, the CaO content of the fly ash was less than 10% and SiO_2+Al_2O_3+Fe_2O_3 > 70%, SO_3 < 5% and L.O.I. < 6% (Loss on Ignition), therefore this type of fly ash can be classified as Class F fly ash, according to ASTM requirements and previous studies that have shown that this type of fly ash can be successfully used as raw material in the production of the geopolymer binder [32-35].

The cumulative distribution and the distribution density of the fly ash was established using a HELOS RODOS/L, R5 instrument capable of dry dispersion in the free aerosol jet for laser diffraction and dynamic image analysis (Fig. 1, Tables 2 and 3). As seen, the cumulative distribution of the particle size of the analyzed fly ash shows that more than 99% of the grains are below 215μm and more than 51% below 45μm (0.045mm). The fineness of the raw material used in the production of the alkali-activated fly ash-based geopolymer binder is also an indicator of the later mechanical properties of the material [31].

Table 1. Chemical characterization of the fly ash sample used as main raw material in the development of alkali-activated fly ash-based geopolymer paving blocks

Oxides	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	TiO_2	L.O.I.*
%	46.9	23.8	10.1	10.7	2.7	0.5	0.6	1.7	0.9	2.1

*L.O.I. – Loss on ignition

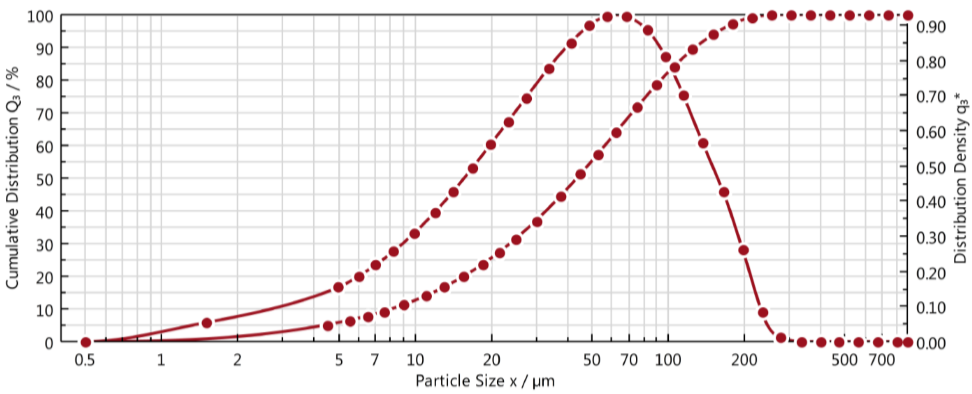


Fig. 1. Cumulative distribution and distribution density of fly ash

Table 2. Cumulative distribution of the particles (fly ash)

x_o (μm)	Q_3 (%)	x_o (μm)	Q_3 (%)
4.50	5.21	75.00	71.63
5.50	6.56	90.00	78.66
6.50	7.91	105.00	84.09
7.50	9.26	125.00	89.41
9.00	11.30	150.00	93.88
11.00	13.99	180.00	97.27
13.00	16.66	215.00	99.29
15.50	19.94	255.00	99.91
18.50	23.75	305.00	100.00
21.50	27.43	365.00	100.00
25.00	31.52	435.00	100.00
30.00	37.01	515.00	100.00
37.50	44.54	615.00	100.00
45.00	51.28	735.00	100.00
52.50	57.29	875.00	100.00
62.50	64.31	-	-

Table 3. Distribution density of fly ash (LOG)

x_m (μm)	q_3 (lg)	x_m (μm)	q_3 (lg)
1.50	0.055	68.47	0.924
4.97	0.154	82.16	0.888
5.98	0.186	97.21	0.812
6.98	0.218	114.56	0.702
8.22	0.257	136.93	0.565
9.95	0.310	164.32	0.462
11.96	0.368	196.72	0.261
14.20	0.429	234.15	0.084
16.93	0.497	278.88	0.011
19.94	0.563	333.65	0.000
23.18	0.625	398.47	0.000
27.39	0.694	473.31	0.000
33.54	0.777	562.78	0.000
41.08	0.850	672.33	0.000
48.61	0.899	801.95	0.000
57.28	0.927	-	-

Alkaline activator

In order to study the influence of the NaOH solution on the mechanical properties of the alkali-activated fly ash-based geopolymer paving blocks, a combination between sodium silicate solution (Na_2SiO_3 solution) and sodium hydroxide solution (NaOH solution) was chosen as alkaline activator for their production. The Na_2SiO_3 solution was commercially purchased and has the following chemical composition: SiO_2 30%, Na_2O 14% and H_2O 56%. Sodium hydroxide 99% purity pearls were used to produce the NaOH solution, which was set to 8M, respectively 10M. In order to achieve the specified molar concentration of the solution 40g x 8M = 320g of NaOH flakes were dissolved in one litre of water for the 8M solution concentration, respectively 40g x 10 = 400g of NaOH for the 10M solution concentration.

Aggregates

Granular class 0/4mm (S - sand) and 4/8mm (CA – coarse aggregates) natural aggregates were used in this study to produce alkali-activated fly ash-based geopolymer concrete paving blocks.

Mix design parameters

The alkali-activated fly ash-based geopolymer paving blocks mix design was based on previous findings. The most important parameters which were taken into account in the production of the material were: ratio of alkaline liquid-to-fly ash by mass, concentration of

NaOH solution, ratio of Na_2SiO_3 to NaOH by mass, curing method, curing temperature, curing time (Tables 4 and 5).

Table 4. Mix design parameters selected for fly ash geopolymer concrete paving blocks

Parameters	Selection
Ratio of alkaline liquid to fly ash (by mass)	0.9
Concentration of NaOH liquid (molars)	8 -10
Ratio of Na_2SiO_3/NaOH solution (by mass)	2.5
Curing method	Oven
Curing temperature (ºC)	70
Curing duration (hours)	24
Testing age (days)	7

Table 5. Details of mix proportions for fly ash geopolymer concrete paving blocks (ratio to fly ash by mass)

Parameters	AAGPB1	AAGPB2
Fly ash (%)	1.0	1.0
AA/FA	0.9	0.9
Na_2SiO_3 solution (%)	0.64	0.64
NaOH solution (%)	0.26	0.26
NaOH concentration (Molar)	8	10
Na_2SiO_3/NaOH (%)	2.5	2.5
S 0/4mm	0.93	0.93
CA 4/8mm	0.93	0.93
S:CA	0.5:0.5	0.5:0.5
Binder/Aggregates	1:1.85	1:1.85

Production of the alkali-activated fly ash-based geopolymer paving blocks

The technology used for the production of the alkali-activated geopolymer paving blocks was based on the principles established for the production of alkali-activated geopolymer concrete and the results obtained in previous studies [30, 31, 36]. Mixing of the materials was performed at constant temperature (20±2°C) and addopting the following the technological scheme (Fig. 2).

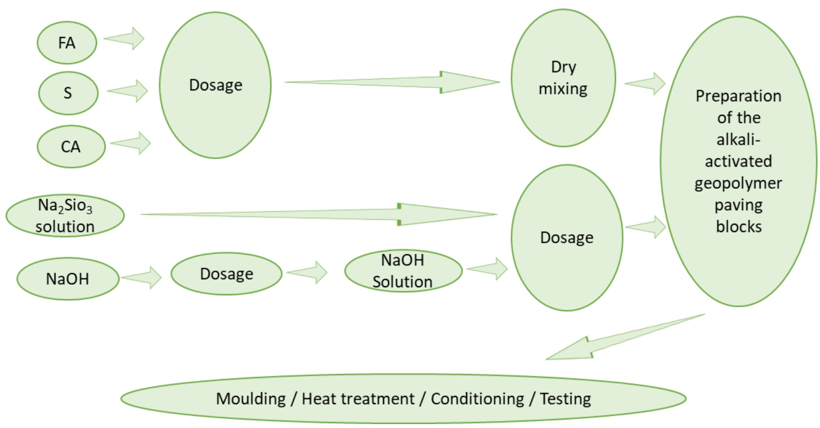


Fig. 2. Technological production of the alkali-activated geopolymer paving blocks

After dosing, the mixing of the dry raw materials was started by mixing the *sand* (S) and the *coarse aggregates* (CA), with the addition of the necessary amount of fly ash. The alkaline activator was then added and the mixing was continued until a homogenous mixture was obtained. After the mixing was finished, the samples were casted into polypropylene moulds, with the corresponding 5 minutes vibration and the proposed heat treatment (70°C/24 hours) was started. After demoulding, the geopolymer paving blocks were stored in the climatic chamber at the temperature T (20±2°C) and relative humidity RH (60±5%). The characteristic tests for their mechanical properties were determined at the age of 7 days (Fig. 3).

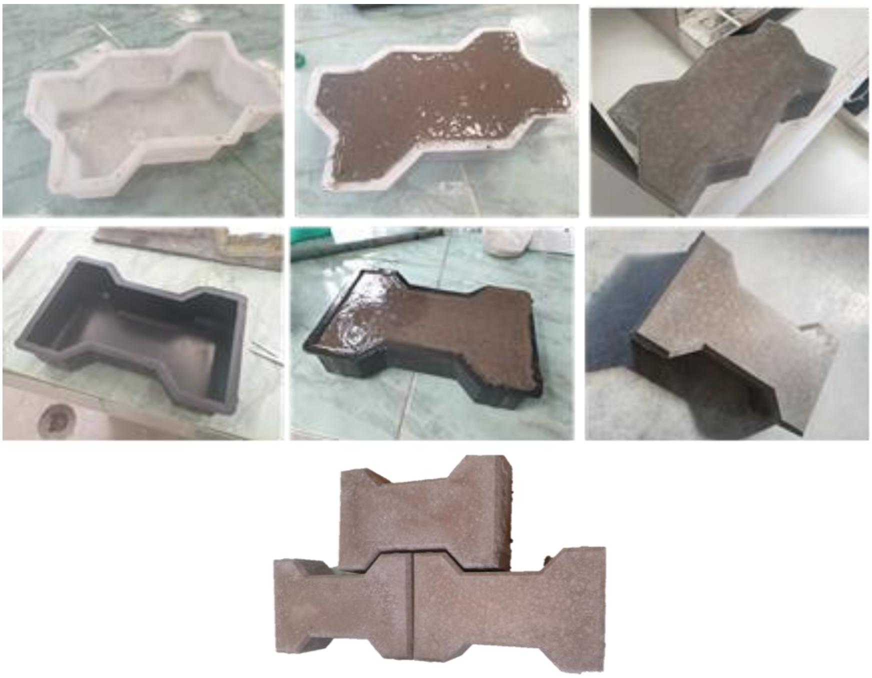


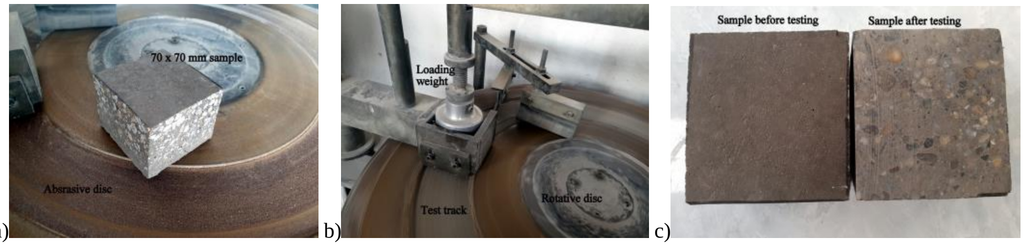
Fig. 3. Production of the alkali-activated fly ash-based geopolymer paving blocks

Methods

The assessment of the mechanical properties of the alkali-activated fly ash-based geopolymer paving blocks was made according to EN 1338:2004/AC:2006: *Concrete paving blocks. Requirements and test methods* [37]. In order to be used for their intended use, the geopolymer paving blocks must comply with certain conditions at the time of their declaration, therefore, this standard was chosen for their assessment because specifies the materials, characteristics, conditions and methods of testing for paving blocks. Test performed for the alkali-activated fly ash-based geopolymer paving blocks and the limits imposed by the standard are presented in table 6 and figures 4 and 5.

Table 6. Tests performed for the assessment of the mechanical properties of the alkali-activated fly ash-based geopolymer paving blocks

Parameters	EN 1338 limit
Total water absorbtion (Annex E)	≤ 6%
Freeze-thaw resistance with de-icing salt (Annex D)	≤ 1.00kg/m^2
Böhme abrasion resistance (Annex H)	≤ 18000mm^3/5000 m^2
Tensile splitting strength (Annex F)	≥ 3.6N/mm^2



a) b) c)

Fig. 4. Böhme abrasion resistance test:
a) Sample dimensions; b) Testing of the samples; c) Sample after testing

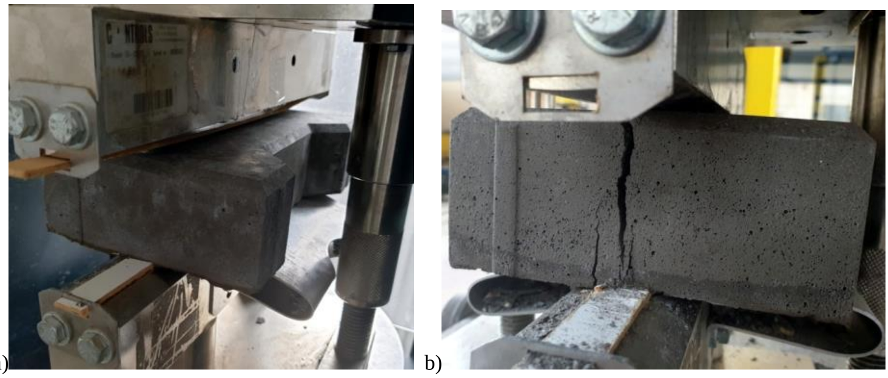

a) b)

Fig. 5. Tensile splitting strength test:
a) Sample testing; b) Sample after maximum load

Results and discussion

By adopting all the conditions and testing methods in order to obtain the mechanical properties of the alkali-activated fly ash-based geopolymer paving blocks the follosving results have been obtained and their assessment with the EN 1338:2004 / AC:2006: Concrete paving blocks. Requirements and test methods standard was analyzed. Also, the mix design properties, in terms of molar concentration of the NaOH solution, was assessed to study the influence of this parameter on the mechanical properties of the paving blocks. All the results are also expressed in accordance with the standard limits for every testing method.

Wathering resistance - Total water absorption

As seen in Figure 6, the total water absorbtion of the alkali-activated fly ash-based geopolymer paving blocks lies beyond the limits of the EN 1338 standard, which indicates that, from this point of view, both the mixtures comply with this limit. Results also show that mixture AAGPB2 (10M NaOH solutions) presents better results than mixture AAGPB1 (8M NaOH solution). This indicates that the molar concentration influences this parameter in terms of mechanical properties.

Wathering resistance – Freeze-thaw resistance with de-icing salt

Figure 7 shows that in terms of freeze-thaw resistance the alkali-activated fly ash-based geopolymer paving blocks lie beyond the limits of the EN 1338 standard, which indicates that, from this point of view, both the mixtures comply with this limit. Mixture AAGPB2 (10M NaOH solutions) presents better results than mixture AAGPB1 (8M NaOH solution), also indicating that the molar concentration influences this parameter in terms of mechanical properties.

Böhme abrasion resistance

Figure 8 shows that in terms of Böhme abrasion resistance of the alkali-activated fly ash-based geopolymer paving blocks only mixture AAGPB2 complies with the limits imposed by the EN 1338 standard, with values lower than 18000 $mm^3/5000mm^2$. This mainly happens because as the molar concentration of the NaOH solution increseases from 8 to 10M the structure of the material is denser, therefore obtaining better mechanical properties from the point of view of the abrasion resistance.

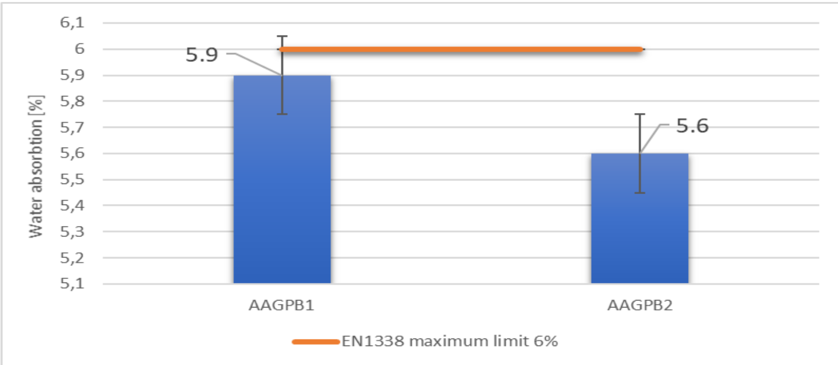


Fig. 6. Weathering resistance – total water absorption of the alkali-activated fly ash, based geopolymer paving blocks

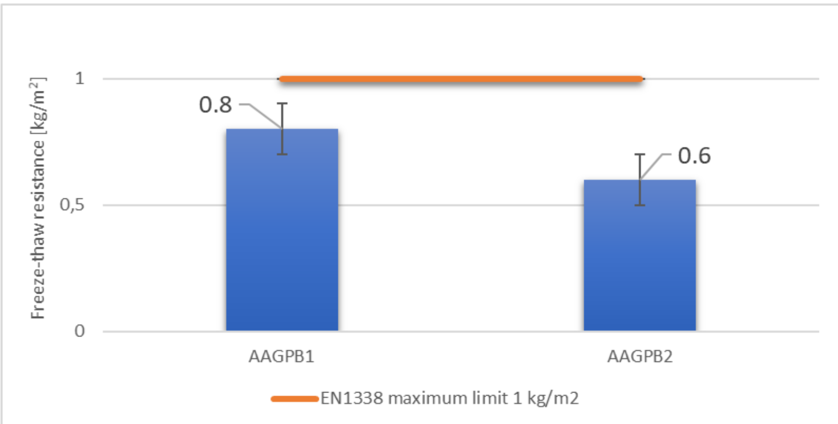


Fig. 7. Weathering resistance – freeze thaw resistance with de-icing salt of the alkali-activated fly ash based geopolymer paving blocks

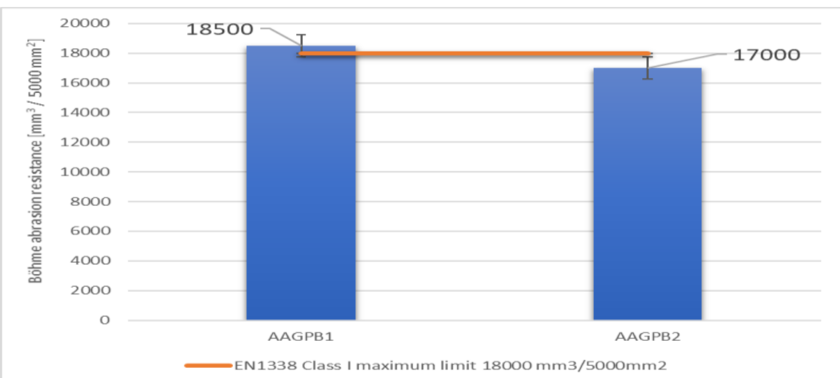


Fig. 8. Böhme abrasion resistance of the alkali-activated fly ash-based geopolymer paving blocks

Tensile splitting strength

In terms of tensile splitting strength, Figure 9 also shows that the molar concentration of the NaOH solution has an influence on the mechanical properties of the alkali-activated fly ash-based geopolymer paving blocks. By increasing the molar concentration from 8 to 10M, mixture AAGPB2 complies with the limit imposed by the EN 1338 standard, with values of the tensile splitting strength above 3.6N/mm^2.

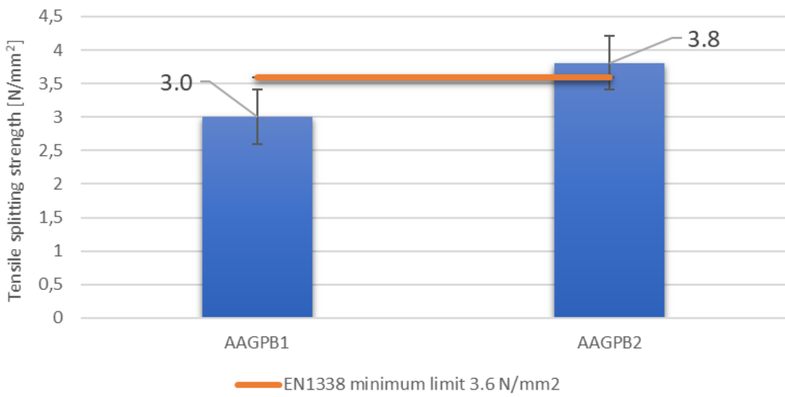


Fig. 9. Tensile splitting strength of the alkali-activated fly ash-based geopolymer paving blocks

Assessment of the mechanical properties of the geopolymer paving blocks

By assimilating the methodology and admissibility conditions specified by standard EN 1338 it can be seen that only mixture AAGPB2 complies with all the limits imposed by the standard (Table 7). The results can be also observed in terms of molar concentration of the NaOH solution. The mixture with 10M concentration showed the best results in terms of compliance with the standard, indicating as in the case of the alkali-activated fly ash-based geopolymer paste, that as the molar concentration of the solution increases, the mechanical properties of the material increases [31, 32].

Table 7. Tests performed for the assessment of the mechanical properties of the alkali-activated fly ash based geopolymer paving blocks

Parameters	EN 1338 limit	AAGPB1	AAGPB2
Total water absorbtion	$\leq$ 6%	✓	✓
Freeze-thaw resistance with de-icing salt	$\leq$ 1.00kg/m^2	✓	✓
Böhme abrasion resistance	$\leq$ 18000mm^3/5000 m^2	X	✓
Tensile splitting strength	$\geq$ 3.6N/mm^2	X	✓

Conclusions

The aim of this research was to present results regarding the design approach and mechanical properties of alkali-activated fly ash-based geopolymer paving blocks using Romanian local raw materials and to study the possibility of assessing their intended use in terms of legislation.

The results obtained on the alkali-activated fly ash-based geopolymer paving blocks produced using Romanian local raw materials are in line with the literature and show that the material properties are notably influenced by several parameters: the physical and chemical properties of the main raw material (fly ash) used as precursor; the chemical properties of the alkaline solution and also, the mix design ratios. The nominated parameters analyzed for mixture AAGPB2 confirm the ability of the material to comply with the assesed standard, demonstrating the possibility of their use as paving elements in the construction market.

While geopolymer technology alone is not sufficient to satisfy economic drivers, the current environmental requirements in terms of stopping carbon dioxide emissions have become reason enough for the construction industry to consider. Despite possible problems and limitations related to the development of alkali-activated geopolymer binder systems (binders, mortars and geopolymer concrete), the results obtained in their production lay the groundwork for validating the possibility of using industrial waste and by-products from local sources in Romania. Moreover, the possibility of integrating geopolymer mixture into construction materials and products has been demonstrated; this is only possible through compositional optimization processes due to the multitude of influences affecting the performance of this type of material.

By analyzing the obtained results presented in the study, the need to continue existing studies was identified due to the many unknowns in the field regarding the geopolymerization process and the properties of the material, all togheter with the improvement in the mix-design of the material, with constant optimization, in order to comply with the current standards in the civil engineering and materials industry.

Acknowledgments

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