



Research paper

Optimization of geopolymer mix composition for structural civil engineering applications

Janusz Kobaka¹, Jacek Katzer², Alberto Gallina³, Karol Seweryn⁴

Abstract: Recently, geopolymers, a type of inorganic non-metallic cementitious materials, have attracted considerable attention as an alternative to ordinary Portland cement (OPC) and as an effective pathway to mitigate energy consumption and minimize CO₂ emissions. The paper proposes a method of geopolymer design to achieve best mechanical properties of the developed material from the civil engineering perspective. Using a ternary plot, the authors selected specific proportions of geopolymer ingredients which predetermine such properties as high workability, high compressive and flexural strength. In the first stage of the research, a mixture of sand, fly ash, and alkaline activators were used to initiate the polymerization process which allowed to form specimens designated for further tests. The promising properties of geopolymers and the lack of access to OPC on the Moon have led to the consideration of using geopolymers as a building material in the construction of future extraterrestrial bases. In the second stage, simulant of the Moon regolith was utilized. The achieved results justify the claim that the proposed formulation method of geopolymers, designed for civil engineering applications, is useful both for terrestrial and extraterrestrial applications.

Keywords: geopolymers, extraterrestrial engineering, ISRU, regolith, lunar soil simulant

¹PhD., Eng., 1) AGH University of Krakow, Faculty of Mechanical Engineering and Robotics, al. Adama Mickiewicza 30, 30-059 Cracow, Poland; 2) University of Warmia and Mazury in Olsztyn, Faculty of Geoengineering, ul. Michała Oczapowskiego 2, 10-719 Olsztyn, Poland, e-mail: janusz.kobaka@gmail.com, ORCID: 0000-0002-3596-3187

²Associated Professor, PhD., Eng., University of Warmia and Mazury in Olsztyn, Faculty of Geoengineering, ul. Michała Oczapowskiego 2, 10-719 Olsztyn, Poland, e-mail: jacek.katzer@uwm.edu.pl, ORCID: 0000-0002-4049-5330

³Associated Professor, PhD., Eng., AGH University of Krakow, Faculty of Mechanical Engineering and Robotics, al. Adama Mickiewicza 30, 30-059 Cracow, Poland e-mail: alberto.gallina@agh.edu.pl, ORCID: 0000-0002-3750-1875

⁴Associated Professor, PhD., Eng., Space Research Centre of the Polish Academy of Sciences (CBK PAN), ul. Bartycka 18A, 00-716 Warsaw, Poland, e-mail: kseweryn@cbk.waw.pl, ORCID: 0000-0002-4372-0900

1. Introduction

Civil engineering in the present day, when the deadline for building a permanent lunar base is set for the end of this decade, doesn't apply anymore to facilities built on the Earth, but has become more generalized, adding a number of variables to the design calculations, such as different gravity conditions, wide temperature range, different atmosphere, or even no atmosphere at all, and applies to much more complicated environments than ever before. NASA's newest and very promising lunar program known as *Artemis* with a concept of *Artemis Base Camp*, as well as planned by European Space Agency *Terrae Novae* program, will be first sustainable foothold on the lunar frontier [1–3]. The differences between the two programs are substantial. The program *Artemis*, named after the Greek goddess of hunting, wildlife, fertility, and the Moon, assumes the establishment of a permanent base on the Moon. The program *Terrae Novae*, which means "New Lands" in Latin, assumes the establishment of a lunar orbital station, which will be a link and base between the Earth and the Moon. However, like the *Artemis* program, the *Terrae Novae* program assumes the creation of a permanent lunar base that will enable research and exploitation of the Moon.

Regardless of the basic assumptions, the both lunar programs will have to deal with a concept of ISRU (*In-Situ Resource Utilization*). The idea of ISRU involves obtaining and processing materials necessary for life and functioning on site (in this case on the Moon), and useable products would be extracted from or made of lunar resources [4–7]. To excavate lunar regolith from the lunar surface, a preliminary infrastructure must be created, including a landing site, a lunar mine [3], and habitats protecting against radiation [8] where people could live and where food would be produced. In addition, a number of devices will be needed to support life processes, obtain oxygen and water, extract regolith, move around, etc. These are very difficult and ambitious tasks, but at the same time necessary for the success of the mission, and permanent human presence on the Moon.

Due to the lack of clinker resources on the Moon, it is impossible to produce commonly used building material on the Earth [9, 10], which is concrete. For this reason, it is necessary to assume the use of other materials that will have similar strength and durability properties. One of such materials, which is predicted to be produced on the Moon, is geopolymers [11–14]. The concept of geopolymer was initially introduced by a French material scientist Joseph Davidovits et al., in the late 1970s, referring to a geosynthesized inorganic polymer [15–17]. It involves the utilization of aluminosilicate materials as precursors to produce three-dimensional gel structures through alkali activation [18]. Geopolymers offer superior performance characteristics over ordinary concrete, these include enhanced strength and fire resistance, reduced permeability, and improved environmental durability [19, 20]. One of the most famous example of a geopolymer structure is building of the University of Queensland's Global Change Institute. The four-story high structure, dedicated for general public use, comprises 3 suspended geopolymer concrete floors involving 33 precast panels, made of slag/fly ash-based geopolymer concrete [21]. Quickly

gaining popularity, geopolymers, also due to their rapid setting properties in comparison to Portland cement and other advantages, may find a number of applications not only on the Earth, but also in extraterrestrial conditions on the Moon and Mars.

The Apollo mission brought to Earth 382 kg of lunar rocks, core samples, pebbles and dust [22]. It is almost impossible for researchers to use this material for testing. Due to lack of access to the original lunar regolith, simulants are used (especially for destructive tests). These materials by definition, have characteristics similar to those of real lunar regolith [23–26]. In recent works [27] researches developed lunar analogues with significant ash content imitating the fine grained dusty fraction of the lunar soil. Lunar regolith simulants, due to different characteristics of the regolith found in seas, highlands and polar regions, often are assigned to such regions. For the purposes of this research, simulant LHS-1 (Lunar Highlands Simulant) was used. It is a commercially available and well-studied lunar regolith simulant that reflects the properties of regolith from the lunar highlands [28–31].

In this study, the authors, using an experiment design based on a ternary plot, tried to establish best proportions of aqueous solutions of two chemical compounds (sodium metasilicate pentahydrate and sodium hydroxide), which play a role of alkaline geopolymer activators. The main aim of the study was to achieve the highest strength results in combination with the LHS-1 lunar regolith simulant.

2. Materials used and the design of the experiment

In order to compose a geopolymer that will meet the requirements imposed on concrete, two types of specimens were prepared: specimens composed of sand and fly ash in equal proportions by weight and specimens composed of lunar soil simulant LHS-1 and fly ash also in equal proportions by weight, together with alkaline activator (see Tables 1 and 2).

Table 1. Composition of mixes based on sand

Designation	Activator (g)	Sand (g)	Fly ash (g)
S01	96	250	250
S02	97	250	250
S03	94	250	250
S04	95	250	250
S05	96	250	250
S06	109	250	250
S07	105	250	250
S08	110	250	250

Continued on next page

Table 1 – *Continued from previous page*

Designation	Activator (g)	Sand (g)	Fly ash (g)
S09	129	250	250
S10	123	250	250
S11	114	250	250
S12	115	250	250
S13	94	250	250
S14	136	250	250
S15	117	250	250
S16	117	250	250
S17	113	250	250
S18	124	250	250

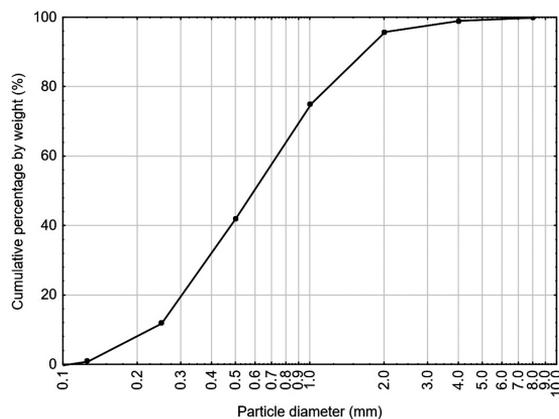
Table 2. Composition of mixes based on LHS-1

Designation	Activator (g)	Fly ash (g)	LHS-1 (g)
LHS01	166	250	250
LHS02	171	250	250
LHS03	169	250	250
LHS04	216	250	250
LHS05	176	250	250
LHS06	184	250	250
LHS07	193	250	250
LHS08	212	250	250
LHS09	199	250	250
LHS10	220	250	250
LHS11	219	250	250
LHS12	213	250	250
LHS13	217	250	250
LHS14	227	250	250
LHS15	224	250	250
LHS16	222	250	250
LHS17	247	250	250
LHS18	232	250	250

Basic parameters of sand used in the experiment were measured (see Table 3), Fig. 1 shows the particle size distribution of the sand. The grain stack consisted of aggregate with fractions of 0–2 mm with the amount of oversize (fractions 2–8 mm) equal to 4%. The aggregate was characterized by high grain size uniformity and continuity of the grain size curve.

Table 3. Properties of sand used in the experiment

Property	Value
Dust content (fractions below 0.065 mm)	1.25%
Grain density	2650 kg/m ³
Bulk density in loose state	1615 kg/m ³
Bulk density in compacted state	1828 kg/m ³
Void ratio in loose state	39.2%
Void ratio in compacted state	30.7%

Fig. 1. Grain size distribution of the sand used in the experiment in a logarithmic scale³

Chemical content of the fly ash used in the experiment, declared by the producer, is presented in Table 4.

Table 4. Chemical content of the fly ash used in the experiment (data sourced from [32])

Chemical content by mass	Value (%)
Chloride (Cl)	< 0.10
Sulfuric anhydride (SO ₃)	< 3.0
Free calcium oxide (CaO)	< 1.5
Total calcium oxide	< 10.0
SiO ₂ , Al ₂ O ₃ , and Fe ₂ O ₃	> 70.0
Total phosphorus	< 5.0

The LHS-1 lunar regolith simulant used in the experiment is highlands simulant type of general purpose. Chosen properties of LHS-1, declared by the producer, are presented in Table 5. Mineralogical composition of the simulant is presented in Table 6.

Table 5. Properties of LHS-1 used in the experiment (data sourced from [32])

Property	Value
Uncompressed bulk density	1.27 g/cm ³
Median Particle size	51 μm
Particle size range	0.04–1000 μm

Table 6. Mineralogical composition of LHS-1 (data sourced from [32])

Component	Wt. (%)
Anorthosite	74.4
Glass-rich basalt	24.7
Ilmenite	0.4
Bronzite	0.3
Olivine	0.2

The alkaline activator applied in order to initiate the formation of geopolymer was water solution of sodium metasilicate pentahydrate ($\text{Na}_2\text{SiO}_3 \cdot 5\text{H}_2\text{O}$) ranging from 0 to 50%, and sodium hydroxide (NaOH) ranging from 0 to 50% (see Table 7).

Table 7. Mass proportions of the alkaline activator ingredients used in the experiment

Designated number	$\text{Na}_2\text{SiO}_3 \cdot 5\text{H}_2\text{O}$ (%)	NaOH (%)	H_2O (%)
1	20	0	80
2	10	10	80
3	0	20	80
4	30	0	70
5	20	10	70
6	10	20	70
7	0	30	70
8	40	0	60
9	30	10	60
10	20	20	60
11	10	30	60
12	0	40	60
13	50	0	50
14	40	10	50
15	30	20	50
16	20	30	50
17	10	40	50
18	0	50	50

The plan of the experiment was developed in a form of a ternary plot with mutual weight proportions of the three alkaline components the activator: water, sodium metasilicate pentahydrate, and sodium hydroxide (see Fig. 2). The amount of water ranged from 50% to 80% which was dictated by the results of preliminary research. Solid components of chemical reagents did not dissolve entirely in solutions with water content below 50%. Specimens containing alkaline activator with water content over 80% were too weak to be demolded.

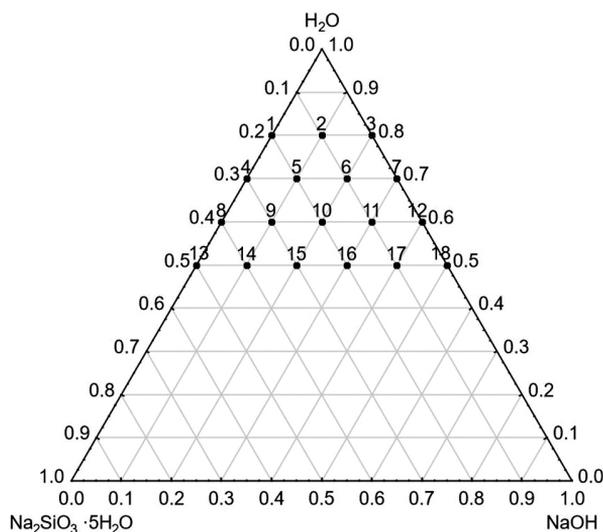


Fig. 2. The plan of the experiment in a ternary plot

The procedure for manufacturing specimens was as follows: dry ingredients such as fly ash, sand, and LHS-1 were mixed in the proportions given above (see Tables 1 and 2). Alkaline water solution was prepared according to the plan of the experiment (see Fig. 2). In order to dissolve the ingredients more efficiently, the water used to prepare the solution was heated to 60°C before adding solid ingredients, which concerned the activators containing less than 30% of sodium hydroxide. There was no need to heat the water for solutions with sodium hydroxide content equal to or greater than 30%. The dissolution of sodium hydroxide in water is a strongly exothermic reaction, and the temperature of the solution can rise even to a boiling point. After preparing the alkaline solution it was mixed with the previously prepared dry ingredients (fly ash, sand, and LHS-1). Subsequent mixing process lasted two minutes. The activator was added gradually until the mixture had the desired consistency. The amount of added activator depended on the consistency of the mixture. The aim was to obtain a plastic consistency that would allow for precise filling of the mold. After gaining the desired consistency, the mixture was placed into the mold in two layers. After each layer was laid, the mixture was compacted by hand with a tamping rod. A total of 36 specimens with dimensions of 160 × 40 × 40 mm were made in order to test relative density, flexural strength and compressive strength. In this way 18 measurement points (see Figs. 2 and 3) for two types of mixtures were created. After the casting process, the filled molds were placed the oven in the temperature of 60°C and left

there. After 24 hours, the specimens were taken out of the molds and placed again in the oven in the same temperature for another 24 hours. The tests of the geopolymer specimens were performed after another 24 hours, when the specimens cooled down.



Fig. 3. Specimens with dimensions $40 \times 40 \times 160$ mm made of geopolymer

3. Results of the experiment

Tests conducted on halves of geopolymer beams, with dimensions of $40 \times 40 \times 160$ mm, showed that the compressive strength of the tested geopolymers, based on sand and fly ash, ranged from 1 to 30 MPa (see Fig. 4). The highest compressive strength values were recorded for the highest concentrations of the activator. Using sodium hydroxide solution resulted in slightly higher compressive strength of the tested geopolymer than specimens created by using solution of sodium metasilicate pentahydrate. The best result was achieved for the solution of NaOH with a molarity of 12.5 M. The lowest compressive strength values were recorded for the lowest solution concentrations.

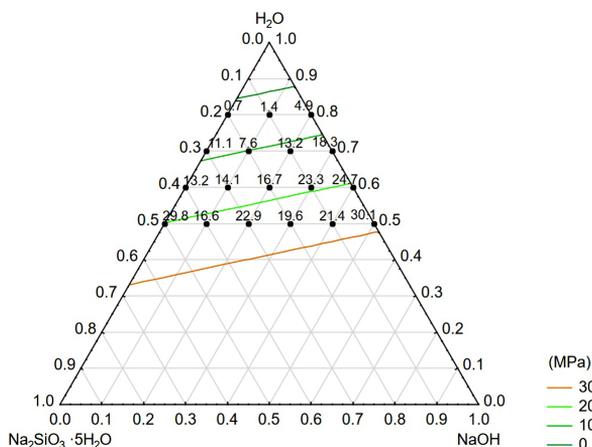


Fig. 4. Compressive strength (MPa) of geopolymer based on sand and fly ash

Bending strength of geopolymer based on sand and fly ash showed similar tendency as compressive strength in the implemented experiment plan (see Fig. 5). The highest values of bending strength were recorded for the highest concentrations of the activator and ranged

from near 0 to 7 MPa. The value of 7.3 MPa was recorded for the specimen with the alkaline activator based on 50% water solution of sodium hydroxide. The specimens prepared with low concentration of alkaline activator were characterized by very low flexural strength. In two cases the sensitivity of the testing machine was insufficient to record the result.

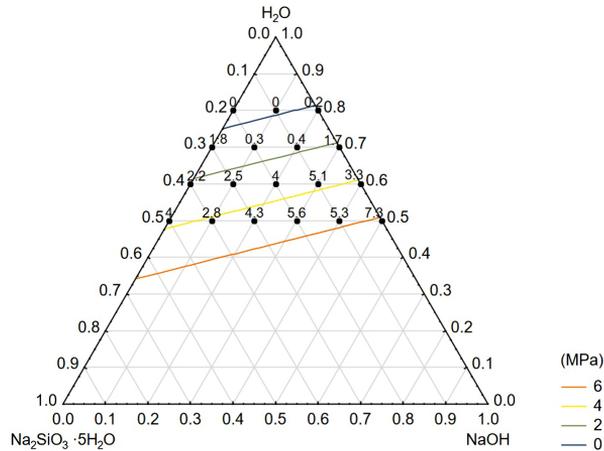


Fig. 5. Flexural strength (MPa) of geopolymer based on sand and fly ash

Relative density of geopolymer based on sand and fly ash was ranging from 1.7 to 2.0 g/cm³ (see Fig. 6). The highest values of the relative density were corresponding to the highest values of both compressive and flexural strength of the tested specimens.

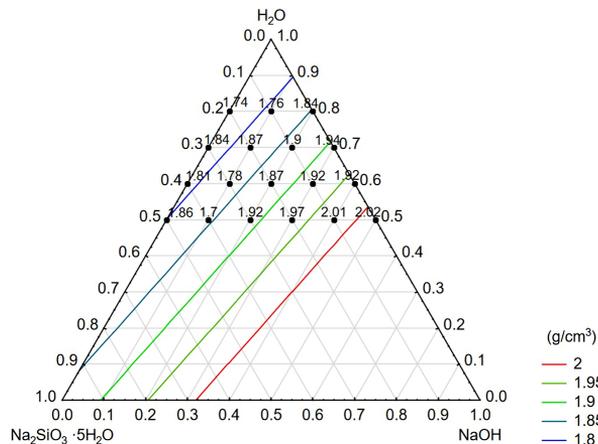


Fig. 6. Relative density (g/cm³) of geopolymer based on sand and fly ash

Compressive strength of the tested geopolymers, based on LHS-1 and fly ash, was ranging from 0.2 to 27.2 MPa (see Fig. 7). The highest values of compressive strength were recorded for the highest concentrations of the used activator similar to the case of a sand and fly ash

based geopolymer. Harnessing the solution of $\text{Na}_2\text{SiO}_3 \cdot 5\text{H}_2\text{O}$, NaOH , and H_2O in proportions of 0.4:0.1:0.5 respectively resulted in the highest compressive strength of 27.2 MPa. The lowest values of compressive strength were recorded for the lowest solution concentrations.

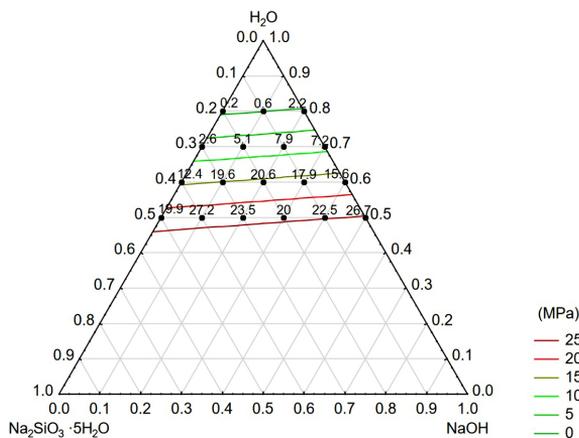


Fig. 7. Compressive strength (MPa) of geopolymer based on LHS-1 and fly ash

Flexural strength of geopolymer based on LHS-1 and fly ash showed similar tendency as compressive strength (see Fig. 8). The highest values of flexural strength were recorded for the highest concentrations of the activator and ranged from 0 to 4 MPa. The highest value of 4 MPa was recorded in three cases, for the specimens treated 50% water solution of alkaline activator, (there were no significant differences due to the type of chemical used). The specimens prepared with low concentration of alkaline activator were characterized by very low flexural strength. In three cases the sensitivity of the testing machine was insufficient to record the result.

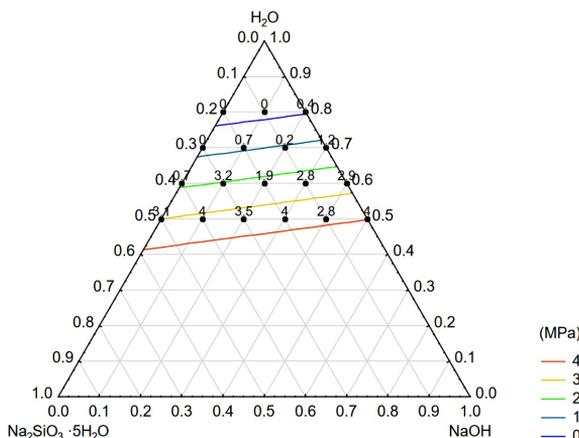


Fig. 8. Flexural strength (MPa) of geopolymer based on LHS-1 and fly ash

Relative density of geopolymer based on LHS-1 and fly ash was within the range of 1.58 to 1.97 g/cm³ (see Fig. 9). The highest values of the relative density were corresponding to the highest values of both compressive and flexural strength of the tested specimens.

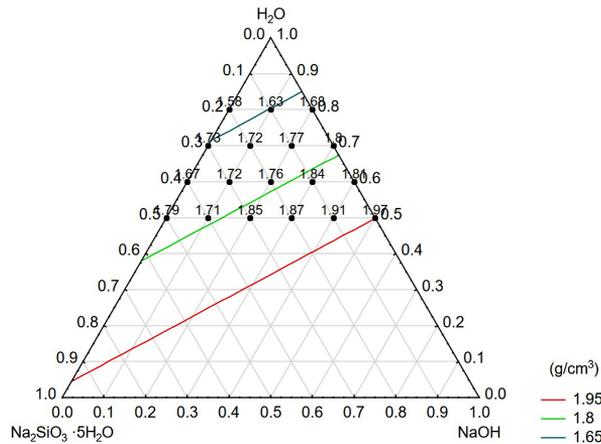


Fig. 9. Relative density (g/cm³) of geopolymer based on LHS-1 and fly ash

4. Conclusions

- The intended goal, which was to obtain geopolymer strength equivalent to that of ordinary concrete, was achieved for both tested materials: the sand-based geopolymer and the LHS-1 based geopolymer.
- The highest compressive strength of 30.1 MPa, as well as the flexural strength of 7.3 MPa, was obtained for the sand based geopolymer at the highest concentration of the alkaline activator, which corresponds to the molarity 12.5 M of sodium hydroxide water solution.
- The highest compressive strength for the LHS-1 based lunar geopolymer was equal to 26.7 MPa, with corresponding bending strength of 4.0 MPa, was obtained for the highest concentration of the alkaline activator of the molarity 12.5 M of sodium hydroxide water solution, which was consistent with the sand based geopolymer result.
- The compressive strength of the geopolymer based on the LHS-1 was only slightly smaller in comparison to geopolymer based on sand. For the activator characterized by the highest concentration, which corresponded to the highest strength, the difference in compressive strength was only 0.4%.
- Geopolymers in question reached full strength after only 3 days. This is a key advantage in comparison to 28 days needed by ordinary concrete.
- The next stage of research on geopolymer intended for extraterrestrial applications should be investigation of the possibility of producing a geopolymer without the compounds contained in fly ash, but with the participation of natural compounds found in the lunar regolith.
- The performed tests were carried out in conditions prevailing on Earth (atmospheric pressure and gravity). The next stage of the research program involves the maturation of

geopolymers in a vacuum chamber. The issue of forming and maturing geopolymers in low gravity conditions will also remain as an important issue.

- At high activator concentrations (50% water solution), there were no significant differences in compressive strength between the composites created using activator with high concentration of $\text{Na}_2\text{SiO}_3 \cdot 5\text{H}_2\text{O}$ and NaOH. However, the use of NaOH as a predominant activator resulted in slightly better compressive strength results (2 to 3%) and higher specimen density (7 to 9%) than the $\text{Na}_2\text{SiO}_3 \cdot 5\text{H}_2\text{O}$ for both sand-based and LHS-1 based specimens.

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Optymalizacja składu mieszanki geopolimerowej w zastosowaniach inżynierii lądowej

Słowa kluczowe: geopolimery, inżynieria pozaziemska, ISRU, regolit, LSS

Streszczenie:

W ostatnich czasach geopolimery, nieorganiczne materiały charakteryzujące się w budowie chemicznej dużymi łańcuchami kopolimerowymi, zaczęły przyciągać uwagę autorów publikacji naukowych jako alternatywa w budownictwie betonowym dla cementu portlandzkiego. Materiały te charakteryzują się mniejszym zużyciem energii potrzebnej do ich wytworzenia i co za tym idzie, zmniejszonym udziałem CO₂ w porównaniu do cementu portlandzkiego. W artykule zaproponowano metodę projektowania geopolimerów w celu uzyskania zoptymalizowanych właściwości mechanicznych z perspektywy inżynierii lądowej. Opierając się na analizie w układzie trójskładnikowym Fereta, autorzy wybrali takie proporcje komponentów geopolimerów, które pozwalają uzyskać wysoką urabialność mieszanki oraz wysokie właściwości mechaniczne, takie jak wytrzymałość na ściskanie i zginanie. W pierwszym etapie badań została użyta mieszanina piasku, popiołu lotnego i alkalicznych aktywatorów w celu zainicjowania procesu polimeryzacji, co pozwoliło na utworzenie próbek przeznaczonych do dalszych testów. Obiecujące właściwości geopolimerów i brak dostępu do cementu portlandzkiego na Księżycu są powodem do rozważania koncepcji użycia tych materiałów przy budowie przyszłych baz pozaziemskich. W drugim etapie badań, w ramach eksperymentu wykorzystano do wytworzenia geopolimeru tzw. symulanty regolitu księżycowego. Wyniki uzyskane z przeprowadzonych badań eksperymentalnych pozwalają na sformułowanie wniosków, że proponowana metoda wytwarzania geopolimerów przeznaczonych do zastosowań w inżynierii lądowej, mogłaby być przydatna zarówno do zastosowań ziemskich, jak i pozaziemskich.

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