

Marta NALEWAJKO

## RESEARCH ON CEMENT-FREE COMPOSITES BASED ON ALKALINE- ACTIVATED WASTE MATERIALS

Marta **Nalewajko** (ORCID: 0000-0002-2458-7147) – *Bialystok University of Technology*

Correspondence address:

Wiejska Street 45E, 15-351 Bialystok, Poland

e-mail: m.nalewajko@pb.edu.pl

**ABSTRACT:** The article presents a review of research conducted on cement-free concretes based on alkaline-activated waste materials. Research is conducted in order to create concretes that are in line with the doctrine of sustainable development. Their main assumption is the reuse of recycled materials in newly produced building materials without compromising their properties. In addition, attempts are made to eliminate Portland cement, replacing it partially or completely with fly ashes or metakaolin. Another modification of concrete consists of replacing natural aggregate with artificial aggregate. The research conducted on lightweight concretes based on fly ashes, and alkali-activated porous ash aggregate is also presented.

**KEYWORDS:** cement-free composites, geopolymer concretes, artificial aggregate, alkaline activation

## Introduction

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According to the Climate Target Plan adopted by the European Commission, The European Union has set itself an ambitious goal of lowering carbon dioxide emission levels by 50-55% by 2030 compared to 1990 levels. The primary objective is to achieve a net-zero-emissions economy by 2050. Accordingly, the European Commission is proposing to introduce regulations regarding greenhouse gas emissions, of which level would be zero percent by then. To achieve this, it is planned to implement the European Climate Law under the relevant regulation. The pursuit of these goals is extremely important for environmental protection and the fight against climate change. The European Union is taking decisive action to become a global leader in the fight against climate change and to preserve the planet for future generations ([KNE, 2021](#)).

The construction sector is consistently aligning itself with the objectives set forth in Regulation (2011), focusing on the sustainable use of natural resources. It introduces guidelines that take into account recycling, the durability of construction objects, and the use of environmentally friendly and reprocessed materials. Although the complete elimination of industrial waste (e.g., from energy, metallurgy, mining and construction facilities) generated by business activities is not possible, the industry is making efforts to reduce its volume and find ways to reuse it. In most cases, industrial waste is not suitable for reuse. However, some of it can find a second life. Research is being carried out around the world to develop technologies for processing industrial waste so that it becomes a valuable raw material in construction production. Aimed at allowing these wastes to retain their properties and sometimes even become better substitutes for natural resources. This approach provides an opportunity to limit a negative impact on the environment and promotes a closed-loop economy that minimises waste of resources. The implementation of these technologies contributes to the sustainable development of the construction sector while contributing to global efforts to protect the environment and combat climate change. These measures provide a greener and more sustainable approach to construction, which is crucial for the future of our planet ([Nalewajko, 2022](#)).

In 2022, Kalinowska-Wichrowska et al. (2022) presented a paper detailing the physical and mechanical characteristics of geopolymers incorporating lightweight artificial aggregate. They conducted a research experiment to investigate the impact of a fly ash-slag mix (FA-S) as a pozzolanic additive on geopolymer properties, determining the most favourable molar concentration of sodium hydroxide solution. Through statistical analysis, considering compressive strength after 28 days, water absorption, and

density tests on GLC, the optimal composition for lightweight geopolymer concrete was established: FA 200 kg/m<sup>3</sup>, FA-S 200 kg/m<sup>3</sup>, 10 M NaOH solution, artificial aggregate fractions of 0–2 mm for 222 kg/m<sup>3</sup>, 1–4 mm for 223 kg/m<sup>3</sup>, and 4–8 mm for 445 kg/m<sup>3</sup>, and for surface impregnation of coarse aggregate, a fraction of 4–8 mm with 125 L of NaOH. Optimisation tests on the influence of FA-S percentage relative to silicate fly ash, as well as the content and molar concentration of NaOH solution, indicated that the best composition resulted in GLC with compressive strengths exceeding 16 MPa. To achieve higher compressive strength, it is recommended to use two activators: sodium water glass and sodium hydroxide. These GLC composites can be applied in insulating materials where exceptional strength is not a prerequisite (Kalinowska-Wichrowska et al., 2022).

Ansari et al. (2023) made a review of the design approaches used in preparing the mix geopolymer (GPC). Its literature discussed three types of approaches used in GPC mix proportioning: the target strength approach, the performance approach and the statistical approach. The target strength approach is the most commonly used by researchers in GPC mix proportioning, where the design focuses on achieving specific compressive strength and workability properties. The performance-based approach, on the other hand, is a relatively new method of GPC mix proportioning, where the design is based on specific performance criteria, not just compressive strength and workability. Using statistical approaches in GPC mix design can save time, money and labour. Of the many statistical methods used by the authors in GPC mix proportioning, the most commonly chosen approach is the following Taguchi due to its simplicity of application and effectiveness. In more recent studies, researchers have begun to use artificial neural networks (ANNs) because of their ability to handle more complex data and provide more precise results in GPC mixture proportioning. It is important to note that the continued development and improvement of design approaches in mixture preparation geopolymer contributes to increasingly better results and efficiency in its use (Ansari et al., 2023).

A review of research related to geopolymers has also made Zaid et al. (2023) except that the research these focused on mixtures geopolymer with the addition of nanomaterials (NMs). In their paper, they showed that nanomaterials exert unique effects on GPC due to their specific chemical and physical properties. Of particular importance is that nanomaterials fill voids at the level of the nano, which speeds up chemical reactions, participates in the action of the pozzolanic and strengthens the transition zone between the molecules. The researchers observed that the strength characteristics of GPC, such as compressive, flexural and splitting strengths, increase with nanomaterial dosages, reaching optimum levels at dosages of about 2-4%. In addition, the durability of the mixture improves significantly at different doses of

nanomaterials, as these materials refine the pores in the structure of the geopolymer, prevent the ingress of harmful substances and thicken the structure, leading to better resistance to external agents. The results indicate the potential of nanomaterials to improve and enhance the properties of geopolymer, which could have important implications for practical applications as well as for developing structural and materials technologies (Zaid et al., 2023).

In 2023, a study was published in which Gopalakrishna and Pasla (2023) analysed the impact of the additive metakaolin in concretes geopolymer based on high-strength recycled aggregates, fly ash and ground granulated blast furnace slag. Various parameters such as workability, compressive strength, tensile strength, modulus of elasticity, water permeability and absorbability were included in the scope of the study. Six different geopolymer mixtures were evaluated. The results of the study showed that the use of metakaolin in these concrete geopolymers, which used high-strength recycled aggregates, fly ash and ground granulated blast furnace slag, yielded excellent and durable mechanical properties. This makes such “green” concrete mixtures suitable for a variety of construction projects. These results suggest that the use of metakaolin can be an effective strategy for improving the strength and durability of concrete geopolymers, especially those based on renewable and recycled materials. This, in turn, can have a positive impact on sustainable construction practices, reducing the amount of waste and natural resources used in concrete production (Gopalakrishna & Pasla, 2023).

In that same year, an article authored by Salas Montoya et al. (2023) explored composite cement incorporating ground granulated blast furnace slag, fly ash, and geothermal silica through alkali activation. The findings revealed that sodium hydroxide triggered an alkali–silica reaction, diminishing strength, whereas sodium silicate and sodium sulfate enhanced strength and facilitated the formation of hydration products. Additionally, the inclusion of fly ash led to a reduction in compressive strength but an improvement in workability, while the incorporation of slag and geothermal waste bolstered strength, consolidating the matrix with the creation of additional hydration products. The study illustrated the viability of utilising waste materials to produce blended cement with low energy expenses and high durability (Salas Montoya et al., 2023).

The new material used for concrete geopolymer is hooked steel fibres. The results of such studies is presented in the article Laxmi et al. (2023). Conducted experiments unequivocally proved that the use of steel fibres with hooked ends positively affects the fracture resistance and strength properties of concrete structures geopolymer.

The study used geopolymer concretes based on fly ash, ground granulated blast furnace slag (GGBS), NaOH, Na<sub>2</sub>SiO<sub>3</sub>, sand and natural coarse

aggregate. To these mixtures, steel fibres with a hooked end with an aspect factor of 67 were added in different doses: 0%, 0.5%, 1%, 1.5% and 2%. Experimental results showed that the addition of steel fibres reduced the workability of the concrete, with a minimum slump of 70 mm and a maximum time of Vee Bee of 8 s for mixtures with 2% steel fibres. However, the addition of fibres significantly improved the compressive strength, tensile strength and flexural strength of the concrete geopolymer. The maximum values achieved were 41.44, respectively. MPa, 4,28 MPa i 5,23 MPa for an optimal fiber dose of 1%. Scanning electron microscope imaging revealed the positive effect of steel fibres in reducing progressive cracks in concrete geopolymer. It was found that properly dosed steel fibres improved the performance of geopolymer concrete, which can contribute to obtaining a material with low CO<sub>2</sub> emissions, which is crucial from an environmental point of view. Findings from the study suggest that the use of steel fibres may be a beneficial solution for reinforcing concrete geopolymer, improving its strength and fracture toughness, and contributing to the development of greener and low-carbon building materials (Laxmi et al., 2023).

Innovative geopolymer concrete was also proposed by Noor Abbas et al. (2023) presenting the results of research on kenaf fibre reinforced geopolymer concrete (KFRGC). Conducted experiments to evaluate the effect of the addition of kenaf fibre (KF) on the properties of the geopolymer concrete. The study included an analysis of water absorption, porosity, water absorption, water penetration, and resistance to chloride ion permeation and chemical attacks. The results showed that the addition of kenaf fibres increased the porosity geopolymer, which resulted in better properties related to water absorption. It was observed that the porosity bias increased with increasing fibre length and volume. Compared with control samples, the largest increase in porosity (12.5%) was observed in samples with 1.5% fibre volume with a length of 40 mm. Similarly, water permeability increased with increasing fibre volume and length in the matrix geopolymer. For example, in samples containing 1.5% fibre, increasing the fibre length from 20 to 30 mm resulted in an increase in water permeability from 7.42 cm to 8.9 cm. However, chloride, sulfate and acid resistance was improved due to the fibre's ability to bridge cracks. Longer fibres (40 mm) affected the acid resistance of the concrete, resulting in a loss of strength of up to 16.3% and 19.2% at 180 and 365 days, respectively. The results of this study showed that the use of kenaf fibres as a fibrous material in the production of environmentally friendly and durable concrete has promising prospects. While kenaf fibres can affect some properties of concrete, they also improve its resistance to certain damaging agents. This makes concrete geopolymer with the addition of kenaf fibre can be a valuable solution for sustainable and durable construction projects (Noor Abbas et al., 2023).

Al-Duais et al. (2023) published an article focusing on the optimisation of alkali-activated binders using natural minerals and industrial waste. The physical and chemical properties of four chosen precursor materials (natural pozzolana, limestone powder, red mud, and silico manganese fume) were characterised. The proportioning of these materials was fine-tuned based on flow, setting time, and compressive strength tests conducted on trial mortars. After achieving an optimal blend of the four precursor materials, natural pozzolan was partially substituted with ordinary Portland cement (up to 30% by weight) to significantly enhance the properties of the alkali-activated binders (AABs). The ratios of activator to precursor (A/P), sodium silicate to sodium hydroxide (NS/NH), sodium hydroxide (NH) molarity, and water to precursor (W/P) were varied within the ranges of 0.3 to 0.6, 1 to 2.5, 8 to 14 M, and 0.35 to 0.55, respectively. The resulting samples exhibited 28-day compressive strengths ranging from 28.5 to 32 MPa, 24.15 to 31.8 MPa, 24.2 to 33.1 MPa, 15.33 to 31.16 MPa, and 19.7 to 34.1 MPa. This study's outcomes facilitated the identification of the optimal proportions of selected precursor materials and the ideal combination of A/P ratio, NS/NH ratio, NH molarity, and W/P ratio for producing high-performance AABs (Al-Duais et al., 2023).

All research conducted on lightweight concrete geopolymer were conducted using either natural aggregates or mixtures of natural aggregates with artificial aggregates. In the research presented below, the use of natural aggregates was eliminated and replaced with artificial aggregates made from fly ash, which is a waste energy product.

## Materials

In order to prepare lightweight concretes based on alkali-activated energy waste raw materials, the following ingredients were used: fly ash, alkali activators of different concentrations (in the range of 2-6 mol/dm<sup>3</sup>) and aggregate Ashes type Certyd with different fractions: 0 – 2 mm, 1 – 4 mm and 4 – 9 mm.

The study focused on silica fly ash, which consisted of spherical, vitrified fine grains from the coal combustion process by the Ostroleka thermal power plant. The properties of silica fly ash shown in Figure 1 are presented in Table 1.



Figure 1. Silica fly ash

Table 1. The properties of silica fly ash

Tested feature	Result [%]
Silica SiO <sub>2</sub>	54.60
Alumina Al <sub>2</sub> O <sub>3</sub>	25.30
Iron Oxide Fe <sub>2</sub> O <sub>3</sub>	4.97
Potassium Oxide K <sub>2</sub> O	2.80
Calcium Oxide CaO	2.14
Magnesium Oxide MgO	1.80
Titanium Dioxide TiO <sub>2</sub>	1.07
Sodium Oxide Na <sub>2</sub> O	0.84
Phosphorus pent oxide P <sub>2</sub> O <sub>5</sub>	0.55
Sulfur trioxide SO <sub>3</sub>	0.37
Bar BaO	0.15
Strontium oxide SrO	0.07
Manganese oxide Mn <sub>3</sub> O <sub>4</sub>	0.06
Loss on ignition	4.37
<b>Total</b>	<b>99.09</b>

The test used an artificial aggregate of the type Certyd as a substitute for natural aggregate shown in Figure 2. Certyd manufactured uses a new technology based on sintering the ashes produced in the process of burning hard coal. This method produces a lightweight, porous ceramic aggregate characterised by high thermal insulation and significant resistance to weathering, chemicals, as well as mould, insects and rodents, the basic properties of which are presented in Table 2. Properties Certyd include it also lacks odor, has a high resistance to crushing and relatively low absorption. In terms of oxide composition, it contains significant amounts of silicon oxide and aluminium. These are oxides that have the ability to react to polymerise with alkalis, such as sodium hydroxide. This, in turn can affect the properties and strength of the concrete in which these aggregates are used.



Figure 2. Artificial aggregate of the Certyd type, fractions 0-2 mm, 1-4 mm and 4-9 mm

Table 2. The basic properties of Certyd

Technical parameters	0/2 mm	1/4 mm	4/9 mm
Bulk density [kg/m <sup>3</sup> ]	900 ± 10%	900 ± 10%	700 ± 10%
Thermal conductivity [W/m·K]	0.18	0.16	0.14
Chloride content [%]	0.00 0.25 0.32	0.00	0.00
Sulfate content soluble in acid [%]		0.25	0.25
Total sulfur content in terms of S [%]		0.32	0.32
Alkali-reactivity (rapid method)	0.00	0.00	0.00
Radioactivity [Bq/kg]	f1 < 1.2 f2 < 240	f1 < 1.2 f2 < 240	f1 < 1.2 f2 < 240

The alkaline activation of lightweight concretes based on waste energy materials used a sodium hydroxide solution, which was referred to as the alkaline solution or activator and shown in Figure 3. The alkaline solution was prepared by dissolving OLTCHIM's solid sodium hydroxide flakes in dis-



tilled water, which caused an exothermic reaction. To cool the solution to room temperature, it was left to stand for 24 hours, after which it was mixed with sodium silicate solution. The mass ratio of the sodium silicate solution to the sodium hydroxide solution ( $\text{Na}_2\text{SiO}_2/\text{NaOH}$ ) was kept constant at 2.5 and presented in Table 3. The alkaline solution was the activator and included ANSER's R-145 water glass solution. This process yielded a suitable activator, which was used to geopolymer lightweight concrete based on waste energy raw materials alkaline activate.



Figure 3. The alkaline solution in the range of 2-6 mol/dm<sup>3</sup>

Table 3. The mass ratio of the sodium silicate solution to the sodium hydroxide solution

Concentration of NaOH solution [mol/dm <sup>3</sup> ]	2	4	6	8	10
The weight of NaOH in 1 kg of solution. [g]	80	140	239	260	314
Mass of water in 1 kg of solution [g]	920	860	761	740	686

## Composition of geopolymer mixtures

A mixture was prepared geopolymer, which uses waste raw materials in the form of silica dust and aggregates aftermarket. Activation of the mixture was carried out alkaline, using an alkaline solution. The mixture preparation process was carried out according to the experimental plan, which included two stages. In the first, no surface impregnation was used for the aggregate aftermarket. In the second, in each trial series, it was decided to uniformly impregnate the aggregate aftermarket with a fraction of 4-9 mm with an alkaline solution.

Before starting the preparation of the mixture, the correct amount of each ingredient was carefully measured. In the second stage, surface impregnation with an alkaline solution of appropriate concentration was applied to the aggregate of 4-9 mm fraction. The impregnation procedure lasted 10 seconds, after which the aggregate was sieved to get rid of any excess solution. The aggregate was then weighed.

In the beginning, the dry ingredients were poured into the mixer drum and mixed for 60 seconds. The mixer was then stopped, and the ingredients were mixed by hand. After restarting the mixer, the ingredients were mixed for another 60 seconds, after which an alkaline solution of the appropriate concentration was added to the drum, and the entire mixture was mixed for another 60 seconds. The mixing process was stopped a fourth time to manually detach the ingredients from the walls of the mixer drum, after which mixing was resumed again for 60 seconds. The last step was repeated twice. The total mixing time was 5 minutes.

Then, into the mixer drum, pour in cement and aggregate with fractions of 0-2 mm and 1-4 mm. All ingredients were mixed for 60 seconds, after which the mixing machine was stopped, and the components were mixed by hand. The mixing process was repeated, which lasted another 60 seconds. Next, an impregnated aggregate of 4-9 mm fraction and an alkaline solution of appropriate concentration were added, and the entire contents were mixed for 60 seconds.

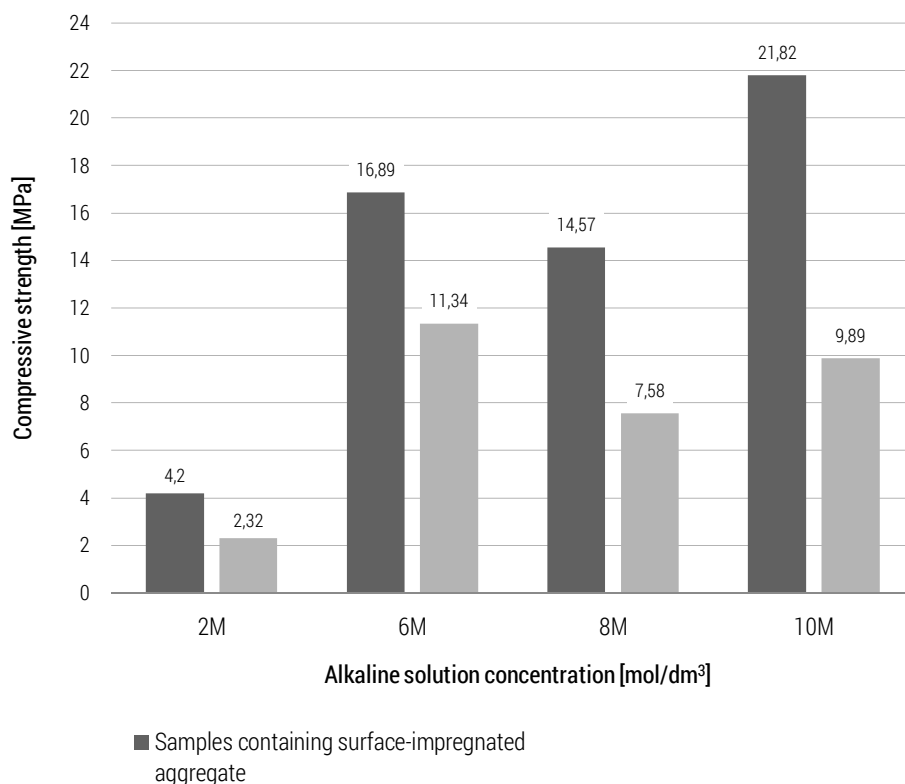
This operation was repeated three times, with breaks for manually separating the ingredients from the walls of the mixer drum. The entire mixing process took a total of 5 minutes.

The finished mixture was placed in 10 x 10 x 10 cm steel molds, conforming to EN 12390-1. Before placing the mixture in the moulds, it was covered with grease to prevent the mixture from adhering. Then, the moulds were compacted with vibropressing for 30 seconds and vibro-vibro-pressing for over 30 seconds. After the moulding process was completed, the samples were left to air-dry for 24 hours. They were then transferred to a dryer, heated to 60°C, for another 24 hours.

After this time, the samples were unmolded and placed over water for 28 days. After this period, they were subjected to analyses in terms of compressive strength, absorbability, apparent density and capillary rise.

## Research results

After 24 hours, the samples were stripped and placed above the water for 28 days. After this period, they were tested for compressive strength and apparent density.



**Figure 4.** Compressive strength of lightweight concrete based on artificial aggregate aftermarket surface-impregnated and, non-impregnated and alkali-activated fly ash

Samples of lightweight concrete based on artificial aggregate aftermarket, which was surface-impregnated and fly ash alkali-activated with an alkaline solution of 2 mol/dm<sup>3</sup> obtained compressive strength results almost twice as high, amounting to from 2.80 MPa – 5,73 MPa. The average compressive strength was 4.20 MPa. For samples alkaline activated with an alkaline solution of 6 mol/dm<sup>3</sup>. Compressive strength ranged from 16.25 MPa – to 18,00 MPa. The average compressive strength was 16.89 MPa. In contrast, samples activated alkaline with an alkaline solution of 8 mol/dm<sup>3</sup> obtained

compressive strength results of approximately 14.20 MPa – 14,85 MPa. The average compressive strength is 14.57 MPa. For alkaline activation with an alkaline solution of 10 mol/dm<sup>3</sup> specimens had compressive strength results ranging from 20.90 MPa – to 24,20 MPa. The average compressive strength was 21.82 MPa. Figure 4 shows the comparison of compressive strength of lightweight concrete based on artificial aggregate aftermarket surface-impregnated and non-impregnated, and based on Alkali-activated fly ash. Basic statistical calculations were performed and showed the results as in Table 4.

**Table 4.** Basic statistical calculations for compressive strength tests

Serial number	Series	Standard deviation [MPa]	Diversity (coefficient of variation) [%]
1	2M SIA	0.1960	8.46
2	2M NSIA	0.6655	3.01
3	6M SIA	0.2083	1.84
4	6M NSIA	0.6430	3.81
5	8M SIA	0.2135	2.82
6	8M NSIA	0.2182	1.50
7	10M SIA	0.5731	5.79
8	10M NSIA	1.4579	6.68

SIA – surface-impregnated aggregate,  
NSIA – not surface-impregnated aggregate.

Among all samples, the lowest level of differentiation is characterised by series 6, containing the 8M activator and Certyd coarse aggregate, which was previously surface-impregnated. This means that these samples are the best, giving the least differentiated results.

In the next step of the analysis, the apparent density of lightweight concrete samples based on raw waste materials was measured, activated with an alkaline solution of 6, 8 and 10 mol/dm<sup>3</sup>, containing surface-impregnated coarse aggregate. Samples of lightweight concrete using artificial aggregate aftermarket, which has been surface-impregnated and fly ash activated with an alkaline solution of 6 mol/dm<sup>3</sup>, reached apparent density values in the range of about 1617.00 g/cm<sup>3</sup> up to 1661.50 g/cm<sup>3</sup>. The average apparent density was 1649.70 g/cm<sup>3</sup>. Samples activated with an alkaline solution of 8 mol/dm<sup>3</sup> with non-impregnated aggregate showed apparent density results ranging from 1645.00 g/cm<sup>3</sup> up to 1663.00 g/cm<sup>3</sup> and the average apparent density was 1652.50 g/cm<sup>3</sup>. On the other hand, samples activated with the

alkaline solution with a concentration of  $10 \text{ mol/dm}^3$  with non-impregnated coarse aggregate reached apparent density values in the range of  $1652.00 \text{ g/cm}^3$  up to  $1669.00 \text{ g/cm}^3$ , with an average apparent density of  $1663.80 \text{ g/cm}^3$ . Figure 5 shows a comparison of the apparent density of lightweight concrete, taking into account the differences between samples based on artificial ash-porite aggregate that has been impregnated and not surface-impregnated.

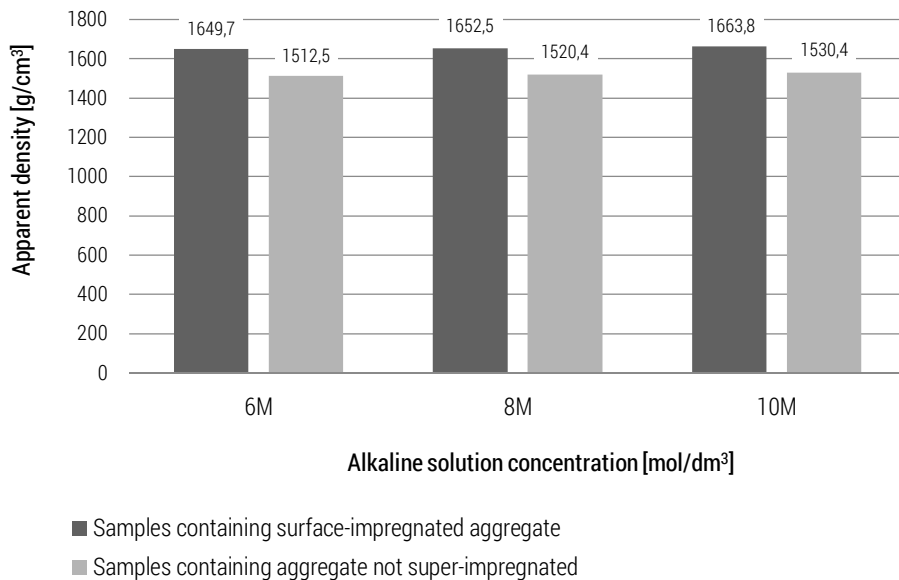


Figure 5. Apparent density of lightweight concrete based on fly ash and artificial aggregate aftermarket surface-impregnated and non-impregnated alkaline-activated

## Conclusions

As a result of the study, confirmation was obtained theses that there is a real possibility of producing lightweight concretes with the expected properties based on alkali-activated waste materials from the energy sector. This kind of innovative approach is a step towards solving the problem of environmental degradation. It allows the implementation of sustainable construction processes.

The analyses carried out clearly prove that lightweight concretes produced from alkali-activated waste materials from the energy sector can be a valuable alternative to traditional lightweight concretes used in construction processes. The study focused on selecting the optimal concentration of an alkaline solution that would allow a coherent combination of fly ash and artificial aggregate while taking into account economic aspects.

As part of the experiments, samples were prepared with various mixtures, including coarse aggregate treated with an activator impregnation process and samples based on non-surface-impregnated aggregate. The analysis of the results of the experiments, the aim of which was to investigate the influence of activator concentration on the properties of geopolymer concrete, clearly showed that surface impregnation of coarse aggregate with an activator results in almost a twofold increase in compressive strength, as well as a slight increase in apparent density.

In light of these results, further research was conducted using coarse aggregate derived from fly ash, which was subjected to a surface impregnation process. This is an important step toward developing greener and more efficient solutions in manufacturing construction materials.

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