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Development of lightweight geopolymer composites containing perlite and vermiculite

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ABSTRACT

Purpose: The aim of this work was to prepare and characterise geopolymer composites containing lightweight aggregates - perlite and vermiculite.

Design/methodology/approach: The geopolymer matrix was prepared on the basis of fly ash, sand and a 6M sodium hydroxide solution with sodium silicate. The properties of the materials were tested 28 days after the preparation of the samples. The following research methods were used to characterise the composites: compressive and flexural strength tests, microstructural tests using a scanning electron microscope, and thermal conductivity were measured.

Findings: The results obtained showed a slight effect of the additives on the strength properties. Lightweight aggregates are characterised by good coherence with the matrix material. Their addition allowed to reduce the density and lowered the thermal conductivity of the materials. The results obtained indicate that the proposed additives can improve the properties of the geopolymer composite for use in the construction industry.

Research limitations/implications: Further research should focus on geopolymer composites with perlite and involve fire-resistant and water-absorption tests.

Practical implications: The production of lightweight building materials brings a number of benefits, such as reducing the density of building elements and, at the same time, the entire structure, which results in a reduction in their weight, as well as lower transport costs. Such elements have better thermal and acoustic insulation, reflected in the parameters of buildings. An additional advantage is the reduced environmental impact through better insulation properties, lower fuel consumption during transport, etc.

Originality/value: The density of the material can be reduced by using lightweight aggregates or obtaining porous material in the foamed process. In the case of geopolymer composites, a number of studies related to foamed materials have been provided, but there is only a few previous research connected with lightweight aggregates such as perlite and vermiculite.

Keywords: Lightweight material, Composite, Geopolymer, Perlite, Vermiculite

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MATERIALS

1. Introduction

Modern civil engineering requires innovative solutions in the area of technology and materials [1,2]. One of the most important directions of innovation development in modern civil engineering is advanced materials, especially lightweight products [2,3]. The use of lightweight building materials in the construction industry brings a number of benefits, including reducing the density of building elements and, at the same time, the entire structure, which results in a reduction in their weight, as well as lower transport costs [3,4]. These elements also have better thermal and acoustic insulation, which becomes the parameters of buildings [5]. Additional requirements of the construction market are environmental benefits that should be associated with using these types of products, particularly a lower carbon footprint compared to traditional building materials [6,7]. The exemplary materials that meet these requirements are lightweight geopolymer composites. In this case, the density of the material can be reduced by using foamed materials or lightweight aggregates. In the case of the geopolymer composite, several investigations related to foamed materials have been provided [8,9], but there is only a few previous research connected with lightweight aggregates [10,11].

Building materials such as concrete or geopolymers are considered lightweight when their density is in the range of 800-2000 kg/m^3; below this weight they are considered ultralightweight [12,13].To achieve this value and, at the same time, have reasonable mechanical properties, different kinds of additives are applied as aggregates to geopolymer paste, such as:

- Industrial waste or by-products, for example, microspheres [13,14],
- Industrial product - artificial aggregates, such as foam glass granules, expanded polystyrene (polystyrene), and others [14-16],
- Natural minerals, including pumice, vermiculite, perlite, expanded clay, etc. [14,17],
- Other lightweight natural components include ground walnut shells, rice husks, or wood products [18,19].

Among these groups, natural minerals seem to be one of the most suitable for geopolymers because of their chemical composition. Examples of aggregates from this group are perlite and vermiculite. Perlite is a naturally occurring volcanic stone based on aluminosilicates (ca. 75%SiO_2 and 15% Al_2O_3), similar to geopolymers [17,20]. It was investigated as raw materials for the geopolymerization process as well as an aggregate, particularly in expanded form for lightweight materials [11,17,20]. Different proportions of perlite have been applied as a lightweight aggregate up to 95% and show that the synthesis of geopolymers incorporating perlite is possible; however, further investigations to optimise the properties of the material [17,21]. In the case of vermiculite, only a few researches have been provided [22-24]. Vermiculite is also a natural mineral with a chemical composition more complex than perlite, except for a relatively large amount of silica and alumina - approximately 40 and 10%, respectively. It also includes magnesium and ferrum compounds (ca. 25% MgO and 10% Fe_2O_3) [22]. However, the particular composition can depend on the place of mineral exploitation.

This work aimed to prepare and characterize geopolymer composites containing lightweight aggregates - perlite and vermiculite. The geopolymer matrix was prepared on the basis of fly ash, sand, and a 6M sodium hydroxide solution with sodium silicate. The properties of the materials were tested 28 days after the preparation of the samples. The research program involves physical properties, mechanical strength tests, microstructural investigation and thermal conductivity.

2. Materials and methods

2.1. Materials

As a raw material for geopolymer production, class F fly ash from Skawina Heat and Power Plant. Previous research shows it has a suitable chemical composition and physical properties for synthesis [6,13].

As a fine aggregate, the sand river was applied. To replace the traditional aggregate, two lightweight aggregates were applied:

- expanded perlite type 180, grain dimensions 0-4 mm (white granulates);
- vermiculite, 2-4 mm (goldish granulates).

The lightweight aggregates were subjected to scanning microscopy observations. The investigation shows different internal structures for these materials. Perlite is the form of spheres covered by an irregular corrugated surface (Fig. 1). Each grain includes small voids, which are pores within the material and are irregular. The structure of vermiculate is more ordered. The layers created by the materials are visible (Fig. 2). The voids inside the materials are arranged in one direction.

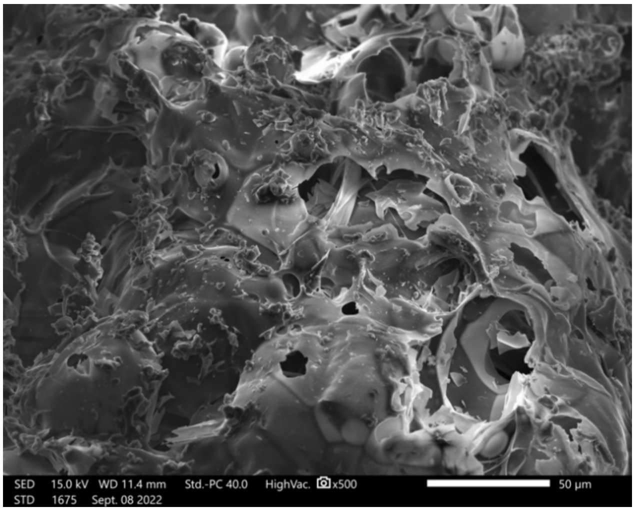

Fig. 1. SEM image of perlite's microstructure

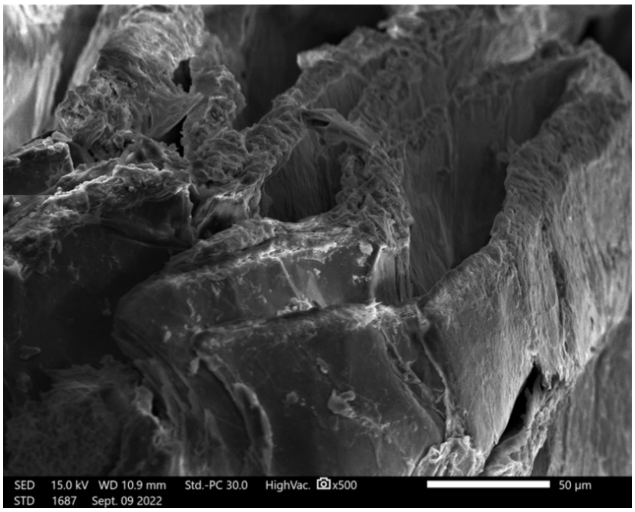

Fig. 2. SEM image of vermiculite's microstructure

2.2. Samples preparation

In the first step, the fly ash was mixed with quartz sand (ratio of 1:1). In the next step, lightweight aggregate was added in amounts of 30% by volume. Two types of composites were created with expanded perlite and vermiculite. Then, a sodium activator – 6 M NaOH solution was added and mixed in a slow-speed mixer to obtain the homogeneous paste. The sodium activator includes technical sodium hydroxide flakes, aqueous sodium silicate solution, and tap water. The prepared compositions were placed in a set of moulds. Three types of moulds were used:

- Flexural strength with dimensions 50 mm × 50 mm × 200 mm (prismatic moulds);
- To compressive strength with dimensions 50 mm × 50 mm × 50 mm (cubic moulds);
- For the thermal conductivity tests with dimensions, 200 × 200 × 25 mm were made (panels-plates).

Subsequently, the vibrating table was used to remove air bubbles from the moulded samples. Finally, the samples were placed at 75°C in a laboratory oven. Each mould set was wrapped in a polyethylene film to avoid too rapid water loss. After 24 h, the samples were unmoulded and stored under laboratory conditions for 28 days – Figure 3.

Fig. 3. Sample preparation: basic steps

2.3. Methods

The density was calculated using a geometric method (samples weight and dimensions of the samples). It was determined on a series of samples dedicated before the compressive strength test. The samples have dimensions of 50 × 50 × 50 mm.

The compressive strength test was provided according to standard: PN-EN 12390-3:2019-07 on samples with dimensions of 50 × 50 × 50 mm. The flexural strength test was performed according to the following standard: PN-EN 12390-5:2019-08) in samples with dimensions 50 mm × 50 mm × 200 mm and the distance between the supports; 150 mm (three-point bending test). Both tests were made on the samples after 28 days using the MATEST 3000 kN test machine. The speed employed in both tests was 0.05 MPa/s. The standard deviation was calculated for all results obtained from mechanical properties.

Microstructural studies were performed with the JEOL JSM-IT200 scanning electron microscope (SEM). Additionally, an energy-dispersive X-ray spectroscopy (EDS) system was applied to analyse the oxide composition for composites. For the research, the material mechanical properties tests were used. Before SEM observation, the samples were coated with gold-plated for good conductivity.

The thermal conductivity was investigated on the NETZSCH HFM 446 machine, for test samples with dimensions 200 × 200 × 25 mm were used.

3. Results and discussion

3.1. Physical properties

The results obtained for the density calculations are presented in Table 1.

Table 1.
Density of the geopolymer composites

No	Sample	Density, kg/m^3
1	Geopolymer with vermiculite	1.824
2	Geopolymer with perlite	790
3	Geopolymer – reference sample	1.815

Both composites have a typical density for lightweight composites dedicated to the building industry [12,13]. The density of the materials obtained is strictly related to the amount of lightweight aggregates. Compared to the data, the obtained values for the expanded perlite were approximately 500 kg/m^3, but with the use of a larger amount of expanded perlite incorporated into the composite [20,21]. The densities obtained for the vermiculate geopolymers were in the range between 700 and 900 kg/m^3 for 20% weight of lightweight aggregate [23]. However, investigations have also been made for this type of material with a density of approximately 1500 kg/m^3 [24].

Moreover, visual observations were made on the samples after the flexural strength test. It confirms the regular distribution of lightweight aggregates in the volume of the geopolymer matrix (Figs 4 and 5).

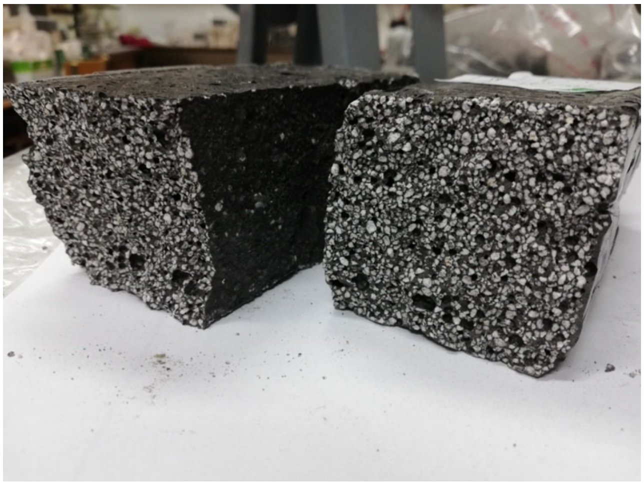

Fig. 4. The appearance of the geopolymer composite with perlite

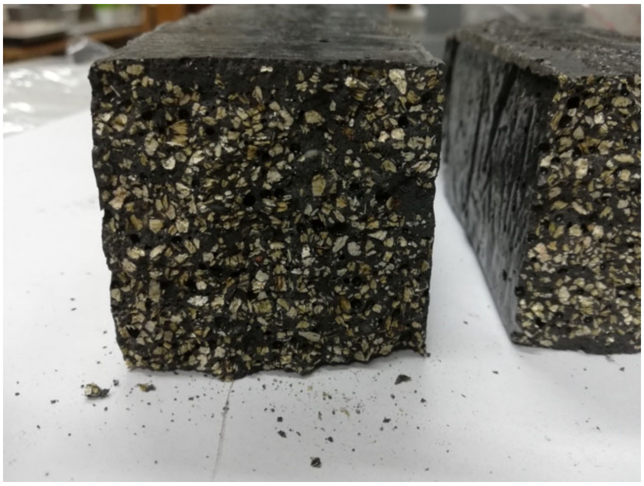

Fig. 5. The appearance of the geopolymer composite with vermiculite

Regular distribution is important because potentially lightweight aggregates or fillers could tend to agglomerate near the surface. It could influence mechanical properties.

3.2. Mechanical properties

The results of the mechanical tests are presented in Table 2.

Table 2.
Mechanical properties of the geopolymer composites

No	Sample	Flexural strength, MPa	Compressive strength, MPa
1	Geopolymer with vermiculite	0.11 (±0.03)	3.75 (±1.25)
2	Geopolymer with perlite	---	2.30 (±0.36)
3	Geopolymer – reference sample	5.90 (±0.70)	42.75 (±6.90)

The flexural strength test for the geopolymer with perlite obtained a value below the machine measurement scale. Overall, better results for mechanical properties were obtained for the geopolymer with vermiculite. It is consistent with the relationship from the literature that higher-density composites have better mechanical properties [10,13]. Compared to the other investigations carried out for perlite geopolymers, these results are in line with research in the literature that shows low mechanical values, even in the range of 246 and 264 kPa, for this material with a perlite content of 80 and 95% expanded by volume [21]. For geopolymers with vermiculite provided, research also finds confirmation in other publications. For this kind of composite with a density ranging between 906 and 1477 kg/m^3, the compressive strength has values between 0.59 MPa and 3.81 MPa and flexural strength was between 0.30 MPa and 1.31 MPa [24]. It is a slightly better result than that obtained for the research provided, taking into consideration density and mechanical properties.

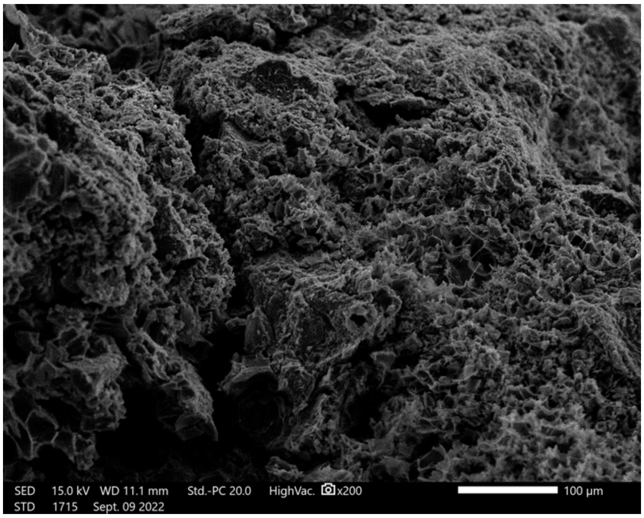

Fig. 6. SEM picture of geopolymer composite with perlite

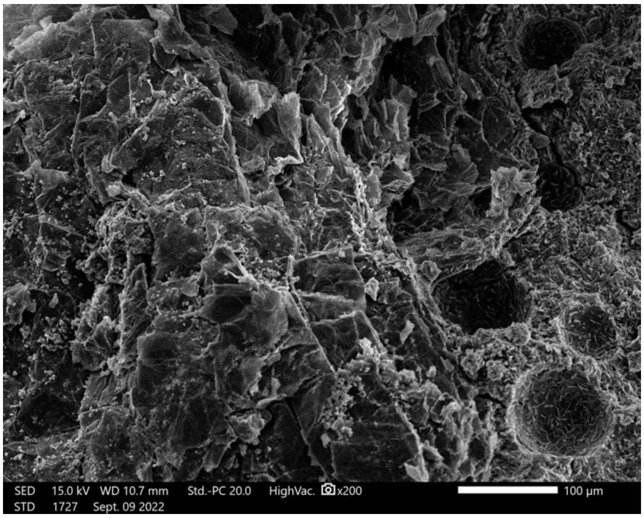

Fig. 7. SEM picture of geopolymer composite with vermiculite

3.3. Microstructure investigation

The important element of the research was the microstructure investigation provided to confirm the coherence of lightweight aggregates with the matrix and to obtain additional information on the chemical compositions. SEM observations were made in the magnification range between 100 x and 5,000x. The exemplary pictures are presented in Figures 6 and 7. The figures show a typical structure for geopolymer material [13,19]. The lightweight aggregates become part of the structure and part of the composition.

Furthermore, for selected points, the EDS analysis was performed. The results obtained are presented in Figures 8 and 9, where the measurement point is marked. A detailed description of the values obtained is presented in Tables 3 and 4, respectively. EDS measurements confirm the occurrence of the elements typical for geopolymer structure, especially silica (SiO_2) and alumina (Al_2O_3) oxides [13,19]. Furthermore, for both samples, a certain amount of Na_2O is visible. It is an effect of the alkali activation process in which sodium compounds were used.

The main difference between these two compositions is the amount of MgO and FeO. It is also worth noting that EDS does not difference between FeO and other Fe oxides, such as Fe_2O_3, because it cannot be pointed at the exact form of Fe that occurs in the composition. The small amount of

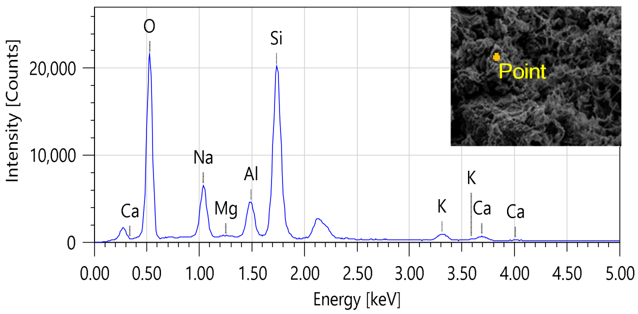

Fig. 8. EDS analyses provided for the geopolymer composite with perlite

Table 3.
Oxide composition based on EDS: geopolymer with perlite

No	Chemical formula	Mass%	Mol%
1.	Na_2O	13.88±0.13	14.30±0.14
2.	MgO	0.42±0.04	0.67±0.06
3.	Al_2O_3	11.62±0.14	7.27±0.09
4.	SiO_2	68.53±0.36	72.82±0.38
5.	K_2O	2.99±0.07	2.03±0.05
6.	CaO	2.56±0.08	2.91±0.09
7.	Total	100.00	100.00

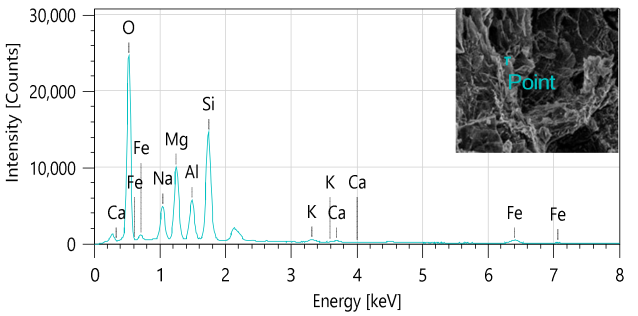

Fig. 9. EDS analysis provided for the geopolymer composite with vermiculite

Table 4.
Oxide composition based on EDS: geopolymer with vermiculite

No	Chemical formula	Mass%	Mol%
1.	Na_2O	9.11±0.10	8.60±0.10
2.	MgO	21.01±0.16	30.47±0.23
3.	Al_2O_3	14.27±0.15	8.18±0.09
4.	SiO_2	47.11±0.29	45.84±0.28
5.	K_2O	1.23±0.04	0.76±0.03
6.	CaO	1.00±0.05	1.04±0.05
7.	FeO	6.27±0.17	5.11±0.14
8.	Total	100.00	100.00

these elements in the geopolymer composite with perlite could come from fly ash. The relatively large amount of these compounds in the geopolymers with vermiculite is related to the chemical composition of vermiculite, that includes relatively large amount of these oxides.

Other compounds, such as K_2O and CaO, occur in small amount and comes from fly ash.

3.4. Thermal properties

The main results of the thermal properties are presented in Table 5.

Table 5.
Thermal properties of the geopolymer composites

No	Sample	Thermal conductivity, W/(m·K)	Thermal resistance, $(m^2{\cdot}K)/W$
1.	Geopolymer with vermiculite	0.95915	0.0362
2.	Geopolymer with perlite	0.35832	0.1007

The thermal conductivity is much better for geopolymer composites with perlite than for vermiculite, but the obtained values are below the expectations when the values of mechanical properties. The best value for the geopolymer with perlite was 0.084 W/(m·K), but in this case, also the mechanical properties were much lower – compressive strength of about 250 kPa [20]. In the case of geopolymers with vermiculite, in the literature, better values were obtained for the 20% weight addition of lightweight materials, 0.2 W/mK [23].

4. Conclusions

The developed composite has the potential for application in the construction industry. The results obtained showed the expected effect of additives on the density and strength properties (reduction compared to the matrix material). Both lightweight aggregates are characterized by good coherence with the matrix material. Adding perlite reduced the density and lowered the thermal conductivity of the material; in the case of vermiculite, the expected results were not obtained, in particular, a relatively high value for the thermal conductivity. The main reason for this behaviour can be the internal structure of a lightweight aggregate that is characterised by some directional voids. The practical application for these compositions required further research, including optimisation of the geopolymer compositions with perlite, and involved fire-resistant and water-absorption tests.

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Additional information

The topic was presented at a conference entitled: ‘Inżynieria Materiałowa - Materiały i Technologie’, Lodz, Poland, 20-23 November 2022.

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