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Research paper

Application of the non-stationary method of thermal diffusivity measurement for the evaluation of the thermal conductivity coefficient of geopolymer mortars

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Abstract: This article presents a study on the thermal properties of geopolymer mortars, which are novel materials with lower environmental impact and higher performance than Portland cement. Geopolymers are formed by the reaction of aluminosilicate sources and alkali activators, resulting in a polymeric Si-O-Al network. The study used the non-stationary method of measuring thermal diffusivity to evaluate the heat transfer coefficient of geopolymer mortars modified with perlite powder, cenospheres and perlite sand, which are porous components that affect the bulk density and thermal conductivity of the composite. The study had two specific objectives: to assess the significance of the factors related to the composition and bulk density on the thermal conductivity coefficient and to test the suitability of the non-stationary method for geopolymers. The study found that all the composites met the RILEM standards for a Class II composite, with a thermal conductivity coefficient below 0.75 W/(m·K). The most influential factor was the dosage of perlite sand, which reduced the composite density and decreased the thermal conductivity. The study concluded that geopolymer composites modified with low-density grain additives are a promising thermal insulation material, but they need more research on their durability.

Keywords: geopolymer mortar, non-stationary method, perlite additive, thermal coefficient

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1. Introduction

To achieve the goal of sustainable development, various kinds of environmental engineering are needed more than ever due to the impact of civilization's progress [1, 2]. Therefore, geopolymers as a novel class of materials, with low carbon footprint, have gained considerable interest in recent years for their potential applications. This is particularly important in the context of sustainable development and limitations of CO_2 emissions [3, 4]. Geopolymers are produced by the reaction of aluminosilicate sources and alkali activators. This reaction involves the dissolution and polycondensation of the aluminosilicate components at ambient or slightly elevated temperatures, resulting in the formation of a polymeric Si-O-Al network with SiO_4 and AlO_4 tetrahedra linked by shared oxygen atoms. Geopolymers could be used as binder for mortars and concretes [5], as they offer several advantages over conventional Portland cement, such as lower carbon footprint, higher durability, and better resistance to aggressive environments [6]. Geopolymers also exhibit high resistance to high temperatures, which makes them suitable for applications in thermal insulation, thermal energy storage, air filtration, ceramic production and composites [7]. Geopolymers have more interconnected pores than Portland cement, which allows for faster release of water vapor and lower internal pressure when heated [8]. The properties of geopolymers depend on various factors, such as the type and proportion of aluminosilicate source, the type, concentration and ratio of alkali activator, and the curing condition [6, 9]. The listed factors affect the course of the geopolymerization process, and thus the structure of the composite [10], and consequently also its thermal conductivity [11].

1.1. Geopolymers thermal conductivity

Geopolymers are a new type of materials that have been explored for their potential use in thermal insulation of buildings. A low thermal conductivity (λ) is a key parameter that indicates the thermal insulating [12]. Geopolymers generally have low λ (< 0.70 W/(m $\cdot$ K)) [13], which is about 50% lower than that of Portland cement (PC) materials [14]. The thermal insulating performance of geopolymers can be further enhanced by introducing pores (chemical creation [15, 16] or mechanical action [17]) or voids in the geopolymer matrix.

The thermal conductivity of geopolymer composites plays a crucial role in determining their performance and suitability for various applications. Numerous factors contribute to the thermal conductivity of these composites, including their composition, processing techniques, and inherent characteristics. Research focusing on composition delves into key aspects such as the type and quantity of filler used, the specific geopolymer binder employed, as well as the incorporation of waste materials as both sources and additives [18–21]. Processing parameters significantly impact the thermal conductivity of geopolymer composites, notably the curing temperature and duration, the method of foaming, and the moisture content [7]. Furthermore, the intrinsic characteristics of the geopolymer also influence thermal conductivity. These characteristics encompass factors like porosity, density, and the mechanical strength of the composite [22, 23].

Given that foamed concrete is a material with significant thermal insulation potential [24,25], researchers have also focused on exploring the possibilities of these composites utilizing geopolymer binders. Many researches concentrate on examining geopolymer foam concrete and the outcomes are quite encouraging. Considering the geopolymer foam concrete that is most often regarded as having low thermal conductivity, it is possible to produce it with λ between 0.1 and 0.4 W/(m · K) [26]. To achieve this, a composite with a density of 500–700 kg/m^3 must be created, which is achieved with a porosity ranging from 50% to 95% [27]. Unfortunately, such high porosity results in a reduction of compressive strength, which in few studies exceeds 10 MPa [27], most often it is within 3–5 MPa [28, 29].

According to the RILEM classification [30], there are three classes of lightweight concretes based on their properties of compressive strength, density and thermal conductivity:

- Class I for structural purposes – density of 1600–2000 kg/m^3, compressive strength above 15 MPa,
- Class II for both structural and insulating purposes – density under 1600 kg/m^3, compressive strength between 3.5–15 MPa, and λ under 0.75 W/(m · K),
- Class III for insulating purposes only – density under 1450 kg/m^3, compressive strength 0.5–3.5 MPa and λ under 0.3 W/(m · K).

1.2. Aim of the study

The aim of this study was to apply the non-stationary method of measuring thermal diffusivity to evaluate the heat transfer coefficient of geopolymer mortars modified with components of significant porosity. The addition of perlite powder, cenospheres and perlite sand was analyzed. Since the literature review indicated a strong correlation between bulk density and thermal conductivity, this relationship of characteristics was also focused on. Two specific objectives were set: to assess the significance of the influence of factors related to the composition and bulk density on the thermal conductivity coefficient and to assess the usefulness of the non-stationary method for testing geopolymers. The verification assumption for the designed composite was to meet the RILEM requirements for a composite classified as Class II.

2. Materials and methods

2.1. Materials

The study examined how three additives that could influance thermal conductivity of the geopolymer composite: perlite powder (PP), perlite sand (PS) and cenospheres (C). The experiment aimed to find the optimal dosage ranges of each component that had a critical influence on thermal conductivity. The composition of 1 m^3 of analyzed mortar was based on 500 kg of precursors mix (fly ash (80%), metakaolin (10%) and zeolite (10%)), 315 kg of alkaline activators (30% sodium hydroxide and water glass), 110 kg of calcium compound and 15 of rheology modifiers. Perlite powder, perlite sand and cenospheres as per the experimental design (Table 1).

The factor analysis considered three parameters of the composite composition: the amount of perlite sand, perlite powder, and cenospheres per cubic meter of composite. Each parameter had three possible values: low, medium, and high. the lowest dosage of all components was assumed to be 0 kg. The upper levels were determined in preliminary studies and for dosing perlite dust and cenospheres, 50 kg per cubic meter was adopted, and for perlite sand, 30 kg per cubic meter was adopted.

A random fractional factorial design with three variables, each with three levels, was applied in the study. Three additional runs with variables at the middle levels were included in the experiment matrix to estimate the method error. The quantity of natural sand required to produce 1 m^3 of the mix was determined for each run. The full design, the variable levels, and the results are presented in Table 1.

Table 1. Experiment plan for composite composition with varying parameters

No.	**Components**			
	Natural sand (NS), kg	**Perlite sand (PS), kg**	**Perlite powder (PP), kg**	**Cenospheres (C), kg**
1	1100	0	0	0
2	400	30	25	0
3	443	15	50	0
4	515	30	0	25
5	560	15	25	25
6	601	0	50	25
7	670	15	0	50
8	715	0	25	50
9	17	30	50	50
C1	560	15	25	25
C2	560	15	25	25
C3	560	15	25	25

2.2. Methods

The samples were tested after 28 days of incubation. The thermal parameters of the samples were measured using a non-stationary method – Isomet 2114 device (Fig. 1). The measuring range of the device includes λ values from 0.015 to 6.0 W/(m · K) and C_v values from 0.04 to 3.00 MJ/(m^3 · K).

The method consisted in analysing the temperature changes resulting from the reaction of the tested material to the flow of thermal impulses generated by the device. Isomet 2114 emits a known amount of heat during measurement, which propagates radially throughout the sample. The results of the measurements were transferred to the (Fig. 1).

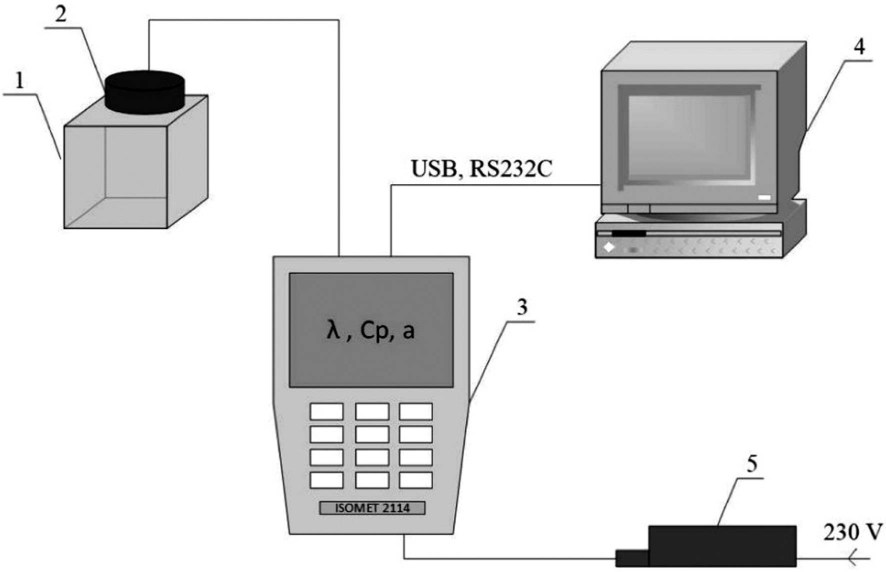

Fig. 1. Scheme of the stand for measuring the thermal properties (1 – tested sample, 2 – probe, 3 – Isomet 2114 device, 4 – computer, 5 – power supply [31, 32])

The dependence of temperature increase with logarithm of time allowed to obtain the thermal conductivity of the tested material directly [33]. Non-stationary methods often provoke polemics and criticism among some researchers, but due to the short time it takes to perform a single measurement, they are gaining more and more popularity.

According to Fourier's law, there is a linear temperature distribution as a function of the distance from the heated surface. In the case of an indeterminate condition, the test specimen is isolated on the side surface and supplied with heat uniformly released on the surface. The heat spreads along the sample, causing it to heat up. It is important to note that the temperature distribution along the sample varies depending on the time and location of the measurement. Thermal diffusivity is defined as the ratio of the thermal conductivity of a material to its volumetric heat capacity This parameter represents how well a material conducts heat relative to its ability to store thermal energy.

3. Results

Thermal properties of modified geopolymer samples were measured by a transient method based on the measurement of thermal diffusivity, which characterizes heat conduction in unsteady conditions. The thermal conductivity λ, the volumetric heat capacity C_v, and the thermal diffusivity a were measured, always performing (at least) 12 series of measurements. The samples for the study were in an air-dry state. The specific heat C_p, expressed in units of J/(kg · K), was calculated as the quotient of the measured volumetric heat capacity C_v and the material density ρ (Table 2).

Table 2. The experiment response

No.	λ **W/(m·K)**	C_v **MJ/(m^3· K)**	C_p **J/(kg·K)**	a **mm^2/s**	ρ **kg/m^3**
1	0.93 ± 0.04	1.74 ± 0.07	989 ± 39	0.53 ± 0.02	1763 ± 16
2	0.47 ± 0.02	1.61 ± 0.01	1240 ± 5	0.29 ± 0.01	1301 ± 5
3	0.48 ± 0.02	1.63 ± 0.01	1160 ± 9	0.30 ± 0.01	1403 ± 14
4	0.49 ± 0.02	1.64 ± 0.01	1210 ± 9	0.30 ± 0.01	1354 ± 8
5	0.53 ± 0.01	1.65 ± 0.00	1132 ± 3	0.32 ± 0.01	1454 ± 20
6	0.47 ± 0.01	1.59 ± 0.01	1001 ± 7	0.29 ± 0.01	1584 ± 6
7	0.63 ± 0.01	1.63 ± 0.01	1129 ± 5	0.39 ± 0.00	1442 ± 17
8	0.58 ± 0.02	1.61 ± 0.02	1058 ± 11	0.36 ± 0.01	1518 ± 4
9	0.24 ± 0.00	1.52 ± 0.07	1331 ± 60	0.16 ± 0.01	1145 ± 19
C1	0.47 ± 0.01	1.56 ± 0.04	1201 ± 29	0.30 ± 0.01	1455 ± 10
C2	0.51 ± 0.02	1.61 ± 0.04	1138 ± 30	0.32 ± 0.01	1412 ± 10
C3	0.45 ± 0.02	2.37 ± 0.08	1170 ± 30	0.31 ± 0.12	1441 ± 8

All of the experimental compositions reached the critical value of thermal conductivity. The results obtained were subjected to a significance analysis of the independent variables. Pareto analysis was selected for the thermal conductivity λ (Fig. 2) and the specific heat C_p (Fig. 3). The analysis results show that the addition of perlite sand (PS), perlite powder (PP) and cenospheres (C) has a significant impact on the thermal conductivity and the specific heat of the composite.

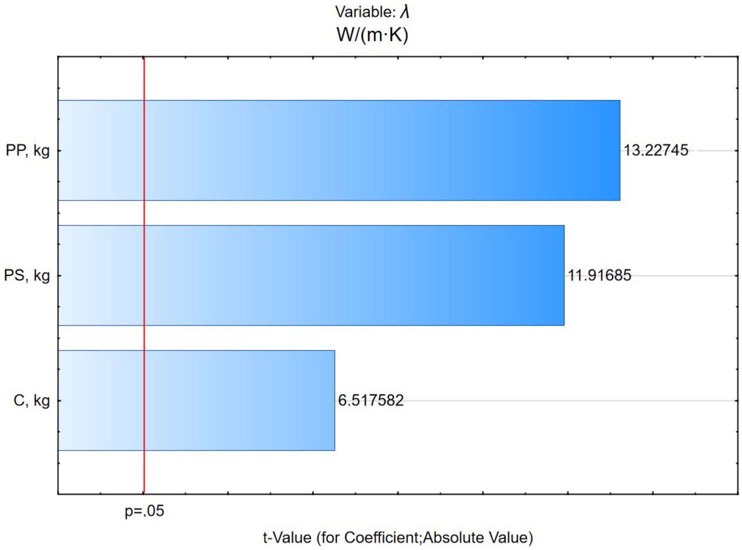

Fig. 2. Pareto analysis of thermal conductivity

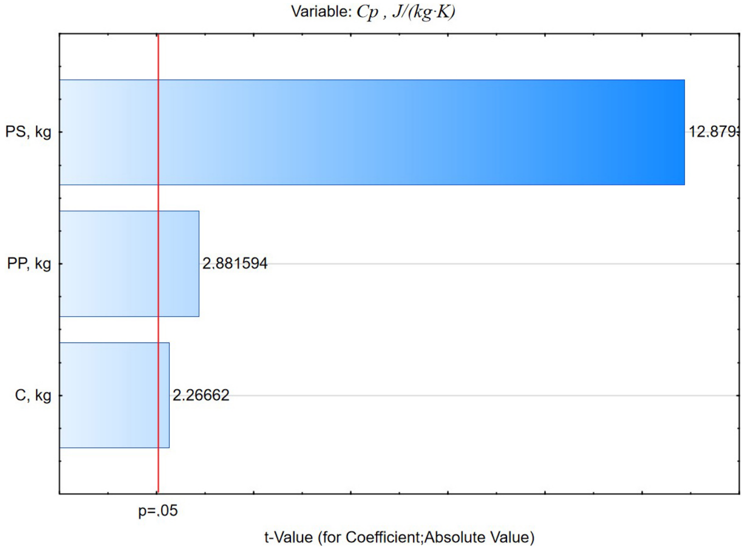

Fig. 3. Pareto analysis of specific heat

As the perlite sand content increases, the composite's thermal conductivity decreases significantly, which is related to the high porosity of the additive. The thermal conductivity is less affected by the cenospheres (Fig. 4) and perlite powder content (Fig. 5).

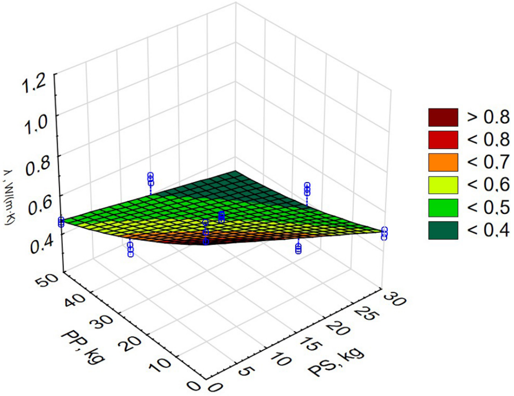

Fig. 4. Thermal conductivity depending on perlite sand and perlite powder content

Figures 6 and 7 show the graphs of all the thermal properties measured in the samples as functions of their density. It was observed that, as the geopolymer density reduced, the thermal conductivity λ also lowered (Fig. 6). Simultaneously, as the density of the modified geopolymers samples rose, the specific heat C_p values (Fig. 6) dropped. The linear regression of λ with ρ indicates a R^2 of 0.89 (Fig. 6), implying that the thermal conductivity is dependent

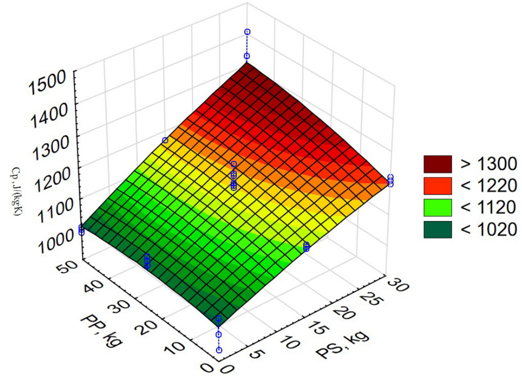

Fig. 5. Specific heat depending on perlite sand and perlite powder content

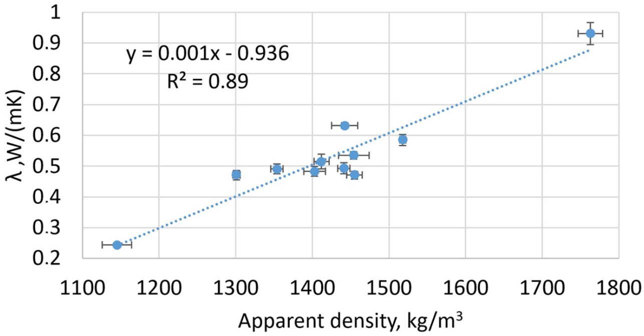

Fig. 6. Graph of the relationship between $\lambda = f(\rho)$ of modified geopolymers

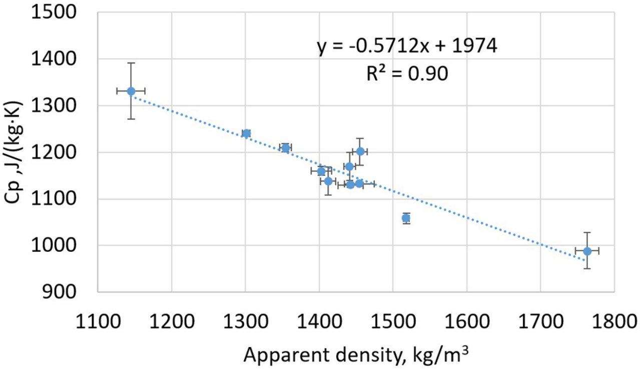

Fig. 7. Graph of the relationship between $C_p = f(\rho)$ of modified geopolymers

on the density of the geopolymers. Furthermore, C_p (Fig. 7) is also strongly associated with the density with R^2 of 0.90. For low densities, the porosity of the geopolymers increases, and consequently λ and C_p decrease considerably.

4. Conclusions

This study aimed to use the non-stationary method of measuring thermal diffusivity to evaluate the heat transfer coefficient of geopolymer mortars modified with porous components. The effects of adding perlite powder, cenospheres and perlite sand were analysed. The literature review suggested a strong correlation between bulk density and thermal conductivity, so this characteristic relationship was also examined. The specific objectives were: to evaluate the significance of the factors related to the composition and bulk density on the thermal conductivity coefficient and to test the applicability of the non-stationary method for geopolymers. The non-stationary method, proved to be effective in investigating the analyzed composites as it allowed for obtaining averaged results independent of the direction of geopolymer formation. The uniformity of the results regardless of the direction tested indicated the homogeneity of the composite and the absence of significant segregation of components, despite the risk associated with the use of modifiers with low volume densities. Further studies were conducted using the stationary method under stabilized conditions, yielding convergent trends in the research outcomes.

The verification criterion for the composite meet the RILEM standards for a Class II composite. The composite achieved a thermal conductivity coefficient below 0.75 $\mathrm{W/(m \cdot K)}$, irrespective of the expected modification. The results indicate that the most statistically significant factor is the dosage of perlite sand, which has the largest grains. The more perlite sand, the lower the composite density, which has a key influence on the thermal conductivity and specific heat. The dynamic measurement method works well for geopolymer composites and allows for the assessment of the key thermal parameters. In conclusion, geopolymer composites modified with low-density grain additives, especially perlite sand, are a promising thermal insulation material. Considering the provision of sufficient compressive strength [34] and workability, the analysed geopolymer composites can be used as mortars with considerable thermal conductivity. However, they require further research on durability.

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Zastosowanie niestacjonarnej metody pomiaru dyfuzyjności cieplnej do oceny współczynnika przewodzenia ciepła zapraw geopolimerowych

Słowa kluczowe: dodatki perlitowe, metoda niestacjonarna, współczynnik przewodzenia ciepła, zaprawa geopolimerowa

Streszczenie:

Geopolimery to nowa grupa materiałów, która wzbudza coraz większe zainteresowanie w ostatnich latach ze względu na ich potencjalne zastosowania w różnych dziedzinach. Jest to szczególnie ważne w kontekście zrównoważonego rozwoju i ograniczenia emisji CO_2. Geopolimery wykazują wysoką odporność na wysokie temperatury, co sprawia, że nadają się do zastosowań w izolacji termicznej, magazynowaniu energii cieplnej, filtracji powietrza, produkcji ceramiki i kompozytów. Właściwości geopolimerów zależą od różnych czynników, takich jak rodzaj i proporcja źródła aluminosilikatowego, rodzaj, stężenie i stosunek aktywatora alkalicznego oraz warunek utwardzania. Celem badań było zastosowanie niestacjonarnej metody pomiaru dyfuzyjności cieplnej do oceny współczynnika przenikania ciepła zapraw geopolimerowych modyfikowanych składnikami o znacznej porowatości. Analizowano wpływ

trzech dodatków, które mogły poprawić izolację termiczną kompozytu geopolimerowego: odpadowego pyłu perlitowego (PP), piasku perlitowego (PS) i cenofer (C). Celem eksperymentu było znalezienie optymalnych zakresów dozowania każdego składnika, które miały istotny wpływ na przewodność cieplną. Zaprawy geopolimerowe charakteryzowały się następującym składem na 1 m^3: 500 kg mieszaniny prekursorów (metakaolin – 50 kg, zeolit – 50 kg i popiółłotny – 400 kg), 110 kg mączki wapiennej, 15 kg modyfikatorów właściwości reologicznych, 315 kg aktywatorów alkalicznych (wodorotlenek sodu – 30% roztwór wodny – 275 kg, szkło wodne – 40 kg), pył perlitowy, piasek perlitowy i cenofery zgodnie z planem eksperymentu. Do analizy czynnikowej wybrano trzy parametry składu: udział piasku perlitowego w kg na m^3 kompozytu, udział pyłu perlitowego w kg na m^3 kompozytu, udział cenofer w kg na m^3 kompozytu. Każda z zmiennych była badana na jednym z trzech poziomów: niższym, średnim i wyższym. Ponieważ przegląd literatury wskazywałna silną korelację między gęstością objętościową a przewodnością cieplną, skupiono się również na tej zależności charakterystyk. Ustalono dwa konkretne cele: ocenę znaczenia wpływu czynników związanych ze składem i gęstością objętościową na współczynnik przewodności cieplnej oraz ocenę przydatności metody niestacjonarnej do badania geopolimerów. Założeniem weryfikacyjnym dla zaprojektowanego kompozytu było spełnienie wymagań RILEM dla kompozytu klasyfikowanego jako klasa II.

Przewodność cieplna kompozytu znacznie spada wraz ze wzrostem ilości piasku perlitowego, co wiąże się z wysoką porowatością dodatku. Do analizy współczynnika przewodności cieplnej λ i ciepła właściwego C_p wybrano analizę Pareto. Wyniki analizy wskazują, że dodatek piasku perlitowego (PS) ma istotny wpływ na przewodność cieplną kompozytu. Zawartość cenosfer i proszku perlitowego nie ma statystycznie istotnego wpływu na przewodność cieplną. Kompozyt osiągnął współczynnik przewodności cieplnej poniżej 0,7 W/(m $\cdot$ K), niezależnie od przewidywanej modyfikacji. Im więcej piasku perlitowego, tym mniejsza gęstość kompozytu, a to ona ma kluczowy wpływ na współczynnik przewodności cieplnej i ciepło właściwe. Metoda dynamicznego pomiaru bardzo dobrze sprawdza się w przypadku kompozytów geopolimerowych i pozwala na ocenę kluczowych parametrów cieplnych. Podsumowując, kompozyty geopolimerowe modyfikowane dodatkami o niskiej gęstości ziarnowej, zwłaszcza piaskiem perlitowym, są obiecującym materiałem termoizolacyjnym. Biorąc pod uwagę zapewnienie wystarczającej wytrzymałości na ściskanie i urabialności, analizowane kompozyty geopolimerowe mogą być stosowane jako zaprawy o znacznej izolacyjności termicznej. Wymagają jednak dalszych badań związanych z ich trwałością.

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