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Lightweight geopolymers made of mineral wool production waste

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Abstract. Reducing industrial slags and developing building materials on its basis is one of the priority areas for the development of the construction industry. The problem of obtaining cellular concrete from mineral wool production waste (lightweight geopolymers) is considered within the framework of the researches, the results of which are presented in the article. The research purpose was to establish the impact of composition of the raw material mixture and technological production features on the physical, mechanical and thermophysical properties of light geopolymers based on mineral wool production waste. Light geopolymers were obtained by preparing a mixture of ground slugs (specific surface is 400±20 m²/kg), alkaline activator (NaOH), water, a gas-forming additive (aluminum powder), fine aggregate and a water-holding additive. The resulting mixture was placed in the form without vibration. Molded products were steamed in order to accelerate hardening. The mobility of mortar mixtures was studied during the work, the average density, compressive strength, and thermal conductivity was studied for hardened samples. The compositions of lightweight geopolymers with an average density from 610 to 1,130 kg/m³, compressive strength from 1.7 to 5.4 MPa, and a thermal conductivity from 0.144 to 0.345 W/m.°C were obtained. The application of the results will contribute to the expansion of the raw material base for obtaining light geopolymers, thereby reducing the amount of waste generated during the mineral wool production. The developed materials can be used for the construction of walling, and also as the insulation of the roof and floor of industrial and civil facilities.

1. Introduction

One of the main development directions of the global construction industry is the production of building materials, products and structures with minimal negative impact on the environment. At the same time, environmental safety in the production and implementation of building materials becomes a determining factor while selecting the raw materials and technologies for their production. The use of industrial slags in the manufacture of building materials helps to reduce CO2 emissions into the atmosphere, and also reduces pollution of soil, water and plants [1–3]. After the invention of geopolymers, the reuse of slags in the production of building materials has become more intense. Slags generated by industrial production and chemical industry (errous and non-ferrous metallurgy slags, ashes, etc.) are milled and tempered with an alkaline activator. The resulting materials have high strength, water and chemical resistance [4-12]. In [13, 14], we confirmed the possibility of using mineral wool production waste (MWPW) in the production of geopolymers.

An equally significant direction in the development of the global construction industry is the production of building materials with low density, sound and heat conductivity, as well as high physical and mechanical properties. Such materials include various types of cellular concrete. Products made of cellular concrete are used for constructing the external and internal walls of buildings, they can be used to insulate the floor, roof, etc [15, 16]. The studies of numerous researchers [2, 3, 7, 17-21] describe the compositions and technology for producing cellular concrete from industrial wastes (lightweight geopolymers). Since the beginning of the 20th century, two main technologies for producing cellular concrete have been known [3, 15]. The first is characterized by foaming of the concrete mix as a result of adding gas-forming components to the composition. These are mainly fine powders of certain metals (Zn, Al, Mg, etc.) [22-24], mixtures of acids with carbonates

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(for example, HCl, H₂SO₄ with MgCO₃, CaCO₃) [1], as well as oxidizing agents (H₂O₂, KMnO₄ and others) [22–25]. The second technology is associated with the use of foaming agents, such as synthetic surfactants [3, 5, 18] or based on protein raw materials [18, 26].

Studies regarding technology for producing lightweight geopolymer materials from MWPW are presented in the article. Up to 30 % of such waste from the mass of finished products is generated during production process [13, 14, 27, 28] The main task in the technology for producing high-quality cellular concrete is the selection of such a composition that ensures the formation of products with the required physical and mechanical properties with minimal technological costs [29]. Mobility optimization of the mortar in the production of cellular concrete is the key to the quality of the final product. The mobility of the developed mortar mixtures is primarily affected by the amount of water in the composition. Excessive amount of water, as well as gas-forming or foaming components, leads to its «boiling» and subsequent settling, and their insufficient amount does not provide the necessary foaming [30–32]. The optimal amount of alkaline activator provides the material with specified strength characteristics [21, 32, 33]. Fine aggregate and water-retaining additives prevent shrinkage of the mortar mixture (at the manufacturing stage) and the final product (after 1 year of operation, the shrinkage of aerated concrete can reach 0.36 %) [1, 3, 34, 35].

The study is aimed at establishing the impact of the composition of the raw material mixture and production technological features on the physical, mechanical and thermophysical properties of lightweight geopolymers based on MWPW.

As a result of the work carried out, the following tasks were solved:

- the effect of alkaline activator and of the water/slag ratio (W/S) on the mobility of the mortar mixture was determined;
- the impact of the mobility of mortar mixture, the amount of alkaline activator and gas-forming additive in the composition on the average density and compressive strength of light geopolymers was determined;
- the correlations between the change in the thermal conductivity of light geopolymer samples and the change in their average density and humidity were obtained.

2. Methods

2.1. Materials

The following materials were used for the research:

- ground MWPW from LLC "Kombinat teploizolyacionnyh izdelij" (Saransk, Russia). The chemical composition of the waste is as follows: SiO_2 41.800 %, CaO 26.228 %, Al_2O_3 13.257 %, MgO 11.412 %, Fe_2O_3 2.392 %, Na_2O 1.171 %, K_2O 0.383 %, TiO_2 0.297 %, SO_3 0.277 %, MnO 0.227 %, Sr_2O_3 0.096 %, NiO 0.024 %, P_2O_5 0.013 %, CuO 0.010 %, Cl 0.009 %, Co_3O_4 0.008 %, percentage of other impurities 2.396 %. The mineralogical composition is mostly represented by the X-ray amorphous phase (>95 %). Waste was sifted through a sieve with a mesh diameter of 0.63 mm and grinded to a specific surface of 400 ± 20 m²/kg;
- fine aggregate, which is MWPW sifted through a sieve with a mesh diameter of 1.25 mm. The chemical composition is given above;
- alkaline activator granulated NaOH dissolved in water. Weight content of the main substance is not less than 99.5 %;
 - gas-forming additive aluminum powder with a covering power of 600±20 m²/kg in water;
- water-retaining additive "Culminal C8360" cellulose ether. Chemical base: methylhydroxyethyl cellulose (MHEC). Viscosity: 33 000–45 000 MPa·s.

2.2. Sample production technology

The raw material mixture for light geopolymers was prepared according to the following technology. Water with an alkaline activator and a water-retaining additive dissolved in it was added to a working mixer. Then, ground MWPW and fine aggregate were alternately loaded and mixed for 3–4 minutes. The mobility of the mortar mixture was determined. At the same time, a suspension made of water and a gas-forming additive (water/additive ratio=10/1) was prepared. The prepared suspension was poured into a working mixer with the previously prepared mixture and mixed for 30 s. The finished mixture was fed into forms that were qualitatively filled without the use of vibration. The molded samples were kept in molds at a temperature of 50 °C and a relative humidity of at least 85 % for 5 hours. Then, samples outside the molds were steamed at atmospheric pressure according to the regime of 3+6+2 h at an isothermal heating temperature of 85±5 °C. The steamed samples were dried to constant weight at a temperature of 25–30 °C and relative humidity of not more than 50 %, after which they were tested.

2.3. Analytical techniques

The mobility of the mortar mixture for obtaining light geopolymers was determined according to the spread using a Southard viscosimeter.

The average density and compressive strength of material samples of each composition was determined by testing 3–5 cubic samples with a face of 150 mm.

The thermal conductivity of the samples was determined by the probe method using the MIT-1 device. The device is equipped with a measuring probe with a diameter of 6 mm and a length of 100 mm. The studies were carried out on cubic samples with a face of 150 mm at a temperature of 25 ± 2 °C. The test samples had a normalized humidity (4 wt. %) and were also dried to constant weight (dry samples). A hole with a diameter of 6 mm and a depth of 80 to 100 mm, into which the probe was immersed, was drilled in the center of the face. Before testing, the samples with the probe were kept at a given temperature for at least 2 hours. The arithmetic mean of the thermal conductivity of three samples of each composition was taken as the final result.

3. Results and Discussion

3.1. Mortar mobility

Below are the results of researchers carried out to determine the impact of the quantitative content of alkaline activator (NaOH) and the water/slag ratio (W/S) on the mortar mixture mobility made of MWPW. During the experiment, 16 compositions of mortar mixtures were tested. The arithmetic mean value of the three test results for each composition is taken as the final spread value according to the Southard viscosimeter (cm). The compositions and experimental data are presented in Table 1.

Table 1. Compositions and experimental data on the mobility of the mortar mixture made of MWPW.

Eve No	Compos	sition, % by weig	ght	- W/S ratio	Spread according to Southard			
Exp. No.	Ground MWPW	NaOH	water	- vv/S ratio	viscosimeter, cm			
1	78.8	1.5	19.7		5.2			
2	78.4	2	19.6	0.25	5.			
3	78	2.5	19.5	0.25	5.1			
4	77.6	3	19.4		5			
5	75.77	1.5	22.73		10.1			
6	75.38	2	22.62	0.30	10.3			
7	75	2.5	22.5	0.30	9.8			
8	74.62	3	22.38		7.8			
9	72.96	1.5	25.54		20			
10	72.59	2	25.41	0.35	21.5			
11	72.22	2.5	25.28	0.33	20.7			
12	71.85	3	25.15		20.1			
13	70.36	1.5	28.14		25			
14	70	2	28	0.40	26.4			
15	69.64	2.5	27.86	0.40	25.9			
16	69.29	3	27.71		24.3			

As a result of the researches conducted, it was found that the mobility change of the mortar mixture (a mixture of ground MWPW, water and NaOH) is mostly affected by the W/S ratio. So, when the W/S ratio of the mixture changes from 0.25 to 0.30, the mobility slightly increases, from 5 cm to 9.5 cm on average (spread according to Southard viscosimeter). A further increase in the W/S ratio to 0.35 leads to a sharp mobility increase up to 20.5 cm on average. With an increase in the W/S ratio of the mortar mixture to 0.40, the mobility increases only slightly (up to 25.5 cm on average).

A change in the amount of NaOH in the composition from 1.5 to 3 % by weight has practically no effect on the change in the mobility of the mortar mixture. Similar results were obtained within the framework of researches carried out by other scientists [3, 7, 11, 12, 18, 31].

3.2. Average density and compression strength

Below are the results of tests carried out in order to determine the effect of the mortar mixture mobility and the amount of gas-forming additives (aluminum powder) on the average density and compressive strength of dry samples made of lightweight geopolymers based on MWPW. Fifteen formulations of three samples each were tested. The NaOH/slag by weight ratio in all formulations is 2.5/97.5. Light geopolymers were obtained

from a mortar mixture with mobility from 9 to 22 cm (according to Southard viscosimeter). The amount of gasforming additive ranged from 0.06–0.12 % by weight of the total amount of all components in each composition.

Figure 1 presents the results of a studying the effect of the mortar mixture mobility and the amount of injected gas-forming additives on the average density and compressive strength of low density geopolymers.

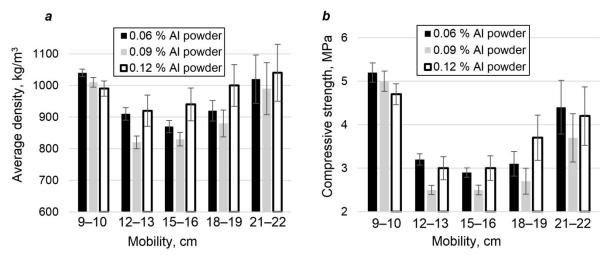


Figure 1. The effect of the mortar mixture mobility and the amount of gas-forming additive on the average density (a) and compressive strength (b) of lightweight geopolymer samples.

According to the data presented in Figure 1, *a*, the average density of light geopolymer samples made of MWPW, and obtained from a mortar mixture with a gas-forming additive in the amount of 0.06 %, decreases from 1,040 to 870 kg/m³ with increasing mobility. The mobility is measured using a Southard viscosimeter and increases from 9.5 to 15.5 cm on average. A further increase in the mobility of the mortar mixture to 22 cm leads to its rapid rise and subsequent subsidence. The average density of samples increases up to 1,020 kg/m³. When the content of the gas-forming additive in the composition of the mortar mixture is 0.09 %, the minimum average density value of geopolymer hardened samples is 820 kg/m³ (the mobility of the mixture is 12.5 cm on average). An increase in the mobility of the mixture up to 22 cm also leads to settling of the cellular mixture and an increase in the density of the samples to 990 kg/m³. The mortar mixture with the content of a gas-forming additive in an amount of 0.12 % intensively foams and settles. This is a consequence of the excess aluminum powder content in the composition.

For light geopolymer samples made of MWPW (see Figure 2, *a*) with the lowest density (≈ 820 kg/m³), the compressive strength is 2.5 MPa on average. The mobility of the mortar mixture for samples of these compositions is in the range from 12 to 16 cm. The aluminum powder content in the composition is 0.09 %. An increase in the mobility of the mortar mixture more than 16 cm, and the content of the gas-forming additive more than 0.09 % leads to a wide spread in the values of compressive strength. Samples have a defective structure in which porous regions alternate with dense interlayers (see Figure 2, b).

Figure 3 presents the results of studying the impact of the alkaline component amount in the mortar mixture composition on the average density and compressive strength of light geopolymer samples. During the experiment, 12 compositions of 3 samples in each were tested. During the manufacture of samples, the same mobility of the mortar mixture was achieved (13–14 cm). The NaOH content in the composition ranged from 2.5 to 4.5 % of the total weight of all components in the composition, and the amount of gas-forming additives was from 0.05 to 0.09 %.

According to the data obtained (Figure 3, *a*), an increase in the NaOH content from 2.5 to 4.5 % in the compositions does not significantly affect the change in the average density of lightweight geopolymer samples. The average density of samples based on MWPW depends to a greater extent on the amount of gas-forming additives in the mortar mixture composition. In order to obtain such cellular concrete with a density of not more than 1,000 kg/m³, the content of aluminum powder in the composition must be at least 0.07 %. Samples of cellular concrete made of MWPW with the lowest density (less than 850 kg/m³) without the use of modifying additives can be obtained using the mortar mixture with a mobility between 12–16 cm. The amount of aluminum powder in the composition should be 0.09 %.

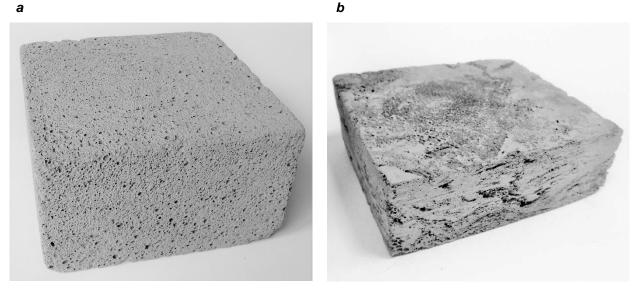


Figure 2. Photo of light geopolymer samples made of MWPW: a – the mobility of the mortar mixture is 13 cm, the amount of aluminum powder is 0.09 %; b – the mobility of the mortar mixture is 19 cm, the amount of aluminum powder is 0.12 %.

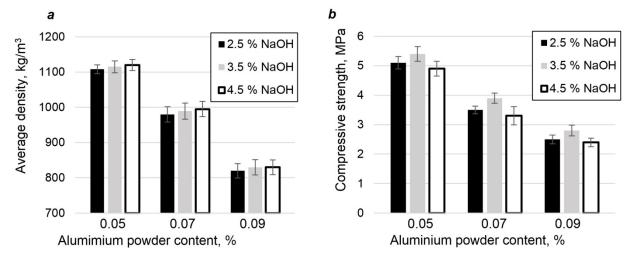


Figure 3. The effect of the alkaline component on the average density (a) and compressive strength (b) of lightweight geopolymer samples.

According to the data presented in Figure 3, *b*, 3.5 % NaOH in the composition of the mortar mixture provides the greatest compressive strength of light geopolymers made of MWPW. With a decrease in the density of the samples from an average of 1,120 to 830 kg/m³, the compressive strength decreases in direct proportion from 5.4 to 2.8 MPa. The obtained data on compressive strength of lightweight geopolymer samples made of MWPW is almost identical to the values obtained when testing samples based on slag of ferrous and non-ferrous metallurgy, ashes, glass wastes (the same method of foaming the mortar mixture and similar average density of the hardened samples) [3, 7, 11, 19, 21–23, 31].

The following can be noticed while comparing the compositions of mortar mixtures for obtaining lightweight geopolymers from MWPW with data coming from other researchers. The amount of water and aluminum powder in the composition of the MWPW mortar mixture providing its maximum foaming without settling, as well as the amount of alkaline component providing maximum compressive strength of the samples at a given density, is lower in comparison with compositions based on fly ash or blastfurnace slag [7, 11, 19, 22]. The optimal W/S ratio of the MWPW-based mortar mixture is on average 0.33, for compositions with fly ash it is around 0.37 [7], and with blastfurnace slag is at least 0.4 [11]. In order to achieve the minimum density of lightweight MWPW-based geopolymers, the maximum amount of introduced gas-forming component (aluminum powder) should not exceed 0.09 %, which is also lower comparing to compositions based on fly ash or blastfurnace slag (>0.1 %) [7, 11, 19, 22]. To achieve high compressive strength of foamed materials and products made of geopolymers based on slags of ferrous and non-ferrous metallurgy, as well as on ashes, a rather large amount of alkaline activator is used: 4.5 % water glass and 10 % NaOH [22, 23], at least 10 % Ca(OH)2 and Mg(NO3)2 [11], up to 10 % NaOH [21], etc. This indicator is at least two times lower for compositions based on MWPW (3.5 % NaOH).

3.3. Thermal conductivity

The thermal conductivity test results of samples made of lightweight geopolymers based on MWPW with various average densities are presented in Figure 4. During the experiment, 8 dry samples and 8 samples with a humidity of 4 % were tested. Samples obtained from the mortar mixture with a mobility of 12–16 cm were selected for testing. The content of the gas-forming component in the composition was in the range of 0.05–0.09 %, and the content of alkaline component was 2.5–3.5 %.

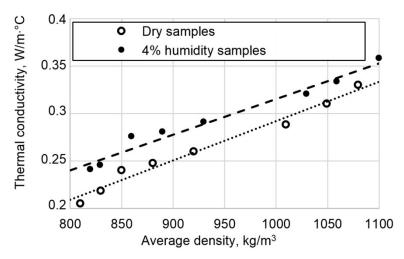


Figure 4. Thermal conductivity of light geopolymers made of MWPW.

According to the data presented in Figure 4, the thermal conductivity of dry samples of lightweight geopolymers made of MWPW increases directly proportionally from 0.208 to 0.332 W/m·°C with an increase in the material density from 800 to 1,100 kg/m³. With an increase in material humidity up to 4 %, the thermal conductivity of samples increases by 15 % on average for compositions with a density of 800 kg/m³ and by 6 % for compositions with a density of 1,100 kg/m³. The resulting changes in the thermal conductivity of lightweight geopolymers according to their density are almost identical to the data presented in the research [3]. The results of many researchers are summarized by the study.

As a result of the conducted researches, it was found that the material density should not exceed 820 kg/m³ in order to obtain light geopolymers made of MWPW with a thermal conductivity of not more than 0.250 W/m \cdot °C. And the material density should not exceed 950 kg/m³ for a desirable thermal conductivity of not more than 0.300 W/m \cdot °C.

3.4. Compositions and properties of modified lightweight geopolymers

The compositions and properties of lightweight geopolymers made of MWPW and modified with fine aggregate and water-retaining additive Culminal C8360 are presented in Table 2. The significance of the introduction of these modifiers into the mortar mixture is described above.

Table 2. Compositions and properties of mounted lightweight geopolymers.												
Components		Composition, weight parts										
Components		2	3	4	5	6	7	8	9			
Ground MWPW	100	100	100	100	100	100	100	100	100			
Fine aggregate	10	15	20	10	15	20	10	15	20			
Alkaline activator	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5			
Water	36.67	37.65	38.75	37.78	38.82	40	38.85	40	41.29			
Gas-forming additive		0.07	0.09	0.05	0.07	0.09	0.09	0.07	0.05			
Water-retaining additive		0	0	0.03	0.03	0.03	0.06	0.06	0.06			
Properties		Indicators for composition										
	1	2	3	4	5	6	7	8	9			
Average density, kg/m ³		1 020	840	1 080	950	670	610	920	1 060			
Compression strength, MPa		3.5	2.7	4.9	3.1	2.2	1.7	2.9	4.5			
Thermal conductivity, W/m·°C (dry samples)		0.303	0.221	0.335	0.273	0.167	0.144	0.259	0.320			

Table 2. Compositions and properties of modified lightweight geopolymers.

According to the data presented in Table 2, the introduction of up to 20 weight parts of fine aggregate into the mortar does not significantly affect the change in average density, compressive strength, and thermal

0.358 0.325 0.258 0.351 0.305 0.197 0.171 0.275 0.338

Thermal conductivity, W/m·°C (4 % humidity samples)

conductivity of lightweight geopolymers. This component also helps to reduce material shrinkage during operation, as well as reduce the cost of the final product.

With the introduction of the Culminal C8360 water-retaining additive, a decrease in the density of geopolymer samples to 610 kg/m³ was detected, as well as a decrease in the thermal conductivity of dry samples to 0.144 W/m·°C, and to 0.171 W /m·°C for 4 % humidity samples. It was also determined that an increase of this additive over 0.06 % in the composition leads to a decrease in compressive strength of lightweight geopolymer samples.

The positive effect caused by the modification of lightweight geopolymers with a fine aggregate and a water-retaining additive was also noted in several studies [1, 3, 34, 35].

4. Conclusion

- 1. Compositions have been developed and technological methods have been described for obtaining lightweight geopolymers (cellular concrete) from mineral wool production waste with an average density of 610 to 1,130 kg/m 3 , compressive strength of 1.7 to 5.4 MPa, and thermal conductivity of 0.144 to 0.345 W/m $^{\circ}$ C.
- 2. In the production of light geopolymers made of MWPW, the mobility of the mortar mixture should be 12–16 cm according to Southard viscosimeter, the amount of alkaline activator (NaOH) in the mortar mixture should be equal to 3.5 %, and the content of aluminum powder should not exceed 0.09 %.
- 3. The introduction of up to 20 % of fine aggregate into the mortar mixture composition in the form of mineral wool production waste fractions of less than 1.25 mm does not significantly affect the change in average density, compressive strength and thermal conductivity of lightweight geopolymers.
- 4. In order to obtain cellular concrete from mineral wool production waste with a density of less than 830 kg/m^3 (up to and including 610 kg/m^3), it is necessary to add a water-retaining additive, for example, Culminal C8360, in an amount up to 0.06 %.
- 5. The use of ground mineral wool production waste in the production of lightweight geopolymers will significantly reduce the amount of expensive components in the mortar mixture (up to 2 times or more for NaOH, more than 10 % for aluminum powder) compared with compositions based on blastfurnace slag and fly ash glass waste (while maintaining the strength and thermophysical properties of the material).
- 6. The conducted researches will contribute to the expansion of the raw material base for the production of lightweight geopolymers, thereby reducing the amount of slags generated during the mineral wool production. The developed materials will find application in the manufacture of building envelopes, as well as insulation of roofs and floors in the construction of industrial and civil facilities.

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Легкие геополимеры из отходов производства минеральной ваты

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Аннотация. Сокращение отходов промышленного производства и разработка на их основе строительных материалов является одним из приоритетных направлений развития строительной индустрии. В рамках исследований, результаты которых представлены в данной статье, рассматриваются вопросы получения ячеистых бетонов из отходов производства минеральной ваты (легких геополимеров). Цель исследования заключалась в установлении влияния состава сырьевой смеси и технологических особенностей получения на физико-механические и теплофизические свойства легких геополимеров на основе отходов производства минеральной ваты. Легкие геополимеры получали путем подготовки смеси из размолотых отходов (удельная поверхность равна 400 ± 20 м²/кг), щелочного активатора (NaOH), воды, газообразующей добавки (алюминиевой пудры), мелкого заполнителя и водоудерживающей добавки. Полученные смеси укладывали в формы без применения вибрации. Для ускорения твердения отформованные изделия пропаривались. В работе у растворных смесей исследована подвижность, а у затвердевших образцов средняя плотность, прочность при сжатии и теплопроводность. Получены составы легких геополимеров со средней плотностью от 610 до 1 130 кг/м 3 , прочностью при сжатии от 1,7 до 5,4 МПа, коэффициентом теплопроводности от 0,144 до 0,345 Вт/м·°С. Применение полученных результатов будет способствовать расширению сырьевой базы для получения легких геополимеров, тем самым сократится количество образующихся при производстве минеральной ваты отходов. Разработанные материалы могут быть использованы при строительстве ограждающих конструкций, а также утепления крыши и пола объектов промышленного и гражданского назначения.

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