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Alkali-activated slag binders from rock-wool production wastes

Шлакощелочные вяжущие из отходов производства минеральной ваты

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Ключевые слова: шлакощелочное вяжущее; отходы производства минеральной ваты; композит; биологическая стойкость

Abstract. The article exposes the results of studies on the production of alkali-activated slag binders from rock-wool production wastes and their composite derivatives, which are resistant to corrosive effects of biological environment. Thermal analysis showed that the structurization in the "rock wool production waste-water-NaOH" system is most efficient with the NaOH/waste ratio of 0.02–0.03, and the main newly-formed phases in the solidified alkali-activated slag composites are low-basic calcium hydrosilicates and analcime. The best values of the physical and mechanical properties of obtained binders are achieved with a waste grinding fineness of 400–450 m²/kg. The compressive strength of the developed composite materials reaches 68 MPa and bending strength is 13.5 MPa with a waste grinding fineness of 300 m²/kg. Composites based on binders made of rock-wool production waste are waterproof and funginert, and individual compounds are fungicidal.

Аннотация. В статье представлены результаты исследований по получению шлакощелочных вяжущих из отходов производства минеральной ваты и композитов на их основе, стойких в условиях агрессивного воздействия биологических сред. Методами термического анализа установлено, что наиболее эффективно процесс структурообразования в системе "отходы производства минеральной ваты-вода-NaOH" протекает при соотношении NaOH/отход равном 2/100–3/100, а основными новообразовавшимися фазами в затвердевших шлакощелочных композитах являются низкоосновные гидросиликаты кальция и анальцим. Наилучшие показатели физико-механических свойств полученных вяжущих достигаются при тонкости помола отхода равной 400–450 м²/кг. Прочность при сжатии разработанных материалов композитов достигает 68 МПа, а при изгибе уже при тонкости помола отхода равной 300 м²/кг равна 13,5 МПа. Композиты на вяжущих из отходов производства минеральной ваты водостойки и грибоустойки, а отдельные составы фунгицидны.

1. Introduction

According to the statistics, the dumps and storage facilities on territory of the Russian Federation alone accumulate over 100 billion tons of solid industrial wastes, which are the pollution source of surface and ground waters, the atmosphere, soil and plants. In addition, hundreds of thousands of land hectares were withdrawn from the economic turnover. However, man-made waste can be used as raw material for obtaining various materials including constructional ones [1–3]. One of the solid industrial waste types is the rock-wool production waste, which compose 15 to 30 % of the finished product mass. According to the statistics, about 5–6 million m³ of such wastes have already accumulated in Russia. Works on the utilization

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of rock-wool production waste are conducted in two directions: returning to the production of mineral wool as an additional source of raw material and obtaining new construction materials [4–7]. The volume of waste involved in both methods remains insignificant and does not solve the utilization problem. At the same time, the chemical and phase composition of this waste type makes it possible to use it as a raw material for the production of alkali-activated slag binder and composite materials based on it [1–4, 7]. A significant number of scientific researches are related to the production of alkali-activated slag binders using industrial waste. However, almost all works include blast furnace slags, various ash, red mud, broken glass, etc. as a waste material [8–25]. There are only a small number of researches related to the development of binders using the rock-wool production waste and composites based on them, and the results obtained in those researches do not meet the requirements for modern building materials [4, 7, 26, 27].

During the exploitation period of buildings and structures construction materials and elements interact with natural and man-made aggressive biological environment, which leads to the reduced strength and other properties. Biological corrosion can be enhanced due to the high humidity, cyclically operating temperatures and other environmental factors. The annual world economic damage from biodeterioration reaches tens of billions of dollars. The appearance of buildings and the ecological situation inside them is becoming worse, and the list of diseases caused by microscopic organisms is expanding [28–35].

Consequently, studies devoted to the development of effective binders based on rock-wool production waste and composites produced using those binders (with improved physical, mechanical and operational properties) are of practical value and are relevant in modern construction materials science.

The main aim of the current researches is development of the binders made of rock-wool production waste and their composite derivatives, resistant to the corrosive effects of biological environment. The following tasks were solved:

- the phase transformations occurring in rock-wool production waste, as well as in composites based on them upon heating were studied;
- the physical and mechanical properties of the astringents and composites developed on their basis, as well as their biological resistance were studied.

2. Methods

The waste of LLC “Kombinat teploizolyacionnyh izdelij” (Saransk) was used as a rock-wool production waste, its chemical composition is as follows: CaO – 29.62–29.93 %, SiO₂ – 43.81–43.88 %, Al₂O₃ – 9.13–9.21 %, Fe₂O₃ – 3.73–3.94 %, SO₃ is 0.23–0.24 %, MgO is 8.33–8.43 %, K₂O is 0.71–0.72 %, Na₂O is 1.33–1.37 %, TiO₂ is 0.46–0.47 %, P₂O₅ – 0.03–0.04 %, SrO – 0.07 %, MnO – 0.16 %, ZnO – 0.002 %, Cr₂O₃ – 0.01 %, percentage of other impurities – 1.8 %, the mineralogical composition: β-SiO₂ – 5.0 %, Ca₂Al₂SiO₇ – 1.2 %, Ca₂MgSi₂O₇ – 23.8 %, amorphous phase – 70.0 % (for waste with more than 0.63 mm fractions) and β-SiO₂ – 5.6 %, Ca₂Al₂SiO₇ – 3.4 %, Ca₂MgSi₂O₇ – 1.0 %, amorphous phase – 90.0 % (for waste with less than 0.63 mm fractions). The shapes of the waste particles of different fractions is shown in Figure 1, according to which it can be seen that the rock-wool production waste of LLC “Kombinat teploizolyacionnyh izdelij” contains particles of slug which are less than 0.5 mm, and also aggregates and spalls.

The sodium hydroxide (NaOH) was used as an alkaline component which meets the requirements of Russian National Standard 55064-2012.

The production method of the alkali-activated slag binder and its derivatives using rock-wool production waste was as follows. The wastes were dried to constant weight at a temperature of 105 °C and then grounded until the specific surface value is from 250 to 500 m²/kg. After that the dosages of the components were weighed. The working mixer was then gradually loaded with the waste and an alkaline component. Stirred for 5-7 minutes. In accordance with All-Union State Standard 310.3-76, a standard stiffness of the mixture was determined. The obtained solution was then vibroformed in order to produce 10×10×30 mm and 40×40×160 mm beam-shaped samples for the further biostability testing, and also cube-shaped with a 20 mm side for studying physical and mechanical properties, and subjected to steam treatment at a 90°C temperature in the 3:8:2 mode. Phase transformations during solidification of composites, strength, water resistance and biostability were considered as controllable properties. The bending and compression strength of the obtained samples were determined two hours after the steam treatment in accordance with All-Union State Standard 310.4-81.

The phase transformations occurring in the waste and composite based on alkali-activated slag binder during heating were studied using a TGA/DSC1 instrument. Samples of waste and composites were ground in an agate mortar with an agate pestle and acetone. The samples were screened through a sieve with an aperture of 90 microns. The balance left on the sieve was bolted again until the whole sample was bolted through a sieve. 0.5 g of the bolted sample was weighed with the accuracy of 0.0001 g and poured into an alundum pot with a volume of 900 mcl. The sample was compacted by tapping the pot on the table. Further, the pot was mounted on a holder and placed in an oven. The sample was heated from 25 to 1000°C at a rate of 10°C/min.

The fouling of alkali-activated slag composites based on the rock-wool production wastes by mold fungi was determined using samples with 10×10×30 mm beads according to All-Union State Standard 9.049-91 by methods 1 (without additional sources of carbon and mineral nutrition) and 3 (on a solid nutritional medium Czapeka-Doksa) with the assessment of fungi resistance and fungicidity, respectively.

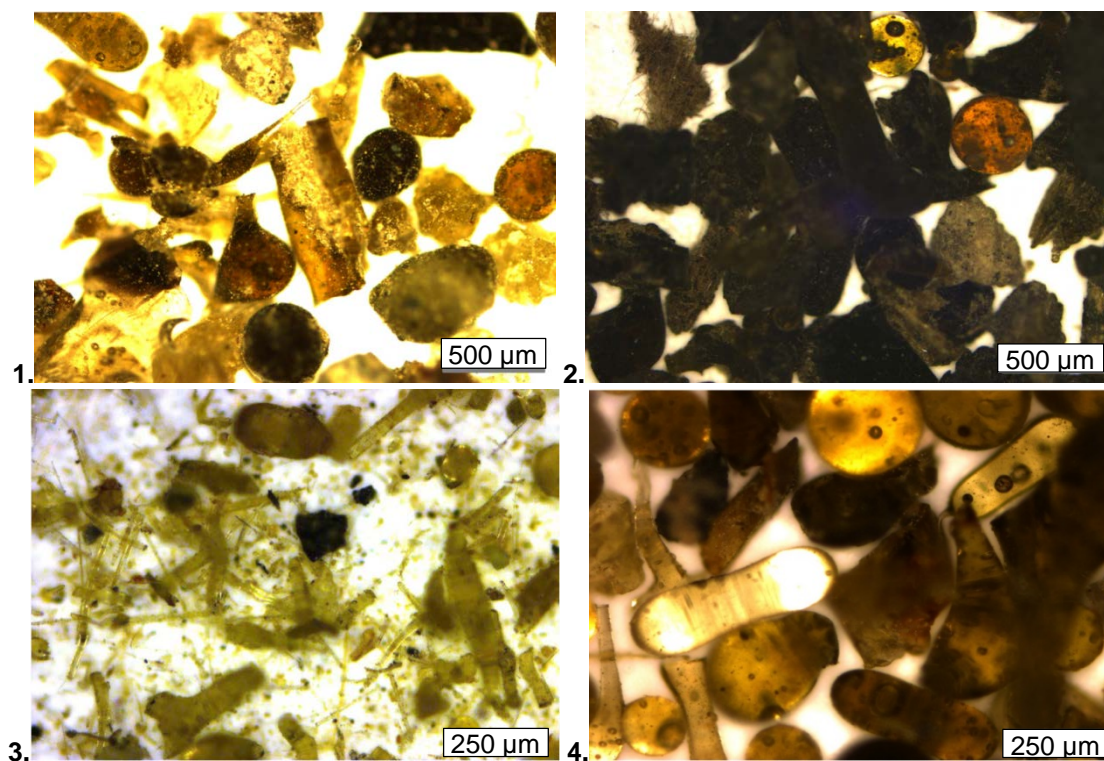


Figure 1. The particle shape of the rock-wool production waste from LLC “Kombinat teploizolyacionnyh izdelij” (Saransk) with the following fractions, mm: 1 – 0.63–1.25; 2 – 0.315–0.63; 3 – 0.16–0.315; 4 – < 0.16

3. Results and Discussion

In order to confirm the possibility of using rock-wool production waste as raw material to obtain alkali-activated slag binders including their individual fractions, studies of phase transformations in the waste of LLC “Kombinat teploizolyacionnyh izdelij” under heating were conducted by the means of thermal analysis methods. The results of the research are presented in Figure 2, lines 1 and 2.

According to the data in Figure 2, lines 1 and 2, the following major phases have been identified in the mineral wool production waste. The first peak in the temperature range from 25 to 100 °C (endo-effect) corresponds to the dissociation of unbound water. The second peak at a temperature of 250–420 °C (exo-effect), most likely corresponds to the burnout of organic impurities both for the 0.63–5 mm fraction and for the 0.08–0.63 mm fraction. The largest amount of this phase is presented in the fine fraction. The third peak in the temperature range of 530–740 °C can be characterized by an exothermic reaction and a sufficiently large mass loss, which most likely corresponds to oxidation and removal of carbonium in the form of carbon dioxide. The largest amount of this phase is also presented in the fine fraction. The endothermic effect and a slight loss of mass in the temperature range from 740 to 780 °C correspond to tiff decarbonization. This peak is more clearly visible on the DTG curve for a fraction of less than 0.63 mm. A large exothermic effect in the temperature range 860–970 °C corresponds to the crystallization process of amorphous phase. It should be noticed that this effect is much larger for a small fraction. As a result of the

quantitative X-ray phase analysis of rock-wool samples heated to 1000 °C, it was determined that their final crystallization product is: diopside – 45.4 %, melilite – 42.0 %, wollastonite – 12.6 %.

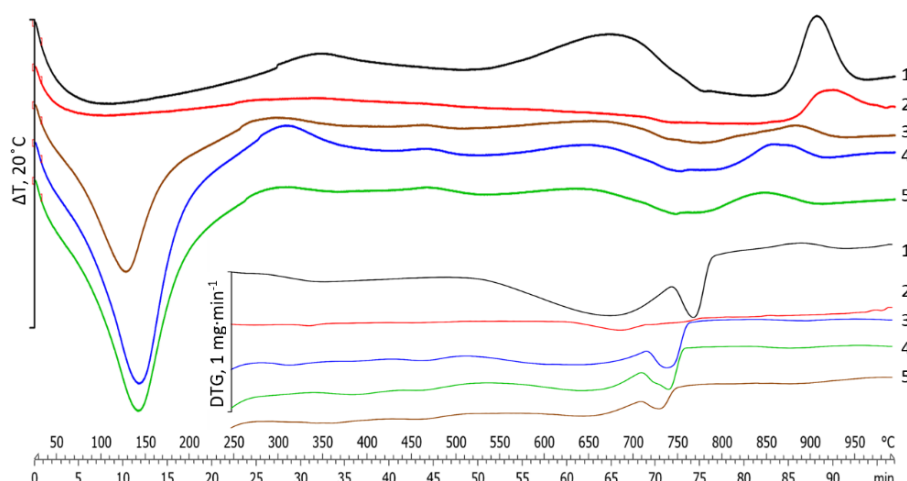


Figure 2. DTA of rock-wool production waste and solidified alkali-activated slag composites based on them:

1 – waste of 0.08–0.63 mm fraction, 2 – the same, 0.63–2.5 mm fraction, 3 – composite based on coarse fraction waste with a 4 % NaOH content, 4 – composite based on a fine waste with a 2 % NaOH content, 5 – the same, with a content of 4% NaOH

The results of the microscopic analysis (Figure 1), the data on the chemical and mineralogical composition, as well as the thermal analysis of the rock-wool production waste of LLC “Kombinat teplozolyacionnyh izdelij” were analyzed. It can be assumed that in the case the wastes are mixed with alkaline solutions they will have astringent properties, and the waste of fraction less than 0.63 mm will provide the increased activity.

In order to confirm all the above mentioned, the researches were conducted. Rock-wool production waste milled to a specific surface of 350 m²/kg were mixed with an alkaline solution (2 % NaOH and 4 % of the waste weight in terms of dry matter). The sample production technology is presented above. Phase transformations occurring in solidified alkali-activated slag composites based on the waste during heating was analyzed using thermal analysis methods. The results are shown in Figure 2, lines 3-5.

As a result of differential thermal studies, the presence of the following major phases formed in solidified alkali-activated slag composites was determined. An intense endothermic effect in the temperature range from 25 to 250 °C, as well as the shift of the exothermic effect with a peak at 905 °C (for waste) to the lower temperature range with a peak at 850 °C (for the composite) indicated the presence of low-basic calcium hydrosilicates in the composition. The intensity of the peaks corresponds with the largest amount of C-S-H (I) in the composite obtained on the basis of fine fraction waste with NaOH content equalling 2 % of the dry components. A slight endo-effect in the temperature range from 350 to 420 °C, as well as a slight exothermic effect in the temperature range from 430 to 520 °C probably correspond to the presence of analcime in the composite [3].

According to the above mentioned research results, it is possible to obtain binders and composites based on mixing the rock-wool production waste with an alkaline solution, which will have an increased strength.

The physical and mechanical properties of the astringents and composites developed on their basis were studied. Milled to a specific surface of 250 m²/kg, rock-wool production waste was mixed with NaOH alkaline solution of various concentrations. The quantity of the solution provided normal consistency of the paste (5–10 mm according to the Vicat apparatus). The methodology of sample production is presented above. The experimental results of analyzing the compressive strength of composites as a function of the quantitative NaOH content are shown in Figure 3

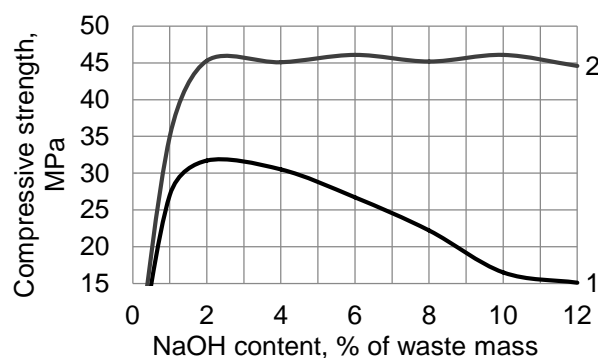


Figure 3. Compressive strength of samples based on alkali-activated slag binders made of rock-wool production waste: 1 – two hours after steam treatment, 2 – one year after steam treatment

According to the conducted research (Figure 3), it was determined that the structuring in the considered system proceeds most effectively at a NaOH/waste ratio of 0.02–0.03 (compressive strength is 32 MPa). One year after the steam treatment, the strength of composites based on alkali-activated slag binders with the rock-wool production wastes is almost identical for compositions containing from 2 to 12 % NaOH (100 % is a waste mass) and is 45 MPa on the average. However, it should be noted that compositions containing more than 4 % of NaOH are characterized by considerable efflorescence. The bending strength of composites is also maximal with a NaOH/waste ratio of 0.02–0.03 (bending strength is 13.5 MPa)

The average density of composites based on developed astringents decreases directly from 2130 kg/m³ to 2030 kg/m³ with increasing the quantity of NaOH in the composition from 1 % to 12 % (100 % is a waste mass).

As is known, one of the major factors affecting the binder effectiveness is its fineness of milling. In order to determine the dependence of the binder effectiveness based on rock-wool production waste and alkaline solution (2 % NaOH), the water requirement of the binder and the compressive strength of solidified composites based on it were determined during the research.

The water requirement of the developed binders (2 % NaOH) is determined to increase proportional from 24 % to 26.5 % with an increase in the fineness of grinding from 250 m²/kg to 400 m²/kg. The further increase of the grinding fineness from 400 m²/kg to 500 m²/kg leads to a slight water requirement increase from 26.5 % to 27.5 %.

According to the obtained data, the optimum fineness of the binder based on rock-wool production waste is 400–450 m²/kg. The compressive strength in this case reaches 62–68 MPa, respectively. With an increase in the fineness of the grind beyond this limit, the growth of effectiveness is negligible. This can be explained by the fact that the ultra-fine grinding causes the grains of the binder to adhere and the alkaline solution interacts not with individual particles, but with the surface of large floccules. In addition, the content of mixing water increases, which increases the amount of pores in the composite. All this leads to inhibition of the composition effectiveness.

Nowadays, much attention is paid to the durability improvement of elements in structures and buildings operating under the influence of aggressive environment. The problem of biocorrosion of building materials, products and structures has recently become urgent. And this is the reason for the research of the alkali-activated slag binder composition effect on fouling as well as the determination of species composition of microorganisms on composite samples. The results of the studies are presented in Table 1.

Table 1. Effect of the alkali-activated slag binder composition on the fouling and the dominant microorganism species on composites

No.	Composition, %			Fungal growth assessment, in points		Characteristics according to All-Union State Standard 9.049-91	Dominant microorganism species on sample
	Waste	NaOH	Water	Method 1	Method 3		
1	77.5	1.6	20.9	0	5	Fungus-proof	Penicillium cyclopium
2	75.9	3.2	20.9	0	5	Fungus-proof	Penicillium cyclopium
3	74.4	4.7	20.9	0	4	Fungus-proof	Penicillium cyclopium
4	72.7	6.3	20.9	0	4	Fungus-proof	Penicillium cyclopium
5	71.2	7.9	20.9	0	3	Fungus-proof	Penicillium cyclopium
6	69.6	9.5	20.9	0	0	Fungicidal	-

As was determined, the alkali-activated slag composites based on the rock-wool production waste and alkaline solution containing from 1.6 % to 7.9 % NaOH are fungus-proof, and the composite samples contain the dominant micromycete species of the genus *Penicillium* (*Penicillium cyclopium*) on their surface after a month of testing in the standard micellum fungus medium. However, with a NaOH content of 9.5 % samples get fungicidal properties.

As articulated earlier, microscopic organisms develop more intensively with increased humidity and optimal temperature conditions. Consequently, in addition to biocorrosion, building materials can also be destroyed due to the water impact. Figure 4 exposes the results of a studying the water resistance of the developed alkali-activated slag composites.

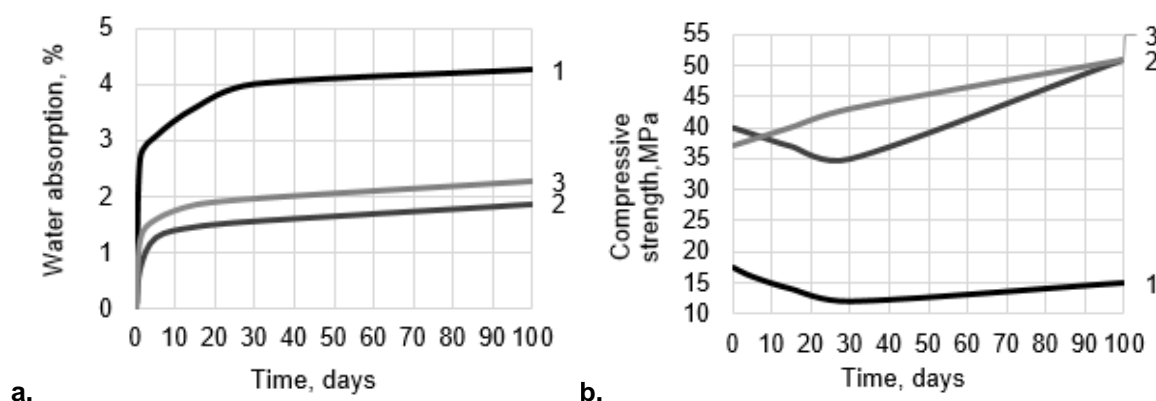


Figure 4. Water absorption (a) and change in compressive strength (b) depending on the duration of water retention of composites based on rock-wool production waste, water and NaOH quantity: 1, 2, 3 %, respectively

Obtained results show that the composites based on binder made of rock-wool production waste have increased water resistance. After exposing the samples in an aqueous medium for 100 days their compressive strength increased by 25 % with a 0.02 NaOH/waste ratio, by 35 % at a 0.03 ratio, and decreased by 15 % with a NaOH/waste ratio of 0.01. However, for all tested compositions there is a tendency to increase the compressive strength, which indicates the acceleration of hydration of these composites in water.

4. Conclusions

1. New building materials with improved physical, mechanical and operational properties were obtained on the basis of rock-wool production waste.
2. Thermal analysis method was used to define the phase transformations occurring in rock-wool production waste, as well as in composites based on them upon heating. The major newly formed phases in solidified alkali-activated slag composites with a NaOH/waste ratio of 0.02–0.03 are low-basic calcium hydrosilicates and analcime.
3. The compressive strength of steamed composites made of binders based on the rock-wool production waste reaches 68 MPa with a waste fineness of grinding of 400–450 m²/kg, and bending strength at a 300 m²/kg fineness of grinding is 13.5 MPa.

4. Composites based on developed astringents are characterized by increased water resistance and fungi-proofness, which makes it possible to recommend their usage in buildings and structures with aggressive biological environment.

5. Taking into account the above-mentioned results of physical and mechanical studies, and also the fact that caustic soda is the most expensive component in the developed material, it is expedient to use less than 0.63 mm waste fractions which are ground to a specific surface of 400–450 m²/kg, and NaOH in an amount of 2–2.5 % of the waste mass. Those materials are resistant to aggressive impact of biological environment.

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