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Features of the synthesis of construction geopolymer composites

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Abstract. Use of clinker-free binders, such as geopolymers and various equivalents based on mineral additives, can significantly reduce the carbon footprint of the construction sector in the environment. The most promising and appropriate benchmark is the disposal of industrial waste of aluminosilicate oxide composition with subsequent mechanical and alkaline activation. For the first time, the microstructure of geopolymers based on aspiration cement dust and tuff has been comprehensively studied. The theoretical prerequisite for the creation of a binder system of such a concept is the synthesis of sufficiently strong and resistant to external manifestations of alkali metals, including the structures of frame aluminosilicates with a hidden crystalline structure. X-ray diffraction analysis of the obtained samples, as well as the results of scanning electron microscopy, electron dispersion spectrometry, differential-thermal analysis, ad infrared spectrometry confirm the presence in the geopolymer paste of products traditionally necessary for the hydration reaction: aqueous aluminosilicates, aluminates and silicates of sodium and calcium, quartz, calcite, feldspars similar to albite and orthoclase, micas, etc. The results obtained on the key results of the conducted studies confirm the high efficiency of the proposed technology and guarantee increased strength and durability of geopolymer concrete.

Keywords: geopolymer; low-carbon footprint, man-made raw materials, clinker-free technology, activation, aspiration dust, structure

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1. INTRODUCTION

In the context of growing demand for cement, innovative approaches are needed to minimize the negative impact of its production on the environment [1]. One such solution is the development of new types of binders and concrete with their use with a reduced clinker content or complete rejection of it [2]. Research in this area shows that the use of non-traditional binders (slag-alkali, geopolymers and others on various bases) can significantly reduce CO_2 emissions into the environment [3]. Such approaches are binary in nature in terms of the result obtained - they not only help to reduce the carbon footprint of the resulting composite, but also improve the physical-mechanical properties ad performances of the final product [4].

In addition, investments in carbon dioxide capture and storage (CCS) technologies are a promising path to so-called "carbon neutrality" [5]. Such technologies are capable of capturing emitted CO_2 at the production stage and storing it in geologically stable formations [6]. Despite the high initial costs, successful implementation of CCS could be a game changer for the cement industry [7, 8].

Coordinated policies at the level of governments and international organizations are also essential [9]. Introducing strict emission standards and supporting sustainable practices in construction could significantly accelerate the transition to cleaner technologies [10]. By combining the efforts of the scientific community, industrial magnates, and the public sector, it is possible to achieve a more sustainable development of the cement industry and reduce its contribution to global warming [11].

The most promising and appropriate benchmark is considered to be the utilization of industrial waste of aluminosilicate oxide composition with subsequent mechanical and alkaline activation [12]. It is recommended to use alkali metal hydroxides of sodium and potassium, sodium (potassium) meta-, di- or orthosilicates, sodium aluminates, and soda ash as an alkaline sealant [13]. The theoretical basis for obtaining a binder system in this concept is considered to be the formation of structures that are sufficiently stable and resistant to such effects, obtained as a result of the interaction of cryptocrystalline structure-forming aluminosilicates of the $R_2O \cdot Al_2O_3 \cdot nSiO_2$ type and finely dispersed solid phases that have aluminate and silicate ions in their structure under alkaline medium conditions [13]. The process of physicochemical reactions in the studied dispersed system "aluminosilicate solid phase - alkaline solution" can be described through a number of key transformations that occur sequentially and in parallel [14].

Firstly, dispersion and destruction of aluminosilicate bonds occurs under the influence of an alkaline environment, which leads to the formation of colloidal solutions [15].

Secondly, small particles of alkaline aluminosilicate and silica sols are converted into larger compounds as a result of their interaction [16].

Thirdly, the implementation of cation exchange ($2Na^+ \leftrightarrow Ca^{2+}$) promotes the formation of new structures, which leads to the emergence of condensation-crystalline formations [17].

Thus, the described processes illustrate the complex mechanism of interactions in this dispersed system, where each of the transformations plays an important role in the formation of the result [18].

The discovered patterns in the formation of the structure and ensuring the properties of geopolymer paste using alkaline activation binders make it possible to obtain concrete composites for various purposes [19]. This includes quick-hardening and acid-resistant mixtures, cellular concretes with a density grade of D150–350, as well as heat-resistant, low-exothermic and high-strength materials of classes up to B140, shrinkage-free and many others [20]. Such possibilities significantly enrich the raw material base for the cement industry [21].

In addition, alkaline binders allow the use of substandard fine sands and fillers with a high content of clay and dust particles, including waste from paste crushing industries [22]. As a result of the research, frost-resistant concretes of grade F1000 and waterproof up to W30 were developed [23]. Their shear strength characteristics, as well as resistance to corrosion and biological influences, are comparable to the same indicators of products made of ordinary Portland cement [23,24]. Conducted scientific analyses show that the outstanding physical and mechanical properties as well as performances of concrete made of alkaline binders allow its use in those areas of construction where traditional cement mixtures are unable to meet the specified requirements [24]. This approach opens up new prospects for the use of modern materials in construction, ensuring the reliability and

durability of objects [24]. New generation concretes demonstrate high efficiency and a wide range of applications, which makes them competitive in the building materials market [25].

Moreover, at the end of the 20th century, regulatory and technical documentation and recommendations regarding raw materials, recipes, indicators and the procedure for preparing slag-alkaline binders and concretes based on them were approved [26, 27]. All this, together with the advantages of this technology, makes us think about the maximum development of clinker-free binders and practical implementation in various segments of construction such precast concrete products and structures, road and monolithic construction, especially in an aggressive sulfate environment, hydromelioration, subway tunnel tubing, etc.

Thus, in light of the increasing emphasis on environmental sustainability and responsible treatment of nature, progress in the study and use of technologies that do not require clinker is becoming not only preferable but critically important for the future of the construction industry. This is particularly relevant in the context of the depletion of the planet's natural resources [26–29]. A research gap lies in the comprehensive study of the microstructure of geopolymers based on aspiration cement dust and tuff.

The goal of this work was to develop a low carbon, non-clinker binder using a technological raw material. The following tasks were achieved:

– study of the characteristics of raw materials as potential components of the binding bond of alkaline clogging;

– selection of the optimal composition of the geopolymer composition using clinker production waste;

– comprehensive examination of the obtained alkaline activate paste microstructure.

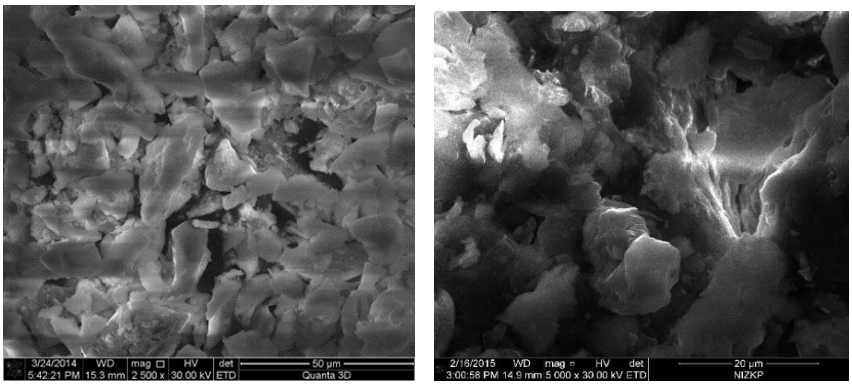
2. METHODS AND MATERIALS

The authors of the work offer their vision in solving the identified issues, and the proposed development results aimed at creating a clinker-free binder system will reveal the positive possibilities of alkaline mixing of finely dispersed additives of various origins. Aspiration dust from a cement plant was used as a dispersed powder of the binder composition. This product is not secondary in cement production and is not returned back to the technological cycle of the wet method of preparing the raw mix, since attempts led to thickening of the sludge, which negatively affects the production technology and properties of clinker. The origin algorithm of aspiration dust is accumulation in a dust precipitation system, extraction and storage on the territory of the plant as it accumulates, followed by removal to nearby agricultural lands. Being in a finely dispersed state, aspiration dust is weathered in the environment, harming all living beings. But a weighty reason to find an application for this waste is that aspiration dust is obtained from a carefully selected and adjusted raw mix of clinker production, in which clay minerals and calcium carbonate (calcite) are burned at t = 400–500°C and at the same time retain an amorphous state. It is also important that it can be used without mechanical activation in its natural form (specific surface area S_{sp} = 220 m^2/kg).

An aqueous solution of low-modulus sodium liquid glass with a density of 1.42 g/cm³ and a silicate modulus of 2.8 (in terms of dry matter Na_2O - 6.2%) was used as an alkaline grout.

To improve the processes of forming the geopolymer paste structure and reducing pore space in the binder, mineral powder from volcanic tuff was added. The choice of the additive of volcanic origin is justified, since, firstly, it can be classified as an aluminosilicate raw material with the presence of a glass phase; secondly, this raw material has a sufficient degree of "ordering" of the structure; thirdly, when interacting with alkali metal cations, the formation of the necessary structures occurs. The matrix's structural elements are connected to them. The specific surface area of the volcanic tuff powder studied varied in the range 480-500 m^2/kg. The reaction activity was observed even when the samples were compressed with water. The strength limit of the compression test was 3.2 MPa.

A "rough" relief of the surface of the grains characterizes the microstructure of the particles of the volcanic tuff additive (Fig. 1) with closed porosity of the internal structure. This confirms the high solubility and activity of ash particles.



a) *b)*

Fig. 1. SEM images of volcanic tufa particles: a) ×2500 b)×5000.

Therefore, the addition of volcanic tuff (10% of the binder) will make up the percentage of aluminosilicates in the system due to the reduction of the concentration of the clinker component. The setting time of the alkaline binder has increased by 40 minutes, which facilitates high-quality preparation and molding of products.

The addition of sodium Na_2SiF_6 silicate (in the investigations was determined a dosage of 6% by mass of Na_2SiO_3) assists in the secretion of gel of polymerized silicon silica $Si(ON)_4$, a Parallel formed NaF increases the strength of the artificial paste.

Preliminary studies [2, 6] have established effective proportions for the combination of the following components: "Aspiration cement dust – volcanic tuff – Na_2SiO_3 – sodium silicate". The binding agent formulation is shown in Table 1.

Table 1. Mix proportions, wt. %.

Dust	Volcanic tuff	Na_2SiO_3	Na_2SiF_6
56	10	28	6

From the binding composition "aspiration dust – volcanic tuff – Na_2SiO_3 – sodium silicofluoride" (Table 1), sample series of 40×40×160 mm were prepared, using standard Volsky sand as the fine aggregate. The specimens were demolded the next day and then placed in a drying oven for heat treatment at a temperature of 80–110 °C for several hours (2–2.5 hours), with the process duration being one week.

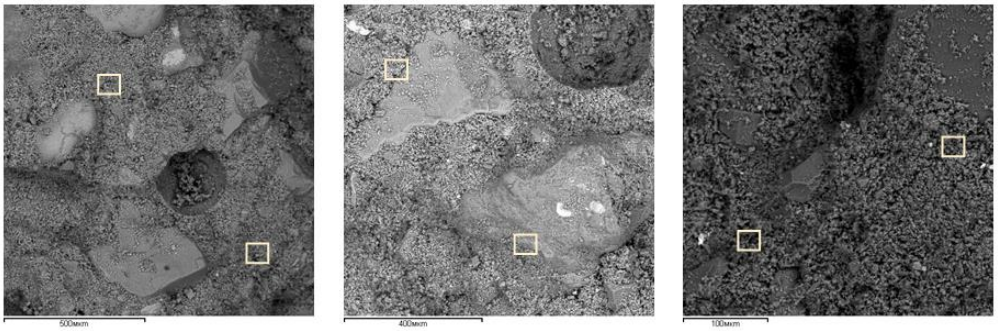
The specific surface area of the volcanic tuff powders was determined using a PSKh-12 instrument (Sudakov–Khodakov device, Russia).

The alkaline activator – as the main carrier of binding properties – was examined using an electronic pH meter with a measurement accuracy of 0.01 pH units; as a result, the hydrogen index of Na_2SiO_3 was pH = 12.6.

The microstructure of the prepared samples was studied using modern equipment; electron probe analysis was conducted using a Vega II LMU scanning electron microscope (Czech Republic), and X-ray phase and structural analyses were carried out using an ARLX'TRA diffractometer (Switzerland). Infrared spectroscopy was also performed using an IR Fourier spectrometer (Japan). The physical and mechanical properties of the binder material samples were determined in accordance with GOST 30744-2001.

3. RESULTS AND DISCUSSION

Analyzing the obtained data, it can be noted that the studied compositions are characterized by a fine-crystalline and heterogeneous crystalline structural organization (see Fig. 2c), with rounded closed pores with a radius of 0.25 mm (see Fig. 2a, b). The close interaction of the cement matrix and fine aggregate particles is clearly visible.



a b c

Fig. 2. SEM images of the studied binder in geopolymer concrete.

Hydrated sodium and calcium aluminosilicates play a key role in the structure formation. They often form dense crystal aggregates reaching sizes of 10–20 μm (Fig. 3, 4). Their component composition, according to semi-quantitative analysis, is presented in Tables 1 and 2 (Analyses 1 and 2). These phases interact with calcite and possibly with thin layers of $Ca(OH)_2$ (Table 2, Analyses 3–5), as well as with fine mica flakes. According to X-ray phase and electron probe analyses, the mica corresponds to muscovite. The measured composition of the flakes (about 20 μm) on average corresponds to the following formula: $(Na_{0.14}K_{0.63})_{0.8}(Fe^{2+}_{0.05}Mg_{0.02}Al^{VI}_{2.01}Ti_{0.01})_{2.1}(Si_{3.01}Al^{IV}_{0.99})_{4.0}O_{10}(OH_{2.00})_{2.0}$ confirming the qualitative composition with the presence of sodium and aluminum.

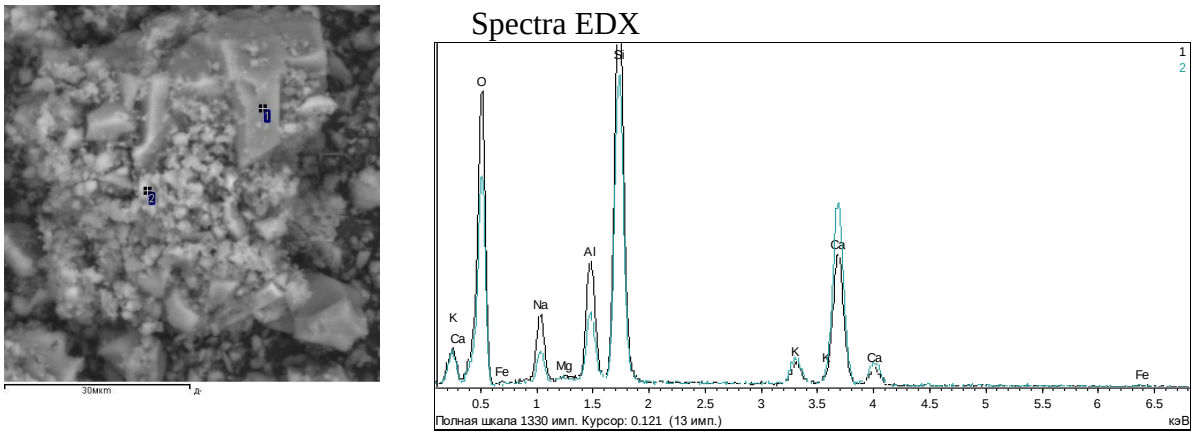


Fig. 3. Ground mass hydrates.

Table 2. Composition of the bulk hydrates, in wt.% (locations of analysis are shown in Fig. 3).

	Na_2O	MgO	Al_2O_3	SiO_2	K_2O	CaO	FeO	Total
1	8.17	0.20	12.53	46.99	2.04	17.72	0.44	88.09
2	4.12	0.20	6.74	35.84	2.56	24.44	0.50	74.41

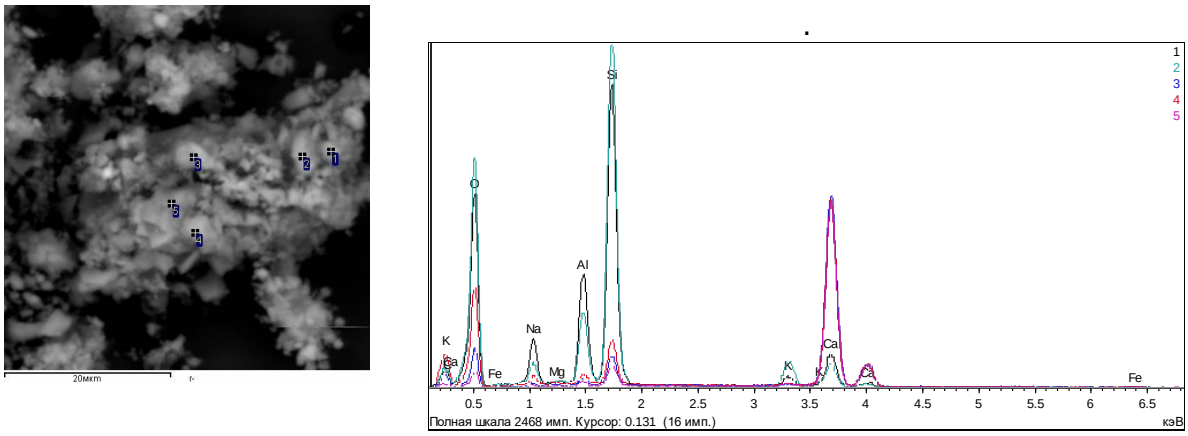


Fig. 4. Phases of the main mass.

Table 3. Composition of the main mass crystals, in wt.% (locations of analysis are indicated in Fig. 4).

	Na_2O	MgO	Al_2O_3	SiO_2	K_2O	CaO	FeO	Total
1	8.65	-	18.77	61.66	1.65	7.62	0.39	98.74
2	4.23	0.22	12.14	71.44	5.01	5.59	0.27	98.90
3	0.90	0.20	1.16	5.99	0.20	44.55	-	53.00
4	2.34	0.52	1.27	4.37	0.25	43.19	-	51.94
5	0.72	-	1.05	4.09	0.30	42.54	-	48.70

The results of X-ray phase analysis revealed the presence of quartz, feldspars close to albite and orthoclase, micas, calcite, and zeolites (Fig. 5).

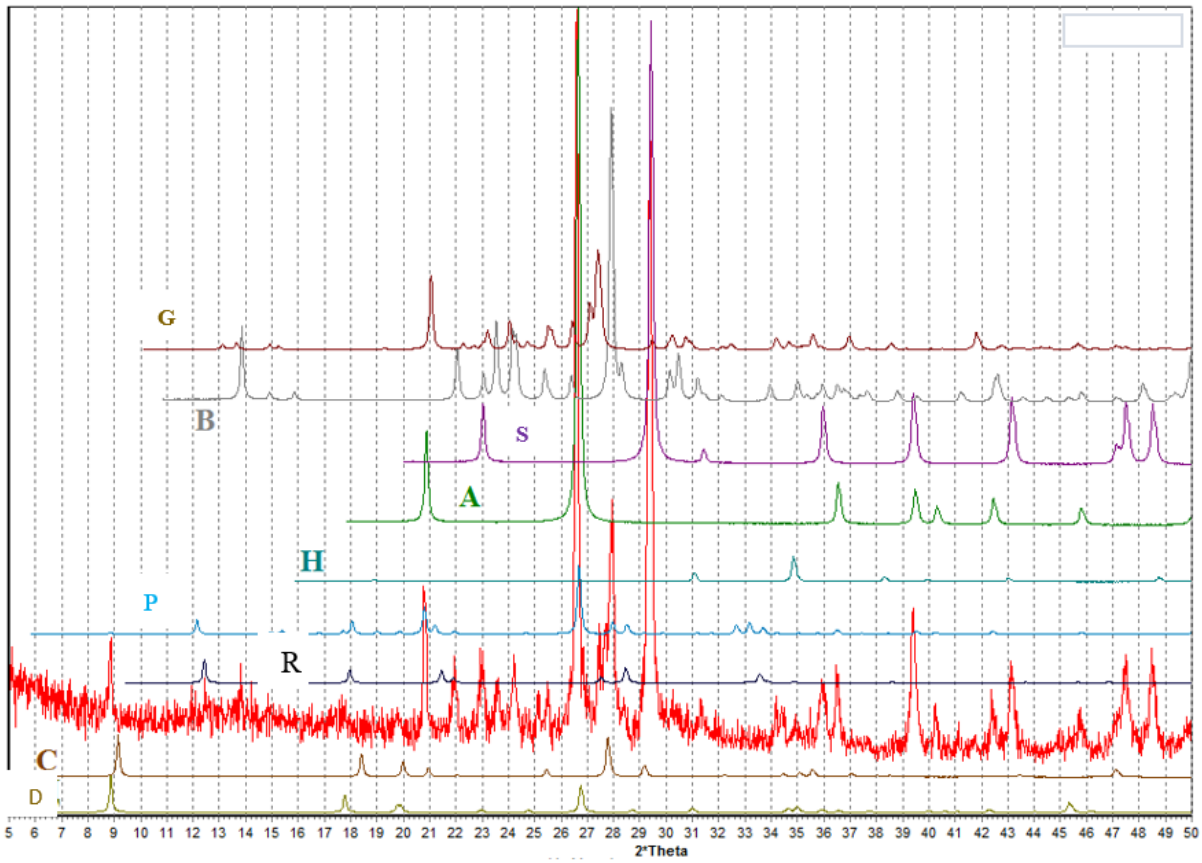


Fig. 5. Diffraction pattern of the sample "aspiration dust – volcanic tuff – Na_2SiO_3 – sodium fluorosilicate" compared with the PDF-2 database data. The comparison phases are given: A – quartz, S – calcite, B – albite, G – potassium feldspar, D – muscovite, H – analcime, P – gismondite, R – garronite, C – paragonite.

X-ray structural analysis shows that the zeolite under study has a structure similar to garronite, whose theoretical formula is $Na_2Ca_5Al_{12}Si_{20}O_{64}{\cdot}27(H_2O)$, although the actual composition may vary significantly. The key reflection of this phase is clearly defined at $2\Theta = 12.4$ (7.10 Å). Electron probe analysis reveals the presence of hydrated amorphous Na-Ca-Si compounds rich in water (see Figure 6 and Table 3). Additionally, phases with a similar composition containing aluminum and resembling zeolites by elemental composition are observed (see Fig. 7 and 8, as well as Table 4). Due to the small particle size and surface roughness of the samples, accurate quantitative measurements are difficult. However, it can be stated that the elemental composition is close to Ca-phillipsite or garronite, which are characterized by a SiO_2/Al_2O_3 ratio close to 2.

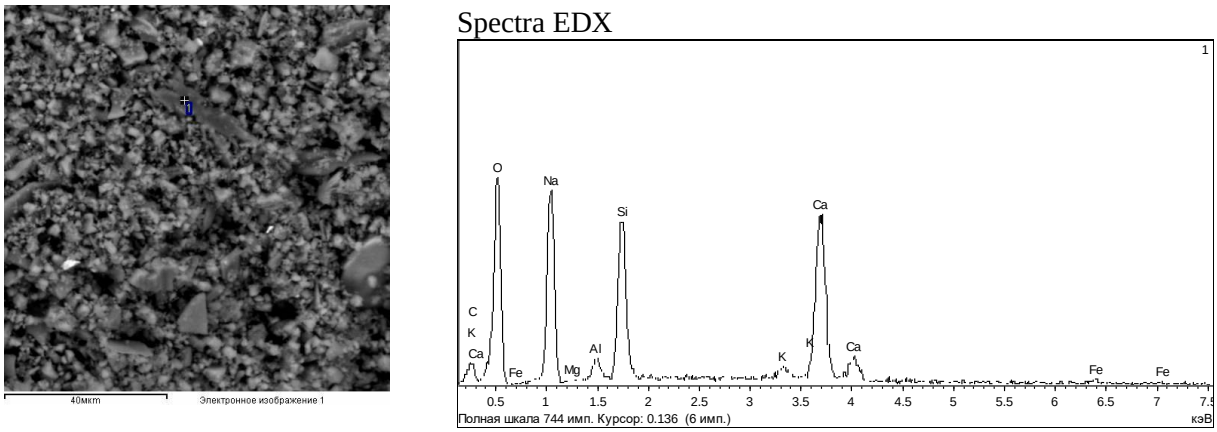


Fig. 6. Hydrated Na–Ca–Si compounds in the structure of the material.

Table 4. Composition of hydrated Na–Ca–Si compounds (shown in Figure 6), in wt.%.

	Na_2O	MgO	Al_2O_3	SiO_2	K_2O	CaO	FeO	Total
1	13.52	0.04	1.43	11.80	0.55	12.31	0.72	40.36

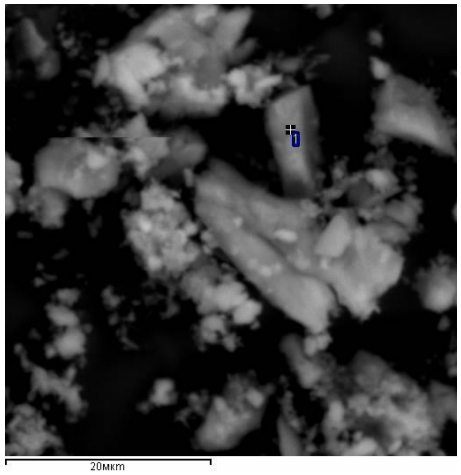


Fig. 7. Hydrated Na–Ca–Si compounds in the structure of the material.

Table 5. Example of the composition of the zeolite phase (shown in Fig. 7), wt.%.

	Na_2O	Al_2O_3	SiO_2	K_2O	CaO	FeO	Summary
1	5.10	17.09	40.93	0.37	6.48	0.12	70.09

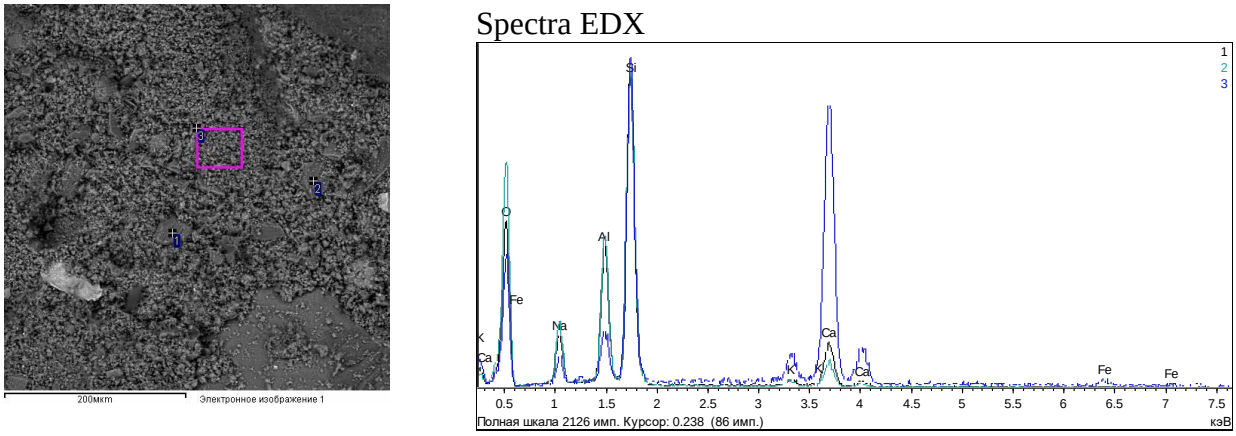


Fig. 8. EDX spectra of crystals close to zeolites.

Crystals of calcium hydroaluminates are developed in the microcavities (Fig. 9).

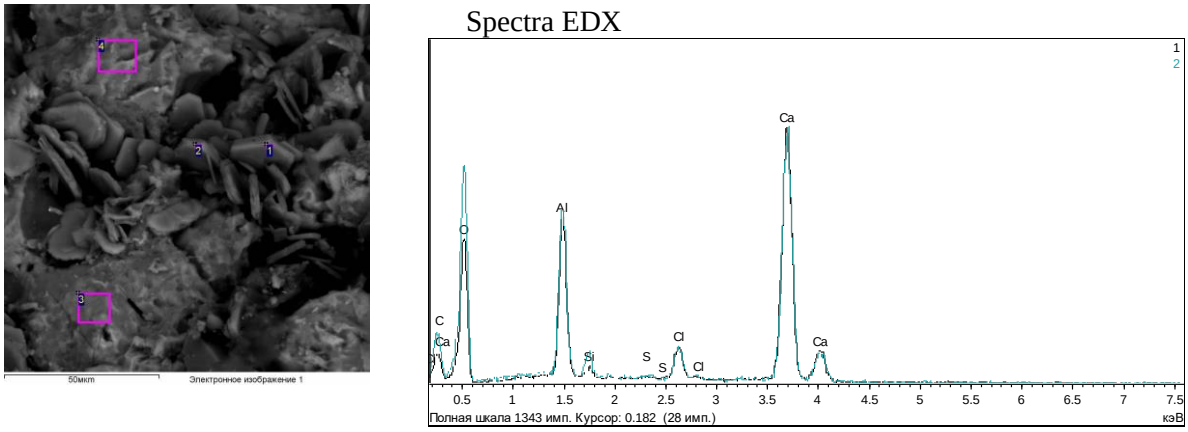


Fig. 9. Crystals of calcium hydroaluminates (1 and 2).

Table 6. Results of the analysis of the aggregate of calcium hydroaluminate crystals (1, 2) and the enclosing mass (3, 4) (the analysis areas are shown in Figure 9), wt.%.

	Na_2O	MgO	Al_2O_3	SiO_2	SO_3	K_2O	CaO	TiO_2	MnO	FeO	Total
1	-	-	17.86	1.25	0.20	-	34.66	-	-	-	53.97
2	-	-	18.41	3.39	0.31	-	33.81	-	-	-	55.92
3	0.40	1.23	5.32	38.14	0.18	0.12	37.36	0.14	0.01	1.23	84.13
4	0.31	1.20	4.32	33.59	-	0.10	35.91	0.01	0.01	0.58	76.03

On the DTA curve (Fig. 10), the formation of the specified compounds is confirmed by the presence of endoeffects: 80–2000 ºC – intense mass loss with heat absorption, Δm ≈ 30–35%, max – 1300 ºC. At temperatures of 220 – 4700 ºC, there is a uniform mass loss, at 330 ºC, an endoeffect is observed. Starting from 470 ºC, there is a more intense mass loss, at 580 – 590 ºC there is a small endoeffect. Starting from 740-860 ºC – there is a very intense mass loss Δm ≈ 30 % with heat absorption, max – 8400 ºC.

Fig. 10. DTA of hydration products “aspiration cement dust – volcanic tuff – Na2SiO3 – sodium fluorosilicate”.

4. CONCLUSIONS

The analysis of the obtained results from electron microscopy, XRF, electron probe analysis, differential thermal analyses, and infrared spectroscopy confirms the presence of the following minerals in the cement stone: hydrated compounds of sodium and calcium aluminosilicates, sodium and calcium silicates, calcium aluminates, calcite, feldspars, quartz, albite, orthoclase, and muscovite.

The formation mechanism of submicron crystalline formations is based on the chemical interaction of crystalline calcite and alkaline hydroaluminosilicate gel. The additive of volcanic nature had a positive effect on the composition of hydration products both in the contact zone and in the interpore space of the geopolymer paste. Amorphous silica in the composition of the volcanic additive lowers the pH of the environment insignificantly, but independently forms a structure of quartz and polymerized silicic acid.

The results of the studies confirm the effectiveness of the development data, which shows:

1. The components of the binder system “aspiration dust – volcanic tuff” exhibit a certain degree of amorphousness, and when mixed with an alkaline activator, the processes of hydration product interaction and geopolymer transformations are intensified.
2. The most effective formulation of the binding bond «Aspirational cement dust - volcanic tuff - Na_2SiO_3 - siliceous sodium» was established, the presence of aluminosilicate with the presence of glass phase promotes the development of own structural formations, matrix binding to structural elements.
3. Comprehensive study of microstructure confirm the presence in of difficult to dissolve and strong joints in geopolymer paste guaranteeing its durability.

Thus, the growing problems of resource and energy conservation in recent decades have encouraged the scientific community and industry to actively develop effective alkali-activated binders. These new materials represent an alternative to traditional cements, which require significant energy costs during production. The research shows that the use of alkali-activated binders, such as geopolymers and various equivalents based on mineral additives, can significantly reduce the carbon footprint of the construction sector in the environment.

All data, models, and code generated or used during the study appear in the submitted article.

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