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Porous aggregate developed with the use of coal-containing clays of the Angren field

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Abstract. Relevance. One of the ways to solve the issues of resource and energy conservation is the production and use of porous aggregates. Porous aggregates allow obtaining effective lightweight concrete for thermal insulation, wall panels, monolithic walls and other load-bearing structures, contribute to the increase of energy efficiency, improvement of thermal insulation, reliability, increase of fire resistance, frost resistance and seismic resistance of buildings, reduction of their weight. Therefore, in the production of porous aggregates, the primary task is the use of industrial waste and products of their processing. Solving these problems leads not only to saving valuable natural resources, but also to solution of environmental problems. **Aim.** Development of compositions and study of properties of porous aggregate based on bentonite clay of Navbakhar deposit and coal-containing clay of Angren brown coal deposit. **Object.** Coal-containing clay of Angren brown coal deposit, bentonite clay of Navbakhar deposit and artificial porous aggregate based on them. **Methods.** Chemical, energy-dispersive X-ray, X-ray phase and infrared spectroscopic analysis, scanning electron microscopy, mathematical modeling, etc. **Results.** The authors have determined chemical and mineralogical compositions of the clays used. Using the mathematical modeling method they developed the regression equations describing the effects of the amount of bentonite clay in the batch, firing temperature and isothermal holding time on the bulk density and water absorption of the porous aggregates. The resulting porous aggregates with a bulk density of 395 to 690 kg/m³ have a compressive strength in a cylinder of 2.74 to 6.46 MPa, respectively. It was found that the aggregates obtained meet the requirements of regulatory documents.

Keywords: mineral rocks, industrial waste, secondary materials, porous aggregate, composition, optimization, batch, coal-containing clay, bentonite clay, glass phase, lightweight concrete

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Пористый заполнитель, разработанный с использованием углесодержащей глины Ангренского месторождения

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Аннотация. Актуальность. Одним из путей решения вопросов ресурсо- и энергосбережения является производство и применение пористых заполнителей. Пористые заполнители позволяют получать эффективные легкие бетоны для теплоизоляции, стеновых панелей, монолитных стен и других несущих конструкций, что способствует повышению энергоэффективности, улучшению теплоизоляции, надежности, повышению огнестойкости, морозостойкости и сейсмостойкости зданий, снижению их массы. Поэтому при производстве пористых заполнителей первоочередной задачей является использование отходов промышленности и продуктов их переработки. Решение этих задач с одной стороны приводит к экономии ценного природного сырья, а с другой – к решению экологических задач. **Цель:** разработка составов и исследование свойств пористого заполнителя на основе углесодержащей глины Ан-

грэнского буроугольного месторождения и бентонитовой глины Навбахорского месторождения. **Объект:** углесодержащая глина Ангренского месторождения бурого угля, бентонитовая глина Навбахорского месторождения и искусственный пористый заполнитель на их основе. **Методы:** химический, энергодисперсионный рентгеновский и рентгенофазовый анализы, инфракрасная спектроскопия, сканирующая электронная микроскопия, математическое моделирование и др. **Результаты.** Определены химический и минералогический составы использованных глин. Методом математического моделирования разработаны уравнения регрессии, описывающие влияние количества бентонитовой глины в шихте, температуры обжига и времени изотермической выдержки на насыпную плотность и водопоглощение пористого заполнителя. Полученные пористые заполнители с насыпной плотностью от 395 до 690 кг/м³ имеют соответственно прочность при сдавливании в цилиндре от 2,74 до 6,46 МПа. Установлено, что полученные заполнители отвечают требованиям нормативных документов.

Ключевые слова: минеральные породы, промышленные отходы, вторичные материалы, пористый заполнитель, состав, оптимизация, шихта, углесодержащая глина, бентонитовая глина, стеклофаза, легкие бетоны

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Introduction

At present, the solution of problems of increasing resource and energy conservation of building materials and products by reducing material and energy costs both in their production and in the construction and operation of buildings is very relevant. This is due to the fact that a significant part of energy costs falls on the stages of manufacturing building materials and products and the operation of construction objects [1–4].

Energy and resource conservation is achieved primarily through the use of energy-saving materials and their production technologies. One of the ways to solve these problems is to reduce energy loss in residential buildings, energy consumption for heating in winter and for air conditioning in summer by increasing the heat transfer resistance of enclosing structures [1, 4–5]. In this regard, lightweight concrete on artificial porous aggregates occupies an important place for their production in modern industrial construction from precast and monolithic products and structures. Porous aggregates make it possible to obtain effective lightweight concrete for thermal insulation, wall panels, monolithic walls and other load-bearing structures, and as a result, the use of lightweight concrete based on them helps to increase energy efficiency, improve thermal insulation, reliability, increase fire resistance, frost resistance and seismic resistance of buildings, reduce their weight, save resources and costs in construction [4–6].

In Uzbekistan, expanded clay gravel is practically the only porous aggregate produced [2]. As is known, various types of raw materials of natural and artificial origin are used to produce expanded clay. Among the natural ones, clay rocks, some stone-like clay rocks – clay shales, argillites, which, when fired at high speed in the temperature range of 1000–1250°C, form light porous materials [7–11].

A distinctive feature of clays for the production of expanded clay is swelling during firing, characterized

by a swelling coefficient that depends on the chemical and mineralogical composition of the raw materials used [7–9, 11, 12]. Therefore, the suitability of clay raw materials for the production of expanded clay is established by studying its properties.

Many studies [7–19] are known related to improving the technological properties of clays, to obtain a porous aggregate, adding various mineral and organic additives and improving the quality of the resulting material. All these studies are aimed at increasing the swelling coefficient of the original raw materials, reducing the firing temperature, increasing the output of expanded clay, improving its quality and reducing the cost. In this direction, the works of scientists from Uzbekistan are also known [20–27], which are aimed at obtaining expanded clay and expanded clay-like lightweight aggregates from local raw materials. For example, in their studies, they used coal-containing clay and loam as raw materials for obtaining artificial aggregates [20, 21], 10% coal-containing clay and 90% dacite porphyry [21], 20% clay and 80% quartz porphyry containing coal industry waste [22], an additive to bentonite clay of loess rocks and waste of the cardboard and paper industry [25], dune sands and oil waste [24], waste – soda production sludge and licorice root processing waste [25–27]. The analysis shows that they achieved a decrease in the firing temperature of expanded clay gravel granules to 1100°C and, at the same time, the obtained material met the strength requirements of regulatory documents [28]. However, due to the complexity of the batch composition, such a porous aggregate is not produced.

Despite this, as it is noted above, in Uzbekistan, porous aggregates are used to produce almost only expanded clay gravel, the quality and volume of production of which cannot ensure the implementation of the industrial construction plan for civil and industrial facilities in the future due to the limited reserves of clay for its production and low mechanical strength. There-

fore, lightweight concretes based on artificial porous aggregates from local raw materials and industrial waste are of particular importance for construction, which allows for a significant expansion of the mineral base and reduction in production costs. Despite this, the primary task in the production of porous aggregates is the use of industrial waste and products of their processing. Solving these problems leads not only to saving valuable natural resources, but also to solution of environmental problems.

The way out of the current situation in the production of porous aggregates on an industrial scale is possible by replacing the scarce clay raw materials (montmorillonite, argillite and other clays) with waste from the Angren coal deposit, containing kaolinite clay and up to 40% coal (coal containing kaolinite clay). Local rocks – bentonite clays, as low-melting and widespread in the Navoi region, are used as the second component of the porous aggregate, which allows for a sharp reduction in energy and resource costs.

Geological characteristics

The Navbakhar bentonite clay deposit is located in the Navbakhar district of the Navoi region, on the southwestern slope of the South Nurata ridge, 12 km north of the village of Kalkanota. Confirmed reserves amount to 7142.6 thousand tons. Industrial horizons of bentonite clays are located in the Eocene section and form the wings of an asymmetric synclinal fold extending for a distance of 3 km along the sublatitudinal [29, 30]. Clay is mined by open-pit mining (Fig. 1, *a*). Bentonite (Fig. 1, *b*) and carbonate-palygorskite clays of the Navbakhar deposit are confined to the Lower Eocene section (Ypresian stage) [31].

In the mineral composition of bentonite clays, montmorillonite predominates, the content of which reaches 80%, and illite is secondary (10–25%). Quartz,

cristobalite, iron hydroxides, calcite, palygorskite, halloysite, alunite, jarosite, etc. are found as impurities. According to their physicochemical properties, they are divided into alkaline and alkaline-earth varieties [32–34]. Bentonite clays of the deposit overlap carbonate-palygorskite clays. To obtain a porous aggregate, we will use alkaline bentonites of this deposit. They dissolve slowly in water, but swell strongly [33, 34].

The Angren brown coal deposit is located in the Akhangaran River valley, southeast of Tashkent, in the Chatkal-Kurama region of the Middle Tien Shan [34]. The deposit is confined to Jurassic deposits of two suites: coal-bearing (lower and middle sections) and kaolin (upper section) [35–38]. It is characterized by a specific structure and lithological-petrological composition of the pre-Mesozoic basement, has a complex of Jurassic formations inherent only to it and is distinguished by multiple activation of Mesozoic Cenozoic volcanic activity [36].

The geological structure of the deposit includes the following deposits: Quaternary (loess-like loams), Paleogene Turkestan and chalky (clays), Jurassic kaolin (variegated kaolins), Jurassic supracol (gray kaolins and argillites), Jurassic coal (intrastratal rocks), Paleogene (limestones), Paleozoic (primary kaolins), Jurassic (coal). The coal deposit is a complex coal bed with a thickness of 7 to 70 m [34]. The area of distribution of the coal-bearing suite within the Angren deposit is 70 km² (Fig. 2). The main coal content of the deposit is represented by a thick brown coal bed consisting of two packs (Fig. 3). The thickness of the entire coal deposit varies from 40 m on the southeastern wing of the syncline to 100 m on the western wing; to the south its thickness decreases to 5 m [36].



a



b

Fig. 1. *a)* Navbakhar deposit of bentonite clay; *b)* bentonite clay

Рис. 1. *a)* Навбахарское месторождение бентонитовой глины; *b)* бентонитовая глина

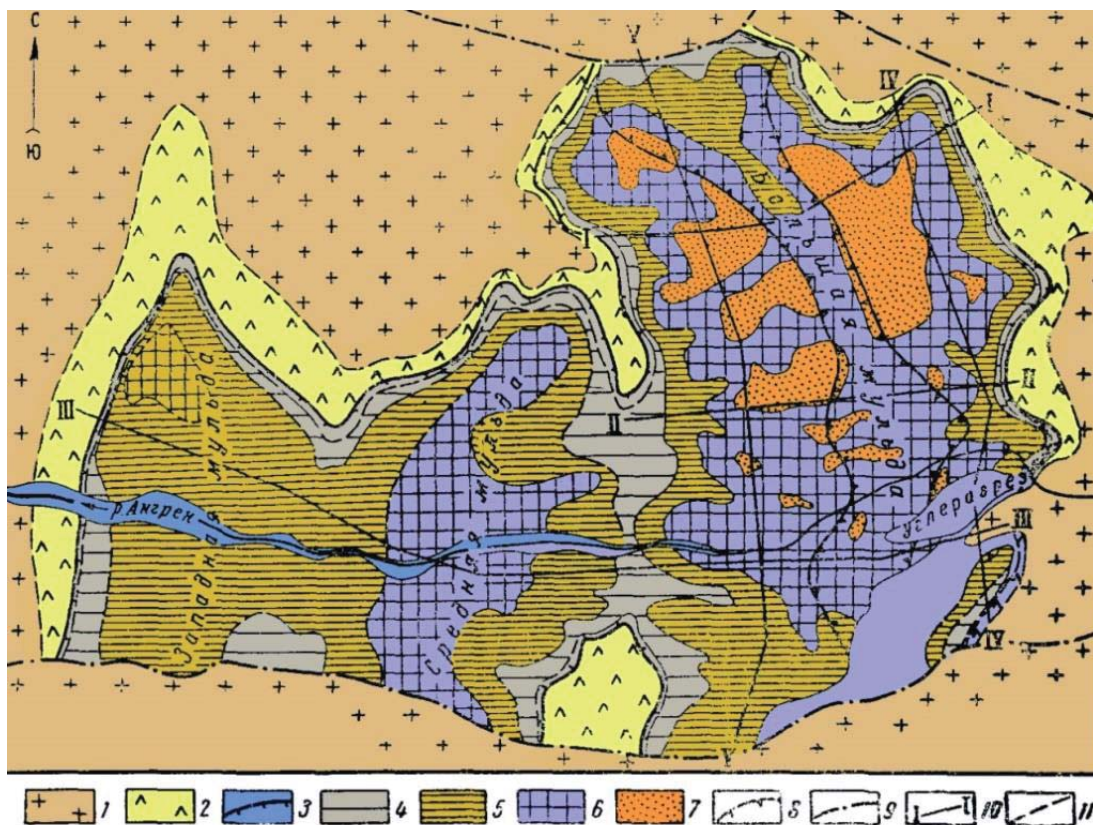


Fig. 2. Angren brown coal deposit. Zoning of formations underlying the kaolin suite and Cretaceous–Paleogene deposits [32]: 1 – complex of volcanic rocks (Pz3); 2 – weathering crust (T-J); 3 – boundary of the distribution of the coal-bearing formation. Zones of equal thickness of coal deposits: 4 – up to 10 m; 5 – 10–30 m; 6 – 30–50 m; 7 – over 50 m; 8 – zone of formation dilution; 9 – lines of violations; 10 – lines of structural-facial profiles; 11 – estimated contours

Рис. 2. Ангренское бурогольное месторождение. Зональность формаций, залегающих под каолиновой свитой и мел-палеогеновыми отложениями [32]: 1 – комплекс вулканогенных пород (Pz3); 2 – кора выветривания (T-J); 3 – граница распространения угольной свиты. Зоны равных мощностей угольной залежи: 4 – до 10 м; 5 – 10–30 м; 6 – 30–50 м; 7 – свыше 50 м; 8 – зона разубоживания пласта; 9 – линии нарушений; 10 – линии структурно-фациальных профилей; 11 – предполагаемые контуры

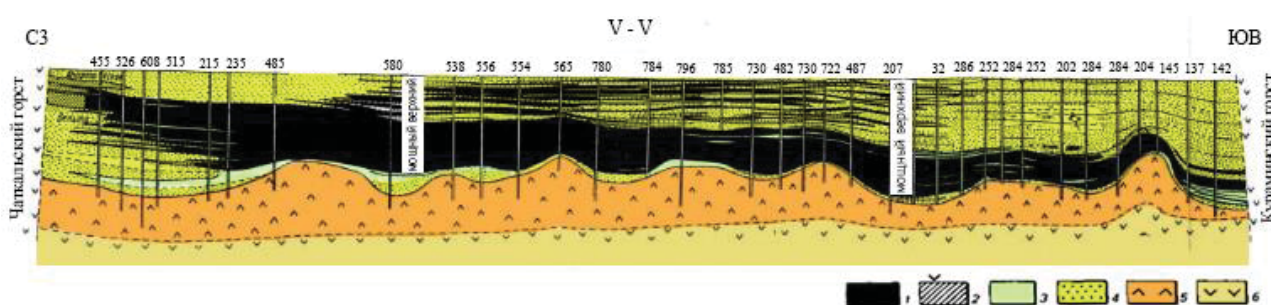


Fig. 3. Geological profile of the Angren brown coal deposit [32]: 1 – coal; 2 – interlayering of coal and rock, clayey coals; 3 – siltstones and clays; 4 – sandstones; granular sandstones; 5 – weathering crust; 6 – effusive rocks of the Upper Paleozoic, not affected by weathering

Рис. 3. Геологический профиль Ангренского бурогольного месторождения [32]: 1 – уголь; 2 – переслаивание угля и породы, угли глинистые; 3 – алевролиты и глины; 4 – песчаники; зернистые песчаники; 5 – кора выветривания; 6 – эффузивные породы верхнего палеозоя, не затронутые выветриванием

The rocks are taken to external and internal waste dumps (Fig. 4). The external waste dumps are located on the left bank of the Angren River, they extend for several kilometers and occupy an area of more than 1,200

hectares. The internal waste dumps of the open pit are intended for the separate storage of gray, variegated and intercoal kaolins, which are raw materials for the ceramic, refractory and aluminum industries [38].



Fig. 4. a) Angren brown coal deposit quarry; b) coal-containing clay

Рис. 4. а) Ангренский карьер бурогоугольного месторождения; б) углесодержащая глина

The state balance of mineral reserves as of 01.01.2016 on the territory of the Republic of Uzbekistan takes into account reserves of expanded clay raw materials – 16 deposits, amounting to 128174 thousand m³ in categories A + B + C1, C2 – 47156 thousand. Off-balance reserves – 20894 thousand m³ [37, 38]. However, in the production of porous aggregates, the primary task is the use of industrial waste and products of their processing. Solving these problems leads not only to saving valuable natural resources, but also to solution of environmental problems. Therefore, the priority problems and tasks that need to be solved, first of all, include the development of methods for obtaining substances with a controlled structure, which will allow the creation of materials with unusual mechanical properties, density, porosity, etc. [39].

The aim of the research is to develop compositions and study the properties of porous aggregate based on bentonite clay of the Navbakhar deposit and coal-containing clay of the Angren brown coal deposit.

Materials and methods

The coal-containing clay was obtained from the Angren brown coal deposit, 62 m below the surface, between two coal seams. Bentonite clay was obtained directly from the Navbakhar bentonite clay deposit located in the Navoi region. The chemical composition of the clays used in the studies was determined at the Institute of General and Inorganic Chemistry of the Academy of Sciences of the Republic of Uzbekistan.

The elemental composition of the studied clay samples was obtained using a Zeiss EVO MA 150 scanning electron microscope (SEM) equipped with an Oxford Instruments X-act instrument for energy-dispersive X-ray (EDX) analysis at an accelerating voltage of 20 keV. These studies were conducted at the Center for Advanced Technologies of the Ministry of Higher Education, Science and Innovation of the Republic of Uzbekistan. X-ray phase analysis was per-

formed using a Rigaku MiniFlex 600 diffractometer with a copper anode.

IR spectroscopic studies were carried out on a SHIMADZU infrared Fourier spectrometer (range 400–4000 cm⁻¹, resolution 4 cm⁻¹). The spectra were interpreted using basic software that implements automatic measurement of spectra, has means for graphic display of spectra and their fragments and forms work with the user's spectrum library.

DTA and TGA analysis was performed on a SHIMADZU DTG-60 instrument (Japan). The studies were carried out in an argon environment of 80 ml/min, the heating rate was 10°C/min.

Optimization of the compositions of the aggregate mixture, sintering technological modes, was carried out using the method of mathematical planning of experiments [40, 41]. To describe the studied properties, a second-order multifactorial plan with three variable factors was used.

$$Y = b_0 + b_1x_1 + b_2x_2 + b_3x_3 + \dots + a_nx_n^2,$$

where Y is the optimization parameter; x_1, x_2, x_3 are the variable factors; b_0, \dots, b_n are the coefficients of the regression equation.

Preparation of materials

Raw clays were dried in a laboratory drying chamber at a 105°C for 24 hours. The dried clays were ground in a ball mill, after which they were sifted through a 0.14 mm sieve.

To prepare a batch of porous aggregates granules, dry clays were mixed in the required proportions in a laboratory mixer until a homogeneous mass was obtained. Then 20–22% water was added to this mass and mixed until a homogeneous plastic dough was obtained. Granules of porous aggregate were prepared in a laboratory extruder. The rod coming out of the extruder is cut with a knife into pieces of 5–10 mm long. Raw grains were dried in a drying cabinet at 110°C to a constant mass for 5 hours or more. The dried aggregate

grains were fired in a chamber furnace heated to 1100°C. The characteristics of the porous aggregate were determined in accordance with current standards [28].

Results and discussion

Characteristics of clays

The term clay refers to a natural material consisting mainly of fine-grained minerals, which is usually plastic in adequate water content and hardens when dried or fired [42, 43].

Clay minerals such as kaolinite, smectite, chlorite, micas are the main components of clay raw materials and are formed in the presence of water. They are known as hydrous layered silicate containing silica, alumina and water with variable amounts of inorganic ions such as Mg^{2+} , Na^+ , Ca^{2+} , which are either in the interlayer space or on the planetary surface. Clay minerals are also characterized by the presence of two-dimensional sheets of tetrahedral (SiO_4) and octahedral (Al_2O_3) [44].

Analysis of the chemical composition of the studied clays (Table 1) showed that the studied samples have approximately the same silica content, while the metal oxide content varies. In both clays, silicon and aluminum oxides are the main rock-forming elements. The $SiO_2:Al_2O_3$ ratio when recalculated for coal-containing clay is 2, and for bentonite clay is 3.52. The significant iron content in bentonites indicates that it belongs to the montmorillonite mineral type. As is known [45], iron and magnesium can enter the montmorillonite lattice, partially replacing aluminum. Magnesium oxide in the studied clays is 0.21 for coal-containing clay and 2.19 for bentonite clay, respectively. The amount of potassium oxide in the clays is 0.815 and 2.53.

As is known [7, 8, 11–17, 46–51], artificial lightweight aggregates are formed by rapid heating at high temperature of materials with the ability to expand. The structure of the artificial porous aggregates during firing is formed due to the formation of gas and very viscous liquid phase. After cooling, the liquid phase turns into glass and forms voids [44–46]. As a result of firing, the material is exposed to high temperatures due to the decomposition of organic and inorganic components, gases in the form of CO , CO_2 , SO_2 , H_2O , O_2 and N_2 can be released. An increase in temperature and gas concentration (gas pressure) leads to an expansion of the volume of the material, known as swelling.

SEM image of the surface of the coal-bearing clay sample (Fig. 5) shows that it is rough and porous. EDX analysis of its profile (Fig. 6) shows a strong signal of elemental silicon and aluminum exhibiting an optical absorption band with a maximum at 1.75–1.8 keV. There are also weak signals related to other chemicals (Ca, Ti and Fe). Table 2 presents the mineral components of the coal-bearing clay sample obtained by EDX analysis.

Analyzing the obtained results of the material and mineralogical composition of coal-containing clay, it can be assumed that a low amount of gas-forming substances during firing may not give the expected results in swelling. Also, the coal contained in this clay, due to the high melting point of this clay, will not be able to form the required porosity of the porous aggregate. However, scientists argue that this does not necessarily mean that dry granules will not swell, since a continuous increase in temperature partially hides the real possibility of expansion, since the samples do not have time to complete swelling before melting begins [50]. To improve the pyroplasticity of clay and increase the swelling coefficient, it is necessary to add additives containing a sufficient amount of gas-forming substances and the clay must reach pyroplasticity at the moment of gas formation.

According to the SEM image (Fig. 7), the surface of the bentonite clay sample is rough and porous. EDX analysis of its profile (Fig. 8) shows a strong signal of elemental silicon and aluminum, which also demonstrate an optical absorption band with a maximum at 1.75–1.8 keV. There are also weak signals related to other chemical substances (Ca, Ti, Na, K, Mg and Fe). Table 2 shows the mineral components of the coal-containing clay sample obtained by EDX analysis.

Analyzing the obtained data from the study of Navbahor bentonite clay, we can conclude that this clay can even be used as an independent component for obtaining an artificial porous aggregate.

X-ray phase analysis of the studied clays showed (Fig. 9, a) that the X-ray diffraction pattern of coal-containing clays shows peaks characteristic of kaolinite, quartz, and a small amount of illite and calcite. Clay minerals in coal shales consist mainly of kaolinite and illite [51], whereas marine shales contain abundant layers of illite or illite-smectite and only a small amount of kaolinite.

Table 1. Chemical composition of clay samples

Таблица 1. Химический состав проб глин

Name of clay Наименование глины	Content in %, per air-dry substance/Содержание в %, на воздушно-сухое вещество								
	SiO_2	Al_2O_3	MgO	SO_3	K_2O	CaO	TiO_2	Fe_2O_3	loss on ignition ппп
Coal containing clay/Углеродсодержащая глина	57.7	28.8	0.21	0.165	0.815	1.04	0.948	0.895	9.08
Bentonite clay/Бентонитовая глина	55.3	15.7	2.19	1.24	2.53	3.31	0.8	6.81	12.12

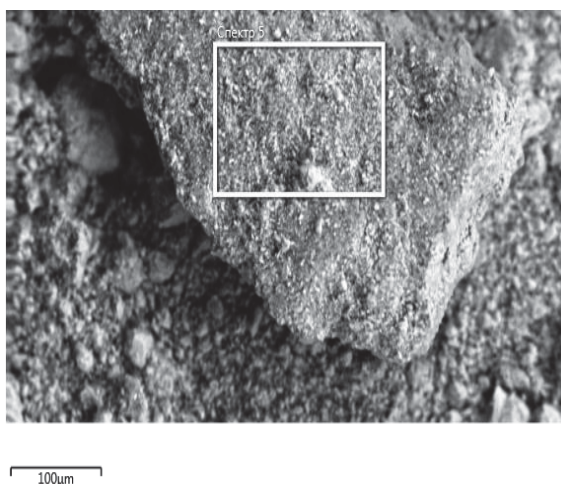


Fig. 5. SEM image of a sample of coal-containing clay
Рис. 5. СЭМ-изображение образца углесодержащей глины

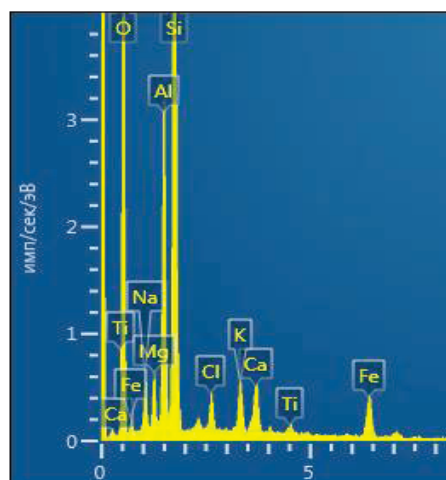


Fig. 8. EDX analysis of a bentonite clay sample
Рис. 8. EDX-анализ образца бентонитовой глины

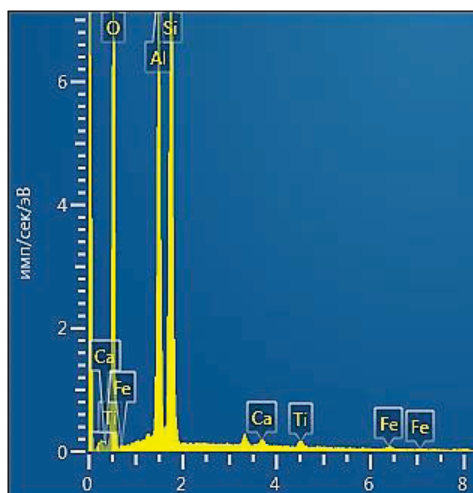


Fig. 6. EDX analysis of a sample of coal-containing clay
Рис. 6. EDX-анализ образца углесодержащей глины

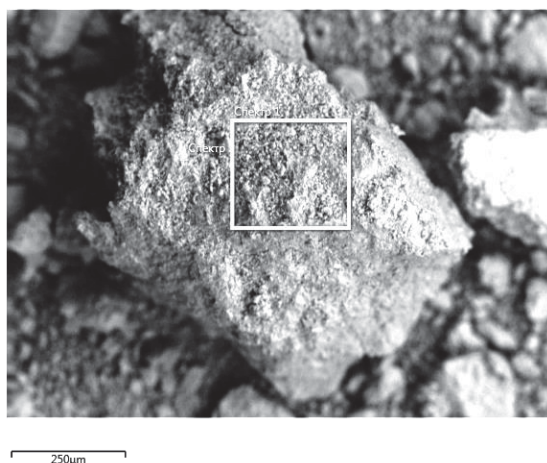


Fig. 7. SEM image of a bentonite clay sample
Рис. 7. СЭМ-изображение образца бентонитовой глины

Table 2. Mineral components of the studied clay samples according to EDX analysis

Таблица 2. Минеральные компоненты исследуемых образцов глин по EDX-анализу

Name of clay Наименование глины	Elements Элементы	Conditional concentration, % Условная концентрация, %	Mass, % Масса, %
Coal-containing clay Углесодержащая глина	C	0.10	5.06
	O	9.47	54.81
	Al	2.33	15.06
	Si	3.19	23.75
	Ca	0.05	0.34
	Ti	0.08	0.57
	Fe	0.06	0.40
Bentonite clay Бентонитовая глина	O	5.97	49.88
	Na	0.25	2.35
	Mg	0.16	1.73
	Al	0.93	8.77
	Si	2.67	25.77
	Cl	0.14	1.34
	K	0.26	2.18
	Ca	0.27	2.22
	Ti	0.06	0.54
	Fe	0.54	5.21

The kaolin group clay minerals consist of 1:1 dioctahedral layered structures with the general composition of $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$. The polytypes are kaolinite, dickite and nacrite [52]. The crystal system type and class of kaolinite known as kaolinite 1A mineral has been identified. The results of polyquantitative X-ray phase analysis show that the content of clay minerals in the sample is 79.46%, indicating that clay minerals are the major component. Kaolinite is the dominant component of clay minerals with an average content of 79.26%.

Illite is a dioctahedral 2:1 layered silicate commonly found in soils and sedimentary rocks. The term illite is used for 2:1 minerals with a non-expanding layer and a

wide range of chemical compositions [52]. The quartz content of this clay was 20.54%.

From the analysis of the oxide content in the Navbakhar bentonite clay (Table 1) it can be seen that SiO_2 and Al_2O_3 also dominate in this clay. Unlike the coal-containing clay, its content of other oxides is much higher. The results of the X-ray phase analysis of this clay showed (Fig. 9, b) that the X-ray diffraction pattern of the clay contains diffraction maxima related to the montmorillonite mineral, a widespread clay mineral from the smectite group of the layered silicate subclass. The most widespread smectite mineral is Ca-montmorillonite, which means that the charge deficit of the layer is compensated by the interlayer calcium cation and water [53]. The montmorillonite content was 74%. In addition to the montmorillonite mineral (74%), the X-ray diffraction pattern of this clay also contains diffraction lines of quartz, calcite and traces of palygorskite. The Navbakhar deposit is considered the only deposit in the republic where three varieties of bentonite clay were discovered together: alkaline, alkaline earth and palygorskite [29, 30].

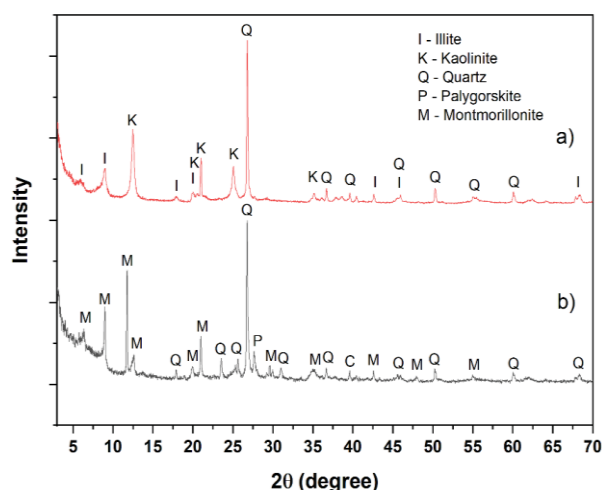


Fig. 9. Xray diffraction patterns of coal-containing clay (a) and bentonite clay (b)

Рис. 9. Рентгенограммы угля содержащей глины (a) и бентонитовой глины (b)

Fig. 10 shows the infrared spectra of the studied clay samples. In this work, the assignment of the IR bands of the mineral spectra was used according to literature data [54–61]. In the IR spectrum of the coal-containing clay (Fig. 10, a), the bands at 3696, 3650 and 3620 cm^{-1} are characteristic of the kaolin group. The bands at 3696 cm^{-1} and about 3620 cm^{-1} arise from internal surface OH groups [54–56]. The bands at 793 (Si-O) and 698 cm^{-1} (Si-O) are also diagnostic for kaolinite. A wide OH stretching band in the region of 3620 cm^{-1} in combination with a doublet of 831 cm^{-1} , 750 cm^{-1} (Fig. 10, a) indicates illite [57, 58]. These

bands arise as a result of deformation of Al-Mg-OH [59]. The spectrum also included negative bands of kaolinite, however, the doublet of the valence bands of quartz $\{797, 778\}\text{ cm}^{-1}$ given in the literature [56, 60] could not be detected.

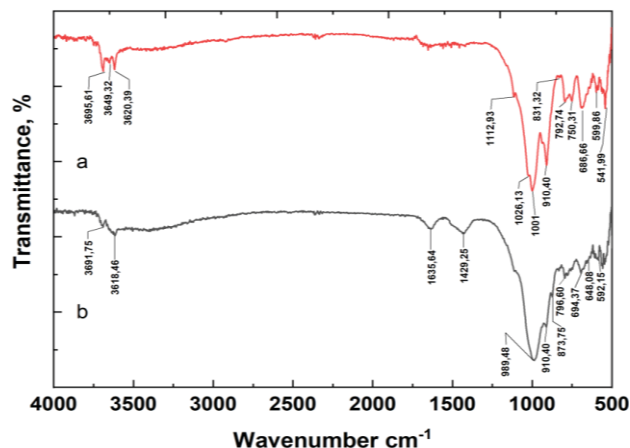


Fig. 10. Infrared spectra of coal-containing clay (a) and bentonite clay (b)

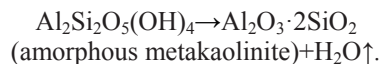
Рис. 10. Инфракрасные спектры угля содержащей глины (a) и бентонитовой глины (b)

The IR spectrum of Navbakhar bentonite clay is shown in Fig. 10, b. The main component of bentonite is montmorillonite, which is the most widely known representative of the smectite group. Based on a comparative analysis of the spectra (Figs. 10, a and 10, b) of kaolinite and montmorillonite, the band at about 3695 cm^{-1} (absent in the spectrum of montmorillonite) was attributed to the intra-surface OH groups of kaolinite (these groups are absent in the structure of montmorillonite), the band at 3620 cm^{-1} , which is common to the spectra of kaolinite and montmorillonite, was attributed to internal OH groups [59]. In the spectrum of the sample, an intense band is observed at 1634 cm^{-1} , attributed to the OH deformation of water. The doublet at 797 and 777 cm^{-1} indicates the presence of quartz impurity in the sample [56, 61], which is confirmed by X-ray phase analysis.

The studied clays were also subjected to thermal analysis. The analyzer temperature range was set at $28\text{--}1000^\circ\text{C}$ with a heating rate of 10°C per minute in an argon atmosphere. Thermogravimetric and differential thermal analysis (TGA/DTA) of the studied clays at these temperatures (Fig. 11, 12) showed that their thermal decomposition occurs in three stages.

The first decomposition of the coal-containing clay (Fig. 11), occurs in the temperature range from 30 to 497°C , meaning the loss of adsorbed water and organic matter. According to TGA, the mass loss during the first decomposition is 4.84%.

The second decomposition was observed at temperatures from 497 to 874°C, which means dehydroxylation of kaolinite. In this case, structural OH is lost in the form of water, since kaolinite is converted to metakaolinite, which is expressed as follows [62]:



As is known [62], at a temperature of 573°C a polymorphic transformation occurs and α -quartz transforms into β -quartz. Since this polymorphic transformation is a reversible process, only volumetric changes occur in the mass of the sample without a change in mass. The mass loss in the temperature range from 497 to 874°C was 5.6%.

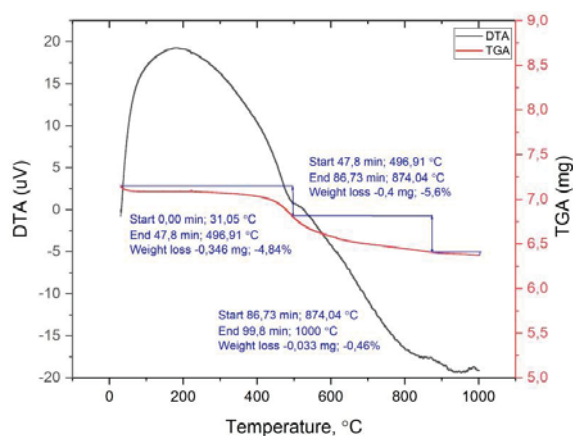


Fig. 11. Thermogravimetric and differential thermal (TGA/DTA) analyzes of kaolinite clay

Рис. 11. Термогравиметрический и дифференциально-термический (ТГА/ДТА) анализы каолиновой глины

The third decomposition was observed from 874 to 1000°C, at which crystallization of amorphous silica occurs with the formation of primary mullite or pseudomullite: Si-Al spinel with a mullite-like composition [62]. In this temperature range, the mass loss was 0.46%.

TGA/DTA analysis of Navbakhar bentonite clay (Fig. 12) showed that thermal decomposition also occurs in three stages. The first decomposition is indicated by endothermic dehydration (loss of sorbed moisture and interlayer free water), which occurs in the temperature range from 29 to 109°C. According to TGA, the mass loss during the first decomposition is 5.3%.

The second and third decompositions were observed at temperatures from 109 to 523°C and from 523 to 1000°C. According to [63], at temperatures of 200–235°C, inter-packet water is removed. Around 700°C, dehydroxylation and formation of the amorphous phase of metamontmorillonite occurs, and be-

tween 850 and 1000°C, solid-phase structural decomposition and crystallization of cordierite, mullite, Mg-spinel, quartz, and cristobalite occurs [62]. The mass loss was 4.64 and 3.68%, respectively, in the second and third decompositions.

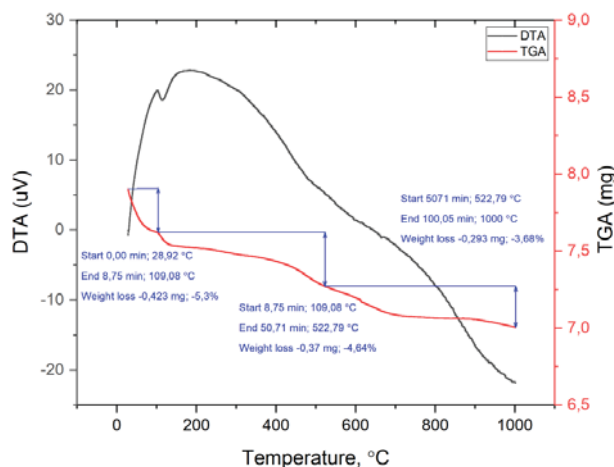


Fig. 12. Thermogravimetric and differential thermal (TGA/DTA) analyzes of bentonite clay

Рис. 12. Термогравиметрический и дифференциально-термический (ТГА/ДТА) анализы бентонитовой глины

The obtained results of the studies of the chemical and mineralogical composition of clays made it possible to proceed to the optimization of the composition of the batch for obtaining a porous aggregate.

Optimization of the batch composition for obtaining porous aggregates

The optimization of the composition and process parameters was performed using the method of mathematical planning of experiments. In this case, a second-order multifactorial plan was used. The bulk density and water absorption of the porous aggregates were used as the optimization parameter. The amount of bentonite clay in the batch, the firing temperature and the isothermal holding time were adopted as variable factors, the main levels of which were adopted based on the results of previous tests [2, 22] and literature data [47].

The intervals and levels of variation of the adopted variable factors are given in Table 3.

The dried aggregate grains were fired in a chamber furnace heated to 1100°C. It should be noted that when the furnace door is opened, the temperature inside the furnace drops to 800–850°C. The layer thickness is 40 mm. After loading with special dishes, the furnace door was closed back. Active combustion of coal and other organic substances occurs within 2.0 minutes. Raw grains are subjected to a sharp impact, the so-called "thermal shock". The temperature in the furnace

was increased to 1100°C for 30 minutes. Isothermal holding (firing) at this temperature is 20–40 minutes depending on the dependence provided in the experimental design matrix. After the firing process, the hot grains were taken out of the furnace for further cooling in air to room temperature.

Table 3. Intervals and levels of factor variation

Таблица 3. Интервалы и уровни варьирования факторов

Factors Факторы	Variation intervals Интервалы варьирования	Factor levels Уровни факторов		
		basic основной	upper +1 верхний +1	lower -1 нижний -1
X ₁ - amount of bentonite clay in the charge X ₁ - количество бентонитовой глины в шихте, %	20	70	90	50
X ₂ - firing temperature, °C X ₂ - температура обжига, °C	50	1050	1100	1000
X ₃ - isothermal exposure time, min X ₃ - время изотермической выдержки, мин	10	30	40	20

After conducting a series of experiments provided in the matrix of experiments, calculating the significance of the coefficients and checking the adequacy, the following regression equations were obtained:

- for the bulk density of the porous aggregate:

$$Y_{\rho_0} = 425 - 159X_1 - 24X_2 - 17.5X_3 + 1.25X_1X_3 - 1.25X_2X_3 + 235X_1^2 - 60X_2^2 - 62.5X_3^2; \quad (1)$$

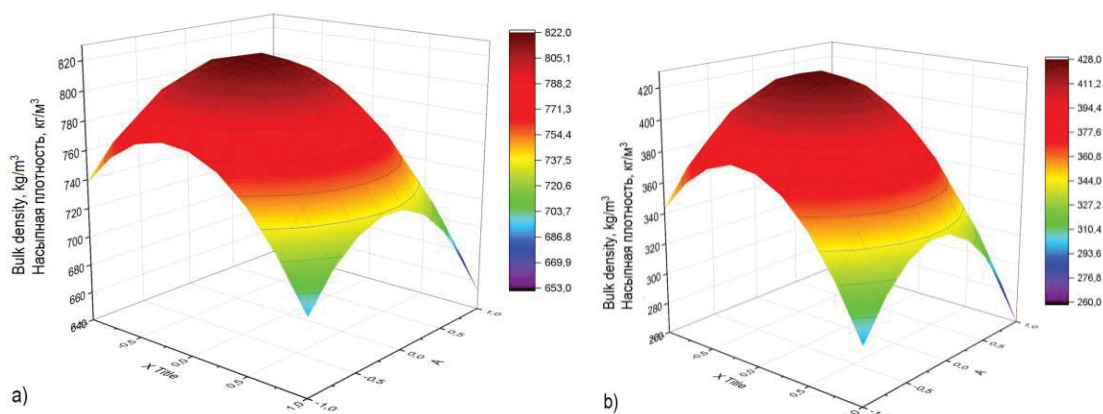


Fig. 13. Impact of isothermal holding time (X_2) and firing temperature (X_3) on the bulk density of the porous aggregate with the content of bentonite clay in the charge (X_1) at: a) $X_1 = +1$ (90%) and b) $X_1 = -1$ (50%)

Рис. 13. Влияние времени изотермической выдержки (X_2) и температуры обжига (X_3) на насыпную плотность пористого заполнителя при содержании бентонитовой глины в шихте (X_1) при: а) $X_1 = +1$ (90%) и б) $X_1 = -1$ (50%)

- for water absorption within 24 hours:

$$Y_w = 13,33 + 11,2X_1 - 2,67X_1^2 + 5,33X_2^2 + 2,33X_3^2; \quad (2)$$

Based on the obtained formulas (1), graphs were constructed (Fig. 13) describing the influence of variable factors on the studied indicators. The coefficients calculated based on the results of the experiment show the significance of the influence of the adopted factors. Analysis of the obtained formulas (1) and graphs showed that the most influential factor on the bulk density of the porous aggregate was the consumption of bentonite clay in the batch (X_1), since in absolute value its coefficient is the largest. This means that with an increase in the content of bentonite clay in the composition of the batch, the bulk density of the porous aggregate decreases. According to the influence, the remaining factors are located $X_2 > X_3$. In the paired influence, factors X_1 and X_2 turned out to be insignificant. In the paired value, the influence of the coefficient of factors X_1 and X_3 , also X_2 and X_3 turned out to be equal, but with opposite signs. This means that with an increase in the values of X_1 and X_3 , the average density of the aggregate will increase. And for X_2 and X_3 in a paired value, to increase the bulk density, one of the factors must be negative.

The obtained formulas (1) and (2) allow to regulate the bulk density and water absorption of the porous aggregate in a wide range. Table 4 presents the physical and mechanical characteristics of the obtained compositions of artificial porous aggregates. The results of the studies show that an increase in the amount of bentonite clay, respectively, with a decrease in the amount of coal-containing clay in the composition of the charge leads to a decrease in the average density and strength when compressed in a cylinder. However, the water absorption of the porous aggregate also increases.

The surface color of the porous aggregate also depends on the ratio of components and the firing temperature (Fig. 14). As the coal containing mixture increases, the surface color changes from dark pink to light gray and has a large-porous structure. The surface of the aggregates also depends on the composition and firing temperature. The surface of the aggregates with a high content of bentonite clay (composition No. 2) has a glassy surface (Fig. 14, a), and the surface of the aggregates with a high amount of coal containing clay (composition No. 1) is light gray and has a matte surface. Apparently, such a matte surface is associated with a higher sintering temperature of kaolin clay compared to the sintering temperature of bentonite clay. This allows us to assume that such an aggregate surface will lead not only to higher adhesion to the cement stone, but also to an increase in the strength of light-weight concrete.

Table 4. Physico-mechanical characteristics of the compositions of artificial porous aggregates

Таблица 4. Физико-механические характеристики составов искусственных пористых заполнителей

Composition no. № состава	Component ratio, % Соотношение компонентов, %		Characteristics of aggregate fired at 1100°C Характеристики заполнителя, обожжённого при 1100 °С		
	Bentonite clay Бентонитовая глина	Coal-containing clay Углеродсодержащая глина	Bulk density, kg/m³ Насыпная плотность, кг/м³	Compressive strength in a cylinder, MPa Прочность при сжатии в цилиндре, МПа	Water absorption, % Водопоглощение, %
1	50	50	686	6.46	8
2	70	30	395	2.74	16

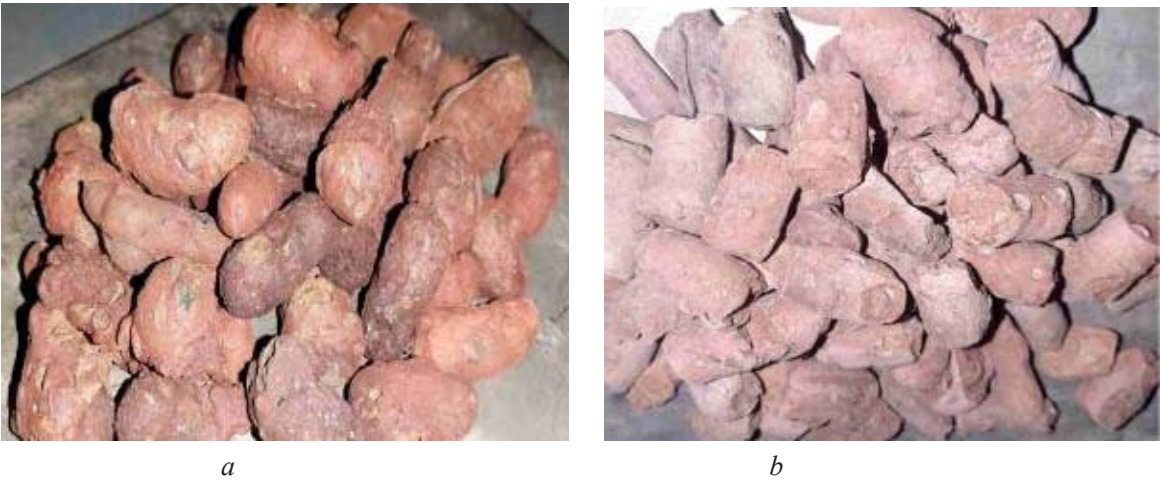


Fig. 14. General view of porous aggregates (compositions according to Table 3, respectively)
Рис. 14. Общий вид пористых заполнителей (составы, соответственно, по табл. 3)

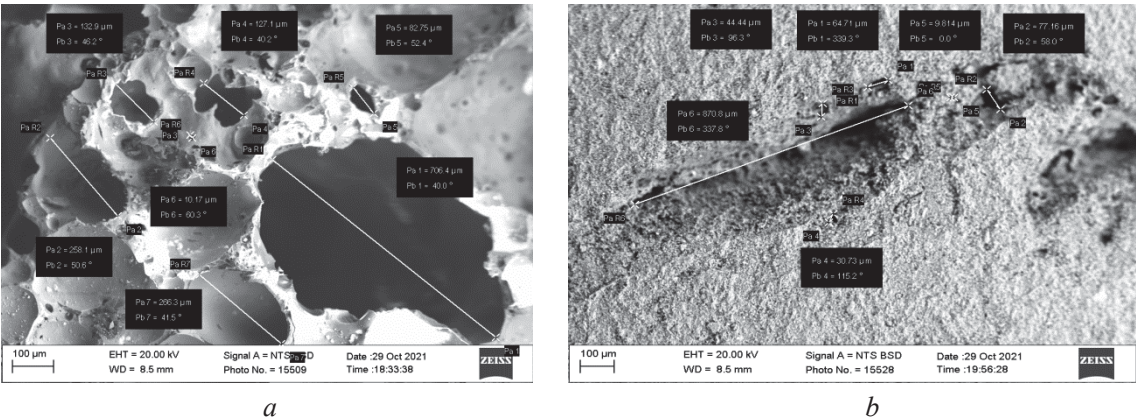


Fig. 15. Images of the porous structure of porous aggregates of the compositions, respectively, according to Table 3: a) no. 2; b) no. 1
Рис. 15. Изображения пористой структуры пористых заполнителей составов, соответственно, по табл. 3: а) № 2; б) № 1

Fig. 15 shows SEM images of the porous structure of the obtained lightweight aggregates. Samples made of 70% bentonite clay and 30% carbon-containing clay (Fig. 15, a) have high porosity, where the opening and closing of pores of different sizes are uniformly distributed throughout the volume. The pore sizes range from 46 to 706 μm . The presence of open pores causes an increase in the water absorption of these aggregates.

The analysis of the microstructure of the chip of the sample made from the composition consisting of bentonite clay and coal-containing clay in a ratio of 1:1 by weight (Fig. 15, b) shows that one long pore with a length of 870 μm was found, and the size of the remaining pores fluctuates between 9.8 and 77 μm . The bulk of the pores had sizes from 30 to 65 μm . The formation of such a fine-pored structure can be explained by the fact that such pores were formed due to the burnout of coal particles and gas-forming substances, the content of which is less compared to composition 2. The bulk of the pores were closed, respectively, which leads to low water absorption compared to the samples prepared according to composition No. 2. The resulting porous aggregate meets the requirements of regulatory documents.

The indicators of technical and economic efficiency of production of the developed porous aggregate [64], which is 82738 sum/ m^3 cheaper compared to expanded clay produced by the expanded clay plant in Gazalkent, Tashkent region, have been determined. Using the developed porous aggregate, compositions of heat-insulating and structural lightweight concrete of classes B-5 and B-20 and additives of superplasticizer Poly-

plast SP-3 have been developed, in the production of small wall blocks which allowed to reduce cement consumption by 20%, and the curing period in summer conditions – by 4.7 times.

A patent for a utility model (FAP 20220172) of the Intellectual Property Agency under the Ministry of Justice of the Republic of Uzbekistan was obtained for the compositions and technological parameters for obtaining the developed porous aggregate.

Conclusion

Involvement of the Angren coal deposit waste containing kaolinite clay and up to 30% coal (coal containing kaolinite clay) in the production of porous aggregates allows increasing the raw material base and reducing damage to the environment. The chemical and mineralogical compositions of the clays used were determined. The influence of technological factors on the bulk density and water absorption of the aggregate was studied using mathematical modeling. The obtained equations made it possible to identify patterns in the influence of the amount of bentonite clay in the batch, the firing temperature and the time of isothermal holding on the bulk density and water absorption of the porous aggregate. The obtained porous aggregates with a bulk density from 395 to 690 kg/m^3 have, respectively, compression strength in a cylinder from 2.74 to 6.46 MPa.

It was established that porosity and bulk density depend on the composition and technological parameters of firing of the developed aggregate. It was established that the obtained aggregates meet the requirements of regulatory documents.

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