



Investigation of structure and properties of expanded clay waste with the purpose of their use in the construction industry

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Abstract. This paper describes the structure and properties of large-tonnage expanded clay gravel waste, which is generated as dust from the cyclone, or clay dust (when drying granules), and expanded clay dust from filters (when leaving the kiln, from the cooler and screen). The mineralogical and phase composition of expanded clay dust and its hydraulic activity were determined. Clay dust from cyclones corresponds to the specific surface of 2500 cm²/g, and expanded clay dust from filters - 6800 cm²/g, which allows us to recommend it for use as a fine mineral additive to cement mortars and concretes. Due to the fact that the bulk density of clay dust is about 1000 kg/m³, and expanded clay dust - 6300 kg/m³, it allows to recommend it in the form of facilitating raw material additives in the production of wall and partition products. The results of X-ray phase analysis of clay dust from cyclones showed the presence of significant residues of unburnt clay (more than 80%), and the analysis of expanded clay dust from filters showed the presence of solid high-temperature phases consisting of: Quartz (SiO₂) – 50.28%, Albite C-1 (NaAlSi₃O₈) – 14.82%, Microcline maximum (KAlSi₃O₈) – 15.73%, Lime (CaO) – 2.77%, Calcite (CaCO₃) – 5.11%, Alunogen (Al₂(SO₄)₂(H₂O)₂₂) – 11.29%. Hydraulic activity of clay and expanded clay dust according to the methods of GOST R 56593 and GOST 30744 showed that it is extremely low, especially for clay dust (0.3 MPa). On the contrary, the characteristics of samples of clay-alkali compositions showed sufficient strength (for clay dust up to 6.8 MPa, and expanded clay dust up to 3.6 MPa), which allows them to be recommended as a component of slag-alkali binder for cement-soil bases of highways.

Keywords: expanded clay gravel, clay dust, expanded clay dust, specific surface area, X-ray phase analysis, thermal analysis, hydraulic activity

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1. INTRODUCTION

One of the thermal insulation materials, widely used in the Russian Federation for more than 80 years, is expanded clay gravel. The most important advantage of this bulk insulation material, used mainly for thermal insulation of attic floors and flat roofs, is non-combustibility, which does not require the device of additional fireproof ties. Also expanded clay gravel is used in fairly large volumes in the production of wall expanded clay concrete blocks, backfilling of wells, the arrangement of bases for foundations of low-rise buildings, the arrangement of drainage systems, etc [1, 2]. The volume of expanded clay gravel production in the Russian Federation in recent decades has remained at a fairly stable level and is about 2.8 - 3.0 million m³ per year [3]. A new trend in recent decades is a slight decrease in the output of expanded clay gravel with a density of 400-500 kg/m³ and a significant increase in the output of lightweight and especially lightweight expanded clay gravel with a density of 250-300 kg/m³, as well as expanded clay sand of various fractions (which are widely used in special purpose solutions for well plugging, etc.) [4]. In the Republic of Bashkortostan, expanded clay gravel is produced by Ufa Gypsum Company LLC, with an annual production volume of about 180 thousand m³ per year. Production is equipped with a drying drum and a 40 m long rotary shaft kiln.

A serious problem in the production of expanded clay gravel is the formation of waste in the form of dust, which is collected in dust cleaning systems (cyclones, filters), and the volume of waste at large clay plants can be up to 7-8 tons per day [5]. The resulting expanded clay dust is partly added to the raw clay and returned to production, but is mostly dumped. Nevertheless, according to a number of both classical authors and modern studies, expanded clay dust can be successfully used in the production of various construction materials [6-8].

2. METHODS AND MATERIALS

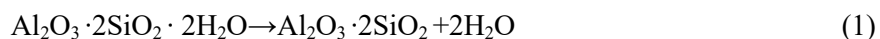
The technology of expanded clay gravel production in Ufa Gypsum Company LLC consists of the following main stages:

Stage 1. Homogeneous puddle clay is made from the clay delivered from the quarry, into which organic burning additives (sawdust) are introduced to improve the swelling, after which raw material pellets of 5 (7) - 20 mm in size with rounded shape and flat surface are formed, pellets humidity is about 20-25%;

Stage 2. Drying of pellets in the drying drum at the temperature of gases from gaseous fuel combustion about 400-450°C. During the drying process, free water evaporates and pellets are dehydrated to 8-12% moisture content and organic additives are burned out, chemically bound water also starts to evaporate [9];

Stage 3. Pellet firing in a 40 m long rotary kiln, during which the expanded clay granules go through the following main stages [10, 11]:

- complete dehydration of clay minerals (removal of chemically bound water) at 450-800°C to form metakaolinite (clayite, i.e. dehydrated clay) according to the formula:



- direct firing of expanded clay granules at temperatures ranging from 950 to 1000°C, which decomposes anhydrous metakaolinite $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ into Al_2O_3 and SiO_2 , after which an artificial mineral - mullite $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ is formed;

- swelling of expanded clay granules at a temperature of 1050-1200°C.

Stage 4. Cooling of the one that came out of the kiln and has a temperature of 950-1050°C in drum grate coolers.

Stage 5. Screening of expanded clay on vibrating screens to produce fractionated expanded clay gravel, which is then delivered to the finished product warehouse.

The production is equipped with cyclones, on which the dust from the drying drum is deposited, which represents particles of dried and partially hydrated clay, as well as particles of feedstock - unburnt clay and oil sludge (up to 0.2%) [11]. Hereinafter this material will be called dust from cyclones or clay dust (Fig. 1a).

Expanded clay dust is precipitated on the filters, which are particles of clay completely fired at 1150-1200°C, and formed at the kiln outlet, from the cooler and from the screen. Hereinafter, this material will be referred to as filter dust or expanded clay dust (Fig. 1b).

The volume of clay dust generated by the cyclone is 5-7 m³ per day, i.e. the annual volume of formation of this waste is about 3750-4000 m³. The volume of expanded clay dust formed on the filter is about 15 m³ per day, i.e. the annual volume of formation of this waste is about 1750-2000 m³ per year.



a) clay dust from the cyclone



b) expanded clay dust from the filter

Fig. 1. General view of waste from expanded clay production.

In order to determine the possible areas of use of this secondary raw material in the industry of construction materials in the laboratory of the Architectural and Construction Institute of USPTU a set of studies of its physical and mechanical properties and mineralogical composition was conducted.

The grain composition of clay dust from the cyclone according to the results of sieving on sieves is presented in Table 1.

Table 1. Grain composition of clay dust from the cyclone.

Name of residues	5.0	2.5	1.25	0.9	0.63	0.315	0.16	0.14	0.08	0.071	Passage through the sieve 0.071
Particular, g	0	0	0	0.01	0.07	3.38	9.86	3.54	8.31	2.45	72.38
Particular, %	0	0	0	0.01	0.07	3.38	9.86	3.54	8.31	2.45	72.38
Full, %	0	0	0	0.01	0.08	3.46	13.32	16.86	25.17	27.62	100

The grain composition of expanded clay dust from the filter according to the results of sieving on sieves is shown in Table 2.

Table 2. The grain composition of expanded clay dust from the filter.

Name of residues	5.0	2.5	1.25	0.9	0.63	0.315	0.16	0.14	0.08	0.071	Passage through the sieve 0.071
Particular, g	0	0	0	0	0	0	0.05	1.07	0.19	98.69	0
Particular, %	0	0	0	0	0	0	0.05	1.07	0.19	98.69	0
Full, %	0	0	0	0	0	0	0.05	1.12	1.31	100	0

Bulk density and true density of materials were determined according to the method of GOST 8735 (it.8 and it.9).

Specific surface area and average particle size were determined using the device PSX-12 (Fig. 2) – a meter of specific surface area and average particle size of powders. The losses on ignition (hereinafter – l.o.i.) were also determined.



Fig. 2. PSX-12 device.

The results of the true density, bulk density and specific surface area of clay and ceramic dust are summarized in Table 3.

Table 3. Results of laboratory tests of clay and expanded clay dust samples.

Name	Specific surface area, cm^2/g	Average particle size, μm	True density, g/cm^3	Bulk density, kg/m^3	l.o.i., %
Clay dust from the cyclone	2582	9.5	2.44	1009.6	10
Expanded clay dust from the filter	6781	3.5	2.53	629.8	5

3. RESULTS AND DISCUSSION

X-ray phase analysis of clay and expanded clay dust samples was performed using X-ray diffractometer BRUKERD2 PHASER (Fig. 3). The principle of operation of the diffractometer is based on the diffraction of X-rays from the atomic planes of the crystal lattice of the substance under study. The diffraction pattern is recorded using a detector that allows for high performance. Quantitative analysis was performed in the program “TOPAS” (TotalPatternAnalysisSolution), qualitative analysis was performed in the program “DiffracEva”. The results of X-ray phase analysis of clay dust from the cyclone are presented in Fig. 4 and Table 4, the results of X-ray phase analysis of expanded clay dust from the filter are presented in Fig. 5 and Table 5.



Fig. 3. BRUKERD2 PHASER X-ray diffractometer.

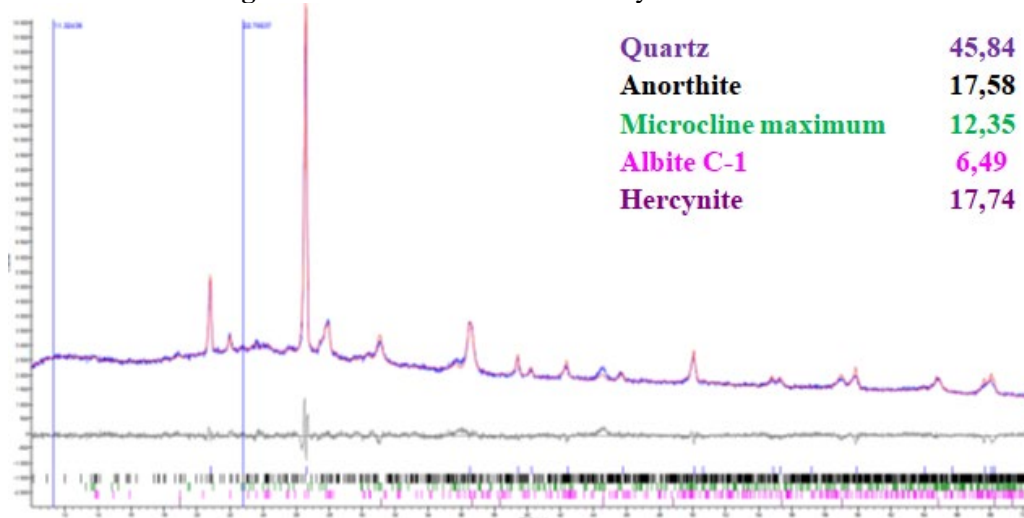


Fig. 4. Results of X-ray phase analysis of clay dust from the cyclone.

Table 4. Results of X-ray phase analysis of clay dust from the cyclone.

Name of mineral	Formula	Quantitative content, %
Quartz	SiO ₂	50.28
Albite C-1	NaAlSi ₃ O ₈	14.82
Microcline maximum	KAlSi ₃ O ₈	15.73
Lime	CaO	2.77
Calcite	CaCO ₃	5.11
Alunogen	Al ₂ (SO ₄) ₂ (H ₂ O) ₂₂	11.29

As it can be seen from Fig. 4 and Table 4, the mineralogical composition of clay dust from the cyclone is represented by the following minerals: Quartz (SiO_2) – 50.28%, Albite C-1 ($\text{NaAlSi}_3\text{O}_8$) – 14.82%, Microcline maximum (KAlSi_3O_8) – 15.73%, Lime (CaO) – 2.77%, Calcite (CaCO_3) – 5.11%, Alunogen ($\text{Al}_2(\text{SO}_4)_2(\text{H}_2\text{O})_{22}$) – 11.29%.

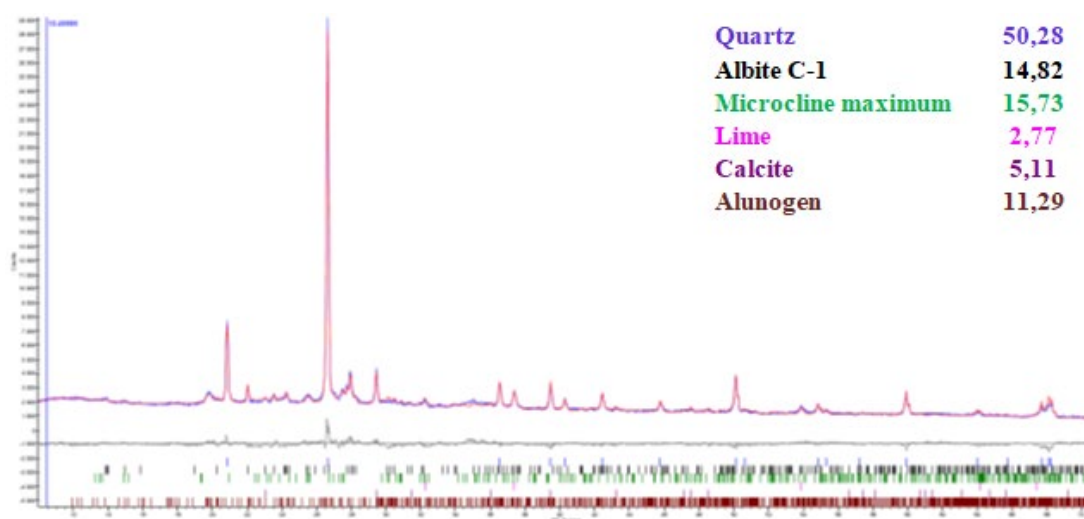


Fig. 5. Results of X-ray phase analysis of expanded clay dust from the filter.

Table 5. Results of X-ray phase analysis of expanded clay dust from the filter.

Name of mineral	Formula	Quantitative content, %
Quartz	SiO_2	45.84
Anorthite	$\text{CaAl}_2\text{Si}_2\text{O}_8$	17.58
Microcline maximum	KAlSi_3O_8	12.35
Albite C-1	$\text{NaAlSi}_3\text{O}_8$	6.49
Hercynite	FeAl_2O_4	17.74

As it can be seen from Fig. 5 and Table 5, the mineralogical composition of expanded clay dust from the filter is represented by the following minerals: Quartz (SiO_2) – 45.84%, Anorthite ($\text{CaAl}_2\text{Si}_2\text{O}_8$) – 17.58%, Microcline maximum (KAlSi_3O_8) – 12.35%, Albite C-1 ($\text{NaAlSi}_3\text{O}_8$) – 6.49%, Hercynite (FeAl_2O_4) – 17.74%.

The thermal analysis was performed using a STA 449 F1 Jupiter® synchronous thermal analysis (TG-DSC) instrument with a QMS 403 CF Aeolos mass spectrometer (Fig. 6). The results of thermal analysis of a sample of clay from the Shaksha deposit used by Ufa Gypsum Company for the production of expanded clay are presented in Fig. 7, the results of thermal analysis of clay dust from the cyclone are presented in Fig. 8.



Fig. 6. STA 449 F1 Jupiter® synchronous thermal analysis (TG-DSC) instrument with QMS 403 CF Aeolos mass spectrometer.

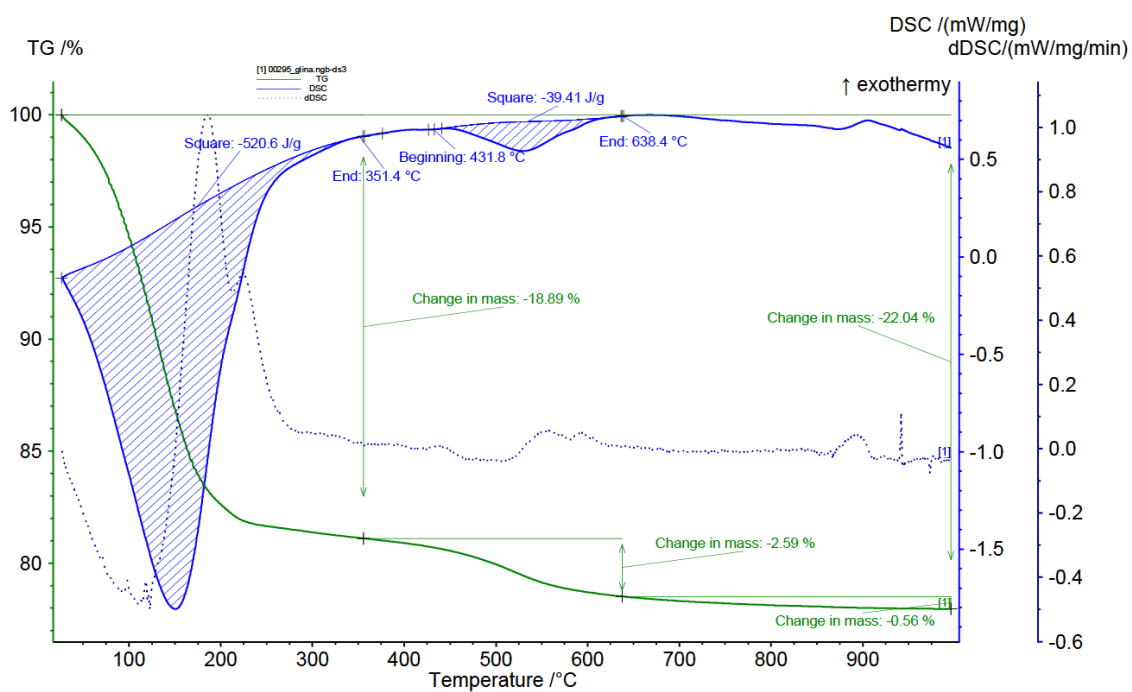


Fig. 7. Results of thermal analysis of clay from the Shaksha deposit.

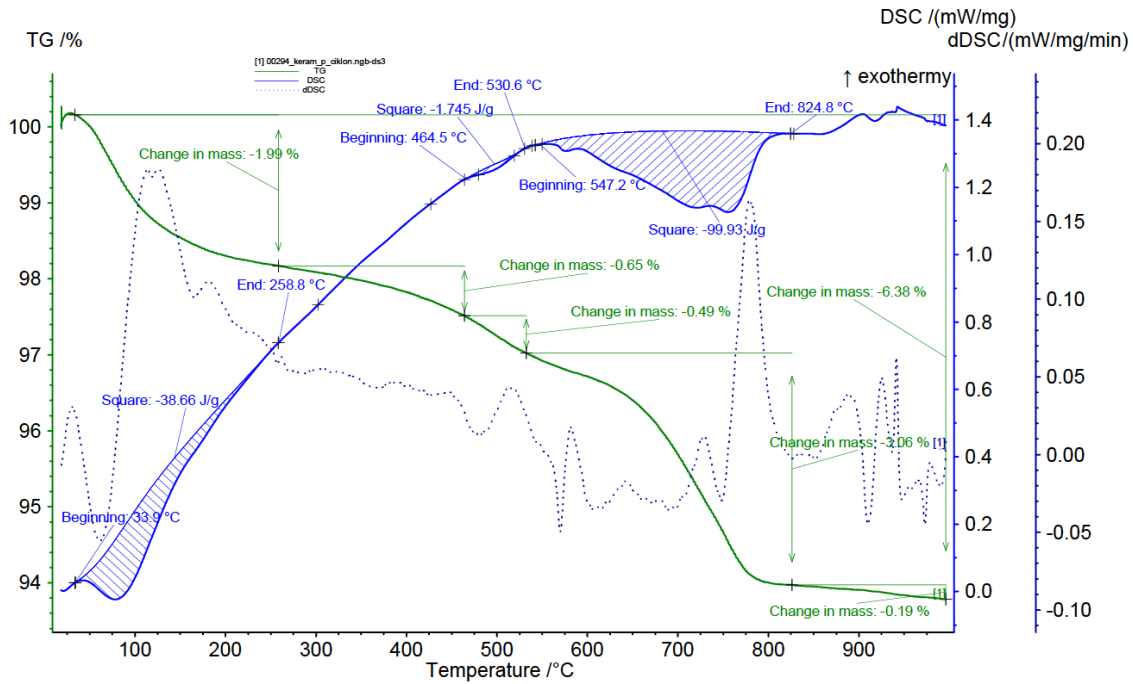


Fig. 8. Results of thermal analysis of clay dust from the cyclone.

As it can be seen from Fig. 7 and 8, the evaporation of free water from clay occurs in the temperature range from 20 to 350°C. The complete dehydration of clay minerals occurs in a much wider range, from 430 to 820°C.

Determination of hydraulic activity of clay and expanded clay dust according to the methods of GOST R 56593 and GOST 30744 showed that the compressive strength of samples made with the use of clay dust after 28 days of normal hardening is equal to zero, the compressive strength of samples made with the use of clay dust is also extremely low and is about 1 MPa, the test results are presented in Table 6.

Table 6. Determination of hydraulic activity of clay and expanded clay dust.

Name	Compressive strength at 28 days of age, MPa	Flexural strength at 28 days of age, MPa	Initial setting, min	Setting end, min	Normal density, %
Clay dust from the cyclone	0.3	0	-	-	0.45
Expanded clay dust from the filter	1.2	0	145	310	0.54

The results of determining the strength characteristics of specimens made of clay and expanded clay dust with mixing with alkali solution are presented in Table 7.

Table 7. Results of determination of strength characteristics of specimens made of clay and expanded clay dust, with mixing with NaOH alkali solution (at a concentration of 6-7 mol).

Name	W/C	Flexural strength, MPa (after HMT)	Compressive strength, MPa (after HMT)
Clay dust from the cyclone	0.55	2.1	6.8
Expanded clay from the filter	0.55	0.7	3.6

The average density and porosity were determined according to the methods of GOST R 52129 (item 7.4 and item 7.5).

The essence of the method of determining the average density is to determine the density of powder after compacting it in a mold of 100 cm³ under a load of 40 MPa. The lower part of the mold is placed on a pallet, weighed, and then the upper part is placed on it. Powder in portions of 60-80 g is transferred into the assembled mold, distributed layer by layer and poked with a knife or spatula, filling it 15-20 mm below the top edge, and lightly pressed with a liner. The mold with powder is placed on the bottom plate of the press, smoothly bring the compacting load to 40 MPa and maintain it for 3 min. After that the load is removed and the mold with the liner is transferred to a baking tray. The liner and the upper part of the mold are removed, the excess powder above the lower part of the mold is cut off with a knife, the outer parts of the mold and the tray are cleaned with a soft brush. The lower part of the mold with powder and the tray are weighed.

The average density of the powder ρ_m , g/cm³, is calculated by the formula:

$$\rho_m = \frac{m - m_1}{V} \quad (2)$$

According to the results of two parallel tests it was found that the average density of expanded clay dust was 0.857 g/cm³.

Porosity of mineral powder is determined by calculation on the basis of predetermined values of true density and average density and calculated by the formula:

$$V_{\text{nop}} = \left(1 - \frac{\rho_m}{\rho}\right) \cdot 100 \quad (3)$$

According to the calculation results, the porosity of expanded clay dust was as follows:

$$V_{\text{nop}} = \left(1 - \frac{0.857}{2.53}\right) \cdot 100 = 66\% \quad (4)$$

As it can be seen from the test results, clay dust from the cyclone is characterized by a specific surface of 2600 cm²/g, i.e. somewhat coarser than Portland cement, the specific surface of which averages from 3800 to 4500 cm²/g. Expanded clay dust from the filter is characterized by a specific surface of 6800 cm²/g, i.e. much more dispersed than Portland cement.

The results of X-ray phase analysis (XPA) show that the mineralogical composition of clay dust from the cyclone is represented mainly by clay minerals - aluminosilicates and quartz (SiO₂). The presence of calcium oxide (CaO) and calcium hydroxide (CaCO₃) can be explained by powdering of granules to prevent their sticking, as well as the presence of calcareous inclusions in the clay composition [12].

Thermal analysis of clay samples of Shaksha deposit, delivered to the laboratory in the state of natural humidity, showed that in the temperature range from 20 to 350 °C evaporation of free water from the clay, in the technological process, this temperature range corresponds to the stage of drying in the drying drum. In the range from 430 to 640°C, complete dehydration of clay minerals occurs, i.e. removal of chemically bound water [13]. The thermal analysis of clay dust specimens showed a

similar result, with the temperature range of hydration shifted to higher temperatures: from 540 to 820°C.

As it can be seen from Tables 6 and 7, clay dust from the cyclone (which is dried fine clay) does not have binding properties, has a high water demand (normal density is 45%), but at the same time has a certain activity when mixing with alkali solution.

Expanded clay dust with filter does not possess certain binding properties (hydraulic activity) and, according to GOST R 56592, belong to inert mineral additives (IMA). Conducted studies to assess the possibility of using expanded clay dust as a component of lime-clay (lime-puzzolanic) binder by making samples containing 20% quicklime and 80% expanded clay dust, showed that the compressive strength of the manufactured specimens does not exceed 15 kg/cm² (1.5 MPa), which is lower than the minimum possible grade M50, i.e. expanded clay dust also does not possess pozzolanic activity.

Based on the laboratory studies of clay and expanded clay dust – a secondary resource of Ufa Gypsum Company LLC - potentially possible directions of their use in construction technologies were determined.

Expanded clay dust, which is an inert mineral additive, can potentially be used as:

- inert mineral admixture-filler for concrete and mortars, the requirements for which are regulated by GOST R 56592. In terms of dispersibility expanded clay dust meets the requirements of it. 5.1.4 of GOST R 56592 (residue on the sieve 0.315 - not more than 10%). Complicating technological factor in this case is the high water consumption of expanded clay dust (is 54%), in this regard, the feasibility of using expanded clay dust as an inert mineral admixture for concrete and mortars should be determined experimentally;
- mineral filler in accordance with GOST 32021 in the production of dry construction mixtures;
- mineral filler for construction polymers (such as those used in the manufacture of vinyl siding);
- as a component of complex mineral binder according to GOST 23558 for soil strengthening.

The use of expanded clay dust as a mineral powder for asphalt and organomineral mixtures, the requirements for which are regulated by GOST R 52129 and GOST 32761, is not possible, as the indicator “porosity” expanded clay dust does not meet the requirements of Table 1 of GOST R 52129 and Table 1 of GOST 32761 (not more than 40%).

Clay dust from cyclone does not possess binding properties (hydraulic activity) or pozzolanic properties in natural conditions. Clay dust (as well as ordinary clay) has high water consumption and cannot be used as an inert mineral admixture in concrete or mortars. Possible uses of clay dust from the cyclone are:

- return to the technological process as an additive to clay raw material used in the production of expanded clay gravel to reduce its moisture content;
- as clay soil for road base or land fill, backfilling of foundation slots;
- as a component of slag-alkali binder, but this direction is currently limited by the technological difficulties of manufacturing of building products from this type of binder and the lack of their mass production.

4. CONCLUSIONS

Laboratory studies of expanded clay dust from electrostatic precipitators – expanded clay waste production of Ufa Gypsum Company LLC - have shown the fundamental possibility of its use as an inert mineral additive-filler for concrete and mortars, mineral filler in the production of dry building mixtures [14], mineral filler for polymers for construction purposes, as a component of complex mineral binder according to GOST 23558 for soil reinforcement. The obtained results generally agree with the results of research from other authors [15-17].

The scope of application of clay dust from cyclones – another waste product of expanded clay production of Ufa Gypsum Company – is limited to slag-alkali binders [18-20], as well as use as clay soil for road base or landfill, backfilling of foundation slots.

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