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## Properties of Verkhnealkunsky deposit mudstones used as raw materials to manufacture clinker tiles

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**Abstract.** The article writes about the development of mudstone-based raw material compositions used to manufacture clinker tiles with improved physical and technical properties. Clinker tiles are one of the popular roofing materials. Compared to other roofing covering options, clinker tiles have higher strength, more resistance to aggressive environments, and better aesthetic characteristics. Clinker tile production is relevant due to the fact that currently architects and building constructors are showing great interest in such an elite roofing material, but due to the economic situation, this roofing material is no longer imported to Russia, with an empty market niche having been formed. Many Russian brick factories are planning to install additional technological lines to manufacture ceramic tiles, but the current demand is to produce clinker tiles. The main unresolved issue concerns raw materials since traditional raw materials – loams and clays – are very rarely suitable for the production of low-temperature sintering clinker tiles. Having studied a large amount of academic literature and carried out the necessary laboratory studies, the authors of this article, under the guidance of the Chair of the Construction Materials Department, Prof. V.D. Kotlyar, pay serious attention to such raw materials as mudstones from the Verkhnealkunsky deposit of the Russian Republic of Ingushetia. The article provides a general description of mudstones, including their chemical and mineral composition as well as their structural features for the production of clinker tiles. It has been experimentally established that the degree of their grinding has a significant influence on technological properties of both mudstones and the end products. Based on raw material mixtures with a grain composition of 0-0.16 and 0-0.08 mm, it is possible to manufacture clinker tiles using the extrusion molding method without introducing corrective additives. The data obtained will make it possible to develop technological regulations to manufacture clinker tiles based on Verkhnealkunsky deposit mudstones.

**Keywords:** mudstones, clinker tiles, mineral composition, structure, water absorption, strength

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## 1. INTRODUCTION

Currently, not a single factory which manufactures walling, cladding and roofing products operates in the Russian Republic of Ingushetia, and not a single deposit of clay raw materials is being developed there [1]. One of the reasons to close old factories was the initial focus on traditional clay raw materials – loess and loam – which, due to the regional geological peculiarities, are of low quality and, accordingly, the manufactured products were not of high quality as well and, therefore, not in demand among consumers [2]. Ingushetia is a region of Russia, which is rich in various mineral resources and raw materials [3]. It used to have three loam deposits with total reserves of about 2 million cubic meters explored to manufacture ceramic bricks – the Nesterovskoye, Ekazhevskoye, and Karabulakskoye deposits.

The low quality of these loams also determined the low quality of the bricks produced [4], as a result of which all three factories, unable to withstand the competition and new economic realities, were closed. Therefore, focusing on loam as a raw material for the production of clinker tiles, where raw materials must have special properties, is not promising.

It is a difficult task to select a potential deposit for organizing the production of clinker tiles in the Russian Republic of Ingushetia, despite the large forecast reserves of raw materials, since there are many limiting factors and conditions [5, 6], e. g.:

- raw materials must have the necessary chemical and mineral composition as well as technological properties;
- raw material reserves must be of least 1.0-1.5 million cubic meters;
- the deposit's mining allotment site should not fall into the protection zones of rivers, lakes, nature reserves, power lines, gas pipelines, monuments of historical and architectural heritage, natural monuments, and other existing restrictions;
- the potential deposit should be located near a road and preferably paved for uninterrupted delivery of raw materials at any time of the year;
- mining conditions for the deposit development must be favourable – there should be no water content, large overburden, or interlayered rocks of various compositions.

We have identified a large occurrence of mudstones to the south of Verkhniy Alkun village (the Republic of Ingushetia, Russia), which is located relatively close to the developed industrial centres of the Eastern Caucasus [7]. The manifestation of mudstones is located 200 meters from the left bank of the Assa River near the asphalt road leading to the regional centre of Dzheirakh. An analysis of the geological structure of the Ingushetia's territory showed that Verkhnealkunsky deposit mudstones are promising raw materials to manufacture clinker ceramic tiles and belong to the Lower Cretaceous (137 – 67 million years ago) [8].

## 2. METHODS AND MATERIALS

To conduct complex studies, a representative technological sample weighing 500 kg was selected, formed from point samples without plant inclusions and soil. The selected and formed sample characterizes this mudstone deposit as fully as possible. Before testing, the sample was dried to an air-dry state. Then it was crushed in a hammer crusher for clay raw materials to the specified fractions in laboratory conditions. After the crusher, the raw materials were sifted on a laboratory screen with a given cell size. In this way, crushed argillite was obtained and stored in plastic bags until testing began.

Laboratory tests were carried out in the following order:

- study of material composition;
- determination of technological post-firing and firing properties of raw materials;
- testing of obtained laboratory samples;
- analysis of the resulting data.

The research is based on the current Russian and international regulatory documents, well-known methods, and recommendations of leading research institutes [9-11].

Chemical and mineral composition of mudstones was studied using a complex of methods. To determine their chemical composition, the following regulatory documents were used: Russian State

Standard GOST 21216-2014, NSAM method No. 138-X, Russian State Standard GOST 2642-2018 “Refractories and refractory raw materials. Method for determination of potassium and sodium oxides”, and Russian State Standard GOST 33850-2016 “Soils. Determination of chemical composition by X-ray fluorescence spectrometry” (Spectroscan MAX-GVM).

The mineral composition of mudstones was determined according to Russian State Standard GOST 21216-2014 “Clay raw materials. Test methods” (clause 5.7 – Determination of mineral composition) using X-ray diffraction and thermal analysis methods [12]. X-ray phase studies were carried out applying an ARL X’TRA device with a slit width of 2-4-1-0.5. Recording conditions on the ARL X’TRA diffractometer were as follows: interval – 5-70°; speed – 5-6 °/min; voltage – 40 kV; current – 30 Ma [11, 12]. Diffraction patterns were interpreted via appropriate techniques, by comparison with similar studies, as well as using international databases. Thermal analysis was performed on a NETZCH STA 449 °C device with samples heated to 1,100 °C. The results obtained were interpreted basing on appropriate techniques and published data.

Plasticity was determined according to the Russian State Standard GOST 21216-2014 method using raw materials crushed to a certain fraction. It is natural to note that the technological properties of mudstones are different, depending on the degree of grinding [13].

The mechanical bending strength of the dried raw material was determined using the VNIISTROM (Scientific Centre for Ceramics) methodology. Water absorption, flexural and compressive strength were studied applying generally accepted methods.

The samples were formed by the plastic molding method. Laboratory samples were cylinders with a diameter and height of 50 mm, cube-shaped samples of 50x50x50 mm, and beam-shaped samples of 160x40x20 mm in size. After molding, the samples were dried at room temperature for 24 hours. Next, they were placed in a drying cabinet until completely dry at a temperature of 105 °C. The dried samples were fired in a laboratory oven with an average temperature rise rate of 3 °/min and held at maximum temperature for 1.5-2 hours. Next, the samples were cooled to a temperature of 100-150 °C for 12-14 hours.

### 3. RESULTS AND DISCUSSION

The chemical composition of Verkhnealkunsky deposit mudstones was determined in the laboratory of the Department of Construction Materials (Don State Technical University, Rostov-on-Don, Russia).

Table 1 presents the results of an averaged chemical silicate analysis of the Verkhnealkunsky deposit mudstone sample. The table also shows the content of water-soluble salts measured in our study.

**Table 1.** Chemical analysis of mudstones, % by weight.

Imps	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub> gen.	CaO	MgO	SO <sub>3</sub>	K <sub>2</sub> O	Na <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>	TiO <sub>2</sub>
7.14	60.39	16.89	4.91	3.02	1.86	0.19	3.60	1.73	0.16	0.10

**Table 2.** Content of water-soluble salts, mEq.

pH	Anions				Cations			Sum of cations and anions, mEq
	CO 3	HCO 3-	C I-	SO 4--	Ca+ +	Mg+ +	Na ++ K+	
8.0	0.07	1.22	1.04	3.12	1.01	0.32	4.12	10.90

According to Russian State Standard GOST 9169-21 “Clay raw materials for the ceramic industry. Classification”, mudstones, in terms of their chemical composition, can be classified as semi-acidic raw materials with a silica content (about 60%),  $\text{Al}_2\text{O}_3$  (about 17%) and a high content of colouring oxides. Potassium oxide is largely predominant over sodium oxide with their total content of 5.0-5.5%. This indicates the possibility of low-temperature sintering, which is a favourable outcome for the production of clinker tiles [14].

Fig. 1 shows an X-ray diffraction pattern of the Verkhnealkunsky deposit mudstone sample while Fig. 2 represents the corresponding thermogram.

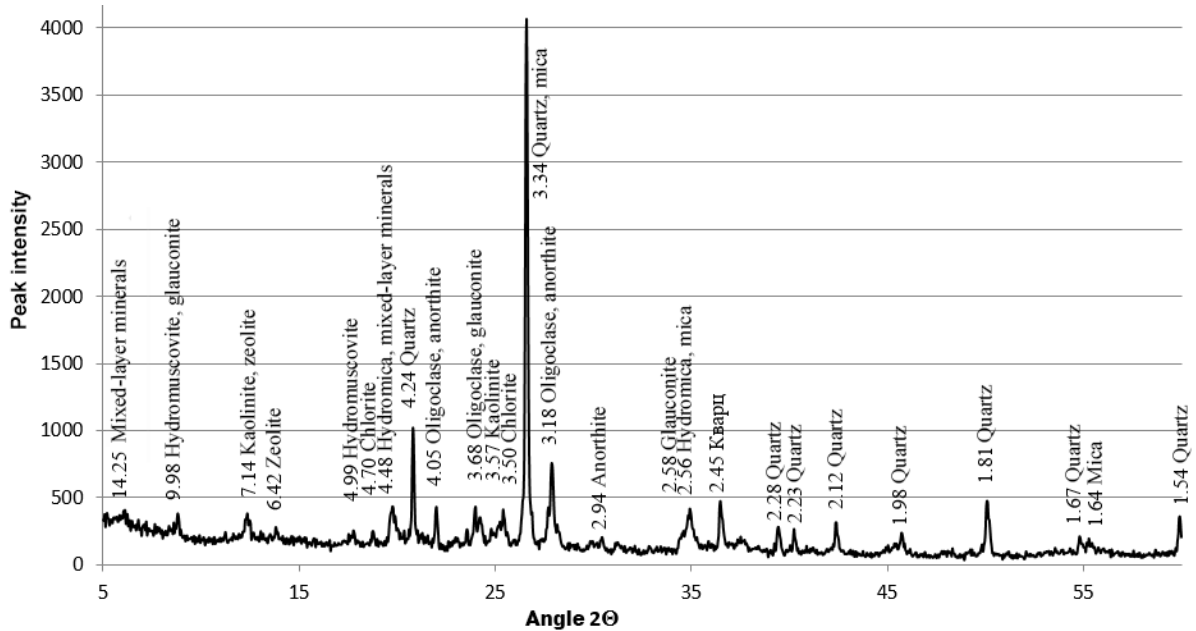


Fig. 1. X-ray diffraction pattern of the Verkhnealkunsky deposit mudstone sample.

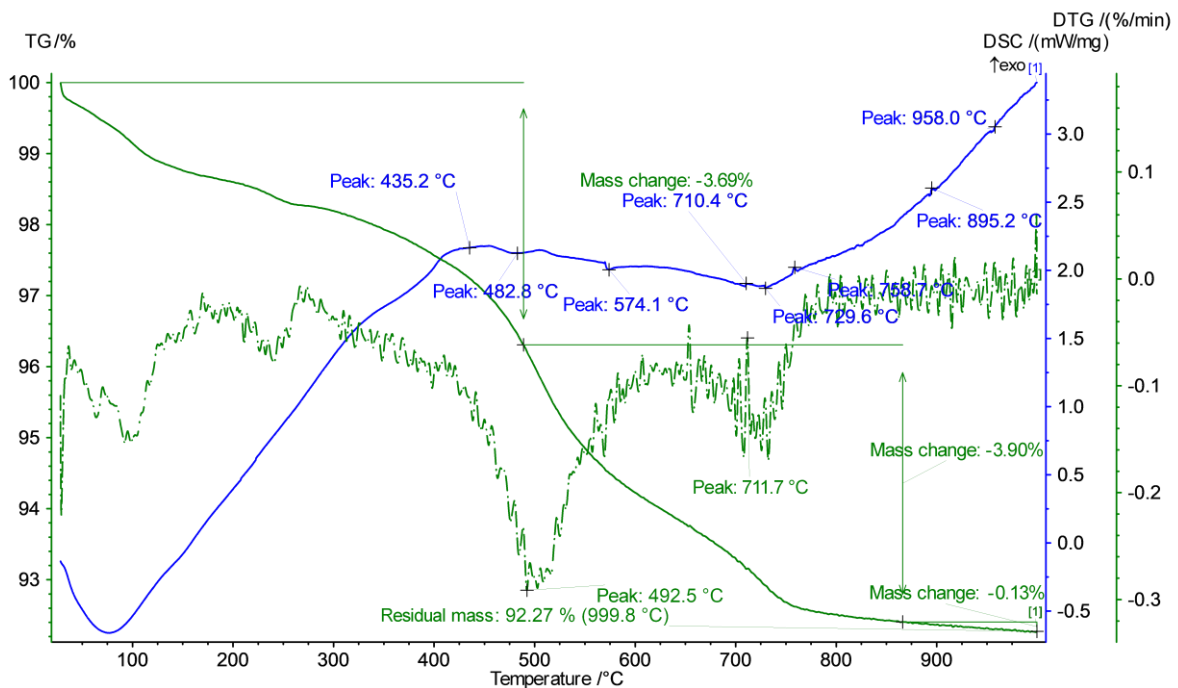


Fig. 2. Thermogram of the Verkhnealkunsky deposit mudstones.

In terms of their clayey components, mudstones can be characterized as those having polymineral, i.e. hydromica-kaolinite, mineral composition. However, mixed-layer minerals are also present. The predominant minerals are various hydromica minerals with the presence of kaolinite in minor amounts. The peaks of minerals from the chlorite group are clearly visible. Chlorite belongs to the group of hydromicas and consists of alternating mica- and brucite-like layers which form the 2:1:1 tetramorphic platelet structure. Due to strong bonds within and between the platelets, chlorites usually do not expand when hydrated. They are always found mixed with other clay minerals. Chlorites are similar in particle size to illite clay minerals [15]. Chlorite does not have a constant chemical composition, and therefore different varieties of chlorite are distinguished, which have their own names, as discussed above. Quartz, which is a terrigenous impurity, is also diagnosed. Due to its high crystallinity, its diffraction peaks (3.34; 1.813; 1.539; 4.24; 2.45; 2.28; 2.123; 1.668; 2.23; 1.975 Å), have significant intensity, even with a small content in the rock. Feldspars represented by oligoclase and anorthite are noted. The total content of clay minerals is 60-65%.

Thermal analysis makes it possible to determine the mineral composition of rocks with a certain degree of reliability based on the processes occurring during heating, as well as to predict the firing schedule of products for ceramic technology.

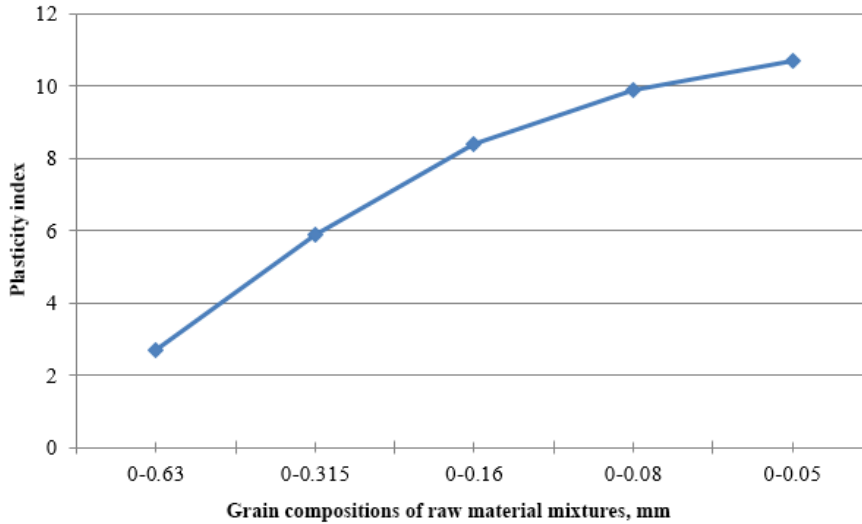
Analyzing the DTA results obtained, the following properties can be noted. The thermogram shows vague endo-effects at temperatures of 100 °C and 260 °C, associated with the release of free molecular water and a slight loss of mass. A small exo-effect at a temperature of 435 °C is related to the burnout of organic matter. The endoeffect in the temperature range between 470 °C and 530 °C is due to the release of constitutional water and partial restructuring of hydromica and micaceous minerals, and, to a lesser extent, kaolinite. Small endoeffects and a gradual loss of mass are observed in the temperature range from 700 °C to 1,100 °C, which is linked to the release of hydroxyl structural water from various types of clay minerals and the complete destruction of their structure. The differential thermal analysis results confirm the predominantly hydromica composition of mudstones with a small kaolinite admixture.

Assessing the chemical and mineral composition of mudstones as well as their structural features, we can assume that the Verkhnealkunsky deposit mudstones are potentially suitable for producing clinker tiles at a relatively low firing temperature. This is confirmed by a fairly high content of alkali metal oxides.

Based on the results of previous studies, it is known that the technological properties of mudstones largely depend on the grain composition during raw materials' grinding and preparation. To study the pre-firing and firing technological properties, we crushed the mudstones to fractions of 0-0.05 mm; 0-0.08 mm; 0-0.16 mm, 0-0.315 mm, and 0-0.63 mm [16, 17].

It has been established that the degree of grinding of mudstones has a significant effect on plasticity – the finer the mudstones are crushed, the greater their plasticity. This pattern is clearly visible in the graph presented in Fig. 3.

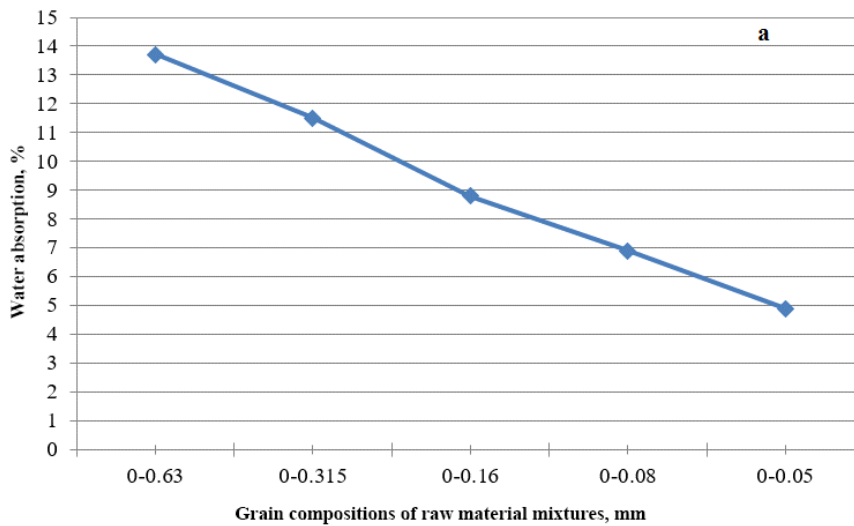
Thus, with a degree of grinding to a grain composition of 0-0.63 mm, the plasticity index of argillites is only 2.7 units, i. e. they are non-plastic. With the degree of grinding to a grain composition of 0-0.315 mm, the plasticity index increases to 5.9 units, i. e. they are already low-plasticity raw materials. With a degree of grinding to a grain composition of 0-0.16 mm, they move into the group of moderately plastic raw materials with a plasticity index of 8.4 units. Finally, with a degree of grinding to grain compositions of 0-0.08 and 0-0.05 mm, their plasticity index is, respectively, 9.9 and 10.7 units. For plastic (extrusion) molding of tiles, raw materials with a plasticity index of 8-10 units and higher are suitable. We have noticed that when the Verkhnealkunsky deposit mudstones are aged in humid conditions, the plasticity index and structural strength of the corresponding raw material mixtures increase, especially if they are irrigated with water with a slightly alkaline reaction.

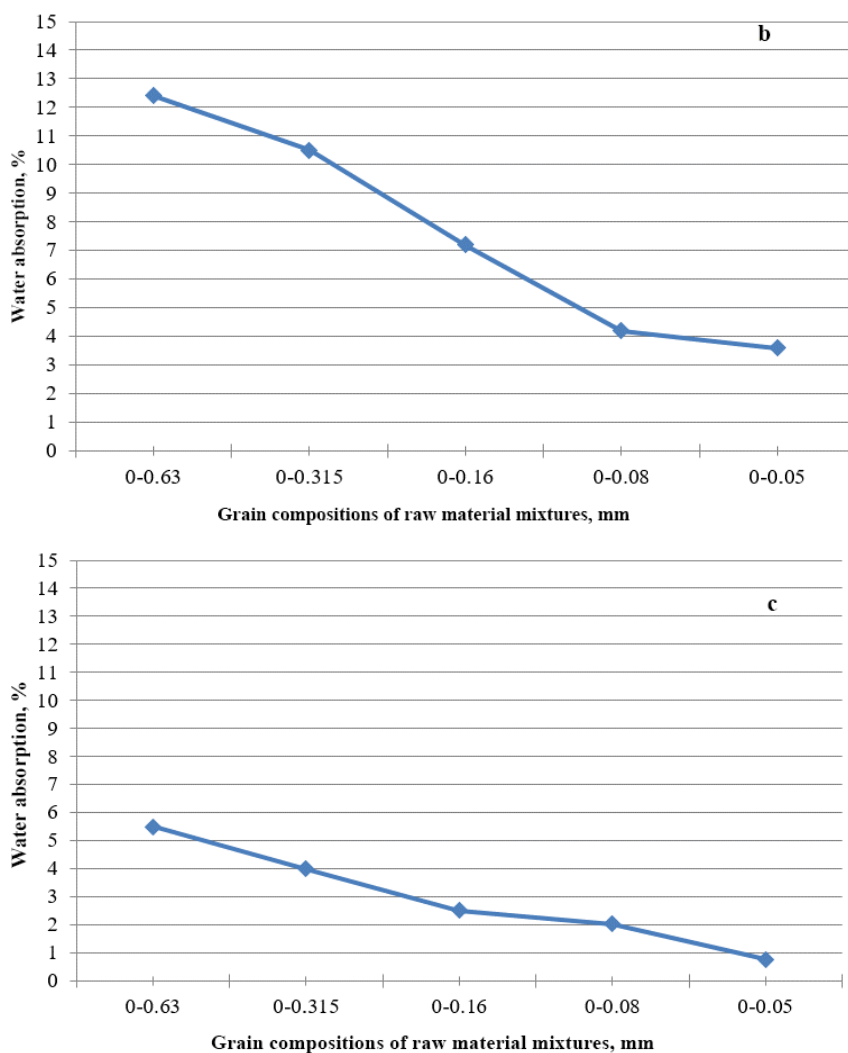


**Fig. 3.** Dependence of plasticity index of the Verkhnealkunsky deposit mudstones on their grain composition.

Important characteristics of any construction materials and products are their physical and mechanical properties. The main indicators of clinker tile samples fired at various temperatures are water absorption, water resistance, and bending strength.

The water resistance and biological resistance of tiles can be guaranteed if their water absorption is less than 5%. Based on the chemical and mineral composition of the Verkhnealkunsky deposit mudstones and the results of preliminary experiments, and the fusibility of the raw material, we selected the firing temperature range to optimize the technological parameters from 950 to 1050 °C (Fig. 4).



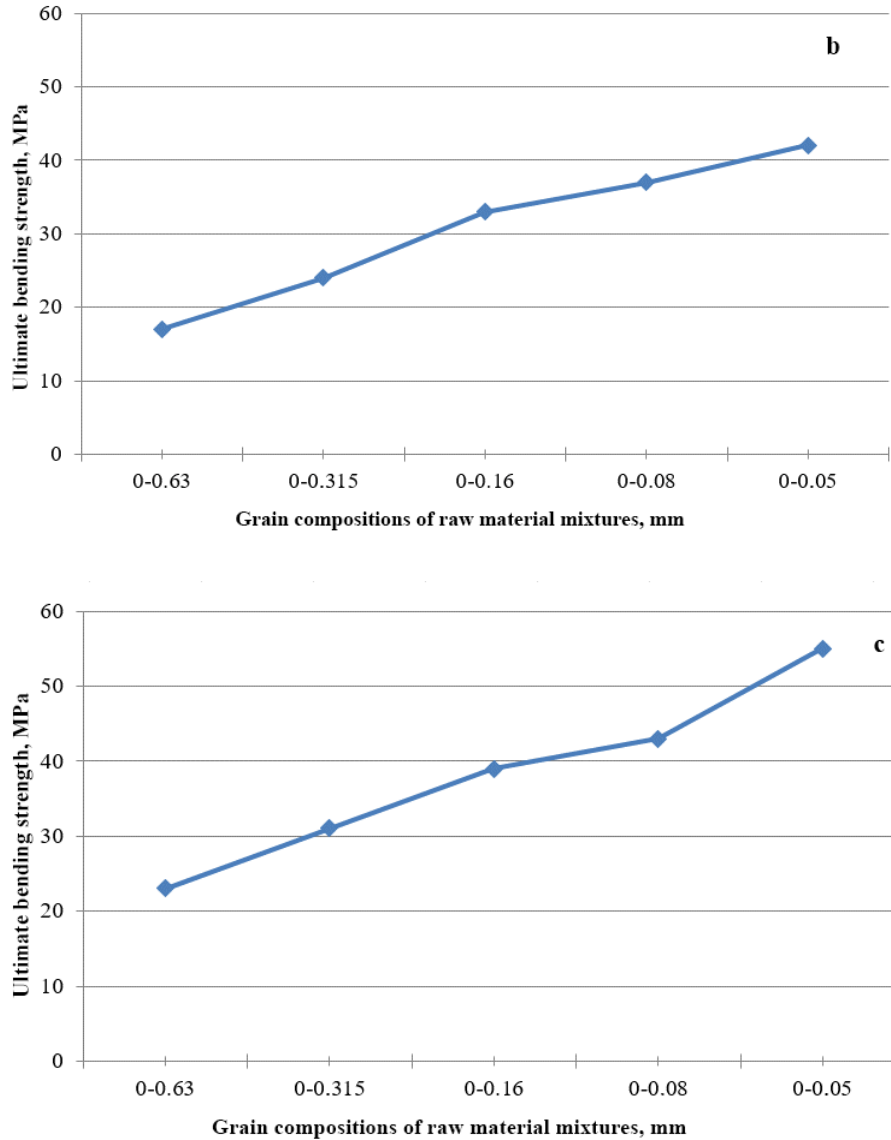


**Fig. 4.** Dependence of water absorption of fired samples on the grain composition of crushed mudstones at different firing temperatures (a – 900 °C; b – 1,000 °C; c – 1,050 °C).

As can be seen from the research results, at a firing temperature of 950 °C, water absorption indicators of less than 5% are achieved only for a grain composition of 0-0.05 mm – 4.94%. These values are at the limit of the required ones and cannot be accepted for technology development. At a firing temperature of 1,000 °C, water absorption indicators of less than 5% are achieved for grain compositions of 0-0.05 mm and 0-0.08 mm – 3.60% and 4.82%, respectively. These values are within the required range and can be adopted for the development of technology to manufacture clinker tiles based on the Verkhnealkunsky deposit mudstones. At a firing temperature of 1,050 °C, water absorption rates of less than 5% are achieved for all grain compositions except 0-0.63 mm. So for a grain composition of 0-0.315 mm, water absorption is 3.98%, for a grain composition of 0-0.16 mm, water absorption is 2.51%, for a grain composition of 0-0.08 mm, water absorption is 2.02% and for a grain composition 0-0.05 mm water absorption is only 0.77%.

Considering that it is planned to manufacture clinker tiles with water absorption ranging from 2% to 5%, the following main technological parameters can be identified. At a firing temperature of 1,000 °C, mudstones must be crushed for grain compositions of 0-0.05 mm and 0-0.08 mm; at a firing temperature of 1,050 °C, mudstones must be crushed to grain compositions of 0-0.08 mm, 0-0.16 mm and 0-0.315 mm.

The dependence of the ultimate bending strength of fired samples on the grain composition of crushed mudstones at different firing temperatures is presented in Fig. 5.



**Fig. 5.** Dependence of the ultimate bending strength of fired samples on the grain composition of crushed mudstones at different firing temperatures (a – 900 °C; b – 1,000 °C; c – 1,050 °C).

Indicators for ultimate bending strength of samples above 30 MPa are achieved at a firing temperature of 950 °C for grain compositions 0-0.05 mm and 0-0.08 mm, at a firing temperature of 1,000 °C – for grain compositions 0-0.05 mm, 0-0.08 mm and 0-0.16 mm, at a firing temperature of 1,050 °C – for all grain compositions except 0-0.63 mm. These indicators correlate with indicators for water absorption and are quite good. The high bending strength of the ceramic stone itself allows the production of tiles with reduced thickness and, accordingly, weight, which is an important competitive advantage – the lower weight of the tiles requires lower production costs and is more attractive for building constructors.

Having analyzed the pre-firing and firing technological properties of the Verkhnealkunsky deposit mudstones, the following conditions can be accepted as most suitable for the production of clinker tiles: mudstone grain composition of 0-0.08 mm with a firing temperature of 1,000 °C, grain composition of 0-0.16 mm with a firing temperature of 1,050 °C, including intermediate indicators for grain composition and firing temperature within the specified limits.

#### 4. CONCLUSIONS

In the course of laboratory tests, we examined the mineral and chemical composition of the Verkhnealkunsky deposit mudstones from the Russian Republic of Ingushetia, and the grain composition of these raw materials. The chemical composition of the Verkhnealkunsky deposit mudstones can be classified as semi-acidic raw materials with an  $\text{Al}_2\text{O}_3$  content of about 17% and silica content of about 60%, with a high content of colouring oxides. As far as the mineral composition is concerned, the clayey component can be characterized as polymineral, more specifically, hydromica-kaolinite. However, mixed-layer minerals are also present.

Tests aimed at determining pre-firing properties showed that the studied mudstones are a very promising raw material to manufacture clinker tiles. Their degree of grinding has a significant influence on technological properties. Raw materials with a grain composition of 0-0.16 mm and 0-0.08 mm make it possible to manufacture clinker tiles using the extrusion molding method without introducing corrective additives [18]. For grain compositions of 0-0.16 mm and 0-0.315 mm, it is necessary to provide for the introduction of natural or artificial plasticizing additives. When grinding mudstones to a fraction of 0-0.05 mm, it is necessary to introduce lean additives, which can be waste from drying and firing of tiles. An analysis of the firing technological properties of raw material compositions based on the Verkhnealkunsky deposit mudstones showed that a grain composition of 0-0.08 mm with a firing temperature of 1,000 °C or a grain composition of 0-0.16 mm with a firing temperature of 1,050 °C can be used. Table 3 presents the main physical and mechanical properties of fired samples based on the recommended grain compositions of 0-0.08 and 0-0.16 mm.

**Table 3.** Physical and mechanical properties of samples.

Raw materials	Grain composition, mm	Firing temperature, °C	Water absorption, %	Bending strength, MPa
Argillite of Verkhnealkunsky deposit	0-0.08	1,000	4.85	37
	0-0.16	1,050	2.51	39

Based on the results obtained, we can conclude that the Verkhnealkunsky deposit mudstones, with their huge reserves, are quite suitable raw materials to manufacture clinker tiles. We consider the most preferable method of producing ceramic tiles to be the extrusion molding method with low molding moisture content of the raw materials and additional pressing of the products. This production method is beneficial due to its economic efficiency, and in this way it is also possible to achieve higher strength of raw and fired ceramic stone, which will significantly reduce the thickness and, accordingly, the weight of the tiles themselves.

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