



Wall materials based on low-grade loams and industrial waste

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Abstract: Clay raw materials used in Kyrgyzstan for the ceramic industry characterized as low-plasticity, highly sensitive and saline. In addition, the high content of carbonates in loams leads to the production of low-quality bricks. In order to obtain ceramic bricks with high-quality performance characteristics, it is necessary to use innovative approaches in the preparation of clay raw materials. The raw used materials were local loess-like loams from the Orok deposit and ash from the Bishkek thermal power plant and a surfactant – sodium naphthenate. The loam and ash subjected to mechanochemical activation by joint grinding in activator-mixer and adding sodium naphthenate together with mixing water. The dried cylindrical samples fired in the temperature range of 900, 950 and 1000 °C.

The results of the studies showed that mechanochemical activation of ash-clay raw materials increases the plasticity of the clay-ash mass by 140 %, while reducing the molding moisture and sensitivity coefficient.

The sintering process is intensified: at a firing temperature of 950 °C and 60 % ash, it is possible to obtain a shard with a density of 1.57 g/cm³ and water absorption of 17 %. The compressive strength is 16.3 MPa.

Amorphization and destruction of raw material particles contribute to an increase in the glass phase content. As a result, open pores are tightened, forming a strong monolithic structure.

The obtained samples based on mechanochemical activation with the addition of 60 % ash have the M150 grade and frost resistance of F 25.

Keywords: loams, ceramic mixture, mechanical-chemical activation, brick, compressive strength, water absorption, fire shrinkage, microstructure, firing temperature

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1. INTRODUCTION

Among the wide variety of building materials and products, ceramic bricks do not lose their relevance in the modern world. The development of the construction industry in Kyrgyzstan determines the need for high-quality competitive wall ceramic material. Ceramic bricks are characterized by high strength, resistance to atmospheric influences and durability, which makes them an ideal material for construction. The production of such bricks could reduce dependence on imported building materials. However, brick manufacturing enterprises use local loams, characterized as carbonized, sanded and saline with low plasticity. Ceramic material based on such raw materials has low strength and density, which negatively affects operational properties, such as efflorescence and frost resistance. And low plasticity makes it difficult to mold the ceramic mass.

To obtain high-quality construction ceramics, it is necessary to use new technologies for processing low-grade clay raw materials.

An increase in the plasticity of the ceramic mass can be achieved by adding various surfactants [1-3] and modifiers [4-6], plastic clays [7-9], organic, chemical and other additives [10-12] to the ceramic mass, and drying properties – leaners [8,13,14] and various modifiers [15, 16]. The authors have found that the introduction of coal mining waste contributes to the improvement of the technological and physical-mechanical properties of the ceramic material [13, 17, 18].

In order to save natural raw materials and utilize waste, the authors proposed using quarry waste in brick production [19, 20]. This technology solves the environmental problem associated with waste accumulation increases the strength and performance characteristics of ceramic bricks. However, at the same time, the mass of the brick increases.

The authors proposed to use waste from the cellulose industry in ceramic production. However, the products were characterized by low strength, which does not allow them to be used in the construction of load-bearing walls [21, 22].

Scientists have discovered the possibility of obtaining ceramic bricks with the addition of wastewater sludge [23-25] and rice husks [26]. In work [23], wastewater and textile industry were added waste to the ceramic mass. Brick grade 150 with low water absorption was obtained. However, when fired, the products characterized by high shrinkage.

As can be seen from the literature review, the use of industrial waste is gaining momentum in ceramic production.

One of the multi-ton wastes in the world is ash and slag waste. The largest amount of this waste, according to the analysis [27], is India - 112 million per year, of which 38 % used in the production of building materials. The authors then provided the following analysis: China – 100, the USA – 75, Germany – 40, Russia – 26.7 million tons per year, the use of which in secondary production is 45, 75, 85 and 18 %, respectively. The involvement of ash and slag waste in the ceramic industry is an important area in the field of resource conservation and ecology.

Every year, Kyrgyzstan accumulates about 1.6 million tons of ash and slag. Ashes from hydro-removal The Bishkek Thermal Power Plant (BTPP) occupies about 2 hectares within the city limits and poses an environmental threat, polluting the soil, groundwater, air, etc.

The use of BTEC ash in the production of ceramic material as an additive-modifier can not only replace natural raw materials, but also solve the problem of ash waste disposal.

The authors found that adding ash to the raw material mixture in an amount of 20 % reduced efflorescence on the samples and decreased their weight [15]. In the work [28], the possibility of obtaining ceramic materials based on compositions of mineral binders (clay and zeolite rock) with ash-containing waste from the combustion of solid fuels (ash microspheres) was considered.

Ash and slag material or hydro-removal ash plays the role of a leaner in ceramic material, and during firing – a burnout additive [15]. However, ash-ceramic products are characterized by low density and high water absorption, which, in turn, leads to a decrease in their frost resistance. [29] In addition, the presence of a large amount of carbonates in clay raw materials causes the formation of spalls (bumps) and cracks in the fired product.

Therefore, it is necessary to pay special attention to the preparation of ash-clay raw materials at the initial stage of the technological scheme of brick production.

Despite a large number of scientific studies in the ceramic industry, the issues of obtaining ceramic bricks in the Kyrgyz Republic (KR) with high-quality performance remain open characteristics.

The harmful effects of carbonates can be eliminated by long-term grinding of clay raw materials [30, 31]. A significant reduction in calcite grains occurs with mechanical activation for 20 minutes [31]. Mechanical activation helps reduce large carbonate inclusions and neutralizes their effects.

Mechanical activation of clay raw materials is a process in which the raw materials subjected to intensive mechanical action in order to improve their physical and chemical properties. The works of such scientists as V.V. Boldyrev, P.A. Rebinder, B.V. Deryagin, N.A. Avvakumov, V.D. Krotov, A.N. Kuznetsov, G.I. Frumkin, L.N. Storozhenko, Tatsky and others have proven that mechanical activation of loam helps to increase the reactivity of clays and improve the quality of final products, such as ceramic bricks.

Mechanical activation or activation of raw materials by grinding is a method of intensifying physical and chemical processes [31-33]. It is based on changing the reactivity of solids under the influence of mechanical forces [34, 35].

The aim of this work was to improve the performance characteristics of ceramic bricks based on local loams and ash waste from the BCHPP.

To achieve this goal, the following tasks were solved:

- study of the characteristics of raw materials;
- analysis of physical and mechanical characteristics;
- analysis of the structure formation of ceramic material;
- determination of operational properties of ceramic bricks.

2. MATERIALS AND METHODS

Local loess-like loams from the Orok deposit and ash from the Bishkek Thermal Power Plant (BTPP) were used as raw materials, the chemical and granulometric composition of which is given in Tables 1 and 2. An alkaline waste from chemical production, sodium naphthenate (SN), was used as a surface-active substance (SAS).

Table 1. Chemical composition of raw materials.

Raw materials	Chemical composition, % by weight									
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO+M gO		SO ₃	K ₂ O	Na ₂ O	Ppp	Σ
Loam	52	13.03	4.81	14.31		1.72	3.05	1.06	9.94	99.92
Ash BTEC	51.57	21.87	3.70	3.09	1.24	1.47	0.52		16.54	100

Table 2. Granulometric composition of raw materials.

Raw materials	Fractions, mm					
	less than 0.005	0.005-0.01	0.01-0.25	0.25-0.5	0.5-1	1-2
Loam	13.6	34.01	45.24	3	1,1	0.15
Ash BTEC	6.6	11.48	51	7.36	15.36	8.2

The loams of the Orok deposit are loess-like, dense, coarsely dispersed, lean, and low-plastic (plasticity index 5.7). The content of water-soluble salts is 1.66 %. The sensitivity coefficient is 2.5 (highly sensitive to drying). The mineral composition represented by the following components in %: quartz – 30-35; feldspar – 20-25; carbonates – 15-20; iron hydroxide – 5; micas and chlorites – 10; organic impurities – 5-7.

Ash from the BCHPP represented by glassy and crystalline phases. The crystalline phase of the ash consists of clayey matter amorphized by roasting and grains of detrital material of quartz, calcium carbonate and magnesium, as well as dicalcium silicate, calcium aluminate, mullite, etc. released from the melt [6].

According to its chemical composition (Table 1), ash is characterized by a high content of silica and alumina.

Ash particles are mainly characterized by a rough surface, which promotes their dense aggregation [6]. There are also particles that are spherical aggregates with a tendency to globularization.

The specific surface area and granulometric composition of ash depend on the type of fuel burned, combustion conditions and the method of ash removal outside the boiler house or thermal power plant. Since ash is a product of the burning of the mineral part of the fuel, the composition of the latter primarily determines the properties of ash. The studied ash of the BCHPP is a loose material of black-gray color with an average density of 870 kg/m^3 , a specific surface area of $2240 \text{ cm}^2/\text{g}$, which classifies it as medium-dispersed. The content of water-soluble salts is 0.28% (unsalted raw materials) [6].

The plasticizing additive sodium naphthenate is an alkaline waste in the form of an aqueous solution of a petrochemical plant. In appearance, the reagent is a light-moving liquid of dark brown color with a density of $1.01\text{-}1.03 \text{ g/cm}^3$, mass fraction, %: petroleum acids – 42; mineral oils – 56; sodium salts – 2.

Mechanochemical activation was carried out as follows. Ash from BTEC in an amount from 0 to 90 % was added to loam and subjected to joint grinding for 3 minutes in an activator-mixer operating on the principle of a centrifugal impact mill at a speed of 800 revolutions per minute. Then surfactants were introduced in an amount of 0.1% together with mixing water of 22-24%. The activation time and the amount of additive were selected empirically [29].

After aging for 3 days, cylindrical samples formed using the plastic molding method and dried at $105\text{-}110 \text{ }^\circ\text{C}$ to constant weight. Then the samples were fired in a laboratory muffle furnace in the temperature range of 900, 950 and $1000 \text{ }^\circ\text{C}$ with a holding time of 1-1.5 hours.

The physical and mechanical properties of ash-ceramic samples were determined using standard laboratory methods in accordance with current GOSTs.

3. RESULTS AND DISCUSSION

The technological properties of the samples were determined on the following compositions: loam + ash (INIT); loam + ash + MA (MA); loam + ash + MA + surfactant (MCA).

The results of the studies showed that the plasticity of the ceramic mass decreases with an increase in the addition of ash (Fig. 1, a) and increases with the combined mechanical activation of loam with ash (MA). The addition of surfactants to the mechanically activated ash-clay mixture (MCA) plasticity increases even more noticeably. When adding 60 % ash with MCA, plasticity increases from 2.5 to 6 (i.e. by 140 %) compared to the original sample, then drops sharply.

The molding moisture increases with the increase of ash addition and decreases with MA (Fig. 1, b). However, with MCA, the addition of ash up to 60 % reduces this indicator by 4 %.

During combined MA, crushed particles of loam and ash, including unburned coal, spherulitic, amorphized clay, etc., colliding and grinding with each other and with loam particles contributed to the formation of reactive sections of particles. And during MCA (subsequent activation of surfactants), the peptizing effect of ash was completely eliminated: intermolecular forces increased due to intensive adsorption of surfactants on reactive sections, caused by the grinding of particles.

As is known, crushed powders are always more active adsorbents than large grains. That is, this MCA has a hydrophobic-plasticizing effect. In addition, the introduction of surfactants contributed to the improvement of moisture distribution processes in the clay-ash mixture.

With MCA, the sensitivity coefficient is 0.15 at a maximum ash content of 90 % (Fig. 1, c). With an ash content of 60 %, the sensitivity coefficient of the samples is 0.52, based on MA 0.6, and with MCA it is 0.18.

From the analysis of the presented results, we found that we consider it appropriate to use joint MCA for further research.

To assess the sinterability, the physical and mechanical characteristics of ash-ceramic samples fired at temperatures of 900, 950 and $1000 \text{ }^\circ\text{C}$ were determined based on the initial (INIT) and mechanically activated compositions with subsequent activation of surfactants (MCA). Were determined the compressive strength, water absorption and total shrinkage.

In Fig. 2 it can be seen that shrinkage decreases with the addition of ash for samples based on the initial compositions and increases with increasing firing temperature. Fire shrinkage increases with MCA and decreases significantly with the addition of ash.

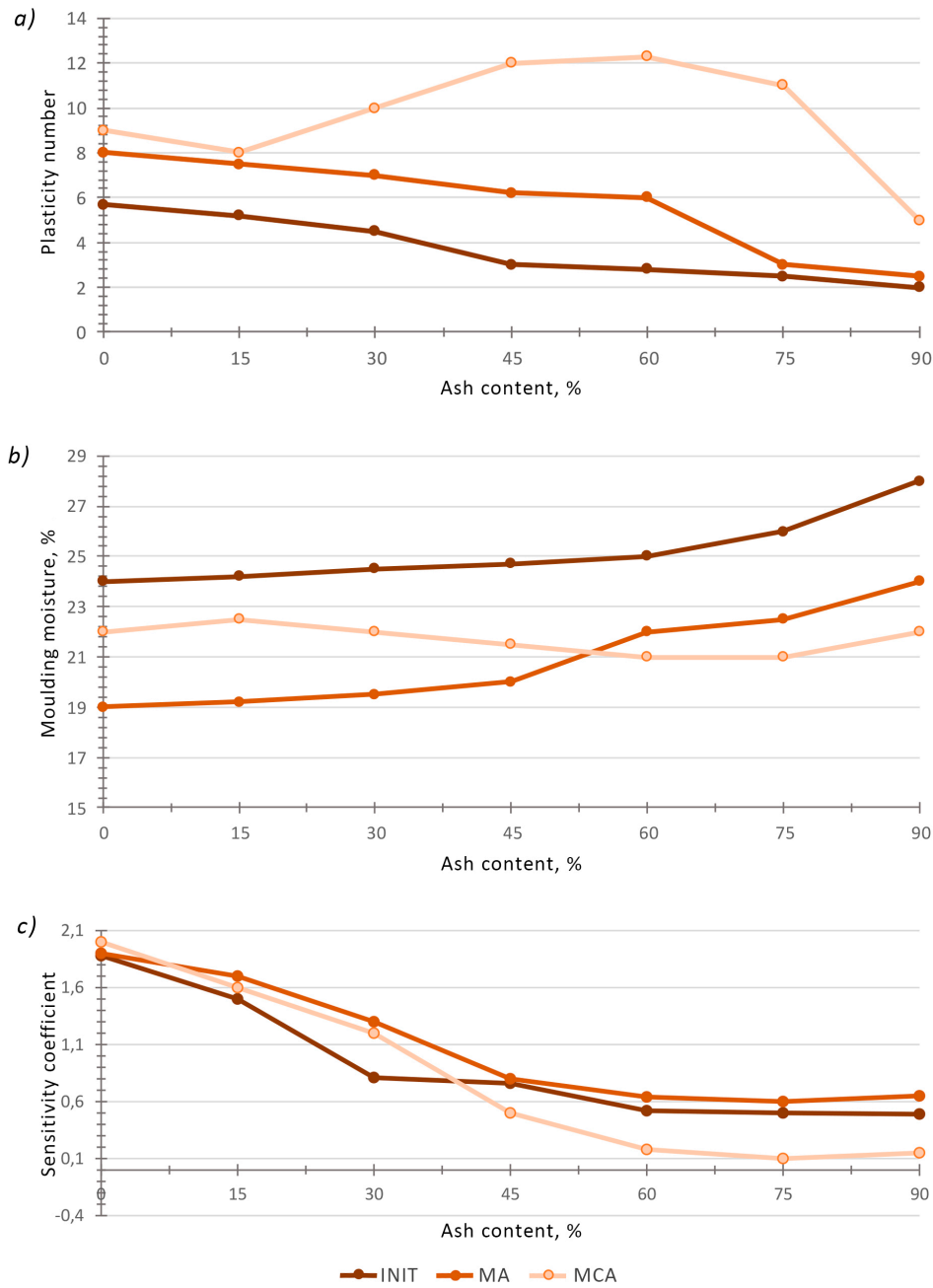


Fig. 1. Effect of ash content on the technological properties of ash-clay samples with different types of activation: a – plasticity index; b – moulding moisture; c – sensitivity coefficient.

The presented results show (Fig. 2) that MCA intensifies the sintering of ceramic material. Thus, fire shrinkage changes significantly depending on the increase in firing temperature from 900 to 1000 °C. However, when adding ash up to 60 % and MCA at a firing temperature of 950 °C, the shrinkage is 0.45, and at a temperature of 1000 °C - 0.41 %, i.e. insignificantly. Therefore, at 950 °C it is possible to obtain a sintered shard.

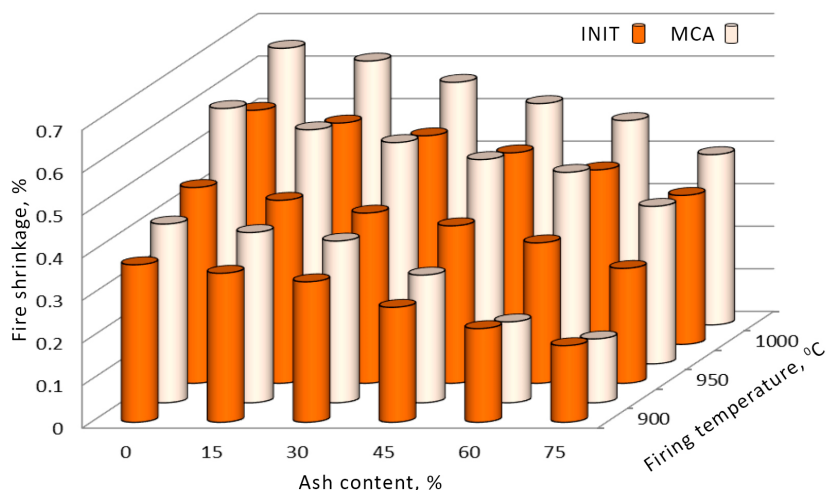


Fig. 2. The influence of mechanochemical activation and firing temperature on fire shrinkage at different ash contents.

The sintering of the samples is indicated by the values of density, water absorption and compressive strength. From Fig. 3 and 4 it is evident that with the addition of ash these indicators deteriorate: a decrease in density and an increase in water absorption observed (Fig. 3, a, b), and, consequently, an increase in porosity.

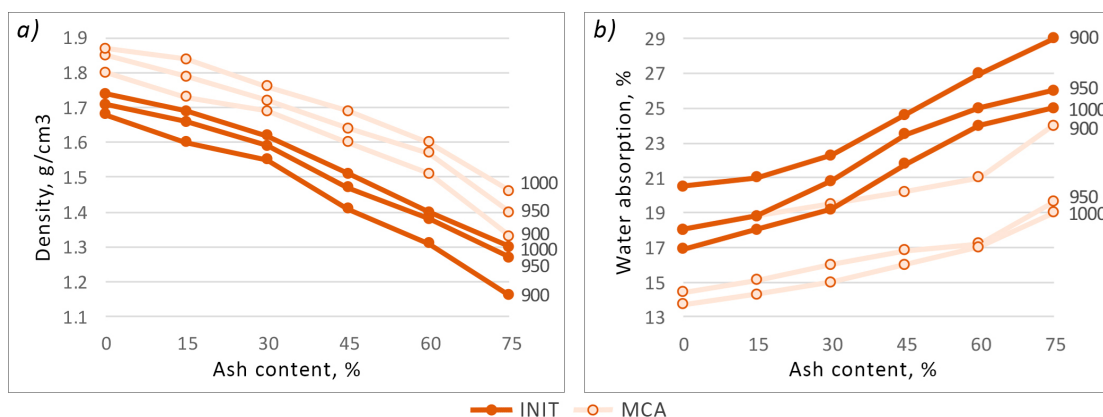


Fig. 3. The effect of mechanochemical activation and firing temperature on water absorption – a, and density – b with different ash content.

The decrease in density of the original ash-ceramic samples (Fig. 3, a) is due to increased gas formation due to the rupture of spherical and carbon particles contained in the ash during firing. The water absorption values of these samples, on the contrary, increase (Fig. 3, b).

The density of the samples based on MCA increases significantly. Joint mechanical activation contributed to the destruction of spherical coal particles, and the surfactant additive – to the decrease of the molding moisture, which contributes to the reduction of pores. The water absorption curves change inversely. The addition of ash up to 60 % at the firing temperature of 950 °C shows an active increase in density from 1.38 to 1.57 g/cm³ and a decrease in water absorption of the samples from 25 to 17.2 % based on MCA in comparison with the samples based on the initial mixtures.

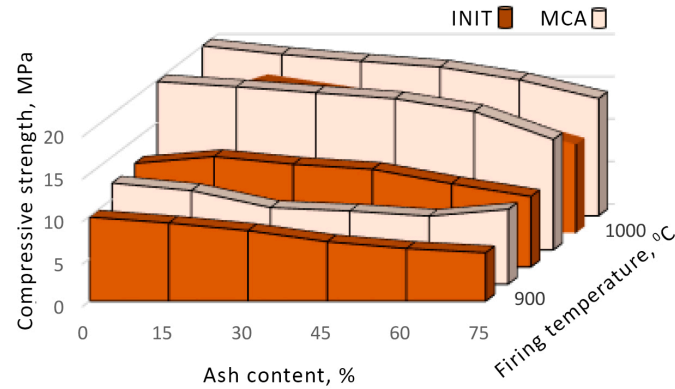


Fig. 4. The effect of mechanochemical activation and firing temperature on compressive strength.

The improvement of the physical and mechanical characteristics of the ash-ceramic samples based on MCA. In the Fig. 4, the strength values of the ash-ceramic samples based on the initial mixtures decrease with an increase in the ash additive and increase with MCA and the firing temperature. Thus, with an ash additive of up to 60 %, the compressive strength of the samples based on the initial mixtures is 6.2 MPa at 900 °C, 9.8 MPa at 950 °C and 13 MPa at 1000 °C. The compressive strength of the samples based on MCA: 8, 16.3 and 16 MPa at 900, 950 and 1000 °C, respectively. A further increase in ash to 75 % leads to a slight decrease in strength.

Based on the above, it can be stated that to obtain ceramic material of the M150 brand, it is possible to select a composition of 40 % loam and 60 % BTEC ash and a firing temperature of 950 °C.

Micrographs of the samples (Fig. 5) showed a decrease in open pores and a decrease in the number of closed pores.

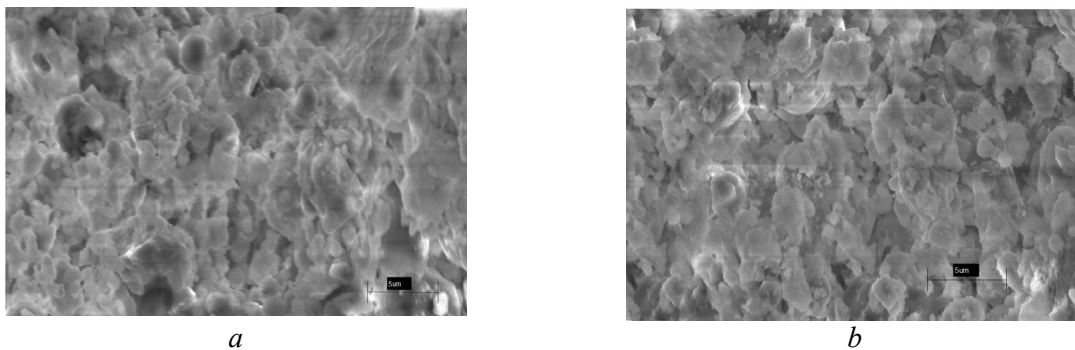


Fig. 5. Photographs of the microstructure of shards based on a) original and b) MCA at a magnification of 1600x, fired at 950 °C.

In the microphotographs of samples based on the initial clay-ash mixture (Fig. 5, a), particles of spherulitic structure with sizes from 0.581 to 2.678 µm and open and closed pores from 0.838 to 5.711 µm are clearly visible.

MCA of the mixtures allowed us to obtain a ceramic material with a denser structure (Fig. 5, b). As can be seen from the fracture, the spherulitic particles and open pores decreased to 1.087 µm.

Thus, the MCA of the clay-ash raw material contributed to the amorphization and partial destruction of the ash particles of spherulitic structure. As a result, the content of the glass phase as a cementing substance increased, which contributed to the tightening of open pores, forming a strong monolithic structure [29].

The results of X-ray phase analysis of the ceramic shard obtained from both the original and MCA (mechanically and thermally activated) rational mixtures (Fig. 6) indicate the amorphization of particles.

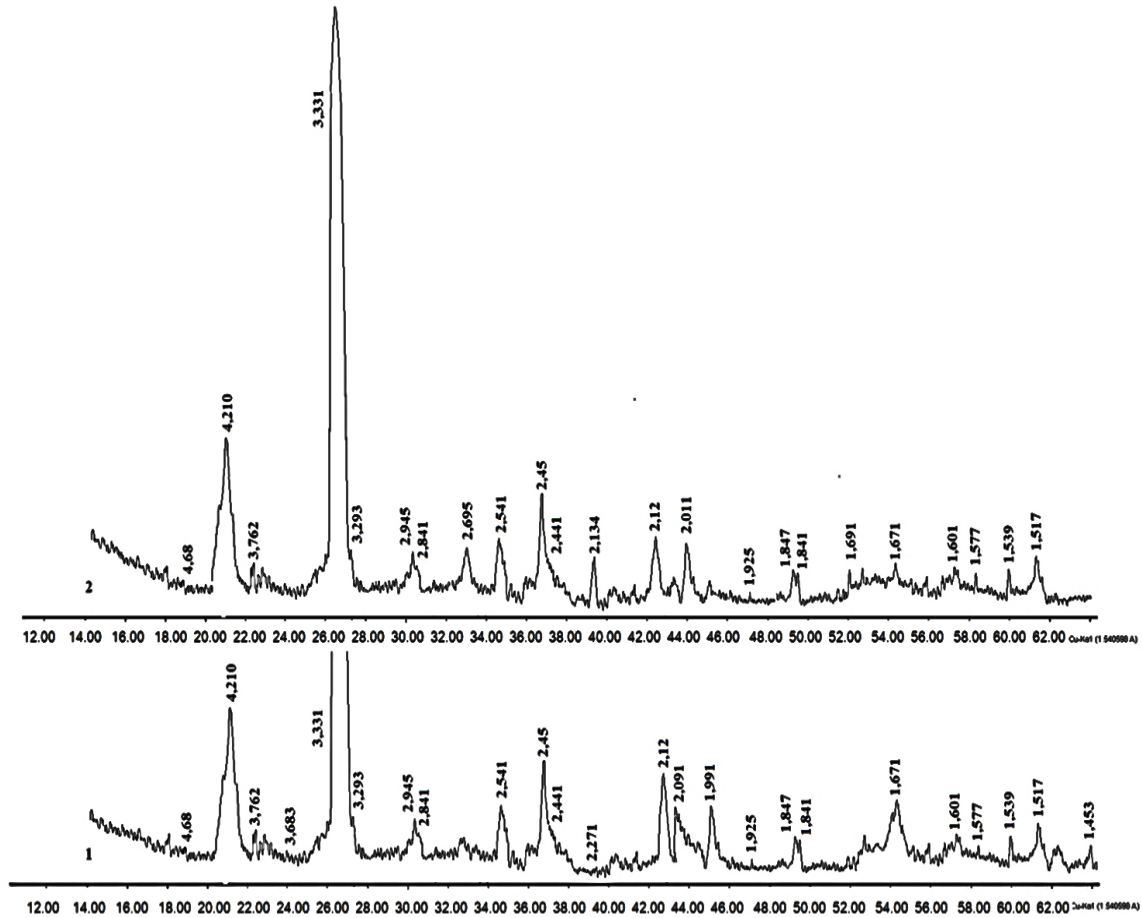


Fig. 6. X-ray diffraction patterns of fired samples at 950 °C based on loam with ash – 1; MAH loam with ash – 2.

As seen in Fig. 6, 2 the reduction of quartz in the MCA alumina-containing raw material indicates its partial crosstabulation due to the disruption of quartz structure during the activation of loams. Quartz introduced by the ash melts completely, as it has already been subjected to thermal exposure. MAH, in turn, enhances reactivity because preliminary thermal and mechanical activation promotes an effective modification of the crystal lattice and the material's reactivity.

The X-ray patterns show that calcium lines disappear because MCA significantly disrupts the crystal lattice of carbonates, forming free bonds, while fine grinding and uniform distribution of the material throughout the mixture facilitate complete binding of free calcium into minerals such as feldspars and anorthite. Thus, in the X-ray diffraction pattern of the fired loam-ash composition at 950 °C (Fig. 6, 1), the following were detected: mullite (3.762; 2.541; 1.841; 1.517 Å), anorthite (2.945 Å), quartz (4.68; 4.210; 3.331; 2.45; 2.12; 1.991; 1.671; 1.577; 1.539; 1.453 Å), feldspars (2.841; 2.441; 1.925; 1.601 Å), hematite (2.441 Å), and weak lines of calcite (3.683; 3.293 Å).

In the samples based on MCA mixtures (Fig. 6, 2), an increase in the amount of mullite is observed through additional peaks (2.695; 1.691 Å) and anorthite (2.134 Å) at higher angles. At the same time, free calcium oxide and calcium aluminate are absent in the material. The increase in feldspars results from reactions of clay minerals, silica, and calcite, and the crystallization of feldspars may proceed via different pathways, for example, from the amorphous phase, whose increase can be judged by halo changes. Quartz dissolution observed at peaks 2.12; 2.011; 1.671; 1.577; 1.539 Å with a shift toward lower angles.

Finished samples based on MCA clay- ash raw materials with the addition of 60 % ash had the M150 grade with frost resistance of 25 cycles. The reduction of pores, in turn, increased the density and, as a consequence, the frost resistance of the ceramic material.

4. CONCLUSION

In a result of the research, we have come to the following conclusions:

1. The addition of loam moss with ash significantly increases the plasticity of the clay-ash mass by 140 %, while reducing the molding moisture and sensitivity coefficient.
2. MCA intensifies the sintering process: at a firing temperature of 950 °C and 60 % ash, it is possible to obtain a shard with a density of 1.57 g/cm³ and water absorption of 17 %. The compressive strength is 16.3 MPa.
3. MCA of clay-ash raw materials contributed to amorphization and partial destruction of ash particles of spherulitic structure. As a result, the content of glass phase as a cementing substance increased, which contributed to the tightening of open pores, forming a strong monolithic structure.
4. The obtained samples based on MCA with the addition of 60 % ash have the grade M150 and frost resistance F 25.

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