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New technologies and compositions for recycling non-ferrous metallurgical waste into acid-resistant ceramics without using traditional natural raw materials

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Abstract: This study investigates non-ferrous metallurgy waste – specifically, the clay fraction of zircon-ilmenite ore gravity separation tailings (ZIGT) – with the aim of using it as both a clay component and a non-plastic additive (chamotte derived from ZIGT) in the production of acid-resistant ceramic tiles. It was found that samples made solely from ZIGT (without any additives), fired at temperatures of 1250–1300 °C, do not meet regulatory requirements for acid resistance. Introducing 40 wt. % chamotte into the ceramic body was found to be optimal for producing acid-resistant tiles at 1300 °C that comply with all requirements of GOST 961-89 Acid-Resistant and Thermo-Acid-Resistant Ceramic Tiles, grade KSh (chamotte-based acid-resistant tiles). Increasing the chamotte content beyond 40 wt.% reduces the clay binder fraction, which in turn lowers the plasticity index (to below 11), causing cracks to form in the samples during shaping. The phase composition of four tile samples with varying ZIGT and chamotte contents was analyzed. *X*-ray diffraction patterns of the samples fired at 1300 °C revealed prominent peaks corresponding to mullite, cristobalite, quartz, and hematite, which were also confirmed by IR spectroscopy. The formation of mullite is crucial in the production of acid-resistant ceramics, as mullite is the primary phase determining the operational properties of the material. As a result, new ceramic compositions were developed and acid-resistant tiles were obtained from non-ferrous metallurgy waste without the use of conventional natural raw materials.

Keywords: clay fraction of zircon-ilmenite ore gravity tailings (ZIGT), chamotte, acid-resistant ceramic tiles, metallurgical waste, phase composition, mullite, cristobalite, quartz, hematite.

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Новые технологии и составы по рециклингу отходов цветной металлургии в производство кислотоупоров без применения традиционного природного сырья

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Аннотация: В работе исследованы отходы цветной металлургии – глинистая часть «хвостов» гравитации циркон-ильменитовых руд (ГЦИ), в целях ее использования в качестве глинистого компонента и отощителя – шамота из ГЦИ, для получения кислотоупорной плитки. Установлено, что образцы из ГЦИ (без применения отощителей), обожженные при температурах 1250–1300 °C, не соответствуют нормативным требованиям по кислотостойкости. Введение в керамическую массу 40 % шамота является оптимальным для получения обжигом при 1300 °C кислотоупоров, которые по всем показателям отвечают условиям ГОСТ 961-89 «Плитки кислотоупорные и термокислотоупорные керамические», марка КШ (кислотоупорные шамотные). Увеличение содержания шамота более 40 % способствует снижению доли глинистого связующего, в результате чего уменьшается число пластичности (до менее 11) керамической массы, и на образцах при формовании появляются трещины. Исследован фазовый состав 4 образцов плиток, отличающихся содержанием компонентов – ГЦИ и шамота. На рентгенограммах образцов, обожженных при температуре 1300 °C, основные интенсивные линии принадлежат муллиту, кристобалиту, кварцу и гематиту, что подтверждают ИК-спектры. «Муллитизация» при производстве кислотоупоров имеет важное значение, так как эксплуатационные показатели определяет именно муллит. Таким образом, разработаны составы и получены кислотоупоры из отходов цветной металлургии без употребления традиционного природного сырья.

Ключевые слова: глинистая часть «хвостов» гравитации циркон-ильменитовых руд (ГЦИ), шамот, кислотоупорная плитка, металлургические отходы, фазовый состав, муллит, кристобалит, кварц, гематит.

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Introduction

Physicochemical processes involving phase transformations play a critical role in the formation of stronger, acid-resistant minerals. These processes result in changes to morphological properties, crystalline phases, and texture, which together largely determine the performance characteristics of acid-resistant ceramics. For this reason, special attention is paid to these transformations during the thermal treatment of acid-resistant products. Industries such as chemical manufacturing, ferrous and non-ferrous metallurgy, and other environmentally hazardous sectors are increasingly focused on expanding the production of accessible acid- and heat-resistant materials. Accordingly, it is essential to develop such products not from expensive, conventional natural raw materials, but from low-cost industrial waste.

The expansion of metallurgical production has led to the transformation of vast areas into industrial zones, affecting not only densely populated regions but also agricultural land, designated residential construction sites, areas intended for industrial infrastructure, and even water protection zones [1—4]. As a result, the environmental situation in industrially urbanized areas has significantly deteriorated.

The specific nature of metallurgical enterprises contributes to the harmful interaction of industrial waste with the environment, adversely impacting the health of the local population. Additionally, the large volumes of metallurgical waste lead to soil contamination, pollution of nearby water bodies, and degradation of ecosystems [5; 6]. In terms of environmental pollution, metallurgical enterprises rank second only to the fuel and energy sector.

Some ecologists and researchers suggest that the growing environmental threat stems from a shortage of reducers — organisms known as decomposers — which can no longer effectively break down industrial waste into microcomponents, owing to the rapid accumulation of technogenic materials [7]. These waste materials differ significantly in chemical composition from natural substances, and the quantity of microorganisms (such as reducers) capable of decomposing them is clearly insufficient [7—9]. From the standpoint of integrated materials science, the development of zero-waste technologies is essential. These technologies regulate the reuse of industrial by-products in the manufacture of value-added products. Such recycling helps reduce subsidies for geological exploration and mining operations, while simultaneously freeing up large areas from the adverse effects of anthropogenic activity.

In the 21st century, metallurgy involves not only competitive, cutting-edge technologies, but also the generation of hazardous waste and the creation of aggressive industrial environments. In such conditions, acid-resistant materials (ARM) are used to protect personnel from direct exposure to toxic, corrosive agents and to prevent the degradation of wall and floor surfaces. These materials are most commonly represented by ceramic products — such as pipes, bricks, tiles, and various shaped components — that demonstrate exceptional resistance to a wide range of aggressive media.

In [10], the authors reported that replacing acid-resistant bricks with acid-resistant tiles reduces raw material consumption by a factor of 2.5—3.0. In addition, the thinner tile profile shortens both drying and firing operations. Acid-resistant tiles serve as a stable and long-lasting barrier, preventing contact between workers and harmful components of hostile industrial environments. These tiles can effectively replace expensive metal equipment and apparatus, as they are not only significantly more affordable but also corrosion-resistant [11].

The production of high-quality acid-resistant ceramics requires aluminosilicate raw materials rich in aluminum oxide — over 20 wt. % Al_2O_3 for clay components and over 25—30 wt. % Al_2O_3 for non-plastic additives. However, such materials are either scarce or depleted in many regions of Russia [6; 11]. Moreover, no funding is currently allocated for geological exploration aimed at discovering new raw material sources [6], which underscores the rationale for substituting natural resources with industrial waste. Additionally, recycling technogenic feedstock into value-added products contributes to achieving environmental safety goals, as outlined in EU Directive 2008/98/EC [6].

Thus, the depletion of key natural raw materials used in the manufacture of acid-resistant ceramics has prompted the rational substitution of conventional inputs with technogenic industrial waste. This not only facilitates the production of various ceramic goods, but also supports the protection of ecological systems.

The objective of this study is to investigate the feasibility of recycling and repurposing the clay fraction of zircon-ilmenite ore gravity separation tailings (ZIGT) — a technogenic by-product of non-ferrous metallurgy — as a clay binder in acid-resistant ceramic compositions, and to analyze the phase composition of ceramic tiles obtained by firing these materials.

Materials and methods

To analyze the elemental composition and microstructure of the raw materials, a JSM 6390A scanning electron microscope (Jeol, Japan) was used. Petrographic analysis was performed using immersion liquids, transparent thin sections, and polished sections, examined under MIN-8 and MIN-7 optical microscopes. The qualitative mineral (phase) compositions of the samples were determined using a Dron-3 automated X-ray diffractometer (Burevestnik, Russia) with CuK_α radiation and a β-filter, as well as a Spekord 75JR spectrophotometer (Carl Zeiss, Germany).

The basic physicochemical and technological properties of the ZIGT (zircon-ilmenite ore gravity separation tailings) were determined and analyzed in accordance with standard laboratory procedures and data from previous studies [12—15]. These properties included particle size distribution, plasticity, shrinkage, drying sensitivity, water permeability, sinterability, refractoriness, water absorption, mechanical strength, and others.

ZIGT, a by-product of non-ferrous metallurgy, was used as the clay binder in the production of acid-resistant ceramics, while chamotte derived from ZIGT, pre-fired at 1200 °C, was used as a non-plastic additive. Their oxide and elemental compositions are presented in Tables 1 and 2, while their microstructure and mineralogical composition are shown in Figs. 1 and 2.

The clay component (ZIGT) is formed as a homogeneous slurry with a moisture content of 37—45 % and a density of 2.36—2.42 g/cm^3 after screening and mechanical processing of the mined ore [6; 16]. A review of the literature and numerous studies have shown that ceramics shaped from homogeneous clay feedstock exhibit 10—15 % higher strength compared to those produced from clay taken directly from the quarry [16—19.

Based on its Al_2O_3 content, ZIGT is classified as a raw material with semi-acidic properties and an elevated content of colorant oxides ($Fe_2O_3 > 3$ %, see Table 1). According to its particle size distribution (58 % < < 0.001 mm, see Table 3), it is classified as a fine-grained (highly dispersed) material. In terms of plasticity, it is medium-plastic (plasticity index of 21—23); in terms of drying behavior, it is moderately sensitive; in terms of refractoriness, it is highly refractory ($t = 1580$ °C); and in terms of sintering behavior, it exhibits high sinterability, with a sintering interval of 120—150 °C.

Table 1. **Average oxide composition of the components**

Таблица 1. Усредненный оксидный химический состав компонентов

Material	Oxide content, wt. %							
	SiO_2	$Al_2O_3 + TiO_2$	Fe_2O_3	CaO	MgO	R_2O	ZrO_2	LOI*
ZIGT	58.77	22.43	6.74	1.28	1.54	1.58	0.82	7.04
Chamotte	61.02	25.18	7.58	1.54	1.89	1.93	0.86	–

* LOI – loss on ignition; $R_2O = Na_2O + K_2O$.

Table 2. **Elemental c™omposition of the components**

Таблица 2. Элементный химический состав компонентов

Material	Elemental composition, wt. %									
	C	O	Na	Mg	Al + Ti	Si	K	Zr	Ca	Fe
ZIGT	2.18	50.78	0.26	0.54	15.45	24.8	0.32	0.48	0.2	4.87
Chamotte	–	48.22	0.31	0.71	17.18	27.14	0.35	0.54	0.22	5.18

The main clay mineral in ZIGT is kaolinite (Fig. 2), which differs significantly from natural, kaolinite-rich clays. The elevated iron oxide content ($Fe_2O3 > 3$ %) promotes the formation of poorly crystalline kaolinite, as partial substitution of aluminum ions (Al^{3+}) by iron ions (Fe^{3+}) occurs within the mineral structure. This substitution leads to the formation of a solid solution in which iron ions are strongly retained in the crystal lattice and cannot be removed without structural degradation [16, 20—23]. Based on the kaolinite crystallinity index (0.68), ZIGT is classified as Class III. According to the author of monograph [20], the lower the structural order of kaolinite, the more it enhances the material's reactivity.

Thermal treatment of ZIGT at 1200 °C yielded chamotte, which is commonly used in ceramic materials as a non-plastic additive. Chamotte stabilizes firing shrinkage, helps establish a robust structural framework,

Table 3. **Particle size distribution of the clay component (ZIGT)**

Таблица 3. Фракционный состав глинистого компонента (ГЦИ)

Fraction content, wt. %	>0.063	0.063–0.01	0.01–0.005	0.005–0.001	<0.001
Particle size, mm	0.8	8.1	12.1	21.0	58

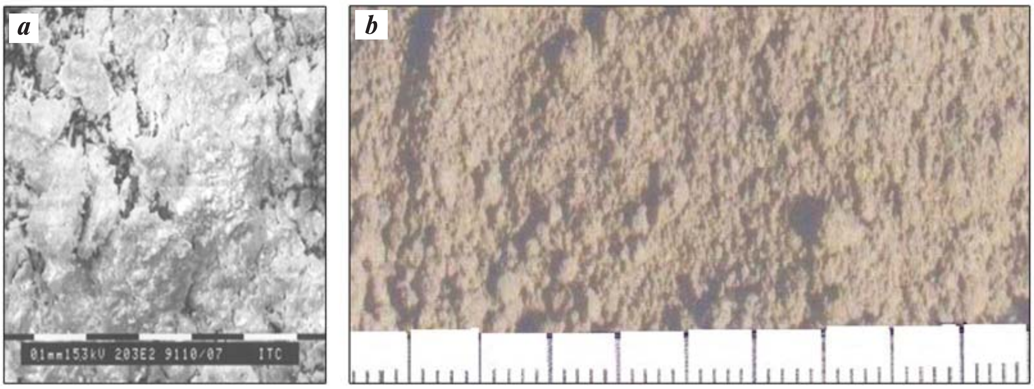

Fig. 1. Microstructure of raw materials: ZIGT (***a***) and chamotte derived from ZIGT (***b***)

Magnification: 1000$^\times$ (***a***), 10$^\times$ (***b***)

Рис. 1. Микроструктура сырьевых материалов: ГЦИ (***a***) и шамота из ГЦИ (***b***)

Увеличение 1000$^\times$ (***a***) и 10$^\times$ (***b***)

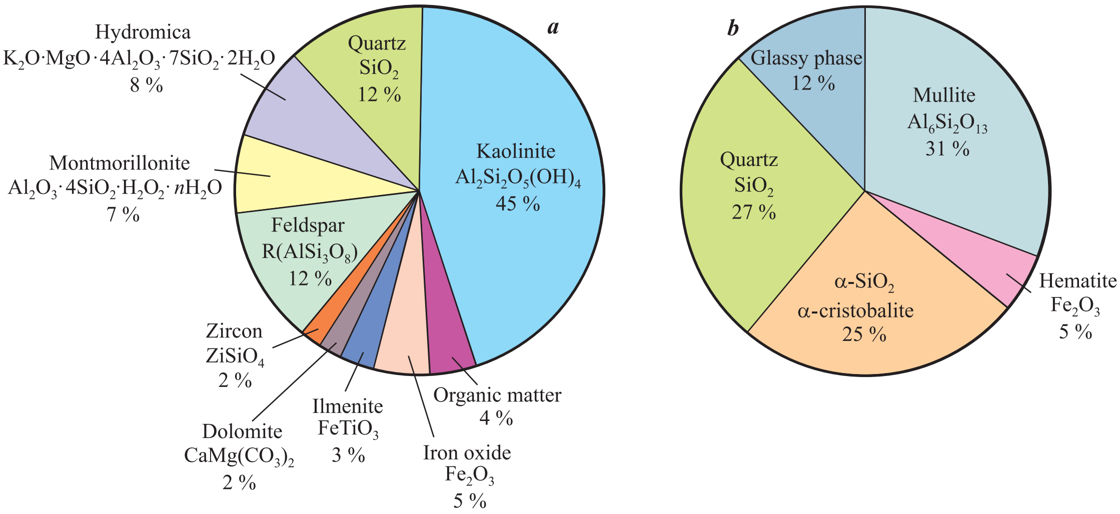

Fig. 2. Mineralogical composition of the raw materials: ZIGT (***a***) and chamotte derived from ZIGT (***b***)

Рис. 2. Минералогический состав сырьевых материалов: ГЦИ (***a***) и шамота из ГЦИ (***b***) and controls the plasticity of the ceramic body. As shown in Table 1, firing increased the aluminum oxide content of the clay material from 22.43 % to 25.18 %.

Production of acid-resistant tiles and their technical characteristics

Square acid-resistant tiles of type PK-1, measuring 100×100×20 mm, were produced using classical ceramic technology. ZIGT and chamotte were ground to a particle size of no more than 1 mm and thoroughly mixed (see Table 4). The ceramic samples were shaped by plastic forming at a charge moisture content of 22—24 %. After pressing, the tiles were dried to a moisture content below 5 %, and then fired at 1250 and 1300 °C. Their physical and mechanical (technical) properties are presented in Table 5.

Fig. 3 shows the *X*-ray diffraction (XRD) patterns of samples *1* and *4* fired at 1300 °C, while Fig. 4 presents their IR spectra. Sample *1* is included for comparison purposes, whereas sample *4* demonstrates optimal technical performance. It should be noted that potential impurities or poorly crystallized mineral phases, whose identification cannot be confirmed due to their low content, are not included in the diffractograms.

Results and discussion

The data presented in Table 5 show that samples based on composition *1*, when fired at 1250—1300 °C, do not meet the GOST acid resistance requirements. The addition of 20—40 wt. % chamotte to the ceramic body significantly improves the drying behavior of the semi-finished products (see table 4). However, after firing at 1300 °C, the samples comply with all GOST standards for acid-resistant tiles (see Table 5).

Composition *4*, which contains 40 wt. % chamotte, can be considered optimal, as it exhibits superior performance compared to samples *1*—*3* (Table 5). In selecting the optimal composition, it is also important to account for the plasticity of the clay binder, since its reduction causes the plasticity index of the ceramic body to drop below 11, resulting in cracking during forming..

X-ray diffraction analysis of samples *1* and *4* confirmed the presence of mullite, cristobalite, quartz, and hematite, which was further supported by IR spectroscopy (see Figs. 3 and 4). In the Al_2O_3—SiO_2 system, both mullite ($3Al_2O_3 \cdot 2SiO_2$) and sillimanite ($Al_2O_3 \cdot SiO_2$) can form. These phases exhibit similar crystal structures, as mullite is regarded as a structurally disordered or defective form of sillimanite. Consequently, their XRD patterns are nearly indistinguishable, although they can be differentiated by their IR spectral signatures [17; 20; 23—26].

The crystallization of α-cristobalite typically originates from amorphous silica released during mullite formation. Due to its high thermal expansion, cristobalite increases the permeability of acid-resistant ceramics while simultaneously reducing their mechanical strength [12; 14—16; 19; 20].

Previous studies [2; 29; 30] have demonstrated that hematite promotes the formation of iron-rich glass phases in ceramic materials, which serve as a cementing matrix within the ceramic body. During firing, a glassy phase of variable composition forms in the samples, depending on the firing temperature, as confirmed by IR spectra (Fig. 4). This phase enhances structural disor-

Table 4. **Sample compositions, plasticity of the ceramic body, and characteristics of semi-finished products after drying**

Таблица 4. Составы образцов, пластичность шихты и показатели полуфабрикатов после сушки

Component	Component content, wt. %			
	Sample *1*	Sample *2*	Sample *3*	Sample *4*
ZIGT	100	80	70	60
Chamotte	–	20	30	40
Plasticity index of ceramic body	22	17	14	11
Characteristics of semi-finished products after drying				
Shrinkage of dried tile, %	6.8	6.1	5.3	4.8
Drying time to constant shrinkage at t = 100÷120 °C, h	1.2	1.1	1.0	0.8
Compressive strength of dried body at 5 % residual moisture, MPa	7.8	5.2	3.9	2.7
Water permeability, 10^{-4} m^2/h	1.80	2.89	3.50	4.30

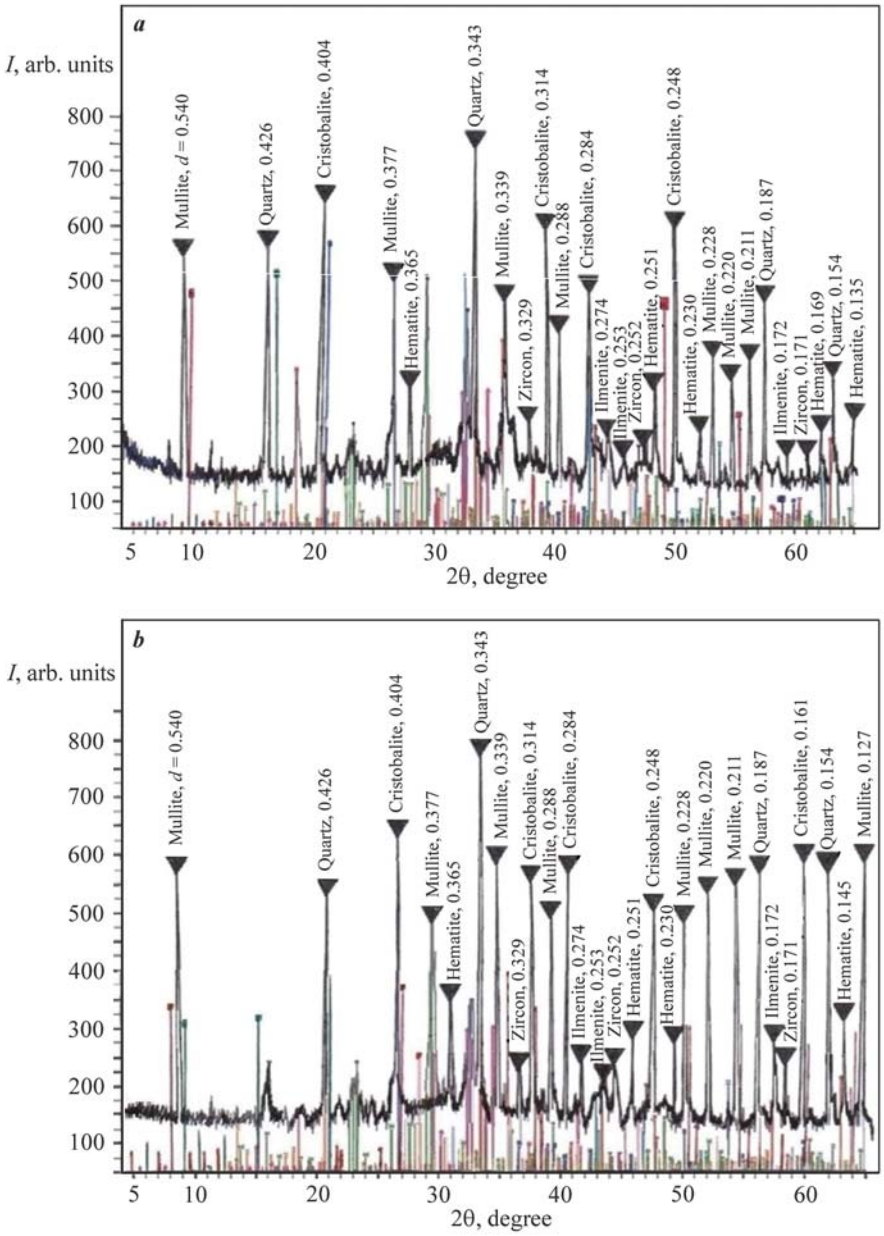

Fig. 3. *X*-ray diffraction patterns of samples *1* (***a***) and *4* (***b***)

d, nm – interplanar spacing between planes of the same family of parallel crystallographic planes

Рис. 3. Рентгенограммы образцов *1* (***a***) и *4* (***b***)

d, нм –межплоскостное расстояние между плоскостями одного семейства параллельных плоскостей

der during thermal treatment, elevates internal stresses, weakens the crystal lattices of individual mineral phases (and may even partially dissolve them), and can inhibit the development of specific phases such as mullite [11; 31]. The crystallization of structurally imperfect mullite is initiated at temperatures above 1100 °C.

Even in limited amounts, the glassy phase facilitates sintering of the ceramic body by promoting particle adhesion into a consolidated matrix, and may accelerate the overall densification and firing process.

Conclusions

1. The study revealed that fired samples made from the clay fraction of zircon-ilmenite ore gravity separation tailings (ZIGT) at temperatures of 1250—1300 °C did not meet the acid resistance requirements of GOST 961-89. The addition of 20—40 wt. % chamotte to the ceramic body significantly improves the drying behavior of the semi-finished product (except for strength), and after firing at 1300 °C, the resulting tiles

Table 5. **Physical and mechanical properties of acid-resistant tiles**

Таблица 5. Физико-механические показатели кислотоупорных плиток

Property	Sample *1*	Sample *2*	Sample *3*	Sample *4*	Regulatory requirements*
Firing temperature 1250 °C					
Water absorption, %	3.8	3.8	3.7	3.6	<5
Acid resistance, %	96.5	96.9	97.4	97.7	>98
Compressive strength, MPa	57.2	58.1	59.3	60.1	>50
Flexural strength (static), MPa	32.4	32.9	33.6	34.3	>25
Frost resistance, cycles	42	44	46	47	>20
Thermal shock resistance, thermal cycles	4	4	5	6	>5
Firing temperature 1300 °C					
Water absorption, %	2.3	2.3	2.2	2.2	<5
Acid resistance, %	97.8	98.2	98.5	98.7	>98
Compressive strength, MPa	63.8	65.4	67.8	69.4	>50
Flexural strength (static), MPa	38	39	41	42	>25
Frost resistance, cycles	58	63	67	69	>20
Thermal shock resistance, thermal cycles	6	6	8	9	>5

* According to GOST 961-89 “Acid-resistant and thermal acid-resistant ceramic tiles”, grade KSh (acid-resistant chamotte-based tiles).

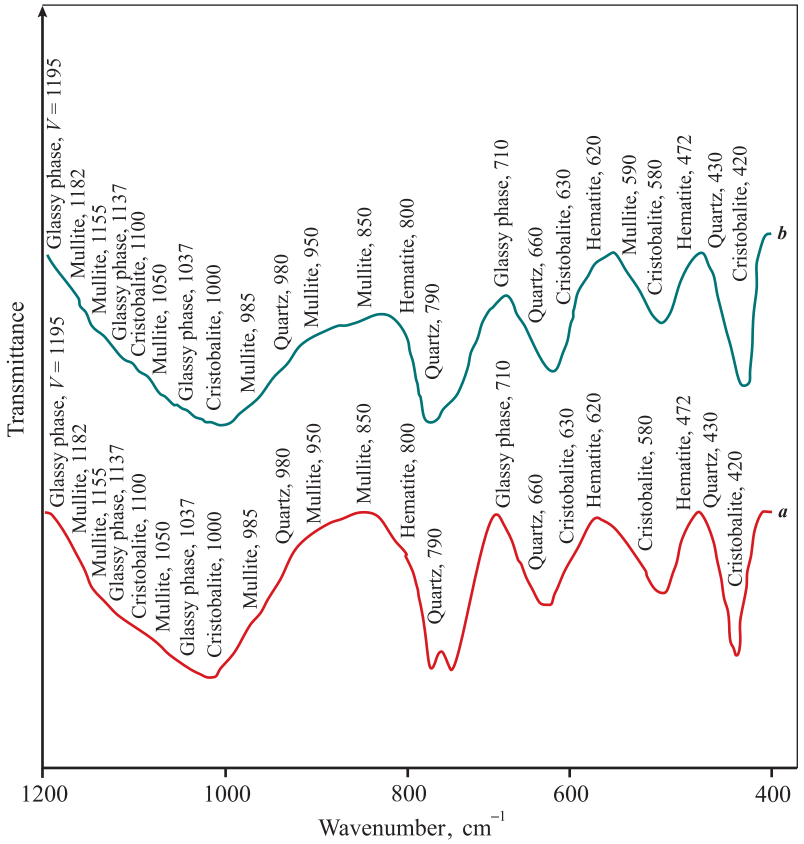

Fig. 4. IR spectra of samples *1* (***a***) and *4* (***b***)

Рис. 4. ИК-спектры исследуемых образцов *1* (***a***) и *4* (***b***) meet all regulatory standards for acid-resistant ceramic tiles.

2. Sample composition *4* was identified as optimal, as it fully complies with the GOST standards for acid-resistant tiles. The plasticity of the clay binder must also be taken into account, since its reduction causes the plasticity index of the ceramic body to drop below 11, resulting in cracking during forming.

3. *X*-ray diffraction analysis of compositions *1* and *4* revealed that the dominant peaks correspond to mullite, cristobalite, quartz, and hematite, which was confirmed by IR spectroscopy. The presence of a glassy phase was also detected in these samples, and it has a negative effect on the mineral structure.

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