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В роботі проаналізовані існуючі методи оцінки придатності глинистої сировини для виробництва архітектурно-будівельної кераміки та обґрунтована необхідність їх удосконалення в напрямку розроблення показника, заснованого на аналізі мінерального складу глин. З використанням композицій системи «каолінит – монтморилоніт – гідрослюда – кварц», які моделюють склад полімінеральних глин, встановлений вплив глиноутворюючих мінералів і домішок кварцу на властивості клінкерних керамічних матеріалів. Визначено, що позитивний вплив на водопоглинання матеріалів чинить монтморилонітові складова композицій, а на механічну міцність і морозостійкість – каолінова. Збільшення вмісту кварцу у складі композицій від 30 % до 50 % приводить до зростання водопоглинання матеріалів, зниження їх механічної міцності і морозостійкості до рівня, неприйняттого для керамічного клінкеру. Встановлено, що для отримання клінкерної кераміки вміст кварцу в композиціях не може перевищувати 40 %.

Розроблена діаграма мінерального складу, яка наочно ілюструє співвідношення основних породоутворюючих мінералів у глинах із вмістом в них 30 % і 40 % кварцу, допустимі для отримання сучасної клінкерної продукції. На діаграмі виділені області мінерального складу глин, придатних для виробництва фасадного, тротуарного і дорожнього клінкеру марок М200–300, а також дорожнього клінкеру марки М400 за температури випалу 1100 °С. Діаграма мінерального складу доповнює існуючі показники придатності глинистої сировини для виробництва архітектурно-будівельної кераміки і може служити додатковим критерієм їх технологічної якості. Діаграма може застосовуватися для аналізу придатності глинистої сировини для виробництва керамічного клінкеру за умови визначення лише мінерального складу глин без встановлення їх хімічного і гранулометричного складу

Ключові слова: архітектурно-будівельна кераміка, клінкерні матеріали, полімінеральні глини, критерії технологічної якості, промислове використання

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AN IMPROVEMENT OF CRITERIA FOR ASSESSING THE QUALITY OF CLAY RAW MATERIAL FOR ARCHITECTURAL AND CONSTRUCTION CERAMICS

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1. Introduction

The construction materials industry is an important component of any economy, which significantly affects the rate of development of other industries. The modern con-

struction materials industry is a complex area that includes many independent subsectors, one of which is the production of construction ceramics. The most widespread types of construction ceramics, which are widely used in modern construction technologies, are ordinary brick, large-size

hollow blocks, as well as architectural and facade ceramics in the forms of facing and clinker products.

In the production of architectural and construction ceramics, various types of mineral raw materials are used, including low-melting and refractory clays, kaolinities, bentonites, spondyl clays, mudstones, quartz-feldspar materials, and sands. However, the main raw material is low-melting clayey rocks, which are ubiquitous minerals as the raw material base for architecture and construction. These clay rocks belong to the quaternary sediment system and cover the surface of the Earth almost continuously [1]. Low-melting clays are polymineral natural mixtures of a highly variable composition, largely polluted with quartz, carbonate, feldspathic, ferrous, and other impurities, owing to the different geological conditions of their formation. Due to the contamination with impurities, the clays are often characterized by unfavorable technological properties because of the variability of the composition, which means poor reproducibility of properties even within one section of the field. This adversely affects the stability of production and the properties of ceramic products. This circumstance leads to the need to develop and improve ways to assess the suitability of mineral clay for the production of different types of architectural and construction ceramics based on analyzing various indicators of their composition. In this regard, research on the dependence of the properties of ceramic materials on the mineral composition of clays is essential.

2. Literature review and problem statement

Currently, to assess the technological quality of clay raw materials for the production of construction ceramics, diagrams are used that reflect the interrelation of the composition of clays with the areas of their use. As indicators of the composition, the granulometric and chemical compositions of clays and, to a limited extent, the mineral composition are widely used.

In study [2], a diagram was proposed, which was later called the Winkler diagram, as based on analyzing the particle size distribution of clays for four types of coarse ceramic products. Judging by modern publications, this diagram is still used in the analysis of brick clays [3] and raw materials for the production of ceramic blocks and tiles [4, 5]. However, the Winkler diagram has the disadvantage that in constructing it, the statistical sampling was only 47 samples (for individual products only 4 samples), which does not make it possible to consider it representative.

The Winkler diagram was later refined in [6], highlighting the clay area for products that do not require frost resistance (the so-called pottery clay), as a result of which the Kalnins diagram was obtained. The Winkler and Kalnins diagrams were tested on a more representative sample (150 samples of coarse ceramic clays and charges) by the author of [7]. As a result of these tests, a diagram of the particle size distribution of Schmidt was proposed, the disadvantage of which is the limitations of the considered ceramic products to only three types.

Since the publication of the H. Schmidt diagram, the products themselves as well as the requirements for their properties and the technology for the preparation of clay raw materials using modern equipment have changed significantly, which makes it possible to process low-quality clay. This required clarification of the existing diagrams, which

was done in [8] using 118 samples of clays of the modern period. As a result, a diagram was obtained of the particle size distribution by Sh. Vogt, which identified areas of the composition of clays suitable for modern ceramic products, including hollow and clinker ceramics.

From the point of view of the chemical composition of clays, the result of their industrial systematization by this indicator is represented by the Augustinik diagram [9] and its modern version according to Sh. Vogt [8]. At the same time, such systematization of clays is necessary but insufficient, since it does not take into account their mineral composition, which directly affects the phase composition and properties of the finished ceramic material.

An alternative to overcoming this problem can be mineral compositions of clays proposed in [8], optimal for obtaining high-quality products and obtained by excluding samples from the author's database that had problems with product quality. The disadvantage of this option is that these compounds are the desired average mineral content and, therefore, do not take into account the composition of natural polymineral clays, which give a negative result on the properties of the products of their roasting. This does not give a complete picture of the influence of the mineral composition of clays on the properties of ceramic products.

To eliminate this drawback, the authors of [10] experimentally established a direct relationship between the mineral composition of low-melting clays and the properties of different types of ceramic products. Diagrams were constructed with areas of hydromica, kaolinite and montmorillonite content in clays, which additionally included from 30 % to 50 % quartz. The diagrams allow analyzing polymineral clays for their suitability for obtaining wall and facade ceramics. These diagrams are adapted to modern low-melting clays, contaminated with quartz and other impurities, and their significant disadvantage is the absence of areas of mineral composition which correspond to the currently in demand clinker ceramic products. Systematized information on the above indicators of the technological quality of clay is given in Table 1.

Table 1

Criteria for assessing the quality of clay raw materials to produce construction ceramics

Assessment criterion	Authors	Pottery groups
Particle-size distribution	H. Winkler (1954)	Roof tile, solid and hollow bricks
	M. Kalnins (1957)	Roof tile, solid and hollow bricks, pottery ceramics
	H. Schmidt (1973)	Roof tile, solid and hollow bricks
	Sh. Vogt (2004, 2014)	Roof tile, solid and hollow bricks, clinker brick, rustic brick
Chemical composition	A. Augustinik (1975)	Stone ceramics, terracotta, roof tile, bridge clinker, construction brick, expanded clay
	Sh. Vogt (2004)	Roof tile, facade tile, clinker brick, construction and hollow bricks
Mineral composition	Sh. Vogt* 2004	Roof tile, hollow and facing bricks, clinker brick, facade tile, ceramic pipes
	H. Lysachuk, L. Shchukina, V. Tsovma (2013)	Ordinary brick, highly hollow stones, and facade tiles with water absorption from 2 % to 12 %

Note: * – means that this criterion is indicative

In the technology of clinker ceramics, the authors of [11] used an approach based on calculating the composition and amount of the melt produced by the state diagrams of aluminosilicate systems to assess the quality of clays for its production. This approach is not new as it was previously used by the authors of [12] in studying the fluxing ability of fluids for the technology of densely sintered thin construction ceramics. Despite the relatively quick predictive evaluation of raw materials using these methods, their disadvantage is the theoretical nature of the prediction, which requires experimental confirmation for reliability. In addition, this prediction is based only on the chemical composition of the raw material and does not take into account its mineral composition, which is fundamental and responsible for the phase interactions during firing of the materials and their properties.

The analysis of these publications shows that none of the quality criteria for polymineral clays considered covers a complete list of features by which clays can be recommended for various coarse ceramic technologies. The long-standing criteria developed in the second half of the 20th century (see Table 1) do not reflect the state of the modern raw material base of coarse construction ceramics and modern technologies for the processing of clay raw materials. The current criteria are adapted to modern types of polymineral clays and mass preparation technologies, but they are not fully summarized for modern types of coarse ceramic products. In particular, the available diagrams of granulometric, chemical and mineral compositions of clays do not allow predicting their suitability for obtaining simultaneously facing, paving and road clinker. All this reduces the practical value of the existing system of criteria for assessing the technological quality of clays and requires its improvement in the direction of supplementing with indicators that take into account the mineral composition of clays and new types of coarse ceramic products. In this regard, it is expedient to conduct a study on the development of an estimation indicator based on analyzing the mineral composition of clays and making it possible to evaluate their suitability for obtaining various types of modern clinker ceramic materials.

3. The aim and objectives of the study

The aim of the research is to establish the dependencies “composition – property” in relation to the models of polymineral clays and their roasting products for developing a criterion based on them, which would help evaluate the possibility of using clays in the technology of clinker ceramics.

To achieve this aim, the following research objectives are set and achieved:

- to obtain model mixtures of basic clay minerals and impurities in the system “kaolinite – montmorillonite – hydromica – quartz,” reflecting the composition of polymineral clays;

- to conduct, while using model mixtures of clay-forming minerals and quartz, an optimal experiment to study tendencies in the properties of ceramic materials depending on the composition of the mixtures;

- to develop a visual diagram indicating the areas of the mineral composition of clays, suitable for modern clinker production in accordance with the requirements of the standards.

4. Materials and methods for studying the influence of the mineral composition of clays on the properties of ceramic materials

4. 1. Materials, equipment and methodology of the experiment

Tests were carried out using monomineral clay rocks from existing fields in Ukraine: kaolinite clay from the Pologovsky deposit (Zaporizhia Oblast), hydromica clay loam from the Khorolsky deposit (Poltava Oblast) and montmorillonite clay from the Dashukovsky deposit (Cherkasy Oblast). The main impurities of the rock contained quartz, hematite, calcite and feldspar in various quantities. Clay rocks were crushed on laboratory rolls with a gap of 2 mm; they were soaked in water for seven days, and then the crushed clay suspension was passed through sieve No. 0056 to separate the sandy fraction. Enriched dried samples were subjected to the X-ray phase and petrographic tests using a DRON-3M X-ray diffractometer and a MIN-8 optical microscope manufactured by the USSR. The mineral composition of the enriched samples is given in Table 2.

Table 2

The results of studying the mineral composition of enriched clay rocks

Clay rock	Mineral content, %	
	Clay part	Impurities*
Kaolinite clay	Kaolinite – 70	Quartz – 20, others – 10
Hydromica loam	Illite – 60	Quartz – 30, others – 10
Montmorillonite clay	Montmorillonite – 83	Quartz – 10, others – 7

Note: * – means other impurities include carbonates, feldspars, and iron-containing minerals

From the enriched clay samples in the system “kaolinite – montmorillonite – hydromica – quartz,” model mixtures were compiled, which additionally contained quartz as an impurity. The quartz content in the mixtures varied from 30 % to 50 % (hereinafter mass percentages), which corresponds to the real mineralogy of clays for the production of coarse building ceramics [7]. In the case of a lack of quartz, its specified content in the mixtures was provided with a mixture of quartz sand, sifted through a sieve No. 02.

To study model mixtures of different mineral composition, the method of simplex-lattice planning of the experiment was used according to [10]. Fig. 1 shows a simplex lattice, in which the variable factors were kaolinite clay (X_1), hydromica loam (X_2), montmorillonite clay (X_3), and their mixtures. The number of experiments carried out in each case (by varying the content of quartz in mixtures, while studying different properties) was 10 experiments with three parallel tests.

Based on this lattice, cubic polynomial models of the general form were obtained:

$$y = \sum_{i=1}^3 b_i x_i + \sum_{\substack{i=1 \\ i < j}}^3 b_{ij} x_i x_j + \sum_{\substack{i=1 \\ i < j \\ i < k}}^3 \gamma_{ij} \cdot x_i \cdot x_j \cdot (x_i - x_j) + b_{ijk} \cdot x_i \cdot x_j \cdot x_k,$$

where b_i , b_{ij} , γ_{ij} , and b_{ijk} are the coefficients of the polynomial; x_i , x_j , and x_k are coded values of factors (mineral content).

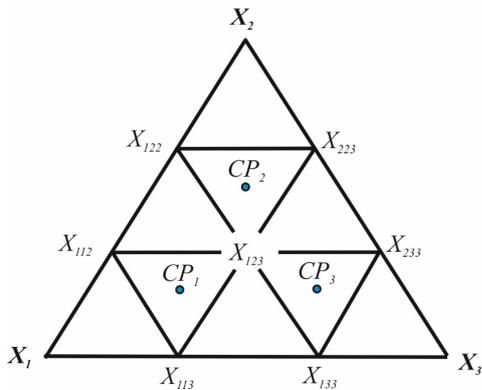


Fig. 1. The plan of the experiment to study model mixtures of clay minerals: X_1 – kaolinite clay; X_2 – hydromica loam; X_3 – montmorillonite clay; X_{112} – 2/3 kaolinite clay, 1/3 hydromica loam; X_{122} – 1/3 kaolinite clay, 2/3 hydromica loam; X_{223} – 2/3 hydromica loam, 1/3 montmorillonite clay; X_{233} – 1/3 hydromica loam, 2/3 montmorillonite clay; X_{113} – 2/3 kaolinite clay, 1/3 montmorillonite clay; X_{133} – 1/3 kaolinite clay, 2/3 montmorillonite clay; X_{123} – 1/3 kaolinite clay, 1/3 montmorillonite clay; 1/3 hydromica loam; CP_1 , CP_2 , and CP_3 – control points

To assess the accuracy of the models obtained, three experiments were additionally carried out at control points (CT) with the coordinates indicated in Fig. 1. The accuracy of the models was evaluated by the R -criterion, which is the relative deviation of the experimental response value from its calculated value:

$$R = \frac{|Y_e - Y_p|}{Y_p} \cdot 100\%$$

where R is the relative deviation of the experimental and calculated response values; Y_e is the experimental value of the response at the control point; Y_p is the value of the response in the control point, calculated by the equation.

The specified accuracy of the models according to the R -criterion was no more than 5 %, which is acceptable for the studied properties of ceramic materials.

Samples for testing in the form of parallelepipeds of 50×25×20 mm in size were obtained by plastic molding in a metallic form. After soft drying, the samples without drying cracks were burned in an electrically heated laboratory muffle furnace at a temperature of 1100 °C with isothermal aging for one hour.

The main regulatory characteristics of clinker ceramics, which determined their compliance with the requirements of the modern standard DSTU B W.2.7-245:2010, harmonized with European norms for clinker products, were studied as responses (Y).

4.2. Methods for studying the properties of the samples

The main indicators of the properties of the ceramic samples, determined in the experiment, were water absorption, ultimate compressive strength, and frost resistance (indirectly through the coefficient of structure).

Water absorption of the samples was determined by saturation in water at room temperature according to DSTU B W.2.7-42-97, whereas compression strength of

the samples was tested on a hydraulic press IP-500 produced by the USSR according to DSTU B W.2.7-248:2011.

To establish the coefficient of structure, the water absorption of the samples was additionally determined by boiling according to DSTU B W.2.7-42-97. The coefficient of structure was determined as the ratio of the water absorption value obtained by the saturation method to the value of this property obtained by the boiling method. Materials with the value of this coefficient more than 0.85 are considered to be frost resistant.

5. The results of studying the properties of the ceramic samples depending on the mineral composition of the model mixtures

After conducting the experiment in accordance with the plan (Fig. 1), ceramic samples were obtained, for which their properties were determined as the arithmetic average of three parallel measurements. As a result of mathematical processing of the experimental data in accordance with the method in [13], cubic polynomial models were obtained for each property of the ceramic materials, describing the effect of the mineral composition of mixtures on the properties of products of their roasting. Tables 3–5 show the coefficients for variables in polynomials as well as the values of the R -criterion, from which it follows that the obtained polynomial models provide the specified accuracy ($R \leq 5\%$).

Table 3

Accuracy coefficients and characteristics of polynomials obtained for model mixtures with 30 % quartz

Polynomial coefficients	The values of the coefficients in polynomials for different responses		
	Water absorption by the samples	Compressive strength of the samples	Structural rate of the samples
b_1	11.0000	37.20000	1.0000
b_2	9.0000	20.0000	0.8500
b_3	2.0000	20.0000	0.8200
b_{12}	4.5023	-13.5068	-0.1801
b_{13}	-2.2511	33.7669	-0.0450
b_{23}	2.2511	18.0090	0.0225
γ_{12}	-4.5023	58.3136	-0.1355
γ_{13}	6.6995	38.0939	0.2688
γ_{23}	2.2781	53.9192	-0.2021
b_{123}	-49.5068	146.1925	-0.2022
Values of the R -criterion in the control points, %			
CP_1	3.20	2.80	2.40
CP_2	4.07	4.90	1.90
CP_3	4.40	3.10	1.20

Table 4
Accuracy coefficients and characteristics of polynomials obtained for model mixtures with 40 % quartz

Polynomial coefficients	The values of the coefficients in polynomials for different responses		
	Water absorption by the samples	Compressive strength of the samples	Structural rate of the samples
b_1	10.0000	37.0000	0.9400
b_2	11.0000	18.0000	0.8200
b_3	4.0000	20.0000	0.7400
b_{12}	-4.5023	-2.2511	-0.0900
b_{13}	-2.2511	9.0045	0.1351
b_{23}	-2.2511	27.0135	0.2251
γ_{12}	-4.4888	51.5872	-0.1353
γ_{13}	-6.7669	-18.0495	-0.0460
γ_{23}	2.2781	35.9371	0.0454
b_{123}	-8.9865	141.6993	-0.3604
Values of the R-criterion in the control points, %			
CP ₁	3.5	2.0	1.7
CP ₂	3.9	2.7	1.5
CP ₃	4.0	3.8	1.1

Table 5
Accuracy coefficients and characteristics of polynomials obtained for model mixtures with 50 % quartz

Polynomial coefficients	The values of the coefficients in polynomials for different responses		
	Water absorption by the samples	Compressive strength of the samples	Structural rate of the samples
b_1	11.0000	23.0000	0.8550
b_2	10.0000	13.0000	0.8050
b_3	6.0000	12.0000	0.7550
b_{12}	2.2511	-1.35·10 ⁻¹⁴	0.1126
b_{13}	-3.55·10 ⁻¹⁵	2.2511	-0.0900
b_{23}	-2.2511	9.0045	-0.0675
γ_{12}	-2.2511	22.4169	-0.0226
γ_{13}	2.2242	-15.7848	0.4490
γ_{23}	2.2646	31.4619	-0.2021
b_{123}	-54.0000	-6.7669	0.5851
Values of the R-criterion in the control points, %			
CP ₁	4.3	2.3	0.8
CP ₂	4.3	4.3	0.9
CP ₃	4.8	4.1	2.7

Fig. 2–4 show a graphical interpretation of the established dependencies in the form of “composition – property” diagrams.

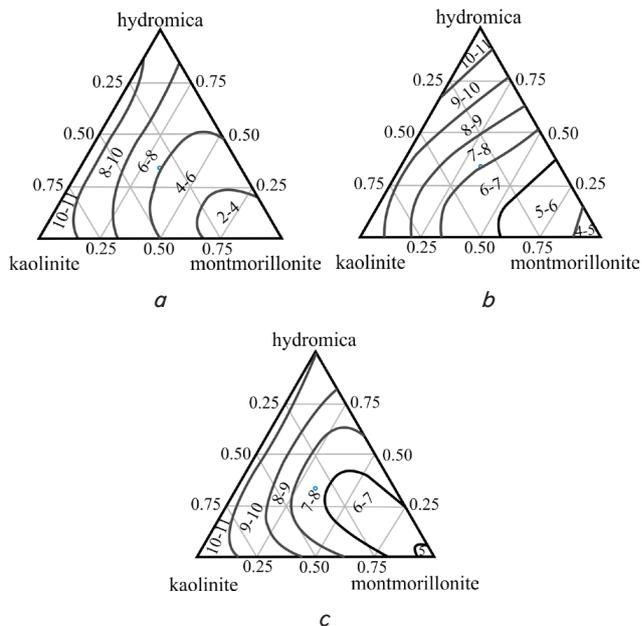


Fig. 2. Dependence of the water absorption (%) of the ceramic samples on the mineral composition of model mixtures with different quartz content: a – 30 % quartz; b – 40 % quartz; c – 50 % quartz

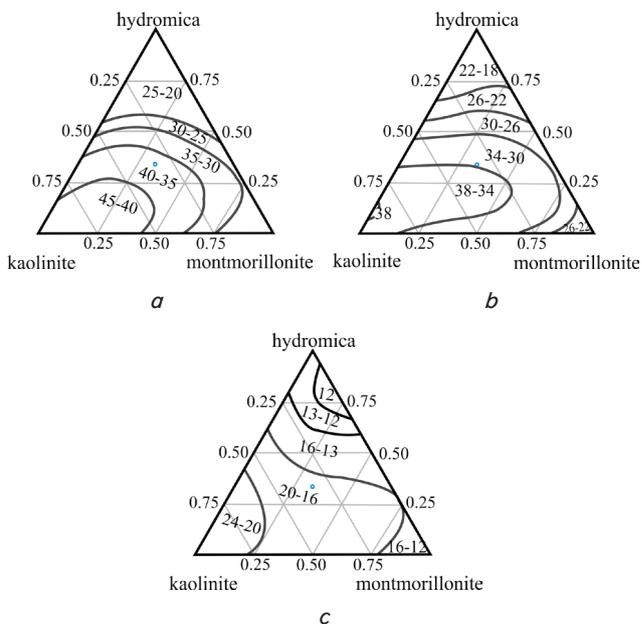


Fig. 3. Dependence of the compressive strength (MPa) of the ceramic samples on the mineral composition of model mixtures with different quartz content: a – 30 % quartz; b – 40 % quartz; c – 50 % quartz

The diagrams “mineral composition of the mixture – material property” make it possible to establish some tendencies in the characteristics of the ceramic samples with varying minerals in clays and determine their optimal ratios for obtaining sintered clinker ceramics.

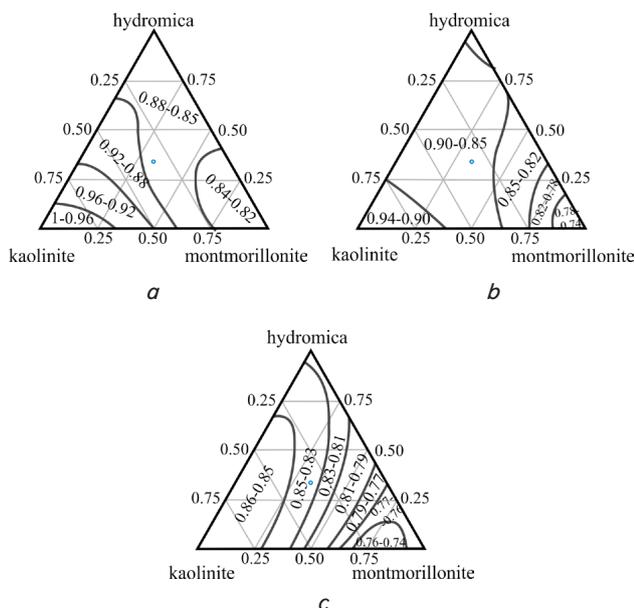


Fig. 4. Dependence of the coefficient of structurality of the ceramic samples on the mineral composition of model mixtures with different quartz content: a – 30 % quartz; b – 40 % quartz; c – 50 % quartz

6. Discussion of the results: analysis of the dependencies of the “mineral composition of clays – properties of materials” as applied to clinker ceramics

The graphic dependences in Fig. 2–4 show that of all the rock-forming minerals, montmorillonite is the most influential in terms of water absorption. An increase in its content in model mixtures leads to a significant decrease in water absorption to values characteristic of clinker materials ($\leq 6\%$). Among the two-component combinations, hydromica-montmorillonite and kaolinite-montmorillonite mixtures are influential.

The effect of clay minerals on mechanical strength is more complex. It is possible to regulate this property of materials with any bimineral mixture; however, the kaolinite component is still the most significant factor. At different compositions of mixtures, the models with the predominant content of kaolinite are characterized by the highest mechanical strength, and the lowest mechanical strength is observed in the model with the predominance of hydromica.

The kaolinite component of the model mixtures has a positive effect on the coefficient of structure, and, consequently, on frost resistance. For mixtures with a predominance of kaolinite, the value of this coefficient is at the level of 0.9. The smallest values of the structural coefficient of 0.72–0.74 are characteristic of mixtures with a predominance of montmorillonite, which do not provide frost resistance of materials.

The most significant influence on the properties of ceramic samples is produced by quartz impurities. An increase in the content of quartz in mixtures leads to a noticeable reduction in areas on the diagrams correspond-

ing to the properties of clinker (water absorption $\leq 6\%$, compressive strength ≥ 20 MPa, structural coefficient ≥ 0.85). This tendency is especially noticeable in the transition from mixtures with 40 % quartz to mixtures with 50 % quartz. With such a quartz content, clinker products cannot be obtained due to the high water absorption of the materials.

The obtained dependences of the influence of the mineral composition of the sand-on clay mixtures on the properties of ceramic materials in conjunction with the requirements of DSTU B W.2.7-245:2010 standard for clinker products made it possible to construct a generalized diagram shown in Fig. 5.

In the diagram, the areas of the mineral composition of clay rocks with 30 % quartz content, which meet the requirements of the standard for different types of clinker, are highlighted in color and numbers. Fig. 5 shows that clinker production is possible on the basis of rather wide bimineral and polymineral combinations of clay minerals, which allows valuating such mixtures as technologically qualitative. The areas of mineral composition indicated in the diagram are suitable for obtaining the entire range of clinker products – from hollow facing clinker brick of the M200 grade to road clinker of the M400 grade.

An increase in the quartz content in clays up to 40 % repeatedly narrows the areas of the admissible mineral composition of clays. In the diagram in Fig. 5, the dashed lines indicate the region of the mineral composition that makes it possible to obtain clinker of the M300 grade with a content of 40 % quartz in clays. As follows from the position of the shaded area in the diagram, the content of rock-forming minerals in such compositions can vary within narrow limits: kaolinite – 30–44 %, hydromica – 0–14 %, and montmorillonite – 53–62 %. The content of quartz in clays at the level of 50 % completely excludes the possibility of obtaining clinker materials based on them.

The diagram in Fig. 5 diagram makes it possible to predict the possibility of using polymineral clay to obtain clinker materials when the content of quartz in them is 30 % or 40 %. To do this for any clay, it is necessary to determine the content of quartz and three rock-forming minerals (kaolinite, hydromica, and montmorillonite). Then clay minerals should be counted by 100 %, and a point of clay composition should be placed on the diagram. Data on the content of quartz and the position of the clay composition point on the diagram make it possible to determine its suitability not only for clinker production in general but for various types of clinker products.

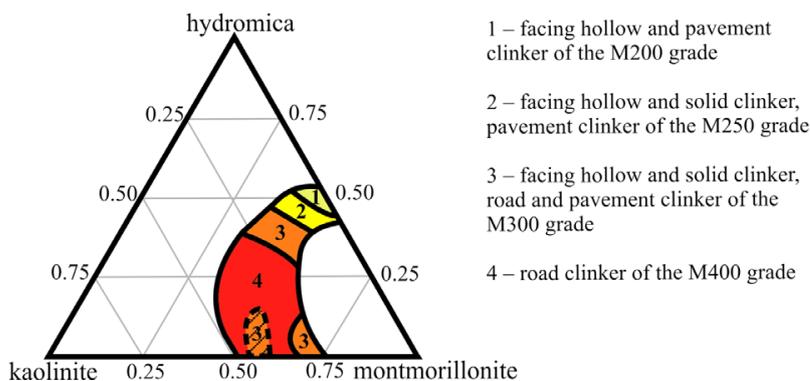


Fig. 5. Areas of mineral composition of clays suitable for the production of clinker construction ceramics at a firing temperature of 1100 °C

The proposed diagram of the mineral composition in combination with the existing criteria for assessing the quality of clays by their composition creates a system of criteria that allow predicting the applicability of polymineral clays for the production of modern architectural and construction ceramics.

The disadvantage of the proposed diagram of the mineral composition is the restriction of the possibility of its use in relation to clinker ceramics obtained at temperatures above 1100 °C. The extension of the temperature range to 1150 °C and 1200 °C as well as the development of a similar diagram for such conditions of firing can be the subject of further research in this direction.

7. Conclusion

1. By the method of wet enrichment of natural clay raw materials with separation of clay-forming minerals, models of polymineral clays with various combinations of kaolinite, hydromica, montmorillonite and quartz in an amount of 30–50 % have been obtained.

2. For compositions of clay minerals containing 30–50 % quartz, polynomial mathematical models have been

obtained, characterizing the dependence of the properties of the calcination products of the compositions on their mineral composition. From the point of view of clinker products, it has been found that montmorillonite has a positive effect on the water absorption of ceramic materials, and kaolinite has a favorable effect on the mechanical strength and structural coefficient. The greatest influence on the properties of materials is produced by quartz impurities; an increase in their content in mixtures leads to an increase in water absorption by the materials, reducing their strength and frost resistance. It is shown that to obtain clinker materials, the quartz content in the mixtures should not exceed 40 %.

3. A generalized diagram of the mineral composition of clays (Fig. 5) was developed to determine the allowable ratios of the clay-forming minerals and quartz in clays to produce clinker ceramics at a roasting temperature of 1100 °C. The areas of the mineral composition of clays shown in the diagram can serve as a criterion for assessing the technological quality of polymineral clay raw materials and complement the existing system of assessment indicators for clays in terms of modern clinker products (facing, paving and road clinker).

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