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# SYNTHESIS OF IMPORT-SUBSTITUTED GLAZES FOR PORCELAIN STONEWARE BASED ON LOCAL RAW MATERIALS

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**Abstract.** *Relevance.* Ceramic glazes for porcelain tiles are strategically important components of building materials within the industrial and innovative development programs of the Republic of Kazakhstan. Porcelain tile production is a high-technology process in which glazes play a key role in forming the decorative and performance properties of finished products. The relevance of this work stems from the need to reduce dependence on imported components and improve the cost-effectiveness of porcelain tile production. *Objective.* Development of import-substituting glazes for porcelain stoneware based on local mineral raw materials of the Republic of Kazakhstan, providing stable technological and operational properties while reducing production costs. At present, porcelain tile production facilities in Kazakhstan have a combined design capacity of several tens of millions of square meters per year, covering a substantial part of domestic demand. *Methods.* The objects of the study were local mineral raw materials used in glaze technology, developed composite glaze compositions, and fired samples of glazed porcelain tiles. The mineralogical and chemical characteristics of feldspar, silica-containing, and clay raw materials were investigated, as well as their influence on melting behavior and glass phase formation. Experimental studies were carried out to select optimal glaze formulations and firing regimes under industrial conditions. *Results.* As a result, glaze coatings with stable technological and performance characteristics suitable for industrial application were obtained using local import-substituting raw materials. It has been established that the use of local raw materials allows for the production of competitive products while reducing the cost of production of porcelain tiles. *Conclusions.* The results provide a scientific and technological basis for the development and implementation of new import-substituting glazes and for expanding the domestic raw material base for porcelain tile production in the Republic of Kazakhstan.

**Keywords:** Porcelain stoneware, engobe, protective glaze, glaze compositions, import substitution

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## ЖЕРГІЛІКТІ МИНЕРАЛДЫҚ ШИКІЗАТ НЕГІЗІНДЕ КЕРАМОГРАНИТКЕ АРНАЛҒАН ИМПОРТТЫ АЛМАСТЫРАТЫН ГЛАЗУРЬДІ СИНТЕЗДЕУ

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**Аннотация.** *Өзектілігі.* Қазақстан Республикасында жүзеге асырылып жатқан индустриялық-инновациялық даму бағдарламалары аясында керамогранитке арналған керамикалық глазурь құрылыс материалдарының стратегиялық маңызы бар құрамдас бөлігі болып табылады. Керамогранит өндірісі – жоғары технологиялық процесс, онда глазурь дайын бұйымның сәндік және пайдалану қасиеттерін қамтамасыз ететін негізгі рөл атқарады. Қазіргі жағдайда глазурь материалдарының едәуір бөлігі шетелден жеткізілетіндіктен, оларды импортты алмастыру мәселесі ерекше өзектілікке ие. *Мақсаты.* Өндірістің өзіндік құны төмендеген кезде тұрақты технологиялық және пайдалану қасиеттерін қамтамасыз ететін Қазақстан Республикасының жергілікті минералдық шикізаты негізінде фарфордан жасалған тастан жасалған бұйымдар үшін импортты алмастыратын глазурь әзірлеу. Қазіргі уақытта Қазақстан Республикасында керамогранит өндіретін кәсіпорындар жұмыс істейді, олардың жиынтық жобалық қуаты жылына бірнеше ондаған миллион шаршы метрді құрайды және ішкі сұраныстың елеулі бөлігін қамтамасыз етеді. Сонымен қатар шетелдік инвесторлардың қатысуымен жаңа инвестициялық жобалар іске асырылуда. Өндірістік қуаттарды ұлғайту және өнім ассортиментін кеңейту жағдайында жергілікті минералдық шикізат негізінде импортты алмастыратын глазурьлерді әзірлеу айрықша маңызға ие. *Әдістері.* Зерттеу объектілері ретінде глазурь алу технологиясына тартылған жергілікті минералдық шикізат түрлері, әзірленген композициялық глазурь құрамдары және глазурленген керамогранит плиталарының синтезделген үлгілері алынды. Жұмыста бастапқы материалдардың минералды-химиялық сипаттамалары, оның ішінде далалық шпатты шикізат, кремнеземқұрамды компоненттер мен сазды материалдар, сондай-ақ олардың балқу және шыныфазаның түзілу процестеріне әсері зерттелді. Керамогранит құрамындағы глазурь жабындарын күйдіру кезінде жүретін физика-химиялық процестер мен фазалық түрленулер талданды. *Нәтижелері.* Алғаш рет жергілікті импортты алмастыратын шикізат негізінде тұрақты технологиялық және пайдалану сипаттамаларына ие, керамогранит өндірісінде өнеркәсіптік қолдануға жарамды глазурь жабындары алынды. *Қорытынды.* Зерттеу нәтижелері өнеркәсіптік өндіріске жаңа импортты алмастыратын глазурьлерді енгізу үшін ғылыми-технологиялық негіз бола алады, сондай-ақ Қазақстан Республикасының глазурь шикізатының ресурстық базасын кеңейтуге мүмкіндік береді.

**Түйін сөздер:** керамогранит; ангоб, протекциялық глазурь, глазурь композициялары; импортты алмастыру

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# СИНТЕЗ ИМПОРТОЗАМЕЩАЮЩЕЙ ГЛАЗУРИ ДЛЯ КЕРАМОГРАНИТА НА ОСНОВЕ МЕСТНОГО МИНЕРАЛЬНОГО СЫРЬЯ

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**Аннотация.** *Актуальность*. Керамические глазури являются важными компонентами производства керамогранита, обеспечивающими декоративные, защитные и эксплуатационные свойства готовой продукции. Для предприятий керамической промышленности Республики Казахстан особую актуальность представляет импортозамещение глазурных материалов, значительная часть которых поставляется из-за рубежа. Расширение производственных мощностей и ассортимента керамогранитной продукции повышает потребность в разработке глазурных составов на основе доступного местного минерального сырья. Использование отечественных сырьевых ресурсов позволяет снизить зависимость от импортных компонентов, сократить производственные затраты и повысить устойчивость керамической отрасли. *Цель*. Разработать импортозамещающую глазурь для керамогранита на основе местного минерального сырья Республики Казахстан, обеспечивающую стабильные технологические и эксплуатационные свойства при снижении себестоимости готовой продукции. *Методы*. Объектами исследования являлись местные виды минерального сырья, экспериментальные глазурные составы и образцы глазурованного керамогранита. Исследованы минералогический и химический составы полевошпатового, кремнезёмсодержащего и глинистого сырья, а также их влияние на процессы плавления и формирования стеклофазы. Проведены экспериментальный подбор компонентного состава глазури и определение рациональных режимов термической обработки. Технологические и эксплуатационные свойства полученных покрытий оценивались по показателям плавкости, растекаемости, качества поверхности и совместимости глазури с керамической основой. *Результаты*. На основе местного минерального сырья разработаны глазурные составы, обеспечивающие формирование покрытий со стабильными технологическими и эксплуатационными характеристиками. Установлено влияние полевошпатовых, кремнезёмсодержащих и глинистых компонентов на плавление глазурной шихты и формирование стеклофазы. Полученные покрытия характеризуются пригодностью для нанесения на керамогранитную основу и потенциальной возможностью промышленного применения. Использование местного сырья способствует снижению себестоимости глазурованных керамогранитных плит и повышению конкурентоспособности готовой продукции. *Выводы*. Разработанные глазурные составы могут служить научно-технологической основой для организации производства импортозамещающих глазурей для керамогранита. Применение местного минерального сырья расширяет отечественную сырьевую базу, снижает зависимость от импортных компонентов и создаёт предпосылки для повышения экономической эффективности производства керамогранитной продукции в Республике Казахстан.

**Ключевые слова:** керамогранит, ангоб, протекционная глазурь, глазурные компоненты, импортозамещение

**Introduction.** Porcelain stoneware is one of the most sought-after modern building materials, widely used in residential, civil, and industrial construction due to its high mechanical strength, wear resistance, low water absorption, and stable decorative properties (Carter and Norton, 2013; Kingery et al., 1976; Reed, 1995). Glaze plays a significant role in determining the performance and aesthetic properties of porcelain stoneware, determining surface quality, color, whiteness, and resistance to chemical attack and abrasive wear (Eppler and Eppler, 2000; Taylor and Bull, 1986).

With the implementation of industrial and innovative development programs in the Republic of Kazakhstan, providing domestic porcelain stoneware manufacturers with competitive raw materials is particularly important. Currently, a significant portion of glazes and components for their production are imported, leading to industry dependence on external supplies, increased production costs, and increased technological risks associated with logistical constraints and price volatility. Therefore, the development of import-substituting glazes based on local mineral raw materials is a pressing scientific and technical challenge (De Noni et al, 2010; Sánchez et al, 2010). Glaze compositions for porcelain tiles are complex multicomponent systems that include feldspar fluxes, silica-containing fillers, aluminosilicates, and modifying additives (Eppler and Eppler, 2000; Taylor and Bull, 1986). Their properties are largely determined by the mineralogical and chemical composition of the raw materials, their degree of dispersion, and the physicochemical processes occurring during heat treatment. When using local raw materials, a special role is played by studying their mineralogical and petrographic characteristics, loss on ignition, melting point, and ability to form a glass phase thermally and chemically compatible with porcelain tiles.

Despite the presence of significant mineral reserves in the Republic of Kazakhstan potentially suitable for glaze production, the issues of their comprehensive evaluation and technological adaptation to the requirements of industrial porcelain tile production remain insufficiently explored. This is due to both the diversity of genetic types of deposits and the lack of systematic data on the influence of local raw materials on the melting processes, phase formation, and performance characteristics of glaze coatings.

This paper examines the synthesis and optimization of import-substituting glazes for porcelain stoneware based on local mineral raw materials. The focus is on the selection of starting components, the development of glaze compositions, the study of the physicochemical processes occurring during their heat treatment, and the evaluation of the technological and performance characteristics of the resulting glaze coatings.

**Materials and basic methods.** For the synthesis of import-substituting glazes for porcelain stoneware, local mineral raw materials of different genetic types were used. The main raw components included:

– feldspars from the Aksoran deposit, acting as fluxing agents and ensuring the formation of the glassy phase;

– clays from the Berezovsky deposit, regulating the aluminosilicate balance and rheological properties of glaze suspensions;

– quartz sands from the Akzhar deposit, used as silica-containing fillers;

– kaolins from the Alekseevsky deposit, intended for stabilizing glaze suspensions and controlling firing processes.

The selection of these raw materials was based on their chemical and mineralogical composition, availability, and potential suitability for application in porcelain stoneware glaze compositions within the framework of import substitution tasks.

To evaluate the properties of the raw materials and the developed glaze compositions, complex of physicochemical and technological research methods was applied. Industrial ready-made frits and opacifying additives were additionally used as components of the glaze batches.

The chemical composition of raw materials and developed glaze compositions was determined by X-ray fluorescence analysis (XRF). The mineralogical and petrographic composition of the initial raw materials was studied using optical microscopy and X-ray diffraction analysis (XRD) (Carter and Norton, 2013; Reed, 1995).

During the development of glaze batch compositions, industrial ready-made frits were used in amounts of 30–40 wt.%, providing stabilization of the oxide composition and reproducibility of glaze properties during firing. To enhance opacity and form a dense white layer, zirconium silicate ($ZrSiO_4$) was introduced into engobe and opaque glaze compositions in amounts of 5–10 wt.%**.** Control of the glaze oxide composition was carried out by XRF, while the influence of the introduced components on the microstructure of the glaze layer was analyzed using scanning electron microscopy (SEM).

Glaze compositions were prepared by wet milling of the initial components until the required degree of dispersion (≤ 60 µm) was achieved, followed by homogenization and magnetic separation. The prepared glaze suspensions were applied onto samples of porcelain stoneware bodies of industrial composition.

Firing of the glazed samples was carried out in the temperature range of 1000–1250 °C with a controlled heating rate and holding at the maximum temperature. After firing, the glaze coatings were evaluated in terms of surface quality, degree of melting, adhesion to the porcelain stoneware body, and susceptibility to defect formation.

The microstructure of the glaze coatings and the distribution of the opacifying phase were examined by SEM. The physicochemical processes occurring during the thermal treatment of the glazes, including glass phase formation, its interaction with the porcelain stoneware body, and possible crystallization processes, were analyzed in order to determine optimal glaze compositions and firing regimes.

**Results.** The chemical composition of the most optimal types of raw materials, selected based on the results of comprehensive studies, is presented in table 1.

Table 1. Chemical analysis of raw materials.

| Local raw materials | Content of oxides, % | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | $Na_2O$ | MgO | $Al_2O_3$ | $SiO_2$ | $K_2O$ | CaO | $TiO_2$ | $Fe_2O_3$ | l.o.i. |
| Berezovsky clays | 0.34 | 0.49 | 36.54 | 56.54 | 2.29 | 0.35 | 1.6 | 1.68 | 10.2 |
| Alek-seevsky kaolins | - | - | 33.28 | 63.6 | 1.47 | 0.23 | 0.4 | 0.84 | 5.5 |
| Akzhar quartz sands | 0.67 | 0.4 | 4.61 | 90.93 | 2.21 | 0.35 | 0.21 | 0.62 | 0.1 |
| Aksoran feldspars | 11.1 | | 17.99 | 69.65 | | 0.47 | 0.48 | 0.32 | 0 |

| Imported materials | $Na_2O$ | MgO | $Al_2O_3$ | $SiO_2$ | $K_2O$ | CaO | $TiO_2$ | $Fe_2O_3$ | ZnO | $Y_2O_3$ | BaO | $ZrO_2$ | $B_2O_3$ | SrO | $HFO_2$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Opaque frit | 2.84 | 2 | 7.37 | 56.35 | 5.23 | 7.8 | - | 0.19 | 0.417 | - | 0.25 | 12.28 | 5.15 | 0.114 | - |
| Transparent frit | 7.48 | 0.19 | 19.93 | 56.36 | 12.48 | 1.88 | 0.11 | 0.16 | 0.119 | - | 0.14 | 0.13 | 1 | - | - |
| Zirconium silicate | - | - | 1.07 | 31.81 | - | - | 0.22 | 0.06 | - | 0.2 | - | 64.02 | - | - | 2.43 |

Chemical analysis allowed us to determine the oxide composition of the raw materials and assess their potential suitability for use in glaze and engobe compositions. However, chemical analysis data do not provide information on the phase state of the components or the oxide species within the minerals. Therefore, X-ray diffraction (XRD) analysis was conducted to clarify the mineralogical composition of the raw materials, identify crystalline phases, and assess their role in shaping the properties of the glazes.

*Aksoran feldspars.* Given their high alkali oxide content, this raw material is a promising fluxing component for the synthesis of porcelain glazes, ensuring the formation of a glass phase and stable glaze coatings during firing. According to geological exploration, the deposit's reserves are sufficient for long-term industrial use in import substitution technologies. The results of the XRD are presented in figure 1 and deciphered in table 2.

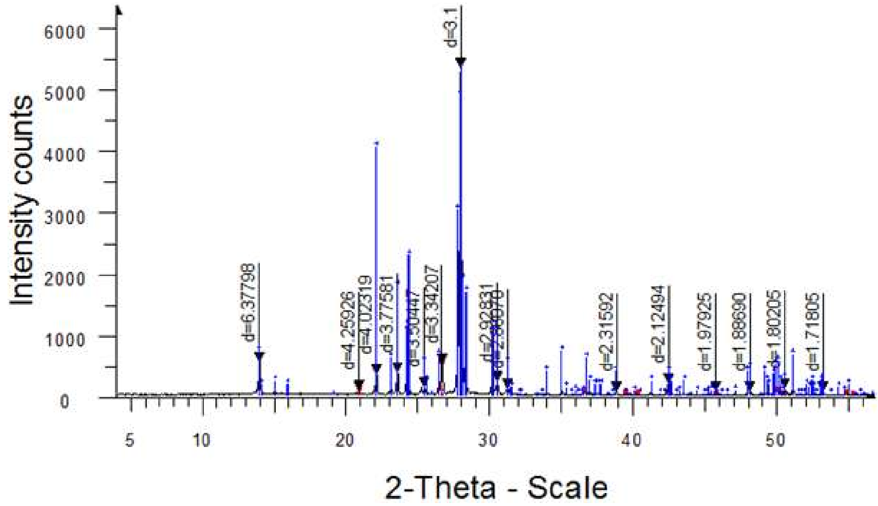



Figure 1. XRD pattern of Aksoran feldspars.

Table 2. Mineralogical composition of Aksoran feldspars according to X-ray diffraction data.

| Mineral | Formula | Concentration, % |
|---|---|---|
| Albite | Na(AlSi3O8) | 89.7 |
| Quartz | SiO2 | 10.3 |

X-ray diffraction analysis of the feldspathic raw material showed that the main crystalline phase is represented by albite Na ($AlSi_3O_8$) (89.7%), with the presence of quartz $SiO_2$ (10.3%). From the viewpoint of oxide composition, this raw material belongs to the $Na_2O–Al_2O_3–SiO_2$ system: albite acts as a source of the fluxing oxide $Na_2O$ and the glass network components $Al_2O_3$ and $SiO_2$, ensuring glass phase formation and stable melting behavior of the glaze. Additional $SiO_2$, associated with the presence of quartz, contributes to increased chemical resistance and surface hardness of the coating, provided sufficient dispersion and dissolution of the quartz phase in the melt, thereby reducing the risk of structural heterogeneity and coating defects.

*Akzhar quartz sands* are used as a high-purity silica component, providing stability of the glass phase and reducing the formation of defects in the glaze coating. To clarify the phase composition and quantitative content of crystalline phases in the studied sand, X-ray phase analysis was performed. The sample's diffraction pattern is shown in figure 2, and the phase composition results are presented in table 3.

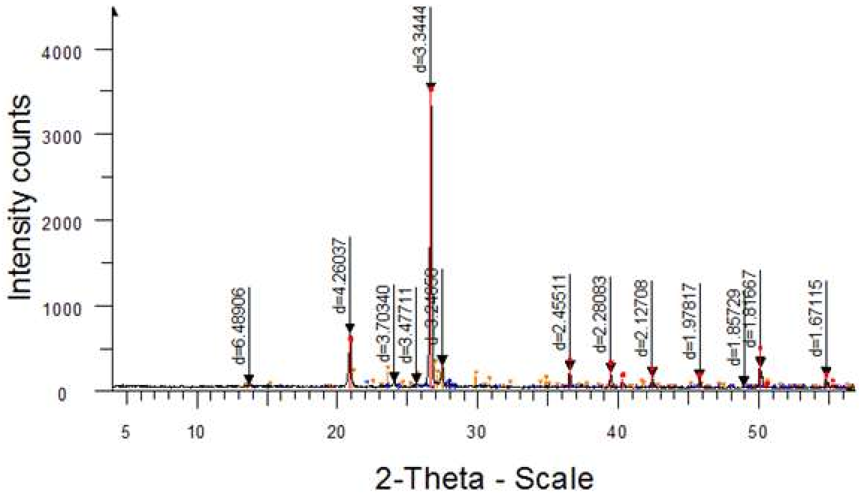



Figure 2. XRD pattern of Akzhar quartz sands.

Table 3. Mineralogical composition of Akzhar quartz sands according to X-ray diffraction data.

| Mineral | Formula | Concentration, % |
|---|---|---|
| Quartz | SiO2 | 92.9 |
| K-feldspar | KAlSi3O8 | 5.5 |
| Albite | Na(AlSi3O8) | 1.6 |

According to X-ray diffraction analysis, the quartz sand is characterized by a high quartz ($SiO_2$) content of 92.9%, which allows it to be classified as a high-silica raw material. Minor phases identified in the sample include potassium feldspar ($KAlSi_3O_8$) in an amount of 5.5% and albite ($NaAlSi_3O_8$) at 1.6%.

From the standpoint of oxide composition, the investigated quartz sand predominantly belongs to the $SiO_2$ system, with minor amounts of $K_2O$, $Na_2O$, and $Al_2O_3$ originating from feldspathic minerals. The presence of potassium and sodium feldspars is subordinate and does not alter the primary function of the sand as a silica-bearing filler.

The high $SiO_2$ content ensures the formation of a strong glass-phase framework in glaze compositions and contributes to increased hardness, wear resistance, and chemical durability of the glaze coating. The minor presence of feldspars provides an additional fluxing effect, enhancing the dissolution of the quartz phase in the glass melt and reducing the risk of undissolved quartz particles under industrial firing conditions (De Noni et al, 2010; Gualtieri; Kingery et al., 1976; Levin et al., 1964).

Thus, the investigated quartz sand represents a promising raw material for use in engobe and glaze compositions for porcelain stoneware, ensuring glass-phase structural stability and improved performance characteristics of the coatings.

*Berezovsky clays* perform the function of a plastic and binding component, ensuring the technological stability of the bodies. The results of the XRD are presented in figure 3 and deciphered in table 4.

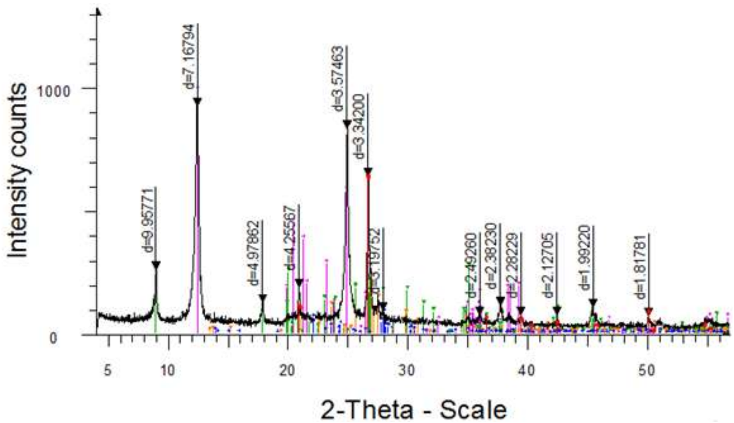



Figure 3. XRD pattern of Berezovsky clays.

Table 4. Mineralogical composition of Berezovsky clays according to X-ray diffraction data.

| Mineral | Formula | Concentration, % |
|---|---|---|
| Kaolinite | Al2(Si2O5)(OH)4 | 80.5 |
| Quartz | SiO2 | 10.6 |
| Mica | KAl2(AlSi3O10)(OH)2 | 5.7 |
| K-feldspar (potassium feldspar) | KAlSi3O8 | 1.8 |
| Albite | Na(AlSi3O8) | 1.3 |

According to X-ray diffraction analysis, the investigated clay is characterized by a predominance of kaolinite $Al_2(Si_2O_5)$ $(OH)_4$, with a content of 80.5%. Associated phases identified include quartz ($SiO_2$) at 10.6%, mica $KAl_2(AlSi_3O_{10})$ $(OH)_2$ at 5.7%, as well as minor amounts of potassium feldspar $KAlSi_3O_8$ (1.8%) and albite Na $(AlSi_3O_8)$ (1.3%).

From the standpoint of oxide composition, this clay mainly belongs to the $Al_2O_3$–$SiO_2$ system with a limited content of fluxing oxides $K_2O$ and $Na_2O$ originating from mica and feldspathic impurities. The high proportion of kaolinite results in an increased $Al_2O_3$ content, which contributes to higher melt viscosity and the formation of a thermally stable glass network in glaze and engobe compositions.

The presence of quartz acts as a silica-bearing filler that regulates shrinkage and structural rigidity during firing. Mica and feldspathic impurities provide a moderate fluxing effect, facilitating the initial stages of sintering and improving the interaction of the clay with other components of the glaze batch.

Thus, the investigated clay represents a technologically valuable raw material for use in engobe and glaze compositions for porcelain stoneware, ensuring stabilization of the rheological properties of glaze suspensions, improved adhesion of the coating to the porcelain stoneware body, and reduced susceptibility to defect formation during firing. In addition, the clay performs the function of a plastic and binding component, ensuring the technological stability of the bodies.

*Alekseevsky kaolins* serve as a source of the aluminosilicate phase, promoting mullite formation and enhancing the thermomechanical properties of the material. The results of the XRD are presented in figure 4 and deciphered in table 5.

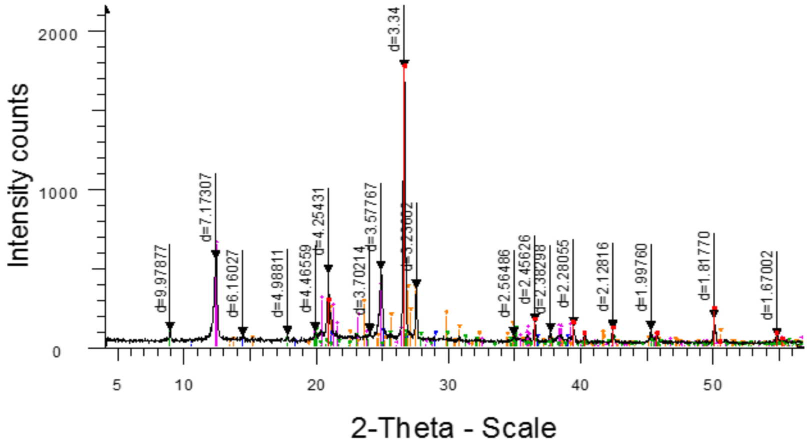



Figure 4. XRD pattern of Alekseevsky kaolins.

Table 5. Mineralogical composition of Alekseevsky kaolins according to X-ray diffraction data.

| Mineral | Formula | Concentration, % |
|---|---|---|
| Kaolinite | Al2(Si2O5)(OH)4 | 57.6 |
| Quartz | SiO2 | 32.4 |
| K-feldspar (potassium feldspar | KAlSi3O8 | 4.8 |
| Mica | KAl2(AlSi3O10)(OH)2 | 5,2 |

According to the results of X-ray diffraction analysis, the investigated kaolin is characterized by a mixed mineralogical composition with a predominance of kaolinite $Al_2(Si_2O_5)$ $(OH)_4$, accounting for 57.6%. A significant proportion of the sample is represented by quartz ($SiO_2$) at 32.4%, indicating an increased silica content of the raw material.

Secondary phases identified include potassium feldspar ($KAlSi_3O_8$) at 4.8%, and mica $KAl_2(AlSi_3O_{10})$ $(OH)_2$ at 5.2%.

From the standpoint of oxide composition, this kaolin belongs to the $Al_2O_3$–$SiO_2$ system with a moderate content of fluxing oxides $K_2O$ originating from potassium feldspar, mica, and potassium hydrogen silicate. The elevated quartz content increases the $SiO_2$ fraction, contributing to the formation of a rigid glass network and enhancing the thermal stability of glaze and engobe compositions.

The high proportion of kaolinite results in a significant $Al_2O_3$ content, leading to increased melt viscosity, improved rheological properties of glaze suspensions, and enhanced adhesion of coatings to the porcelain stoneware body. Minor fluxing components promote more uniform sintering and improve the interaction of kaolin with feldspathic and frit components in glaze systems (Dondi et al., 2014; Sanchez et al., 2010; Lee and Iqbal, 2001).

Within the framework of the conducted research, two types of glaze compositions for porcelain stoneware—engobe and protection —were developed based on an oxide-based calculation methodology (Table 6). The developed compositions ensure the formation of stable coatings and meet the requirements for technological and operational performance characteristics.

Table 6. Glaze batch recipe.

| Material name | Engobe | Protective glaze |
|---|---|---|
| Opaque frit | 37 | - |
| Transparent frit | - | 40 |
| Alekseevsky kaolins | 15 | 7 |
| Berezovsky clays | 12 | 14 |
| Aksoran feldspars | 12 | 16 |
| Akzhar quartz sands | 17 | 23 |
| Zirconium silicate | 7 | - |

The developed test batch glaze was ground by co-milling the raw materials in a ball mill (Figure 5).

Figure 5. Finely ground glaze.

Subsequently, the glaze was applied to an unfired ceramic tile sample using a glazing stamp (Figure 6).

Figure 6. Test specimens.

To evaluate the technological applicability of the developed glaze compositions under industrial production conditions, the firing of glazed porcelain stoneware samples was carried out in the plant's industrial kiln. The firing temperature profile is presented below in the table 7. This approach made it possible to assess the formation of the glaze layer and the coating quality under conditions maximum close to those of serial production.

Table 7. Kiln temperature profile.

| Total firing time - 55 min | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| VT10 | VT12 | M14 | M 16 | M 18 | M 20 | M 22 | M 24 | M 26 | M 28 | M 30 | M 31 | M 33 | VT36 |
| 430 | 450 | 660 | 770 | 830 | 1020 | 1130 | 1200 | 1200 | 1200 | 1100 | 960 | 620 | 470 |
| M010 | M012 | M014 | M016 | M018 | M020 | M022 | M024 | M026 | M028 | M030 | | M033 | |
| 460 | 650 | 790 | 860 | 900 | 1050 | 1160 | 1210 | 1210 | 1210 | 1100 | | 625 | |

The sample after firing is shown in Figure 7.

Figure 7. Fired glaze sample.

Visual inspection of the glazed samples after firing revealed the formation of a smooth and uniform surface without visible defects. No pinholes, cracks, craters, or other signs of defect formation were observed. The glaze layer is characterized by uniform melting and a smooth texture, indicating a properly selected glaze composition and optimal thermal treatment conditions.

For a comprehensive evaluation of the performance properties of the glaze coatings, thermal shock resistance and chemical resistance tests were performed according to international standards ISO 10545-9 and ISO 10545-13, respectively. The surface hardness of the glaze coatings was evaluated using the Mohs hardness scale according to EN 101 (Table 8).

Table 8. Results of physic-mechanical tests.

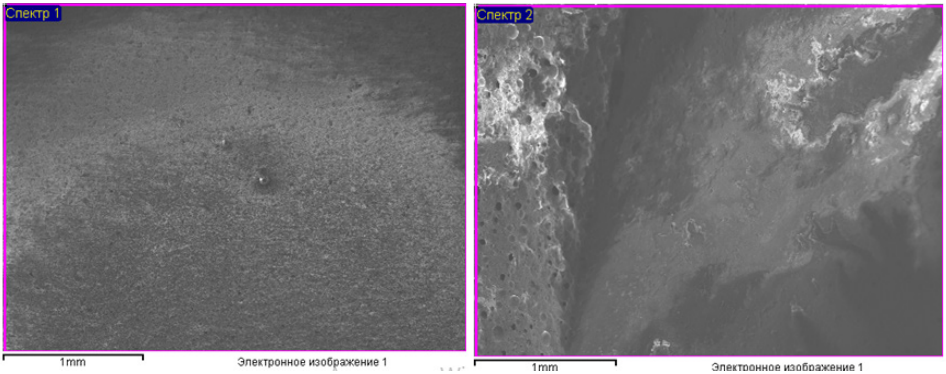
| Test | Standard | Required Performance | Actual Results | Compliance |
|---|---|---|---|---|
| Thermal resistance | ISO 10545-9 | No cracks, chips or peeling of glaze after testing | More than 125 cycles, no defects detected | Corresponds |
| Acid resistance | ISO 10545-13 | Class GA/GLA, no visible surface changes | There are no visible defects | Corresponds |
| Surface hardness (Mohs scale) | EN 101 | Not less than 6 on the Mohs scale | 6 on the Mohs scale | Corresponds |

The results of the physical and mechanical tests demonstrate high thermal stability, chemical resistance, and sufficient hardness of the developed glaze coatings. To identify the structural factors determining the achieved performance characteristics, the next stage of the study involved analyzing the microstructure of the glaze layer using scanning electron microscopy (SEM). To more thoroughly analyze the structural characteristics of the glaze and eliminate the influence of the porcelain tile body (Figure 8), microstructural studies were conducted on pure glaze samples. For this purpose, individual test tablets were produced from the developed glaze compositions and fired at the same temperature as the glazed porcelain tile samples.

a b

Figure 8. Fired glaze samples without shards.
a - Protective glaze (1,2);
b - Engobe (3,4).

The microstructure of the obtained pure glaze samples was studied using scanning electron microscopy (SEM), which made it possible to evaluate the density of the glass phase, the presence of porosity and the distribution of opacifying components without the influence of the substrate (Figure 9).




a b

Figure 9. SEM images of pure glaze coatings after firing.
a — Engobe (Spectrum 1);
b — Protective glaze (Spectrum 2).

The elemental composition of the protective glaze and engobe according to local energy-dispersive X-ray analysis (EDS), is presented in table 9.

Table 9. Elemental composition of protective glaze and engobe according to local EDS analysis, wt.%.

| Engobe | | Protective glaze | |
|---|---|---|---|
| Element | Weight % | Element | Weight % |
| C | 10.72 | C | 20.37 |
| O | 47.29 | O | 53.17 |
| Na | 3.17 | Na | 2.94 |
| Mg | 0.17 | Mg | 0.82 |
| Al | 9.60 | Al | 6.72 |
| Si | 19.83 | Si | 10.85 |
| K | 0.34 | K | 0.68 |
| Ca | 1.62 | Ca | 1.86 |
| Zr | 6,20 | Zr | 0,37 |
| Ba | 1,05 | Ba | 2,22 |
| Total | 100.00 | Total | 100.00 |

Microstructural and elemental analyses revealed differences in the composition and structure of the protective glaze and the engobe, which are determined by their functional purposes.

SEM–EDS analysis of the engobe (Spectrum 1) revealed that oxygen (47.29

wt.%) and silicon (19.83 wt.%) are the predominant elements, confirming the aluminosilicate nature of the coating. Aluminum is present at 9.60 wt.%, indicating the presence of clay minerals that contribute to the mechanical stability and adhesion of the engobe layer to the porcelain stoneware body. Calcium (1.62 wt.%), sodium (3.17 wt.%), potassium (0.34 wt.%), and magnesium (0.17 wt.%) are present as fluxing oxides, promoting liquid-phase formation during firing and improving densification of the coating.

The protective glaze (Spectrum 2) SEM/EDS analysis showed that the protective glaze consists of a homogeneous amorphous silicate matrix dominated by $SiO_2$ (53.17 wt.%). The high CaO content (20.37 wt.%) acts as a network modifier, improving melt fluidity and adhesion to the ceramic substrate, while $Al_2O_3$ (6.72 wt.%) enhances the mechanical strength and thermal stability of the glass network. Alkali oxides ($Na_2O$ and $K_2O$) function as fluxes, promoting densification during firing. SEM micrographs revealed a dense and uniform microstructure without visible pores or microcracks, indicating effective sintering and good phase homogenization. These results confirm the formation of a dense protective glass layer with improved adhesion and thermo-mechanical properties.

SEM images of both types of coatings demonstrate a dense and homogeneous microstructure without pronounced porosity, cracks, or delamination zones. Differences in elemental composition confirm the correctness of the selected formulations and the conformity of the developed glaze compositions to their functional purposes.

The elevated carbon content is associated with the specifics of the analytical method and is most likely due to carbon coating of the sample or residual surface contamination, which does not affect the phase composition of the glaze.

The homogeneity of elemental distribution and the absence of locally enriched zones confirm the proper selection of glaze compositions and are consistent with the results of thermal shock resistance, chemical durability, and surface hardness tests.

The coefficient of thermal expansion (CTE) was also determined on pure glaze samples fired under identical temperature conditions (Table 10). This ensured an accurate evaluation of the thermal properties of the glazes and their comparability with the results obtained for glazed samples.

Table 10. Coefficient of thermal expansion (CTE) of fired pure glaze compositions and the ceramic body.

| Sample | 3α(50-300ºC)$10^{-7}$ºC$^{-1}$ | 3α(50-500ºC)$10^{-7}$ºC$^{-1}$ | 3α(500-600ºC)$10^{-7}$ºC$^{-1}$ | ΔL/L0/600ºC% | Temperature |
|---|---|---|---|---|---|
| Mass | 205 | 258 | 290 | 0,40 | 1210 |
| Protective glaze | 200 | 217 | 261 | 0,43 | 1210 |
| Engobe | 190 | 202 | 225 | 0,36 | 1210 |

The results of the coefficient of thermal expansion (CTE) determination showed that the developed glaze compositions exhibit thermal behavior well matched with the porcelain stoneware body within the investigated temperature range.

For the porcelain stoneware body, the CTE values increase with temperature and amount to $205\cdot10^{-7}$ °C$^{-1}$ in the range of 50–300 °C, $258\cdot10^{-7}$ °C$^{-1}$ in the range of 50–500 °C, and $290\cdot10^{-7}$ °C$^{-1}$ in the interval of 500–600 °C, with a relative elongation $\Delta L/L_0$ of 0.40% at 600 °C.

The protective glaze demonstrates comparable CTE values of $200\cdot10^{-7}$ °C$^{-1}$, $217\cdot10^{-7}$ °C$^{-1}$, and $261\cdot10^{-7}$ °C$^{-1}$ in the corresponding temperature intervals. The relative elongation reaches 0,43%, indicating good thermal compatibility of the protective glaze layer with the porcelain stoneware substrate.

The engobe is characterized by the lowest CTE values—$190\cdot10^{-7}$ °C$^{-1}$, $202\cdot10^{-7}$ °C$^{-1}$, and $225\cdot10^{-7}$ °C$^{-1}$—as well as the minimum relative elongation $\Delta L/L_0$ of 0,36%. These values indicate enhanced thermal stability of the engobe and its ability to effectively compensate for thermal stresses during firing and cooling.

Comparison of the obtained data shows that the differences in CTE values between the porcelain stoneware body, the protective glaze, and the engobe are within acceptable limits, thereby preventing the development of critical thermal stresses. This is confirmed by the absence of cracks and glaze defects after industrial firing and thermal shock resistance testing.

**Discussion.** The results of the conducted studies confirm that the developed engobe and protective glaze compositions exhibit a balanced combination of chemical composition, microstructural characteristics, and thermal properties, which ensures their high performance under industrial porcelain stoneware firing conditions.

Microstructural analysis by SEM performed on pure glaze samples revealed the formation of a dense and homogeneous glass-phase structure without pronounced porosity, microcracks, or delamination zones. For the protective glaze, the presence of a zirconium-containing phase uniformly distributed within the glass matrix was established, as confirmed by local EDS analysis. The presence of zirconium is associated with the addition of zirconium silicate and is directly related to the increased coating density, whiteness, and chemical durability of the protective glaze layer. In contrast, the engobe layer is dominated by an aluminosilicate component, which corresponds to its functional purpose of leveling the ceramic body surface and ensuring reliable adhesion of subsequent layers.

During firing, the development of the glaze structure occurs through successive softening of frit components, formation of a liquid silicate phase, and dissolution of crystalline raw material components. The frit acts as the primary source of the glass-forming melt, while quartz, feldspar, and clay-derived phases participate in the formation of the final aluminosilicate glass network. Quartz contributes $SiO_2$ to the glass structure; however, its dissolution in the molten phase depends on temperature, particle size, and melt viscosity. Feldspar components promote liquid-phase formation through the release of alkali oxides ($Na_2O$ and $K_2O$), which decrease the melting temperature and enhance the mobility of the melt. The $Al_2O_3$ supplied by feldspars and clay minerals participates in the formation of an aluminosilicate network, improving the chemical stability and mechanical strength of the resulting glaze layer.

The alkaline-earth oxides CaO and MgO additionally modify the silicate network by reducing melt viscosity, improving wetting of the porcelain stoneware surface, and promoting stronger adhesion between the glaze and ceramic body. During cooling, the homogeneous silicate melt transforms into a dense glassy phase, while zirconium silicate remains as a finely dispersed crystalline phase. This phase separation mechanism provides effective light scattering, resulting in increased opacity and whiteness of the protective coating. The obtained SEM–EDS results confirm the formation of a compact glass–ceramic structure consistent with the functional requirements of porcelain stoneware glaze coatings.

The obtained coefficient of thermal expansion (CTE) values indicate good thermal compatibility of all elements of the «body–engobe–protective glaze» system. The CTE values of the glaze compositions are slightly lower than or comparable to those of the porcelain stoneware body, which is a favorable factor in terms of residual stress formation during cooling. Such a CTE relationship reduces the risk of crack formation, crazing, and glaze delamination, as confirmed by the absence of defects after industrial firing and thermal shock resistance testing. The obtained results confirm that the achieved performance characteristics are governed by the combined influence of chemical composition, glass-phase structure, and coordinated thermal behavior of the glaze–body system, which agrees with published studies on porcelain stoneware glazes (Chiang et al., 1997; Carty and Senapati, 1998; Sánchez, 2010; Richerson, 2006).

The high resistance of the glaze coatings to thermal shock (more than 125 cycles), the absence of visible surface changes after exposure to acidic media, and a surface hardness of 6 on the Mohs scale indicate the effectiveness of the selected compositions and the correctness of the choice of frits, opacifying additives, and firing regimes. The combination of these results confirms that the achieved performance characteristics are governed not by a single factor but by the complex interaction of chemical composition, glass-phase structure, and coordinated thermal behavior of the system as a whole.

Thus, the developed engobe and protective glazes demonstrate high technological stability and operational reliability under industrial porcelain stoneware production conditions. The obtained data confirm the feasibility of applying the selected formulations and technological approaches, as well as their potential for use within import substitution technologies.

**Conclusion.** In the course of this study, engobe and protective glaze compositions for porcelain stoneware based on domestic mineral raw materials and industrial ready-made frits were developed and comprehensively investigated. The applied approach, which included analysis of microstructure, thermal characteristics, and performance properties, made it possible to objectively assess the technological and functional suitability of the developed compositions.

Microstructural investigations by scanning electron microscopy performed on pure glaze samples revealed the formation of a dense and homogeneous glass-phase structure without pronounced defects. For the protective glaze, a uniform distribution of a zirconium-containing opacifying phase was confirmed, providing high whiteness and chemical durability of the coating, whereas the engobe is characterized by an aluminosilicate matrix that enhances adhesion and reduces defect formation within the glaze system.

The determined coefficients of thermal expansion indicate good thermal compatibility of the «porcelain stoneware body–engobe–protective glaze» system. The coordinated thermal behavior of the components prevents the development of critical residual stresses, which is confirmed by the absence of cracks and defects after industrial firing and thermal shock resistance testing.

The results of physic-mechanical and performance tests demonstrated that the developed glaze coatings exhibit high resistance to thermal shock (more than 125 cycles), chemical resistance with no visible surface changes after acid exposure, and a surface hardness of not less than 6 on the Mohs scale. The combination of these results confirms the correctness of the selected compositions and technological regimes.

Thus, the developed engobe and protective glazes demonstrate high technological stability and operational reliability under industrial porcelain stoneware production conditions. The obtained results confirm the perspective of the proposed solutions for practical application and their feasibility within the framework of import substitution tasks.

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