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Қ. И. Сәтпаев атындағы Қазақ ұлттық техникалық зерттеу университеті

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НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
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MINERAL RAW MATERIALS SELECTION AND ELECTROTECHNICAL USE PORCELAIN SYNTHESIS

Abstract. Clay and nonplastic types of raw materials necessary for electrotechnical porcelain synthesis, as well as wollastonite raw materials for additives were considered from among the minerals of the Republic of Kazakhstan; the compositions, properties, phase transformations during heat treatment of kaolins, white-burning refractory clays, quartz sands, feldspars and wollastonites were studied; a technology was developed for obtaining porcelain for electrical insulation with the use of mineralizing additives – wollastonites; porcelain's structure and phase composition formation processes and its mechanical and dielectric properties were studied; optimal ratios of porcelain mixture components and amount of additives needed to improve the porcelain phase formation process by increasing the mullite crystals formation at lower temperatures compared with traditional porcelain were determined; first proved the possibility of obtaining electrotechnical porcelain with high properties from local natural types of mineral raw materials; the developed electrotechnical porcelain technology introduction will allow to solve the import substitution problem.

Key words: electrotechnical porcelain, mineral raw materials, kaolins, clays, feldspars, quartz sands, wollastonite.

Introduction. Electrotechnical porcelain is the main ceramic material used in the production of a wide range of low-voltage and high-voltage insulators [1-5].

In Kazakhstan, electrotechnical porcelain is not produced, and existing factories for production of household items from porcelain that are close to it in terms of composition and technology are mainly focused on imported raw materials. In recent years, there has been a marked depletion of high-quality raw materials for this industry in the CIS countries. At the same time, Kazakhstan has significant industrial reserves of all types of natural mineral raw materials traditionally used in the production of fine ceramics and electrotechnical porcelain.

In the current industrial and economic situation in the Republic of Kazakhstan, one of the country's priority scientific and technical tasks is accelerated development and introduction of the production of modern electrotechnical porcelain insulators based on domestic mineral resources. The solution of this scientific and practical problem is undoubtedly relevant [6].

The purpose of the work is a comprehensive study of promising selected mineral raw material types, development on their basis the mass composition and obtaining import-substituting electrotechnical porcelain.

Materials and methods. The objects of study are kaolins, white-burning clays, quartz sands, feldspars and wollastonites of deposits of the Republic of Kazakhstan; mass compositions developed on their basis; physical and mechanical properties of the synthesized samples.

The basis of scientific research consists of complex chemical, mineralogical, petrographic and electron microscopic studies of raw materials; study of high-temperature phase-structural changes; chemical analyzes of raw materials and porcelain according to the relevant standard requirements; thermal analyzes using NETZSCH STA 449 F3 Jupiter; elemental analysis using X-Supreme 8000 desktop X-ray fluorescent energy dispersive analyzer; X-ray investigation using DRON-3; electron microscopic studies using JSM-6490LV multipurpose raster microscope (JEOL.Ltd, JAPAN).

Results. Particularly strict requirements are imposed on electrotechnical porcelain in terms of its mechanical strength, breakdown voltage and dielectric losses. These requirements can be satisfied only by dense sintered products with a homogeneous structure and minimum number of pores, obtained on the basis of appropriate types of plastic clay, kaolin, feldspar and silica mineral raw materials with material composition and purity.

The mechanical strength, thermal and chemical resistance of porcelain, including electrotechnical, enhance with increase in the mullite content and improving the glassy phase in it, which is achieved by increasing the kaolin content in the mass [7-9]. The latter at 1000-1200°C decomposes with the release of heat, giving sillimanite at the beginning, and then mullite. These reactions form the basis of all ceramic production with the use of kaolinite.

According to the mineralogical and chemical characteristics, kaolins of one of the large deposits the Soyuznoye, located in the North-Mugodzhar area, for solving the problems of these studies seem to be the most favorable. The deposit is represented by the trias-Cretaceous areal weathering crust over granites and shales. The kaolin useful stratum consists of dense, block types of white and light gray clays [4]. The averaged results from five chemical analyzes of the samples are as follows: SiO_2 – 59.75%, Al_2O_3 – 28.94%, Fe_2O_3 – 0.47%, TiO_2 – 0.32%, CaO – 1.87%, MgO – 0.73%, K_2O – 0.26%, Na_2O – 0.11%, loss on ignition – 8.53%. According to the mineral composition, kaolins are fairly simple: kaolinite – 71-87%, hydromica – 5-7%, quartz – 6-8% and other – 2-4%.

The enrichment of raw materials in the laboratory under the electrolyte-free wet enrichment scheme provided a noticeable increase in the volume fraction of kaolinite in the raw materials and, accordingly, Al_2O_3 (table 1).

On the X-ray diffraction pattern of enriched kaolins (figure 1), unlike natural ones, diffraction maximums dominate and quartz maximums are weakly expressed.

Table 1 – Chemical composition of the Soyuznoye enriched kaolins

Oxide content, mass percent								Δm_{pr}
SiO_2	Al_2O_3	CaO	MgO	TiO_2	Fe_2O_3	K_2O	Na_2O	
50.94	36.52	0.29	0.08	0.514	–	0.17	–	11.94

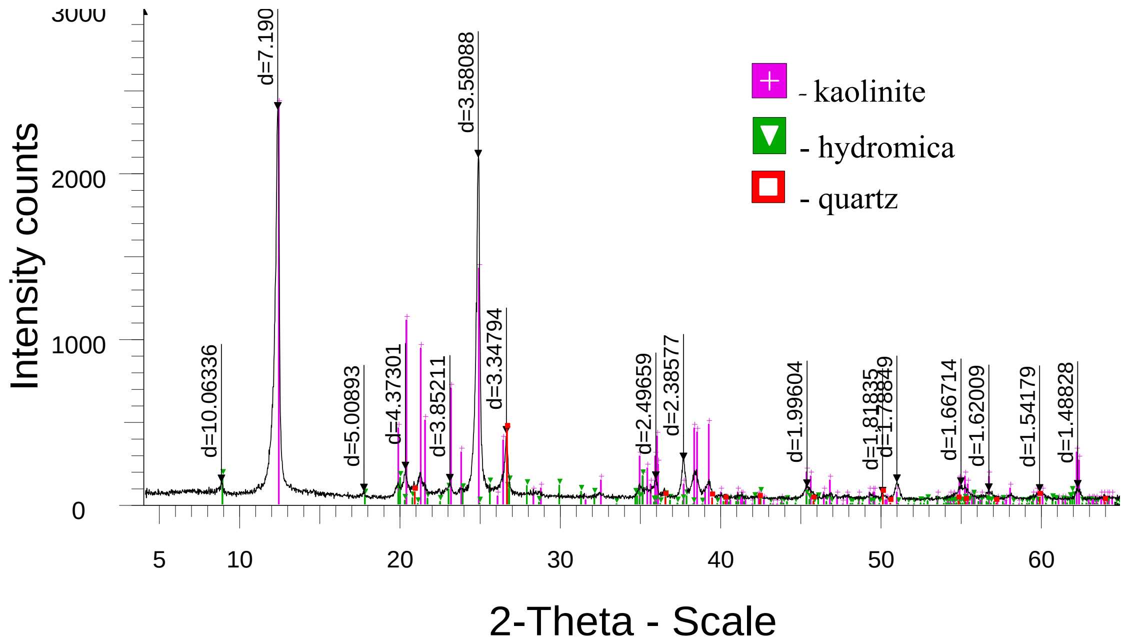


Figure 1 – X-ray diffraction pattern of the Soyuznoye enriched kaolin

The comprehensive studies have shown that the Soyuznoye enriched kaolins are refractory (1690 – 1750°C); the main in the composition (Al_2O_3 – 36,52%) with a very low content of coloring oxides (Fe_2O_3+TiO_2 – 0,514%); kaolinite in the mineral composition; highly dispersed (fractions less than 10 microns – 88.9%); moderate plastic and high-temperature sintering (1400°C). These indicators are fully consistent with the requirements of the State Standard for kaolins for production of electrotechnical porcelain.

In the mass compositions of electrotechnical porcelain, as in other types of fine ceramics, white-burning plastic clays, in addition to kaolin, play an important technological role. The range of functions of such clays includes the forming and molding properties of ceramic masses, mechanical strength in the air-dry state, operational strength and chemical resistance after firing [10-12].

Considerable reserves of white-burning plastic clays are concentrated in the deposits of West, Central and North Kazakhstan. Among them, the Berlinskoye deposit is the most studied and under development, the productive stratum of which is represented by oligocene kaolin clay reservoir. The suitability of its raw materials for production of facing porcelain tiles, chemically resistant bricks, ceramic nozzles and sewer pipes was assessed [13].

In the Berlinskoye enriched clays, kaolinite, montmorillonite, hydromica, and quartz are clearly established (figure 2). Their chemical composition, mass percent: SiO_2 – 49.50; Al_2O_3 – 30.9; Fe_2O_3 – 1.84; TiO_2 – 0.92; CaO – 0.61; MgO – 0.071; K_2O – 0.78; Na_2O – 0.11; SO_3 – 0.021; MnO – 0.008; loss on ignition – 12.87.

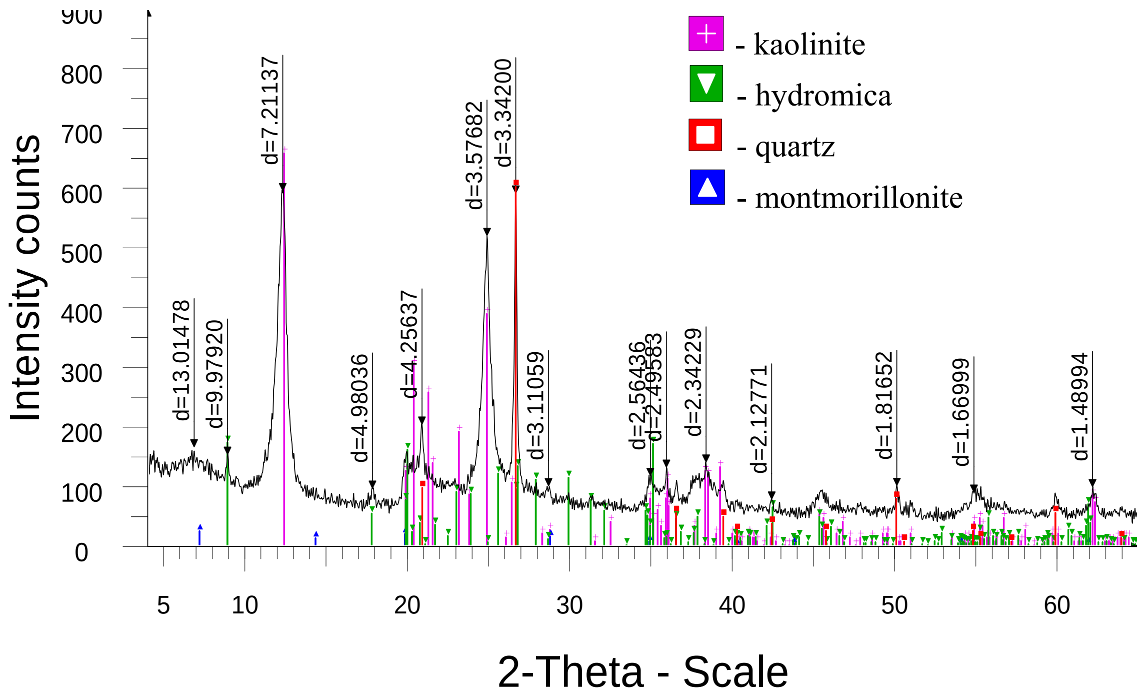


Figure 2 – X-ray diffraction pattern of the Berlinskoye enriched clays

It was established that the Berlinskoye clays after the enrichment are refractory (1630-1790°C); the main in the composition (Al_2O_3 – 32.15%); kaolin-hydromica; medium plastic; medium sintering (1300°C) and highly caking.

Quartz sands from the upper paleogene of the Mugodzhar deposit were selected and investigated as an emaciated component responsible for reducing shrinkage and deformation of products.

Earlier laboratory and technological studies have shown that the Mugodzhar quartz sands are high grade and meet the requirements for production of white cement, sheet glass and technical glass [14].

The average chemical composition of the Mugodzhar sands, mass percent: SiO_2 – 98.07; TiO_2 – 0.05; Al_2O_3 – 0.40; Fe_2O_3 – 0.11; CaO – 0.14; MgO – 0.09; MnO – 0.07; Na_2O – 0.17; K_2O – 0.17. They consist mainly of quartz.

No less responsible is selection of the desired variety of feldspar, introduced as a thickener and fusing agent for formation of the optimal glass phase in the electrochemical porcelain sintering process [15-17].

For this purpose, pegmatite feldspars of the Sarybulak deposit in the area of Mirny village were investigated. The chemical composition, mass percent: SiO_2 – 79; Al_2O_3 – 10.915; Fe_2O_3 – 0.402; TiO_2 – 0.105; CaO – 0.153; MgO – 0.017; K_2O - 5.8; Na_2O – 4.14. The mineral raw materials consist mainly of albite, microcline and less often quartz.

The introduction of various mineralizing additives in the composition of ceramic masses in small quantities leads to improvement in the process of porcelain formation and increase in the appearance of mullite crystals. The latter, in turn, improve the quality characteristics of finished products [18-21].

For the first time, wollastonites of the Upper-Badam deposit were tested as mineralizing additives. The Upper-Badam technological wollastonite sample chemical analysis results, mass percent: SiO_2 – 46.42; Al_2O_3 – 0.11; Fe_2O_3 – 0.53; CaO – 46.09; MgO – 0.5; K_2O – 0.7; Na_2O – 0.20; loss on ignition – 7.73. In the samples studied (figure 3): wollastonite (d/n = 7.66785; 5.44570; 4.05062; 3.83412;), calcite (d/n = 3.83412; 3.03585; 2.89973; 2.47782;) and quartz (d/n = 4.24614).

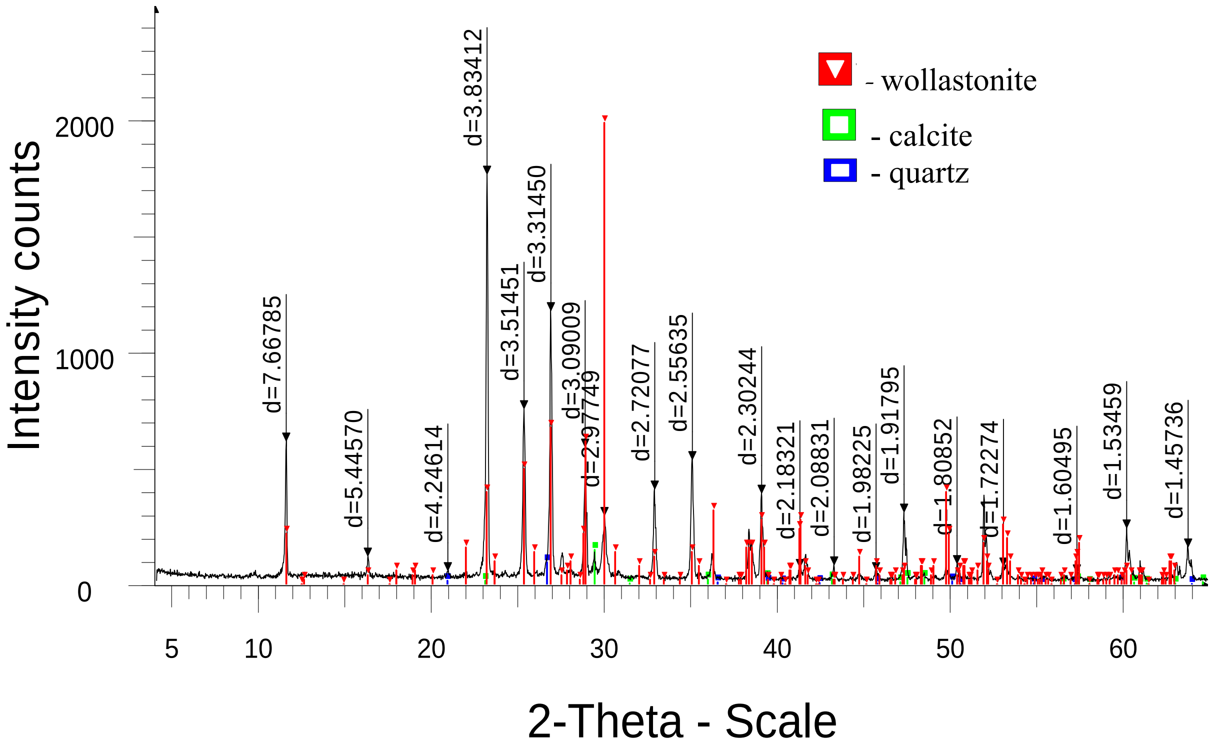


Figure 3 – X-ray diffraction pattern of the Upper-Badam wollastonite

A series of experiments were carried out to find the optimal parameters for synthesizing new porcelain with regard to the results of studying chemical and mineral compositions, technological properties, physicochemical processes and phase transformations during heat treatment of selected domestic kaolin, white-burning clays, quartz sands, feldspars and wollastonites. To determine the role of wollastonite as a mineralizer, compositions with its additives from 1 to 5% were developed.

The samples were made on laboratory equipment using plastic technology. Dried to a residual moisture content of 1%, the test samples were fired at 1280-1320ºC.

The analysis of the change in the samples strength depending on the burning temperature (figure 4) shows that when the masses are fired with the wollastonite addition to 2%, a uniform increase in the strength to a temperature of 1250-1300°C is observed. Then, when the temperature rises above 1300°C, it decreases. The strength of ceramic masses with the wollastonite content from 2 to 3% increases to a temperature of 1200°C, but only to values of 50-52 MPa. Then, as well as in the previous masses, a decrease in the strength characteristics is observed. The porcelain samples with the wollastonite addition from 3 to 5% have low strength indicators, so their values are not reflected in the graph.

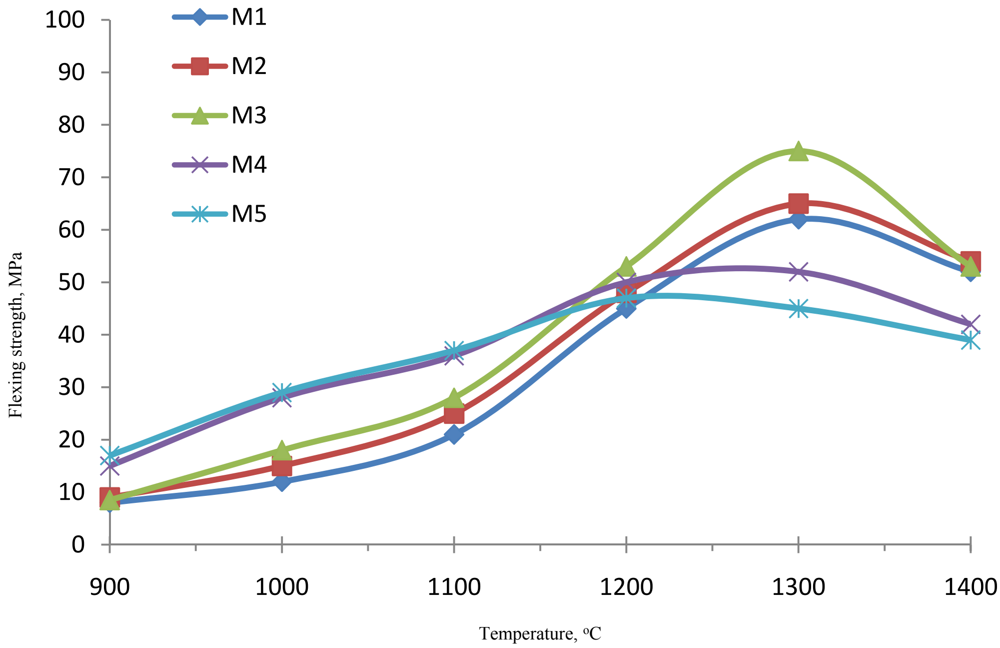


Figure 4 – The change in the porcelain samples strength with various wollastonite content after the firing at various temperatures: M1 – the mass with the wollastonite content up to 1%, M2 – the mass with the wollastonite content up to 1.5%, M3 – the mass with the wollastonite content up to 2%, M4 – the mass with the wollastonite content up to 2.5%, M5 – the mass with the wollastonite content up to 3%

The high strength indexes of the samples based on the masses with the wollastonite up to 2% at a relatively low firing temperature (1290-1300°C) compared with classic porcelain, are explained by the fact that wollastonite in the porcelain mass composition contributes to the formation of a liquid phase with increased reactivity. This accelerates the ceramics structure formation process, increases the content of slow-moving calcium ions in the glass phase.

A higher wollastonite content in the masses – from 2 to 5% narrows the porcelain sintering interval and increases the open porosity degree.

The most favorable ratio of raw components was established: 50-55% clay components (kaolin and clay), 20-25% quartz sands, 25-30% feldspars and 1-2% wollastonite.

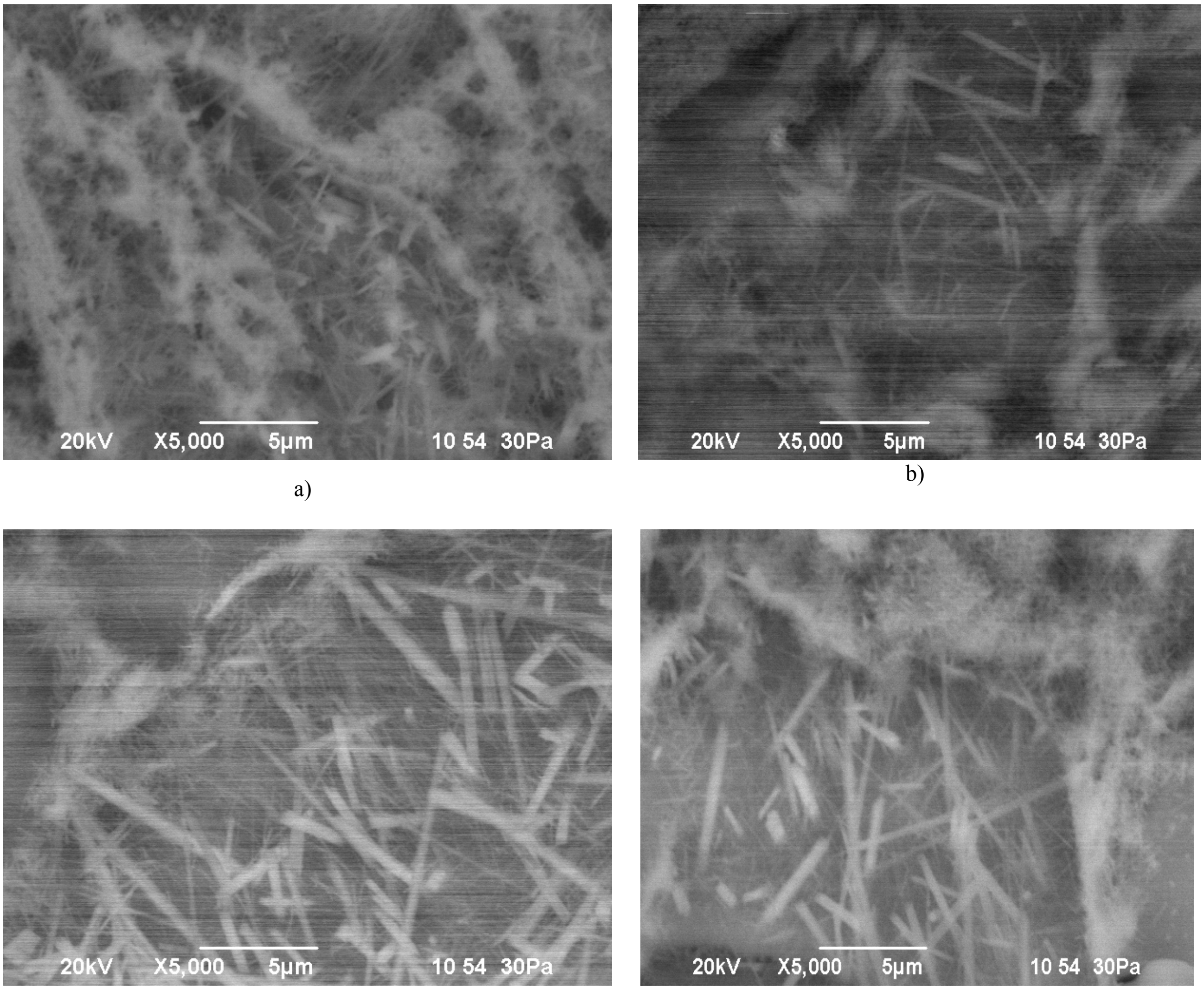
The basic properties of optimal composition porcelain samples were determined (table 2).

The crystalline phases of the calcined samples consist of the mullite grains (d/n = 5.4293; 3.4221; 2.8930; 2.7036; 2.5509; 2.2949; 2.2124; 2.1267; 1.8923; 1.6999; 1.6022; 1.5808) and quartz (d/n = 4.2921; 3.3661; 1.8227).

Table 2 – Physical-mechanical and electrical parameters of the samples

Indexes	Values	Requirements of GOSTs
Fire shrinkage (t=1350°C) / full shrinkage, %	8.2 / 13.2	–
Water absorption, %	0.0	0.0
Density, g/cm^3	2.53	2.45
Flexing strength: Unglazed sample, MPa Glazed sample, MPa	 69.7 75.8	 60 70
Tensile strength: Unglazed sample, MPa Glazed sample, MPa	 31.5 37.8	 30 35
Electric strength at a frequency of 50 Hz, kV/mm	28.9	25
Dielectric permeability at a frequency of 50 Hz	6.5	From 6 to 7

On the electron micrographs of chipped porcelain mass without wollastonite, it can be seen that the sample has a rather dense structure (figure 5 a, b). The main constant phase in which other phases are dispersed is glass. It is clearly visible how the mullite secretions of various habit sprout from the feldspar glass. There are areas penetrated by large well-developed crystals and areas in which there are their frequent weaves.



a) b)

c) d)

Figure 5 – Electron microscopic photographs of the electrotechnical porcelain without (a, b) and with the addition of wollastonite (c, d)

The structure of pieces of the samples obtained with the wollastonite addition is characterized by a more dense structure and greater mullitization, where pores are practically absent. This is apparently due to the fact that the wollastonite in the porcelain mass composition reduces the liquid phase appearance temperature and changes the structure, as well as the feldspar melt properties. The diffusion processes involving the melt are activated, its dissolving capacity grows. As a result, the amount of dissolved primary mullite increases and its transformation into the crystalline phase in the form of secondary mullite. In the electronic photographs of the structure of the mass samples with the wollastonite content up to 2.5%, these mullite crystals are represented mostly in the feldspathic areas in the form of shagreen and thick felt. In general, the samples based on this mass have a good degree of maturity.

Discussion. The mineralogical and petrographic analysis of the selected raw materials types, experimental and technological studies have shown the possibility of obtaining electrochemical porcelain purely on the basis of domestic mineral raw materials.

The technological scheme has been developed for the production of high-performance electrotechnical porcelain of original composition, where kaolins, white-burning clays, quartz sands, feldspars and wollastonites were used as components.

The electrotechnical porcelain products were synthesized and their decisive physical and mechanical properties were determined. The qualitative characteristics of the developed mass compositions meet the requirements for the porcelain of insulating purpose.

Conclusions. The Soyuznoye kaolins, the Berlinskoye refractory clays, the Mugodzhar quartz sands, the Sarybulak feldspars, the Upper-Badam wollastonites fully comply with the requirements set by the State Standards for raw materials for the electrotechnical porcelain production.

The electrotechnical porcelain, produced with the use of plastic and nonplastic raw materials of deposits of the Republic of Kazakhstan, according to its mechanical and electrotechnical properties comply with the requirements of GOST.

The introduction of the developed composition and technology for the electrotechnical porcelain production based on affordable local types of raw materials will provide the creation of domestic production of necessary products and will solve the problem of import substitution.

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МИНЕРАЛДЫ ШИКІЗАТ ТАҢДАУ ЖӘНЕ ЭЛЕКТРТЕХНИКАЛЫҚ МАҚСАТТАҒЫ ФАРФОР СИНТЕЗІ

Аннотация. Қазақстан Республикасының пайдалы қазбаларынан электртехникалық фарфор синтезіне қажетті сазды, иілгіш емес шикізат түрлері қарастырылды; волластонитті шикізат қоспа ретінде таңдалды; каолиндердің, отқа төзімді саздардың, кварцты құмдардың, дала шпаттарының және волластонитті тау жыныстарының құрамдары, қасиеттері және термиялық өңдеу барысындағы фазалық өзгерістері зерттелді; минералдаушы қоспалар – волластониттер қосылған электроқшаулағыш фарфор технологиясы жасалды; фарфордың фазалық құрамы мен құрылымы түзілу процесстері және оның механикалық және диэлектрлік қасиеттері зерттелді; фарфор массасының компоненттерінің оптимальді қатынасы және фаза түзілу процессін жақсартуға қажетті қоспалар мөлшері анықталды; алғаш рет жергілікті табиғи минералды шикізат түрлерінен жоғары сапалы электртехникалық фарфор алу мүмкіндігі дәлелденді; жасалған электрфарфор технологиясын өндіріске ендіру импорталмастыру проблемасын шешуге мүмкіндік береді.

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ПОДБОР МИНЕРАЛЬНОГО СЫРЬЯ И СИНТЕЗ ФАРФОРА ЭЛЕКТРОТЕХНИЧЕСКОГО НАЗНАЧЕНИЯ

Аннотация. Из числа полезных ископаемых Республики Казахстан рассмотрены глинистые, непластичные виды сырья, необходимые для синтеза электротехнического фарфора, а также волластонитовое сырьё для добавок; изучены составы, свойства, фазовые превращения при термической обработке каолинов, беложгущихся огнеупорных глин, кварцевых песков, полевых шпатов и волластонитовых пород; разработана технология получения фарфора электроизоляционного назначения с использованием минерализующих добавок – волластонитов; изучены процессы формирования фазового состава и структуры фарфора и его механические и диэлектрические свойства; определены оптимальные соотношения компонентов фарфоровой массы и количества добавок, необходимые для улучшения процесса фазообразования путем увеличения образования кристаллов муллита при более низких температурах по сравнению с традиционным фарфором; впервые доказана возможность получения из местных природных видов минерального сырья электротехнического фарфора с высокими свойствами; внедрение разработанной технологии электрофарфора позволит решить проблему импортозамещения.

Ключевые слова: электротехнический фарфор, минеральное сырье, каолин, глины, полевые шпаты, кварцевый песок.

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