Study of Shomishkul kaolin for producing ceramic materials

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Abstract. The article discusses the results of research studies of kaolins from the Shomishkul deposit in Karakalpakstan using chemical-mineralogical and X-ray phase analyses. As a result of the tests, it was established that samples of clay minerals from the studied deposits can be used as the main raw material component for the production of ceramic materials, which helps to expand the raw material base for the production of ceramic materials for construction purposes.

1 Introduction

The search for high-quality kaolin raw materials with the necessary physical, chemical and technological properties is an urgent problem in the ceramic industry. In this regard, the search and study of new non-traditional, previously unused clay raw minerals in the production of ceramic products is an urgent task. On the territory of the Republic of Uzbekistan (Karakalpakstan) there are large reserves of kaolins, which can be used in various industries, in particular multi-purpose ceramic materials. Therefore, in the production of high-grade porcelain and earthenware (after enrichment), ceramic tiles, they strive to increase the kaolin content in the mass by reducing the amount of undesirable impurities [1-3].

According to the chemical and mineralogical composition of the studied kaolin rocks with admixtures of muscovite or potassium feldspar, eluvial kaolins can be divided into non-alkaline (normal) and alkaline types. Alkaline kaolins are distributed mainly in certain parts of the formation, normal kaolins contain 0.3-0.5% K₂O, while alkaline kaolins are distinguished by a high content of alkaline oxides (1.7-4.5%) in the form of Na₂O and K₂O [4-5].

2 Experimental

To study the chemical and mineral composition of kaolin raw materials, the sintering process and the ceramic-technological properties of ceramic materials, differential thermal, X-ray diffraction and raster electron microscopic analysis methods were used.

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Chemical analysis of raw materials and prototypes was carried out according to standard silicate analytical methods, specified in accordance with GOST 9169-75 [8].

Determination of melting temperature. The experimental mixtures under study were prepared by careful fine grinding of mixtures of synthesized starting components. Test cones were then pressed into a metal mold to determine melting points. The experimental determination of melting temperatures was carried out in a laboratory tubular furnace with carborundum heaters at an average heating rate of 8-10 °C per minute. Temperature control was carried out by a TPR thermocouple with an accuracy of ±10 °C. The maximum temperature reached in the tube furnace was 1640 ± 10 °C. The melting temperatures of experimental samples of ceramic masses were determined visually using the cone drop method in at least three experiments [9-10].

Phase changes in the studied raw materials and experimental masses were determined by the X-ray phase method. Filter-Ni, length β radiation (αX-ray phase studies of crystalline phases were also carried out on a modern powder diffractometer LABX XRD-6100 (Shimadzu, Japan), controlled by a computer, in the range with a counter rotation speed of 20°/min in the range of 10-80°, using CuK waves 1.5418 Å, current mode and tube voltage 30 mA, 30 kW). Constant rotation speed of the detector is 2°/min in increments of 0.02 degrees. (ω/20-coupling), the scanning angle varied from 4 to 80°. The shooting conditions for all samples were kept constant. In the calculations and identification of phases, we used tables and reference books compiled by the authors of the works, as well as the international American card index [11].

Using scanning electron microscopy, the elemental and phase compositions of minerals and the morphological features of new crystal formations were studied. Studies were carried out using scanning (SEM) CamScan-4 (Cambridge) and VEGA IIXMU (Tescan) and transmission (TEM) JEM 2100 (JEOL, Japan), Tecnai G230ST TEM/STEM (FEI, Hillsboro, OR, USA) electron microscopy [12].

The temperature-phase transformation and stability region of basalt samples were studied using DTA (Labsys EVO Setaram device, heating temperature up to 1000°C, heating rate 20 °C/min). The analysis of the obtained DTA results was carried out using fundamental works on thermal analysis [13].

3 Results and Discussion

In order to use kaolins from the Shomishkul deposit in ceramic materials, chemical and mineralogical analyzes were carried out to determine their chemical composition [6-7]. The results of the analysis of the chemical composition and determination of the physical and mechanical properties of kaolin samples from the Shomishkul (ShomK) deposit are presented in Table 1.

Table 1. Chemical composition of the initial samples of kaolins from the Shomishkul deposit.

<table>
<thead>
<tr>
<th>Sample name</th>
<th>Oxides content, wt.%</th>
<th>LOI, wt.%</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>SiO₂</td>
<td>Al₂O₃</td>
</tr>
<tr>
<td>ShomK -1</td>
<td>66.34</td>
<td>16.61</td>
</tr>
<tr>
<td>ShomK -2</td>
<td>67.20</td>
<td>14.04</td>
</tr>
<tr>
<td>ShomK -3</td>
<td>65.70</td>
<td>17.14</td>
</tr>
<tr>
<td>ShomK average</td>
<td>66.41</td>
<td>15.93</td>
</tr>
</tbody>
</table>

From Table 1 it can be seen that the chemical composition of kaolins samples from the Shomishkul deposit, depending on the aluminium oxide content in the initial state, corresponds to the group of semi-acidic clayey raw materials. Important technological
parameters for the production of building ceramics are the initial and final firing temperatures of raw materials. These values indirectly characterize the energy consumption for obtaining fired material. The phase composition of the studied samples of kaolins samples from the Shomishkul deposits was established by X-ray phase analysis methods, the results of which are respectively shown in Figure 1.

The results of X-ray phase analysis showed that the X-ray diffraction pattern of samples from the Shomishkul deposit mainly shows diffraction maxima related to kaolinite minerals d = 0.720; 0.447; 0.356; 0.282; 0.246; 0.234; 0.199; 0.167 nm, lines corresponding to SiO₂ β-quartz with diffraction lines d = 0.425; 0.334; 0.246; 0.228; 0.224; 0.213; 0.198; 0.182; 0.154; 0.138; 0.137 nm, and albite d= 0.653; 0.375; 0.323; 0.319; 0.299.

Fig 1. X-ray diffraction pattern of a sample of kaolin from the Shomishkul deposit.

X-ray diffraction patterns of the studied kaolin show that the mineral composition consists mainly of the minerals kaolinite, quartz, potassium-sodium feldspar, in addition, a small amount of iron oxide and muscovite are present. The increased content of iron and titanium oxide requires enrichment of the studied kaolin before introducing them into the ceramic mass in the production of porcelain or earthenware; for building ceramics they can be used in their original state. The results of elemental analysis also confirm the results of chemical and X-ray phase analyzes shown in Figure 2 and given in Table 2.

<table>
<thead>
<tr>
<th>Shomishkul deposit</th>
<th>Element</th>
<th>weight. %</th>
<th>Sigma weight. %</th>
</tr>
</thead>
<tbody>
<tr>
<td>O</td>
<td>48.27</td>
<td>0.62</td>
<td></td>
</tr>
<tr>
<td>Na</td>
<td>1.04</td>
<td>0.16</td>
<td></td>
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<tr>
<td>Mg</td>
<td>0.81</td>
<td>0.14</td>
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<tr>
<td>Al</td>
<td>12.86</td>
<td>0.31</td>
<td></td>
</tr>
<tr>
<td>Si</td>
<td>29.60</td>
<td>0.46</td>
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<tr>
<td>K</td>
<td>2.86</td>
<td>0.20</td>
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<tr>
<td>Cl</td>
<td>0.93</td>
<td>0.14</td>
<td></td>
</tr>
<tr>
<td>Ti</td>
<td>1.13</td>
<td>0.22</td>
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<tr>
<td>Fe</td>
<td>2.50</td>
<td>0.38</td>
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<td>Total:</td>
<td>100.00</td>
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</table>
Fig 2. Electron microscopic images and elemental analysis spectra of kaolin sample from the Shomishkul deposit.

The results of DTA of kaolin samples from the Shomishkul deposit are shown in Figure 3.

Fig. 3. Differential thermal curves of Shomishkul kaolin.

The results of DTA of Shomishkul kaolin (Figure 3) showed the presence of four endothermic effects and two exothermic ones. Endothermic effects at 359, 464 and 526°C are associated with the removal of absorbent and chemically bound water. The endoeffect at 869°C reflects decarbonization processes. The appearance of two exothermic effects at 200 and 597°C can be explained by the process of burning out organic impurities present in
the raw material, as well as the transition of low-temperature $\beta$-quartz to high-temperature $\alpha$-quartz.

4 Conclusions

Thus, based on the experimental studies carried out using chemical analytical, DTA, X-ray diffraction, and raster electron microscopic (REM) analysis methods, it was established:

- Samples of kaolin from the Shomishkul deposits of Karakalpakstan, in terms of their chemical and mineralogical composition, meets the requirements for the production of ceramic materials for construction purposes as a plasticizing (clay) component in the composition of ceramic masses for economic and construction purposes.
- The use of new kaolin deposits in Karakalpakstan makes it possible to expand the raw material base for the production of ceramic materials for various purposes.

References

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