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# ORE ENRICHMENT WASTE AS RAW MATERIAL FOR HEAVY METAL SORBENTS

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## ABSTRACT

*The study considered ore enrichment waste from the Ashiktas, Akbakai, and Maikain deposits to create sorbents based on silicophosphates. X-ray phase and electron probe elemental analysis were carried out, which confirmed the presence of silicon, aluminosilicate, and other valuable components suitable for the sorbent's synthesis. The optimal synthesis temperature (600°C) contributes to the sorbents' porous structure development and a sorption capacity increase concerning* $Ni^{2+}$ *ions, allowing them to be used to purify industrial wastewater from heavy metals, which was proven using zeta potential measurements and SEM images.*

Keywords: *technogenic raw materials, ore enrichment wastes, silicophosphates, sorbents, structural analysis.*

## INTRODUCTION

Sorption treatment of wastewater is one of the ways to achieve sustainable development, addressing issues such as clean water and sanitation, as well as responsible consumption and production [1].

Wastewater treatment in Kazakhstan is currently one of the most important problems of the republic [2 - 4]. It requires an integrated approach to the development of sorption treatment technology from various pollutants, including heavy metals. The advantages of sorption treatment are versatility, efficiency, and controllability. It also allows the use of a wide range of porous materials such as active clay, activated carbon, ash, organic sorbents [1], etc.

Activated carbons are the most effective and popular porous materials for water purification [5]. However, the synthesis of active carbons with a nanostructure is expensive, which is associated with their high final cost, fire hazard, and regeneration problems [5, 6]. Synthesis of sorbents from domestic raw materials is economically and environmentally beneficial since waste from ore enrichment at Kazakhstan deposits is used as raw material. New inorganic silicophosphate sorbents can be obtained based on these wastes under acid-thermal synthesis conditions. Currently, there is a tendency to use secondary materials as raw materials for obtaining new sorption-filtering materials [7 - 9].

The most common methods for obtaining sorption materials with a developed specific surface area include the precipitation method from aqueous solutions of salts followed by heat treatment of the obtained compounds [10, 11]. Sorption materials obtained by joint precipitation of silicate and phosphate salts are of practical interest since sorbents based on these individual compounds are widely used in industry.

Several large deposits around the world produce significant volumes of waste rock that can be processed into environmentally useful materials, such as adsorbents, to solve industrial and environmental problems. Mines in countries such as Chile, the United States, and Australia produce copper and iron gangue that often contains reactive minerals such as pyrite. Research suggests that these materials, if processed, have the potential to adsorb harmful substances such as heavy metals and organic dyes in wastewater treatment [12]. Waste from gold mines, particularly in South Africa and China, includes sulfide-rich materials that can undergo oxidation to form compounds suitable for the adsorption of heavy metals [12]. Research suggests that processed gold mining residues can be adapted as low-cost adsorbents for industrial applications, effectively reducing pollutants [13]. The granite and marble quarry wastes such as granite powder, common in Brazil and Italy, have shown promise in the dyes adsorption from industrial effluents. When processed, these by-products can interact with dye molecules through adsorption processes, making them practical for water treatment solutions [13]. Countries such as China and India produce vast quantities of waste and tailings from coal mining. These materials contain clays and silicates that have been reused in studies to remove toxins and heavy metals from polluted waters, suggesting their potential for use in large-scale wastewater treatment [12].

In Kazakhstan, about three-quarters of the gold produced comes from medium-sized gold deposits (gold reserves from 25 t to 100 t) [14, 15]. Such deposits include the Ashiktas and Akbakai deposits.

According to the results of preliminary testing and chemical analysis of the Ashiktas deposit, the average gold grade in the studied samples was 1.94 g $t^{-1}$. The presence of a small amount of silver was also noted, the content of which was 6.24 g $t^{-1}$. The enrichment of this ore is carried out using a flotation scheme with a yield of 2.12 % gold-bearing concentrate with a gold content of 78.52 g $t^{-1}$ and silver of 105.71 g $t^{-1}$ with the extraction of gold of 86.70 % and silver of 35.91 %. Given the low gold content in the ore, after its extraction, a huge mass of enrichment waste is formed, which is currently almost not utilized [16, 17]. In Kazakhstan, during the processing of various types of ore and minerals are sent to landfills, and their volumes are constantly increasing. The annual percentage of processing of man-made mineral formations is only about 11 % of the total production volume, while in world practice this figure is about 70 %. In this regard, the possibility of using man-made mineral formations as raw materials for sorption-filtering materials was studied. The waste used to obtain new silicophosphate sorbents must contain enough silica-containing substance (quartz) [18, 19]. Scientific studies show that the silica content provides more efficient sorption of heavy metals from wastewater [20, 21]. Silicophosphate sorbents obtained from gold mining waste, from polymetallic ores, offer promising opportunities for the adsorption of heavy metals such as copper and lead. To create these sorbents, the waste is subjected to acid-thermal treatment using phosphoric acid and calcination, which increases the porosity and adsorption surface area. Studies show that these modified silicophosphate sorbents can effectively remove heavy metals from water, with the adsorption capacity reaching 2.5 mg $Cu^{2+}$ per gram under optimal conditions. Such methods not only recycle mining waste but also solve environmental problems by offering effective reclamation solutions [22]. Studies have shown that various adsorbents, including silicophosphates and other inorganic materials, are effective in capturing heavy metals such as cadmium, lead, and arsenic. Modified silicophosphates, especially when combined with functional agents, show promising results in ion adsorption due to their high surface activity and reactivity at different pH levels. Key methods include functionalization with oxides or other reactive groups to enhance the sorption capacity and stability under different solution conditions [23, 24]. Surface modifications that create a negatively charged sorbent surface are highly effective in heavy metals adsorption such as lead, copper, and cadmium from aqueous solutions. Commonly used methods include acid activation, chemical functionalization with anionic surfactants, and incorporation of phosphate groups. Acid treatments such as hydrochloric or sulfuric acid replace the exchangeable cations with protons, increasing the surface porosity and exposing more negatively charged silicate structures. This increases the affinity of the material for heavy metals, as seen in modified montmorillonite clays, which effectively adsorb metals due to their increased negative charge and increased surface area. Research shows that these acid-activated clays are particularly useful for the adsorption of ions such as Pb (II) and Cd (II) [25]. Phosphate-modified materials introduce negatively charged phosphate groups, which not only enhance metal binding but also provide stability over a range of pH levels. Such functionalization methods have been shown to improve heavy metal adsorption due to the strong interactions between the phosphate groups and the metal ions [26].

To illustrate the substitution reactions occurring during the sorption of nickel ions ($Ni^{2+}$) on silicophosphate sorbents, the following scheme can be used:

$$R\text{-}M+Ni^{2+}\rightarrow R\text{-}Ni+M^{n+}$$

where R represents the sorbent matrix (e.g., silicophosphate), $M^{n+}$ is the initial ion (e.g., $H^+$, $Na^+$, or $K^+$) present on the sorbent, $Ni^{2+}$ is the nickel ion being adsorbed, R-Ni is the sorbent with the bound nickel ion. If phosphate groups participate in the sorption of nickel ions, the reaction can be represented as:

$$HPO_4^{2-}+Ni^{2+}\rightarrow(HPO_4)Ni$$

The $HPO_4^{2-}$ ion has a negative charge and can bind to positively charged cations such as $Ni^{2+}$ through ion exchange. This process involves the substitution of protons ($H^+$) or other cations (e.g., $Na^+$, $K^+$) associated with phosphate groups for nickel ions. The reaction depends on factors like the structure of the sorbent, its porosity, functional groups, and environmental conditions (e.g., pH, temperature).

During research work, the material and phase composition of waste from the enrichment of non-ferrous metal ores from some deposits in Kazakhstan, such as Ashiktas, Akbakai and Maikain, were studied as raw material sources of silicon compounds with subsequent selection of promising samples based on them for the creation of sorption-filtering materials.

## EXPERIMENTAL

### Materials

The following samples were selected as objects of study: technical sample 1 of gold-bearing ore beneficiation waste (from the Ashiktas deposit); technical sample 2 of oxidized gold-bearing ore (from the Akbakai deposit); technical sample 3 of sulfide gold-bearing ore (from the Akbakai deposit); technical sample 4 of Maikain copper-nickel-zinc ore (from the Maikain deposit). Fig. 1 shows a geographic map indicating the location of the above deposits.

The acid-thermal synthesis of silicophosphate materials based on ore processing wastes was carried out. Based on the content of acid-sorbing components

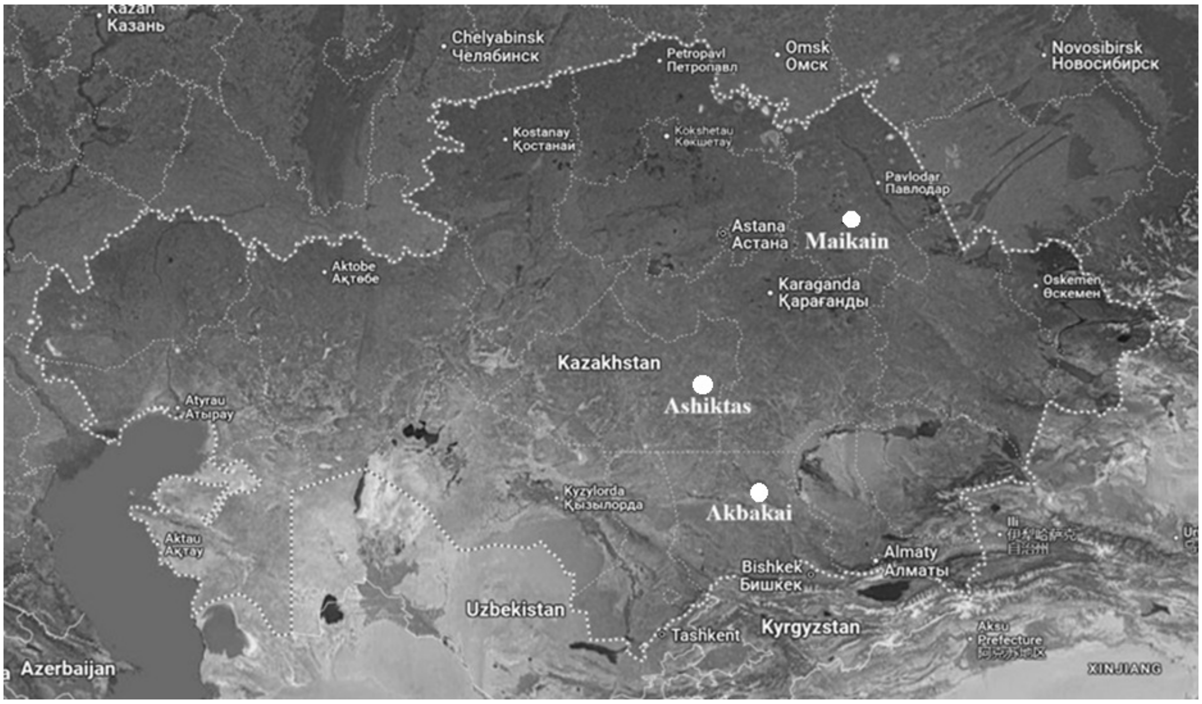

Fig. 1. Geographical location of the Ashiktas, Akbakai and Maikain mines.

were selected the most promising initial samples of wastes from this deposit. Then, the required amount of phosphoric acid (35 % by weight) for synthesis was calculated, considering the content of acid-sorbing components. A wet grinding of each sample with a particle size of 0 -10 μm was carried out followed by drying at 105°C for an hour, and finally, calcination at temperatures of 400°, 600°, and 800°C was made.

## Methods

### *X-ray diffractometric analysis of the phase composition and study of material composition of technical waste samples and obtained sorbents*

The phase composition was examined using X-ray diffraction analysis on an automated DRON-3 diffractometer with $Cu_{K\alpha}$ radiation and a β-filter. The diffraction patterns were recorded under the following conditions: voltage (U) at 35 kV, current (I) at 20 mA, a time constant of 2 s, $\theta$ - $2\theta$ scanning mode, and a detector speed of $2^o$ $min^{-1}$. A semi-quantitative X-ray diffraction phase analysis was conducted on powder samples using the equal hinge plate method and artificial mixtures, allowing for the determination of quantitative ratios of crystalline phases. The diffraction patterns were interpreted with reference to data from the ICDD's PDF2 (Powder Diffraction File) database.

The study of the material composition of products was carried out by electron microprobe analysis using a JEOL-733 electron microscope with an X-ray analyser.

### *Determination of the dynamic capacity of synthesized materials*

The determination of the dynamic capacity of the synthesized materials was studied under the following conditions: the sorbent was loaded into a 10 mm diameter column, then the material was washed with 10 L of distilled water. The column was connected to a pressure tank filled with a nickel salt solution. The solution flowing out of the column was collected in a 1 $dm^3$ Erlenmeyer flask and each portion (50 mL) was analysed for the presence of nickel cations. A qualitative determination was made at first, and after a positive result, a gravimetric analysis was performed.

### *Zeta potential and SEM surface analysis*

The zeta potential of the most effective samples was measured using the Zetasizer NanoZS 90 to determine the surface charge of both the initial material and the developed sorbents. Additionally, surface morphology and structure were analysed using a Scanning Electron Microscope (SEM), model Jeol JSM-6490.

Table 1. Results of semiquantitative X-ray phase analysis of tailings of gold-bearing ore enrichment.

| Waste | Phase name | Chemical composition | Concentration, % |
|---|---|---|---|
| Gold ore of the Ashiktas deposit | Quartz | $SiO_2$ | 96.4 |
| | Muscovite | $KAl_2(AlSi_3O_{10})(OH)_2$ | 3.6 |
| Oxidized Au-containing ore from the Akbakai deposit | Quartz | $SiO_2$ | 78.1 |
| | Albite | $Na(AlSi_3O_8)$ | 12.0 |
| | Muscovite | $KAl_2(AlSi_3O_{10})(OH)_2$ | 3.8 |
| | Calcite | $CaCO_3$ | 6.2 |
| Sulfide Au-containing ore of the Akbakai deposit | Quartz | $SiO_2$ | 56.8 |
| | Albite | $Na(AlSi_3O_8)$ | 39.5 |
| | Muscovite | $KAl_2(AlSi_3O_{10})(OH)_2$ | 5.8 |
| | Dolomite | $CaMg(CO_3)_2$ | 16.4 |
| Polymetallic ore of the Maikain deposit | Quartz | $SiO_2$ | 52.3 |
| | Barite | $BaSO_4$ | 39.5 |
| | Muscovite | $KAl_2(AlSi_3O_{10})(OH)_2$ | 8.2 |

Table 2. Results of semiquantitative X-ray phase analysis of sorbents obtained from waste of the Akbakai sulfide deposit synthesized at different synthesis temperatures.

| Sample | Phase name | Chemical composition | Concentration, % |
|---|---|---|---|
| at 400°C | Quartz | $SiO_2$ | 65 |
| | Muscovite | $KAl_2(AlSi_3O_{10})(OH)_2$ | 17 |
| | Lipscombite | $Fe_3(PO_4)_2(OH)_2$ | 6 |
| | Albite | $Na(AlSi_3O_8)$ | 8 |
| | Dolomite | $CaMg(CO_3)_2$ | 4 |
| at 600°C | Quartz | $SiO_2$ | 68 |
| | Lipscombite | $Fe_3(PO_4)_2(OH)_2$ | 6 |
| | Albite | $Na(AlSi_3O_8)$ | 17 |
| | Dolomite | $CaMg(CO_3)_2$ | 10 |
| at 800°C | Quartz | $SiO_2$ | 74 |
| | Muscovite | $(K)(Al_2)(Si_3Al)O_{10}(OH)_2$ | 10 |
| | Lipscombite | $Fe_3(PO_4)_2(OH)_2$ | 8 |
| | Albite | $Na(AlSi_3O_8)$ | 7 |

## RESULT AND DISCUSSION

### X-ray diffractometric analysis

The conducted X-ray phase analysis of the tailings of gold-bearing ore enrichment at the Ashiktas deposit showed that the main phases of the waste are silicon dioxide and the micaceous mineral muscovite - $KAl_2(AlSi_3O_{10})(OH)_2$ (Table 1). The results of the X-ray phase analysis give evidence that the main components of the waste of ore enrichment at the Ashiktas deposit are quartz $SiO_2$ (96.4 %) and muscovite $KAl_2(AlSi_3O_{10})(OH)_2$ (3.6 %). The results show that the main components of Au-containing ores of the Akbakai deposit are quartz (78.1 % - waste from oxidized ore enrichment; 56.5 % - sulfide ore), aluminosilicate (albite - 12.0 and 39.5 %, respectively) and micaceous (3.8 and 5.8 %, respectively) minerals, as well as carbonates (calcite - 6.2 % and dolomite - 16.4 %). The silicon dioxide content in the waste from polymetallic ore enrichment of the Maikain deposit is 52.3 %, micaceous minerals - 8.2 %. A distinctive feature of these wastes is the presence of a significant amount of natural barite - 39.5 %.

During the study, the best sorption properties were shown by the sorbent obtained from waste of the Akbakai sulphide deposit, obtained by acid-thermal synthesis with 35 % orthophosphoric acid. From the study of the composition (Table 2, 4), it follows that in the crystalline phase, the optimal content of phosphates was found after synthesis, which will allow the sorption of the heavy metal cation, which is confirmed by the results indicated in Table 5.

### Material composition analysis by electron probe method

The obtained results of studying the material composition using electric probe analysis indicate that the acid-sorbing components of the waste from enrichment of gold-bearing ore of the Ashiktas deposit are oxides of aluminium (10.14 %), potassium (3.45 %) and iron (3.85 %) (Table 3). The bulk of the waste is represented by silicon dioxide (81.52 %), which confirms the reliability of previously conducted studies using X-ray phase analysis and allows using it as a silicon-containing component in the synthesis of aluminosilicate phosphate materials. An insignificant content of barium oxide (0.14 ± 0.10 %) is not an obstacle to obtaining silicophosphate compounds based on these wastes, since further acid-thermal treatment of these wastes should lead to the formation of insoluble barium phosphates. The material compositions of the waste from the enrichment of oxidized Au-bearing ore of the Akbakai deposit, sulphide Au-containing ore from the same deposit and waste from the enrichment of copper-nickel-zinc ore from the Maikain deposit were also studied.

Table 3. Material composition of the initial waste samples.

| Gold ore of the Ashiktas deposit | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Spectrum | MgO | $Al_2O_3$ | $SiO_2$ | $K_2O$ | FeO | BaO | Total | | | | |
| Average | 0.70 | 10.14 | 81.52 | 3.45 | 3.85 | 0.14 | 100.00 | | | | |
| Oxidized Au-containing ore from the Akbakai deposit | | | | | | | | | | | |
| Spectrum | $Na_2O$ | MgO | $Al_2O_3$ | $SiO_2$ | $P_2O_5$ | $SO_3$ | $K_2O$ | CaO | $TiO_2$ | MnO | FeO | Total |
| Average | 4.72 | 1.93 | 12.92 | 66.99 | 0.75 | 0.76 | 2.65 | 3.35 | 0.58 | 0.18 | 5.12 | 100.00 |
| Sulfide Au-containing ore of the Akbakai deposit | | | | | | | | | | | |
| Spectrum | $Na_2O$ | MgO | $Al_2O_3$ | $SiO_2$ | $SO_3$ | $K_2O$ | CaO | $TiO_2$ | MnO | FeO | Total |
| Average | 2.42 | 2.20 | 14.30 | 68.16 | 0.30 | 4.16 | 3.88 | 0.44 | 0.11 | 3.86 | 100.00 |
| Polymetallic ore of the Maikain deposit | | | | | | | | | | | |
| Spectrum | $Na_2O$ | MgO | $Al_2O_3$ | $SiO_2$ | $SO_3$ | $K_2O$ | FeO | BaO | Total | | |
| Average | 1.81 | 0.58 | 6.56 | 43.36 | 16.74 | 1.03 | 3.68 | 28.43 | 100.00 | | |

Table 4. Material composition of the obtained sorbents, obtained from waste of the Akbakai sulfide deposit, synthesized at different synthesis temperatures.

| at 400°C | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| Spectrum | $Na_2O$ | MgO | $Al_2O_3$ | $SiO_2$ | $P_2O_5$ | $SO_3$ | $K_2O$ | CaO | $TiO_2$ | FeO | Total |
| Average | 2.25 | 1.75 | 13.89 | 64.99 | 5.98 | 0.50 | 4.03 | 2.52 | 0.60 | 3.48 | 100.00 |
| at 600°C | | | | | | | | | | |
| Spectrum | $Na_2O$ | MgO | $Al_2O_3$ | $SiO_2$ | $P_2O_5$ | $K_2O$ | CaO | $TiO_2$ | FeO | Total | |
| Average | 4.38 | 1.73 | 12.86 | 62.68 | 7.91 | 2.68 | 2.94 | 0.66 | 4.18 | 100.00 | |
| at 800°C | | | | | | | | | | |
| Spectrum | $Na_2O$ | MgO | $Al_2O_3$ | $SiO_2$ | $P_2O_5$ | $K_2O$ | CaO | $TiO_2$ | FeO | Total | |
| Average | 2.06 | 2.63 | 13.93 | 61.87 | 6.86 | 4.27 | 3.15 | 0.69 | 4.54 | 100.00 | |

## Dynamic capacity of synthesized materials

From the obtained results (Table 5) it follows that the dynamic exchange capacity concerning nickel cations $Ni^{2+}$ of silicophosphate obtained based on tailings of sulfide ore enrichment of the Akbakai deposit at 600°C is around 1.7 mg-eq $Ni^{2+}$/g, which is the best result among all obtained sorbents based on ore enrichment wastes.

## Zeta potential and SEM surface analysis

The initial sample has a relatively small negative zeta potential (-4.12 mV). During the synthesis at 400°C and 600°C, the zeta potential drops sharply, reaching more negative values (-17.2 and -18.1 mV, respectively). This may indicate an increase in the negative charge on the surface, which potentially improves the cation exchange capacity.

At 800°C, the zeta potential increases to -13.7 mV. This decrease in the negative charge may be due to changes in the sorbent structure, such as phase transitions or a decrease in the number of active groups. Thus, temperature treatment to 600°C increases the negative charge on the sorbent surface, which probably has a positive effect on its sorption properties for cations.

Figs. 3 - 5 show scanning electron microscopic images of silicophosphate sorbents obtained from sulfides containing gold-bearing ore raw materials of the Akbakai deposit, synthesized at different temperatures (400°C, 600°C and 800°C).

The microstructure of the sorbent synthesized at a temperature of 400°C is shown in Fig. 3. The image with a magnification of X100 shows a porous structure with relatively large, unevenly distributed pores. This result indicates the initial stage of porous structure formation, typical of low-temperature synthesis.

Fig. 4 illustrates the microstructure of the sorbent synthesized at a temperature of 600°C. The image with a magnification of X450 shows a more developed porous structure with many uniformly distributed pores. These changes indicate that increasing the synthesis temperature to 600°C promotes activation of the sorbent

Table 5. Sorption of nickel ions $Ni^{2+}$ on porous silicophosphate obtained from tailings of sulfide ore enrichment at Akbakai deposit at 600°C.

| Volume of solution, ml | Sorbed $Ni^{2+}$ cations, mg-eq | $Ni^{2+}$ cations released from the column, mg-eq | Degree of sorption, % |
|---|---|---|---|
| 50 | 1.003 | 0.000 | 100 |
| 50 | 1.003 | 0.000 | 100 |
| 50 | 1.003 | 0.000 | 100 |
| 50 | 1.003 | 0.000 | 100 |
| 50 | 1.003 | 0.000 | 100 |
| 50 | 1.003 | 0.000 | 100 |
| 50 | 1.003 | 0.000 | 100 |
| 50 | 0.094 | 0.011 | 95 |
| 50 | 0.081 | 0.026 | 80 |
| 50 | 0.081 | 0.027 | 80 |
| 50 | 0.052 | 0.058 | 50 |
| 50 | 0.041 | 0.064 | 40 |
| 50 | 0.000 | 1.003 | 0.0 |

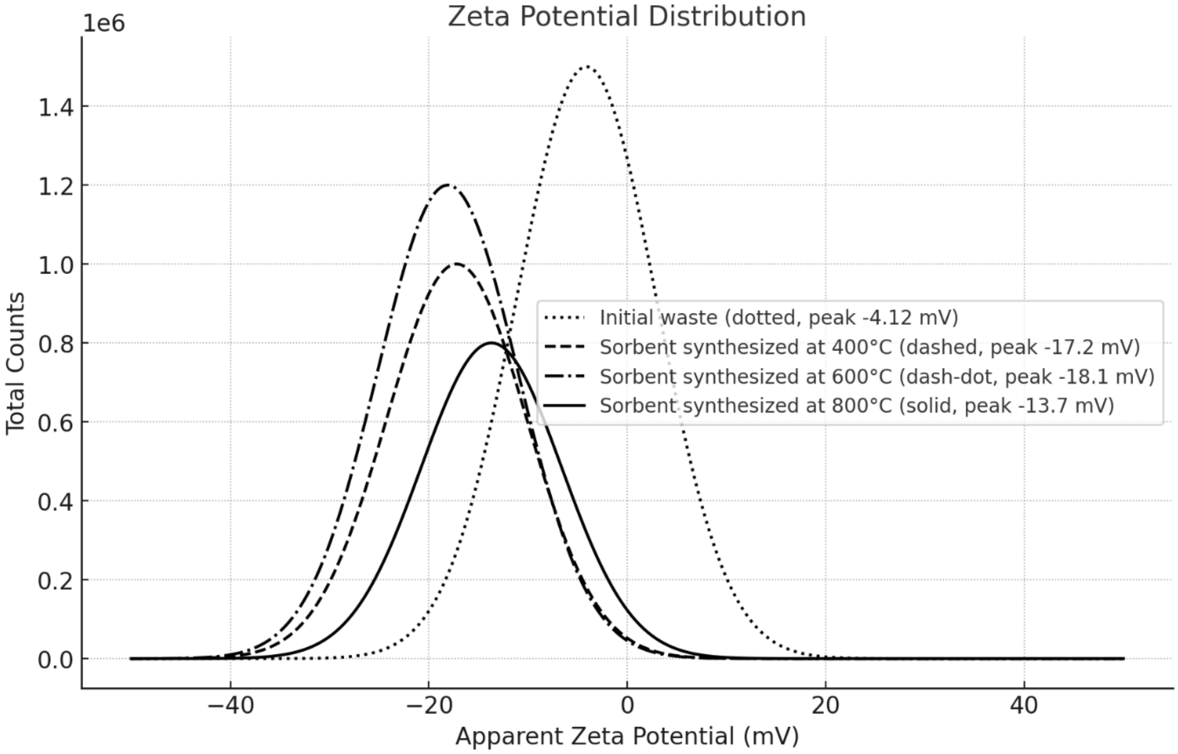

Fig. 2. Zeta potential of the initial substance and sorbents obtained from the tailings of sulfide ore enrichment at the Akbakai deposit.

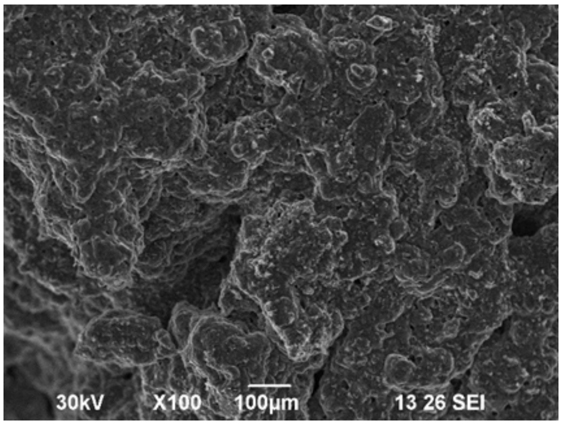

Fig.3. Microscopic images of a silicophosphate sorbent from sulphides containing gold ore raw materials from the Akbakai deposit, synthesized at 400°C.

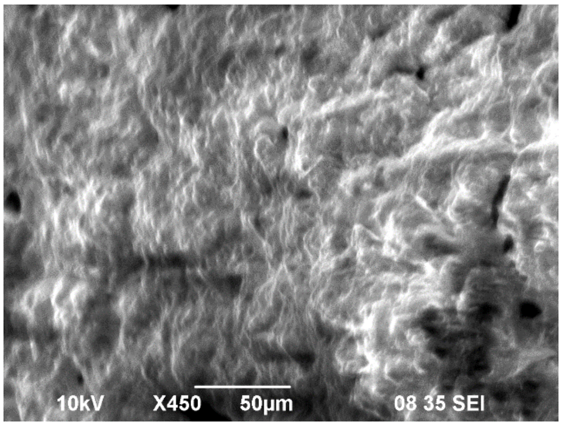

Fig.4. Microscopic images of a silicophosphate sorbent from sulphides containing gold ore raw materials from the Akbakai deposit, synthesized at 600°C.

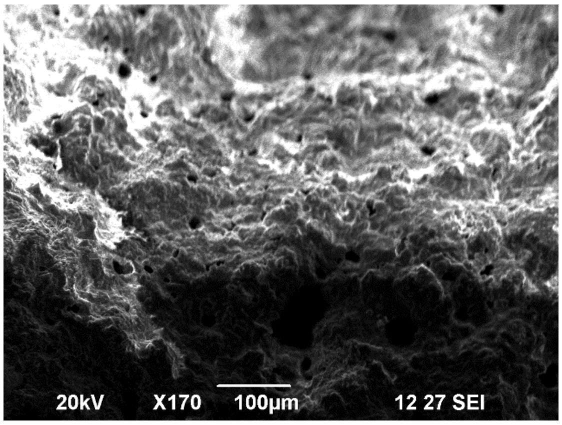

Fig.5. Microscopic images of a silicophosphate sorbent from sulphides containing gold ore raw materials from the Akbakai deposit, synthesized at 800°C.

surface, which probably leads to an increase in its sorption capacity due to the increased specific surface area and developed porosity.

Fig. 5 demonstrates the microstructure of the sorbent synthesized at a temperature of 800°C. The image with a magnification of X170 shows structural changes in the surface, while the porous structure is preserved, but the pores acquire smoother outlines, and the pore density per unit area decreases significantly. This may indicate possible phase changes caused by high temperature, which lead to a decrease in the active surface of the sorbent. This observation correlates with a decrease in the negative zeta potential at a synthesis temperature of 800°C, which can adversely affect its sorption properties. Thus, the results of the microstructural analysis show that the optimal synthesis temperature for obtaining a sorbent with a highly developed porous structure is 600°C.

## CONCLUSIONS

The study demonstrated that ore enrichment waste serves as a promising and sustainable raw material for synthesizing silicophosphate sorbents. The findings highlight the significant potential of heat treatment, particularly at 600°C, in enhancing the sorption efficiency of these materials for nickel ions. This improvement underscores the positive impact of thermal processing on the structural and functional properties of the resulting sorbents.

The applied value of this research lies in its contribution to sustainable waste management and environmental remediation. By repurposing ore enrichment waste, the study addresses the dual challenges of industrial waste disposal and the contamination of water resources by heavy metals. The ability to efficiently remove nickel ions from aqueous solutions paves the way for the development of cost-effective and eco-friendly water purification systems.

This work not only promotes the circular economy by converting industrial by-products into valuable sorbents but also provides a scalable solution for mitigating heavy metal pollution, which is a pressing environmental and public health issue. Future research can further optimize the synthesis process and explore the applicability of these sorbents for a broader range of contaminants, enhancing their utility in diverse water treatment scenarios.

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