EXTRACTION OF SELENIUM FROM THE DUSTS OF BAG DUST FILTERS OF THE KALDO FURNACE

KENZHALIYEV B. K¹, TREBUKHOV S. A², NITSENKO A. V³ & BURABAYEVA N. M⁴

¹*Professor, Vice-Rector for Scientific Work of Satbayev University, General Director of Institute of Metallurgy and Ore Beneficiation JSC, Almaty, Kazakhstan*

²*Associate Professor, Leading Researcher, Satbayev University, Institute of Metallurgy and Ore Beneficiation JSC, Almaty, Kazakhstan*

³*Head of the Laboratory of Vacuum Processes, Satbayev University, Institute of Metallurgy and Ore Beneficiation JSC, Almaty, Kazakhstan*

⁴*Senior Researcher, Satbayev University, Institute of Metallurgy and Ore Beneficiation JSC, Almaty, Kazakhstan*

ABSTRACT

The paper presents the results of the laboratory study to extract selenium from dusts of bag dust filters during the refining production obtained from the Kaldo furnace operation at the Balkhash smelter of Kazakhmys Corporation LLP (the Republic of Kazakhstan). The studies were performed using modern analysis methods (X-ray fluorescence analysis with an Axios PANalytical wavelength-diode combined spectrometer, electron microscopy with a scanning electron microscope equipped with a JEOL GX-8230 microanalyzer, and X-ray analysis using a diffraction spectrometer D8 ADVANCE BRUKER with an X-ray diffractometer) and using vacuum equipment. It was found out that dusts of bag dust filters formed in the Kaldo furnace aspiration system contain up to 30% selenium, mainly in the form of dioxide. The process of transfer of water-soluble selenium compounds to a solution followed by selenium precipitation of with sodium metabisulfite was studied to extract selenium from dusts. The selenium thus obtained was distilled in vacuum. Selenium containing 99.987% of the main component was obtained as a result.

KEYWORDS: *Selenium, Dust, Bag filters, Kaldo furnace, Slime, Vacuum distillation, Extraction, Dissolution & precipitation*

Received: Mar 11, 2020; **Accepted:** Mar 31, 2020; **Published:** May 15, 2020; **Paper Id.:** IJMPERDJUN202050

1. INTRODUCTION

Currently, the main raw material source of selenium is copper electrolyte production sludge; however, the development of technologies to extract selenium from other sources becomes more urgent [1-4], including from gas purification products of copper-nickel and refining production [5-7] as the technologies are introduced to produce copper under the firing-leaching-electroextraction scheme, excluding the electrolysis sludge formation.

One of the main selenium producers in the Republic of Kazakhstan is the Balkhash Smelter of Kazakhmys Corporation LLP [6]. Selenium is obtained at this enterprise as a result of processing sludges received after copper electrolysis. Sludge is processed according to the technology developed by the Swedish Company Boliden in the Kaldo furnace [7]. As a result of the copper electrolyte sludge processing under this scheme, selenium in the Kaldo furnace is oxidized to the tetravalent state, passes into the gas phase and is then trapped in the Venturi system due to its dissolution in the volume of water. The selenious acid obtained is sent to the selenium precipitation with

gaseous sulfur dioxide. The result is technical selenium containing up to 85% of the main component subsequently subjected to vacuum distillation [8].

During the Kaldo furnace operation, all exhaust process gases pass through the gas capture and purification system to prevent environmental pollution [9]. The suction gases from the Kaldo furnace are sent to bag dust filters to collect dust. Dusts from bag dust filter contains up to 42% selenium and precious metals in minor amounts. These dusts are not processed and stored to reduce the circulatory load on the gas duct system of the Kaldo furnace.

The authors of the work studied the physicochemical properties of dusts from bag dust filters of refining production obtained at the Balkhash Smelter of Kazakhmys Corporation LLP and proposed a method to extract selenium from them.

2. RESULTS OBTAINED

The dusts of bag dust filters from the refining production are a finely dispersed friable material of light brown color, partially pelletized, with a moisture content of 1.35%. The bulk density of the material was determined in the laboratory and amounted to 0.92 g/cm³ in its natural state, and 1.21 g/cm³ – with compaction of the material (with a shakedown run). After drying the material at 75°C, the bulk density of the material was 1.07 g / cm³ without a shakedown run, and 1.43 g/cm³ with a shakedown run. The angle of natural repose of the material is 43.5°.

An X-ray fluorescence analysis performed with an Axios PANalytical wave dispersive combined spectrometer determined the element content in the initial dust, wt. %: 29.589 - Se; 1.842 - S; 28.989 - O; 1.065 - Na; 28.029 - Pb; 0.112 - Al; 2.051 - Te; 3.275 - Cl; 3.018 - Ag; 0.073 - Si; 1.977 - Sb; 0.743 - Cu; 0.03 - Fe; 0.587 - As; 1.397 - Bi; 0.02 - Ni; 0.109 - K; 0.056 - Ca; 0.038 - Ti.

Electron microscopic analysis performed with a scanning electron microscope equipped with a JEOL GX-8230 microanalyzer (the results are shown in Figure 1) found out that the material is extremely heterogeneous, the following elements were determined in the initial sample with the content that is different in different areas within the following limits, %: 1.62 ÷ 60.75 Se; 6.16 ÷ 22.68 O; 5.56 ÷ 94.16 Pb; 0.13 ÷ 4.06 Te; 0.42 ÷ 4.09 Cl; 0.28 ÷ 6.01 As; 0.85 ÷ 1.94 Zn; 1.07 ÷ 2.9 Cu; 0.22 ÷ 1.03 Sb; 0.35 ÷ 1.01 Na; 0.11 ÷ 0.79 Mg; 0.81 ÷ 1.47 Si; 0.53 ÷ 1.2 Ag; 0.02 ÷ 0.12 Au. The color intensity in Figure 1 shows the quantitative distribution of elements over the material surface (mapping of the material).

X-ray phase analysis performed with a D8 ADVANCE X-ray diffractometer (BRUKER) showed that selenium in dusts is found mainly in the form of water-soluble impurities - H₂SeO₂ (~91.2%) и SeO₂ (~8.8%). Figure 2 shows the spectrogram obtained from an X-ray diffractometer.

The pressure of saturated selenium vapor sharply increases when the temperature increases, so at 221.3 ° C it is 0.0013 kPa, at 343.7 ° C - 0.133 kPa, at 430.7 ° C - 1.33 kPa, at 546.3 ° C - 13.3 kPa and at 684.8 ° C - 101.08 kPa (a boiling point) [10]. These physical properties of selenium formed the basis for the vacuum distillation refining process [8, 11]. Based on these data, an attempt was made earlier to isolate selenium by direct vacuum distillation of selenium from dusts from bag dust filters of refining production at 450 ° C and a residual pressure in the system of less than 0.67 kPa [12]. In this case, selenium was obtained containing more than 99.5% of the main component, corresponding to the metal intended for export [13]. However, the through extraction of selenium into a commodity metal did not exceed 50% due to the content of volatile selenium compounds (SeO₂) in the starting material.

(a) Snapshot x100 Dust Image; Surface Distribution Intensity: (b) Se; (c) O; (d) Pb; (e) Te; (f) Cl; (g) As; (h) Zn; (i) Cu; (j) Sb; (k) Na; (l) Mg; (m) Si; (n) Ag; (o) Au.

Figure 1: Image of a Dust Surface from a JEOL 8230 Scanning Electron Microscope

Figure 2: Dust Spectrogram Obtained on an x-ray Diffractometer D8 ADVANCE (BRUKER)

A two-stage selenium extraction scheme was tested to obtain a metal containing more than 99.9% of the main component based on the studied physical and chemical properties of the initial dust. The first stage involves the transfer of water-soluble impurities of selenium into a solution followed by precipitation of elemental selenium. The vacuum distillation of the obtained elemental selenium was performed in the second stage.

A sample of the initial dust weighing 1000 g was dissolved in 1000 cm³ of water with dynamic stirring for 30 minutes. Then, the solution was separated from the solid suspension on the filter, the filtrate was sent to precipitate selenium, the precipitate was sent to drying and analyzed for the residual content of selenium and other components.

The loss of dust mass (transferred to solution) was 36.48% (364.8 g) as a result of leaching of water-soluble impurities. The filtrate pH was 1.1. The resulting solution was heated to 70 °C, sodium metabisulfite ($\text{Na}_2\text{S}_2\text{O}_5$) was added there for 3 hours with mechanical stirring to precipitate selenium. As a result, a red α -modification of selenium was isolated from the solution in the amount of 285.3 g where the content of 97.641% of the main component was determined by X-ray fluorescence analysis. It should be noted that the content of the tellurium impurity in the separable selenium that is difficult to be separated by vacuum distillation, amounted to 0.012%.

The residual selenium content was found to be 1.283% in the residue after leaching of water-soluble compounds of selenium by X-ray fluorescence analysis. At the same time, the content of the following elements decreased, including S, Na, Cl, Ca, K, and the concentration of: Pb, Te, Cu, Ag, Sb, etc. increased. Thus, the residue obtained is a product for the extraction of lead and silver, the concentration of which increased after selenium extraction by almost twice and amounted to 56.281% for Pb and 5.188% for Ag.

Based on the study and the analysis results obtained, a material balance of the selenium distribution by products was compiled as a result of water leaching of water-soluble impurities from dusts of bag dust filters, as shown in Table 1.

Table 1: Material Balance of Selenium Distribution by Products as a Result of Leaching of Water-Soluble Impurities from Dusts of Bag Dust Filters

Products Name	Weight, g	Output, %	Se content		Distribution, %
			%	g	
Loaded:					
Source dust	1000	100	29.589	295.89	100
Obtained:					
Dry residue	635.2	63.52	1.283	8.15	2.75
Precipitated Se	285.3	28.53	97.641	278.54	94.14
Total obtained:	920.5	9.05	-	286.69	96.89
Discrepancy:	-79.5	-7.95	-	-9.2	-3.11

36.48% of the initial dust mass, including 97.25% of all selenium contained in dusts, passes into the solution as a result of water leaching of water-soluble compounds contained in dusts of bag dust filters of the refining production. Selenium was precipitated with sodium metabisulfite from the resulting solution; selenium yield was 28.53% of the initial selenium mass. At the same time, 94.14% of all selenium contained in dusts was obtained as a result of precipitation of the latter from solution. The discrepancy for solid products in 7.95% is associated with the transfer into the solution of the compounds of such elements as: Na, S, Cl, Ca and K.

The selenium precipitated from the solution was refined using vacuum distillation technology developed at Institute of Metallurgy and Ore Beneficiation JSC [14]. The distillation was carried out at 420 ° C in an evaporator, 185 ° C in a condenser and a residual pressure in the system of 0.13 kPa. As a result, selenium was obtained with a content of 99.987% of the main component, corresponding to the ST-0 brand [13].

3. CONCLUSIONS

As a result of the study, the physical properties of dusts of the refining production formed at the outlet of the aspiration system of the Kaldo furnace were determined. It is established that dusts are selenium-containing industrial products with up to 30% selenium mainly in the form of water-soluble dioxide.

A two-stage technological scheme to extract selenium from dusts was tested, which includes the conversion of water-soluble selenium compounds into a solution at the first stage, and the refinement of precipitated selenium by vacuum distillation at the second stage. The through recovery of selenium at two stages amounted to 93.86%. Selenium was obtained in the form of ingots containing 99.987% of the main component.

The work was performed as a part of the BR05236406 Scientific and Technical Program implementation funded by the Committee of Science of the Ministry of Education and Science of the Republic of Kazakhstan.

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