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Elemental Assessment of Lead–Bismuth Sludge from Copper Smelting with Emphasis on Tellurium Recovery

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ABSTRACT
Industrial by-products generated during copper smelting are increasingly regarded as promising secondary sources of strategically important and critical elements. Among such technogenic materials, lead–bismuth sludge formed during wet gas cleaning of sulfur-bearing gases represents a potentially valuable reservoir of selenium and tellurium. The present study provides a comprehensive elemental assessment of lead–bismuth sludge obtained from the Almalyk Mining and Metallurgical Complex (Uzbekistan). Prior to analysis, representative sludge samples were subjected to acid digestion using freshly prepared aqua regia, followed by elemental determination using inductively coupled plasma optical emission spectroscopy (ICP-OES). Particular attention was devoted to the distribution behavior of selenium and tellurium due to their technological importance as critical raw materials. The analytical results demonstrated that the investigated sludge is characterized by a pronounced polymetallic composition dominated by lead (20.0%), together with significant concentrations of iron (6.76%), copper (5.34%), and zinc (4.40%). Tellurium was detected at a concentration of 0.33%, indicating its selective accumulation in the lead–bismuth residue, whereas selenium was not detected under the selected analytical conditions. Based on elemental composition data and thermodynamic considerations, the investigated sludge may be considered a promising secondary source for tellurium recovery. The obtained results contribute to understanding the physicochemical behavior of chalcogen elements during copper smelting and wet gas-cleaning processes and may serve as a basis for the development of integrated recycling approaches for technogenic metallurgical waste.

Keywords: copper smelting, secondary tellurium source, critical raw materials, technogenic waste, wet gas cleaning, elemental composition, ICP-OES.

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Introduction

In recent years, waste generated during copper smelting has attracted increasing attention not only as an environmentally hazardous material, but also as a potential secondary source of economically and strategically important elements [[1], [2], [3], [4], [5]]. Among such technogenic products, lead–bismuth sludge formed during wet gas cleaning represents a chemically complex polymetallic system whose composition depends on the mineralogical characteristics of raw materials and the physicochemical conditions of pyrometallurgical processing [[6], [7], [8]].

Particular interest has recently been directed toward selenium and tellurium because these elements are classified as critical raw materials due to the limited availability of primary deposits and their growing demand in photovoltaic technologies, thermoelectric materials, semiconductor manufacturing, and advanced energy systems [[9], [10], [11], [12]]. Copper smelting processes are considered one of the major industrial sources of secondary tellurium recovery because chalcogen elements tend to redistribute into dusts, sludges, and gas-cleaning products during high-temperature processing [[13], [14], [15]].

Despite the growing industrial importance of tellurium, reliable information concerning its distribution in lead–bismuth sludge generated during wet gas cleaning remains limited. Existing studies indicate that selenium and tellurium exhibit significantly different physicochemical behavior under oxidizing smelting conditions, which directly influences their volatility, phase stability, and accumulation in technogenic products [[16], [17], [18]].

Selenium is characterized by the formation of volatile oxide compounds that can migrate with process gases or transfer into liquid phases during gas purification. In contrast, tellurium tends to form more stable and less volatile compounds, favoring its accumulation in condensed solid residues [19]. Consequently, the enrichment degree of selenium and tellurium in individual waste streams may differ substantially from values predicted solely from ore composition [[20], [21]].

The growing demand for tellurium and other critical elements significantly increases the importance of identifying alternative secondary resources suitable for their recovery [[22], [23]]. In this context, technogenic products generated during copper smelting may represent an important raw material base for future extraction of valuable elements.

Therefore, the present study is aimed at evaluating the elemental composition of lead–bismuth sludge obtained from the Almalyk Mining and Metallurgical Complex (Uzbekistan), with particular emphasis on the distribution behavior and selective accumulation of tellurium in wet gas-cleaning residues.

Experimental part

Lead–bismuth sludge samples investigated in this study were obtained from the Almalyk Mining and Metallurgical Complex (Uzbekistan). The material represents a solid technogenic product formed after wet gas cleaning of sulfur-bearing gases generated during copper smelting operations.

Prior to analysis, the samples were dried under laboratory conditions, homogenized, and mechanically crushed to ensure representative sampling. For preliminary characterization, two independent subsamples (S_0) with a mass of approximately 1000 g each were prepared. One subsample was used for primary analytical measurements, whereas the second was employed to evaluate analytical reproducibility.

For elemental analysis, representative portions of approximately 0.5–1.0 g were subjected to acid digestion using freshly prepared aqua regia ($HCl:HNO_3$ = 3:1). The digestion process was carried out in glass vessels at approximately 90 °C for 1–2 h under controlled laboratory conditions until complete dissolution of the solid matrix was achieved. After cooling, the obtained solutions were diluted with distilled water to a final volume of 50 mL and filtered when necessary to remove residual insoluble particles (Fig.1).

Figure 1 - Dissolved sample

The elemental composition of the prepared solutions was determined using inductively coupled plasma optical emission spectroscopy (ICP-OES) performed on a Genesis Spectrum ICP-OES instrument. Calibration of the instrument was carried out using standard reference solutions under standard operating conditions. The analysis included determination of major and trace elements, with particular emphasis on selenium and tellurium as critical elements of technological interest.

All measurements were performed in duplicate, and analytical reproducibility was evaluated based on repeated measurements of independently prepared samples. The obtained analytical data demonstrated satisfactory repeatability for polymetallic technogenic materials. Element concentrations are reported as mass percentages (%).

Special attention was devoted to the determination of selenium because its concentration in the investigated sludge was close to or below the instrumental detection limit under the selected analytical conditions. The applied analytical methodology allowed reliable determination of tellurium and major accompanying elements in the investigated technogenic residue.

Results and Discussion

The elemental composition of the investigated lead–bismuth sludge was determined using ICP-OES analysis (Fig.2). The obtained results confirmed the pronounced polymetallic nature of the studied technogenic material. The analytical data are summarized in Table 1. Lead was identified as the dominant component with a concentration of approximately 20.0%, indicating its significant accumulation in the investigated residue. Considerable concentrations of iron (6.76%), copper (5.34%), and zinc (4.40%) were also detected, reflecting the transfer of volatile metal compounds and fine dispersed particles during copper smelting and subsequent wet gas-cleaning processes.

Bismuth and antimony were detected at concentrations of 0.20% and 0.34%, respectively, while nickel was present only in trace quantities (0.02%). The obtained results additionally confirmed the presence of noble metals in the investigated sludge. Palladium was detected at 0.003%, whereas platinum was identified in minor quantities within the analyzed material.

Particular attention was devoted to the distribution behavior of selenium and tellurium because these elements are considered technologically important critical raw materials. Tellurium was detected at a concentration of 0.33%, indicating its selective accumulation in the lead–bismuth sludge. In contrast, selenium was not detected under the selected analytical conditions, suggesting either its absence in the solid residue or concentrations below the instrumental detection limit.

Repeated measurements of independently prepared subsamples demonstrated satisfactory analytical reproducibility for the investigated polymetallic material.

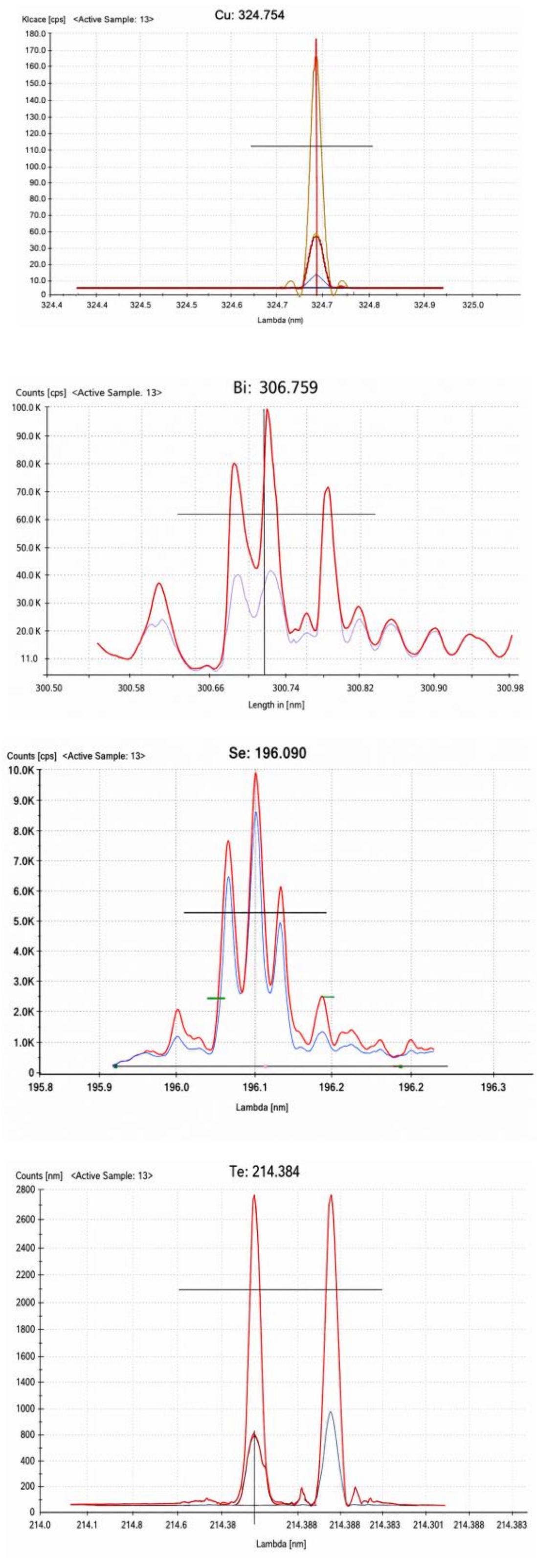

Figure 2 - Representative emission spectra recorded using a Genesis ICP-OES instrument

The obtained results demonstrate substantial differences in the physicochemical behavior of selenium and tellurium during copper smelting and wet gas-cleaning processes. Despite their chemical similarity, these elements exhibit different volatility and redistribution characteristics under oxidizing high-temperature conditions.

According to previously published studies, selenium is generally characterized by the formation of more volatile compounds capable of transferring into gaseous or liquid process streams during gas purification. In contrast, tellurium demonstrates a greater tendency toward accumulation in condensed technogenic products generated during copper smelting.

The observed enrichment of tellurium in the investigated sludge indicates that wet gas-cleaning residues may serve as important secondary resources for tellurium recovery from metallurgical waste. At the same time, the absence of detectable selenium in the solid residue suggests its redistribution into alternative technological streams, including associated dusts and liquid gas-cleaning products.

The present investigation was primarily focused on elemental characterization of lead–bismuth sludge using ICP-OES analysis. Therefore, additional investigations involving X-ray diffraction (XRD), scanning electron microscopy coupled with energy-dispersive spectroscopy (SEM-EDS), and mineralogical analysis are required for direct identification of phase associations and distribution forms of tellurium and accompanying elements in the investigated technogenic material.

Behavior and Distribution of Elements in Lead–Bismuth Sludge

The established elemental composition allows evaluation of the possible distribution behavior of elements in the investigated sludge, taking into account their physicochemical properties and previously published data concerning similar technogenic materials [[24], [25], [26]]. The obtained analytical results indicate that lead-containing compounds represent a significant component of the investigated residue formed during wet gas cleaning of sulfur-bearing gases generated in copper smelting processes.

The presence of bismuth together with elevated lead concentrations indicates accumulation of these elements in the solid fraction of the sludge. Earlier studies have demonstrated that lead and bismuth exhibit similar redistribution tendencies during pyrometallurgical processing and subsequent gas-cleaning operations. The measured tellurium concentration (0.33%) indicates selective retention of this element in the investigated technogenic material. Previous investigations have shown that tellurium demonstrates lower volatility under oxidizing smelting conditions compared with selenium, promoting its accumulation in condensed metallurgical products. Iron, copper, and zinc identified in the investigated sludge are associated with transfer of volatile metal compounds and fine dispersed particles during smelting and gas purification processes. Their presence confirms the polymetallic nature of the investigated residue.

The obtained results demonstrate substantial differences in the distribution behavior of selenium and tellurium during copper smelting and wet gas-cleaning processes. Despite their chemical similarity, these elements exhibit different redistribution characteristics under high-temperature oxidizing conditions. Selenium is generally characterized by formation of more volatile compounds capable of transferring into gaseous or liquid process streams during gas purification. In contrast, tellurium demonstrates a greater tendency toward accumulation in condensed technogenic products due to formation of relatively stable low-volatility compounds. The observed enrichment of tellurium in the investigated sludge indicates that wet gas-cleaning residues may represent promising secondary resources for tellurium recovery from metallurgical waste.

The present investigation was primarily focused on elemental characterization using ICP-OES analysis. Therefore, additional investigations involving X-ray diffraction (XRD), scanning electron microscopy coupled with energy-dispersive spectroscopy (SEM-EDS), and mineralogical analysis are required for direct confirmation of phase composition and elemental associations in the investigated technogenic material.

Implications for Secondary Resource Potential

The results of the analysis indicate that lead-bismuth sludge formed during wet gas cleaning has selective absorption capacity. It cannot be considered a universal matrix for the simultaneous concentration of selenium and tellurium. Nevertheless, the measured concentration of tellurium indicates that this man-made material is a promising secondary resource for tellurium extraction. The absence of selenium in the solid residue confirms its predominant removal in associated technological cycles. In this regard, additional study of alternative flows in the smelting and gas cleaning system is required to identify the main accumulation zones of this element

Table 1 - Elemental composition of lead–bismuth technogenic sludge formed during wet gas cleaning

Element	Content, %
Pb	20.00
Cu	5.34
Fe	6.76
Zn	4.40
Bi	0.20
Sb	0.34
Ni	0.02
Te	0.33
Se	Not detected
Pd	0.003
Pt	1.11

Conclusions

This paper presents a detailed polymetallic characterization of anthropogenic lead-bismuth slag formed during copper smelting. The experimental results confirm that the material under study is a complex multicomponent system. Lead acts as the main carrier matrix, associated with significant concentrations of copper, iron, zinc, as well as a number of impurities and trace elements. During the study, a distinct selectivity in the distribution of target components was observed. The tellurium content was 0.33%, which confirms its preferential accumulation in the lead-bismuth residue, while the presence of selenium was not detected using the analytical methods employed. These results reflect fundamental differences in the physicochemical behavior of selenium and tellurium during high-temperature smelting and subsequent wet gas cleaning.

Based on the data obtained on the elements and previously published studies, it can be concluded that tellurium in the sludge is mainly associated with the oxide phases of lead and bismuth.

Due to its high volatility and increased migration ability, selenium is redistributed into alternative technological cycles. Thus, lead-bismuth sludge exhibits selectivity and is not a universal medium for the simultaneous concentration of both elements.

From a practical point of view, the man-made material under study should be considered as a promising secondary source of tellurium. At the same time, for the effective utilization of selenium, it is necessary to identify and study alternative areas of its accumulation in the smelting and gas purification system. The methodology used and the data obtained allow for a more accurate assessment of the value of secondary raw materials. This opens up opportunities for the creation of rational schemes for the utilization and processing of waste generated during copper smelting.

Conflicts of interest. On behalf of all authors, the corresponding author states that there is no conflict of interest.

CRediT author statement: **M. Tulaganova**: Conceptualization, Methodology, Software; **J. Ismailov**: Data curation, Writing draft preparation; **Z. Matkarimov**: Visualization, Investigation; **G. Alamova**: Supervision; Software, Validation; **S. Matkarimov**: Reviewing and Editing.

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Теллурды мыс балқыту арқылы алуда қорғасын-висмут шламының қарапайым бағалауы

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ТҮЙІНДЕМЕ
Мыс балқыту кезінде түзілетін өнеркәсіптік жанама өнімдер стратегиялық маңызды және критикалық элементтердің перспективалы екінші реттік көздері ретінде жиірек қарастырылуда. Осындай техногендік материалдардың бірі — құрамында күкірті бар газдарды ылғалды тазарту кезінде түзілетін қорғасын-висмут шламы, ол селен мен теллурдың әлеуетті құнды көзі болып табылады. Бұл зерттеу Алмалық тау-кен металлургия

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комбинатында (Өзбекстан) алынған қорғасын-висмут шламының элементтік құрамын кешенді бағалау ұсынылған. Талдау алдында шламның өкілдік үлгілері жаңадан дайындалған патша сұйығымен қышқылдық ыдыратуға ұшыратылып, содан кейін элементтік құрамы индуктивті байланысқан плазмалы оптикалық-эмиссиялық спектроскопия (ICP-OES) әдісімен анықталды. Селен мен теллур маңызды шикізат ретінде технологиялық маңыздылығына байланысты олардың таралу заңдылықтарына ерекше назар аударылды. Талдау нәтижелері зерттелген шламның қорғасынның басымдығымен (20,0%) айқын полиметалдық құраммен, сондай-ақ темір (6,76%), мыс (5,34%) және мырыштың (4,40%) %) айтарлықтай концентрациясымен сипатталатынын көрсетті. Теллур мөлшері 0,33% құрады, бұл оның қорғасын-висмут қалдығында селективті жинақталуын көрсетеді, ал таңдалған аналитикалық жағдайларда селен анықталмады. Элементтік құрам деректері мен термодинамикалық түсініктер негізінде зерттелген шлам теллурды алудың перспективалы екінші реттік шикізат көзі ретінде қарастырылуы мүмкін. Алынған нәтижелер мыс балқыту және ылғалды газ тазарту процестеріндегі халькоген элементтерінің физика-химиялық жағдайын түсінуге ықпал етеді және техногендік металлургиялық қалдықтарды кешенді қайта өңдеу тәсілдерін әзірлеуге негіз бола алады.

Түйін сөздер: мыс балқыту, екіншілік теллур көзі, критикалық шикізат, техногендік қалдықтар, ылғалды газды тазарту, элементтік құрам, ICP-OES.

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Элементарная оценка свинцово-висмутового шлама при выплавке меди с акцентом на извлечение теллура

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АННОТАЦИЯ

Промышленные побочные продукты, образующиеся при выплавке меди, все чаще рассматриваются как перспективные вторичные источники стратегически важных и критических элементов. Среди таких техногенных материалов свинцово-висмутовый шлам, образующийся при мокрой очистке серосодержащих газов, представляет собой потенциально ценный источник селена и теллура. В настоящем исследовании представлена комплексная оценка элементного состава свинцово-висмутового шлама, полученного на Алмалыкском горно-металлургическом комбинате (Узбекистан). Перед проведением анализа представительные образцы шлама подвергались кислотному разложению свежеприготовленной царской водкой с последующим определением элементного состава методом оптико-эмиссионной спектроскопии с индуктивно связанной плазмой (ICP-OES). Особое внимание было уделено характеру распределения селена и теллура в связи с их технологической значимостью как критически важных видов сырья. Результаты анализа показали, что исследуемый шлам характеризуется выраженным полиметаллическим составом с преобладанием свинца (20,0%), а также значительными концентрациями железа (6,76%), меди (5,34%) и цинка (4,40%). Содержание теллура составило 0,33%, что свидетельствует о его селективном накоплении в свинцово-висмутовом остатке, тогда как селен в выбранных аналитических условиях обнаружен не был. На основании данных

	элементного состава и термодинамических представлений исследуемый шлам может рассматриваться как перспективный вторичный источник для извлечения теллура. Полученные результаты способствуют пониманию физико-химического поведения халькогенных элементов в процессах выплавки меди и мокрой газоочистки, а также могут служить основой для разработки комплексных подходов к переработке техногенных металлургических отходов.
	Ключевые слова: плавка меди, вторичный источник теллура, критическое сырье, техногенные отходы, мокрая очистка газов, элементный состав, ICP-OES.
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