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MICROSTRUCTURAL AND ELEMENTAL CHARACTERIZATION OF ZINC PRODUCTION CAKE BY SCANNING ELECTRON MICROSCOPY AND ENERGY-DISPERSIVE X-RAY SPECTROSCOPY

Khabibullaeva Mukhlisa Khayrulla qizi
Student of the Department of Metallurgy, Almalyk State Technical Institute

Abstract. Zinc production cakes generated during hydrometallurgical processing contain valuable metals and represent a potential secondary resource. In this study, the microstructural and elemental characteristics of zinc production cake samples were investigated using scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS), and elemental mapping. SEM analysis revealed a heterogeneous and porous morphology consisting of agglomerated fine particles. EDS results showed that the sample was mainly composed of oxygen (27.2 wt.%), zinc (25.3 wt.%), iron (16.8 wt.%), and sulfur (14.6 wt.%), with smaller amounts of copper, sodium, calcium, silicon, bromine, and magnesium. Elemental mapping confirmed the widespread distribution of Zn, O, S, and Fe, indicating the presence of oxide, hydroxide, and sulfate phases. The results demonstrate that zinc production cake possesses a complex microstructure and contains significant amounts of recoverable metals, highlighting its potential for secondary resource recovery and sustainable waste management.

Keywords: zinc production cake; SEM; EDS; elemental mapping; hydrometallurgical processing; zinc residues; metal recovery; microstructure.

Introduction. Zinc is one of the most important non-ferrous metals widely used in galvanizing, alloy production, chemical industries, and advanced technologies. The growing global demand for zinc has intensified efforts to improve extraction efficiency, reduce metal losses, and promote sustainable utilization of industrial by-products [1]. In hydrometallurgical zinc production processes, significant amounts of solid residues, commonly referred to as zinc cakes, are generated during leaching, purification, and filtration stages. These residues often contain valuable metals and complex mineral phases that can influence both metal recovery and environmental performance [2].

The characterization of zinc production cakes has become increasingly important because their physical and chemical properties directly affect process optimization, waste management, and resource recovery strategies [3]. Among the available characterization techniques, scanning electron microscopy (SEM) is a powerful analytical tool that provides detailed information about particle morphology, surface characteristics, phase distribution, and microstructural features [4]. SEM analysis enables researchers to better understand the formation mechanisms of zinc processing residues and identify potential pathways for recovering valuable components [5].

Despite extensive studies on zinc extraction technologies, the microstructural characterization of zinc production cakes remains insufficiently explored. Many previous investigations have focused primarily on chemical composition and bulk mineralogical analysis, while limited attention has been paid to the relationship between particle morphology, phase associations, and metal distribution at the microscopic level [6]. This lack of detailed microstructural information hinders the development of efficient processing technologies for residue valorization and secondary metal recovery [7].

Recent studies have demonstrated that industrial zinc residues may contain appreciable amounts of zinc, lead, iron, copper, and other economically valuable elements [8]. Furthermore, SEM-based investigations have revealed that these metals are frequently associated with complex heterogeneous structures, making their recovery challenging. Such findings highlight the necessity of detailed microscopic studies to improve the understanding of residue composition and behavior [9].

In Uzbekistan, the development of the non-ferrous metallurgy sector and the increasing emphasis on resource efficiency have created a need for comprehensive investigations of metallurgical wastes and by-products [10]. Zinc-containing residues generated by domestic metallurgical enterprises represent both an environmental concern and a potential secondary resource. Therefore, advanced characterization techniques are required to evaluate their structural features and recovery potential [11].

The objective of this study is to investigate the microstructural characteristics of zinc production cake samples using scanning electron microscopy. Particular attention is given to particle morphology, surface texture, structural heterogeneity, and the distribution of mineral phases observed in the samples [12].

The aim of this paper is to provide a detailed SEM-based characterization of zinc production cake samples and to generate fundamental information that can support the development of more efficient metal recovery technologies and sustainable residue management practices in zinc metallurgy.

2. Materials and Methods.

Materials

The material investigated in this study was a zinc production cake obtained from a hydrometallurgical zinc processing plant. The sample was collected after the filtration stage of the zinc production process, where solid residues are separated from the process solution. The collected cake was stored in sealed polyethylene containers to prevent contamination and moisture exchange with the surrounding environment.

Prior to analysis, the sample was air-dried at room temperature and homogenized to ensure representative characterization. Large agglomerates were gently crushed using a porcelain mortar and pestle. The prepared material was then sieved to obtain a uniform particle size fraction suitable for microscopic examination.

Sample Preparation

For scanning electron microscopy (SEM) analysis, a representative portion of the dried sample was mounted onto standard aluminum specimen stubs using conductive carbon adhesive tape. To improve electrical conductivity and minimize charging effects during electron beam exposure, the samples were coated with a thin conductive layer of gold using a sputter-coating system. The coating thickness was maintained at approximately 10–20 nm to ensure high-quality image acquisition without obscuring surface features.

Scanning Electron Microscopy Analysis

The microstructural characterization of the zinc production cake samples was performed using a scanning electron microscope (SEM). The analysis was conducted under high-vacuum conditions at an accelerating voltage ranging from 10 to 20 kV, depending on the imaging requirements and sample characteristics.

Micrographs were acquired at different magnification levels to evaluate particle morphology, surface texture, porosity, and the distribution of microstructural features. Secondary electron (SE) imaging mode was employed to obtain detailed topographical information, while backscattered electron (BSE) imaging was used to distinguish phases with different average atomic numbers and identify compositional contrasts within the sample.

Elemental Analysis

To determine the elemental composition of selected particles and phases, an energy-dispersive X-ray spectroscopy (EDS) detector attached to the SEM system was utilized. Point analyses and elemental mapping were carried out on representative areas of the sample. The obtained spectra were processed using the instrument software, and the relative concentrations of major elements were determined in weight percentage (wt.%).

The elemental distribution maps were used to identify the association of zinc with other elements such as iron, sulfur, silicon, calcium, and lead. These data provided valuable information regarding the heterogeneity of the zinc cake and the occurrence of different mineral phases.

Data Processing and Interpretation

The acquired SEM images and EDS spectra were analyzed using the microscope's image-processing software. Morphological characteristics, including particle shape, particle size variation, agglomeration behavior, and surface roughness, were qualitatively evaluated. The elemental composition data obtained from EDS analyses were correlated with the observed microstructural features to identify potential mineralogical associations.

The results obtained from SEM and EDS analyses were subsequently interpreted to assess the structural characteristics of the zinc production cake and to evaluate its potential for further processing and metal recovery.

3. Results and Discussion

3.1. SEM Analysis of Zinc Production Cake

The surface morphology of the zinc production cake was investigated using scanning electron microscopy (SEM). The SEM micrograph (Figure 1) reveals a highly heterogeneous and irregular surface structure composed of agglomerated fine particles. The sample exhibits a rough texture with numerous micro-pores and cavities distributed throughout the observed area. Such morphology is characteristic of residues generated during hydrometallurgical zinc processing, where precipitation, filtration, and drying stages promote the formation of compact particle aggregates.

The micrograph also shows significant variations in surface density and particle packing. Some regions appear relatively dense and compact, whereas others display a porous and loosely bound structure. Three representative areas, designated as Spectrum 7, Spectrum 8, and Spectrum 9, were selected for local elemental analysis using energy-dispersive X-ray spectroscopy (EDS). The observed heterogeneity suggests the presence of multiple mineral phases and reaction products formed during zinc extraction and purification processes.

3.2. EDS Analysis

The EDS spectrum obtained from the area marked as Spectrum 9 is presented in Figure 2.

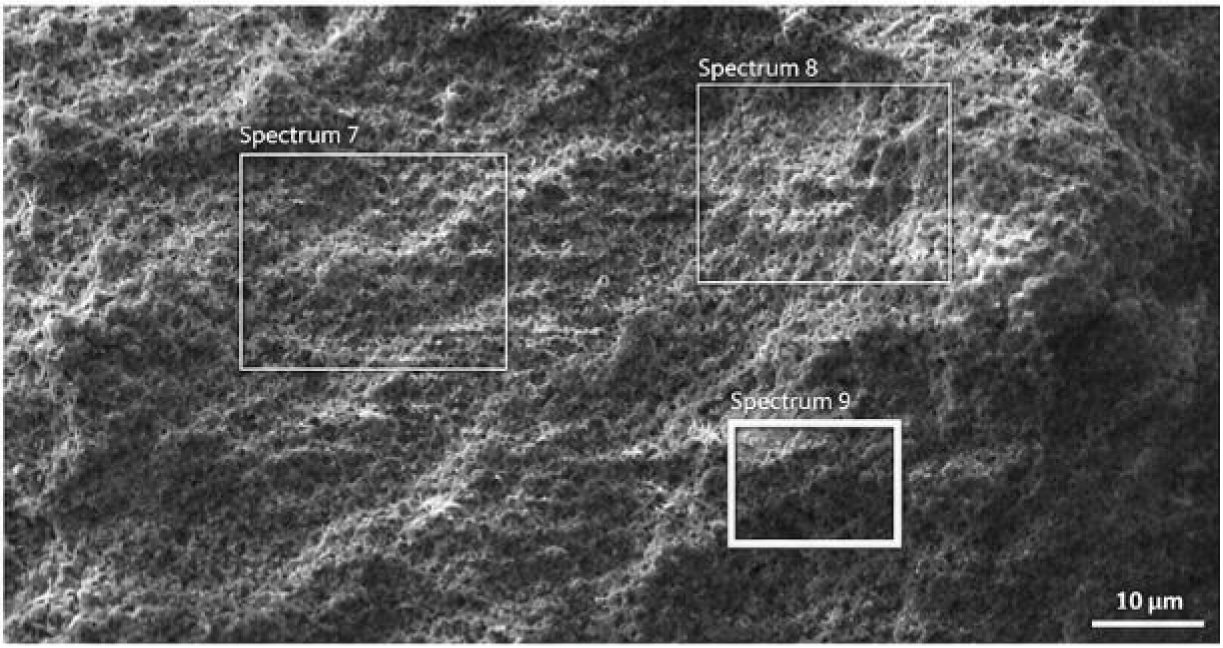

Fig. 1. Scanning electron microscopy (SEM) image of the zinc production cake illustrating the agglomerated particle structure, porous surface morphology, and the areas selected for elemental analysis by EDS (Spectrum 7–9).

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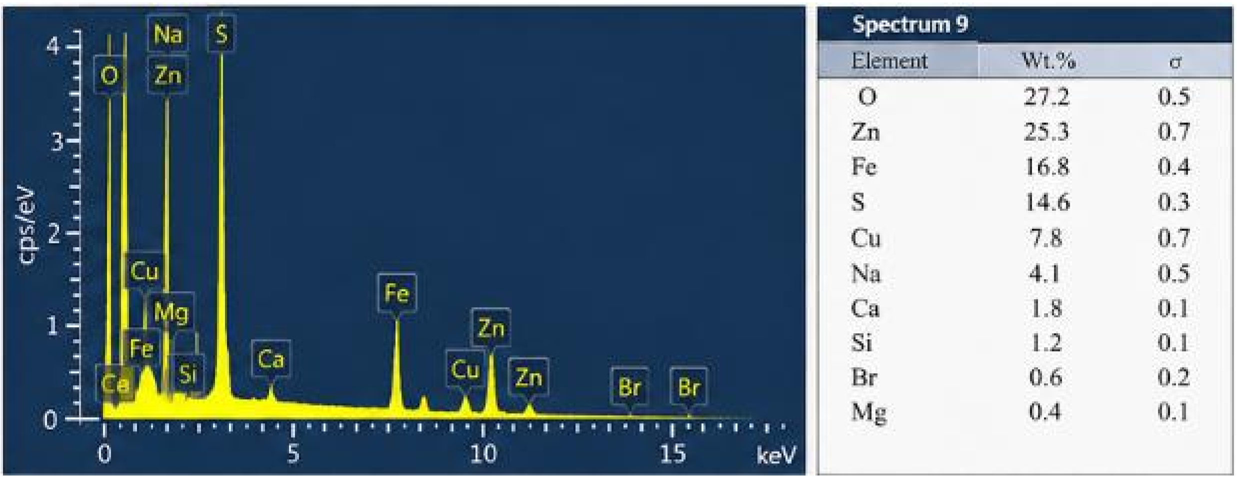

Fig. 2. Energy-dispersive X-ray spectroscopy (EDS) spectrum of the zinc production cake obtained from Spectrum 9, showing the distribution and relative abundance of major elements present in the analyzed region.

The EDS results indicate that oxygen (27.2 wt.%) and zinc (25.3 wt.%) are the dominant constituents of the analyzed region. The high oxygen content suggests that a significant portion of zinc occurs in oxidized forms, including oxides, hydroxides, or basic sulfate compounds. Iron and sulfur are also present in considerable concentrations, accounting for 16.8 wt.% and 14.6 wt.%, respectively. Their coexistence may indicate the presence of iron oxides, iron hydroxides, or sulfate-bearing phases commonly formed during hydrometallurgical processing.

Copper was detected at a relatively high concentration of 7.8 wt.%, indicating incomplete separation of valuable metals during the zinc production process. Sodium, calcium, silicon, magnesium, and bromine were identified in minor amounts, likely originating from process reagents, gangue minerals, or impurities associated with the feed material.

3.3. SEM–EDS Elemental Mapping

The spatial distribution of major and minor elements in the zinc production cake was further investigated using SEM–EDS elemental mapping. Figure 3 presents the distribution maps for O, S, Zn, Fe, Ca, Na, Si, Cu, Br, and Mg across the analyzed surface.

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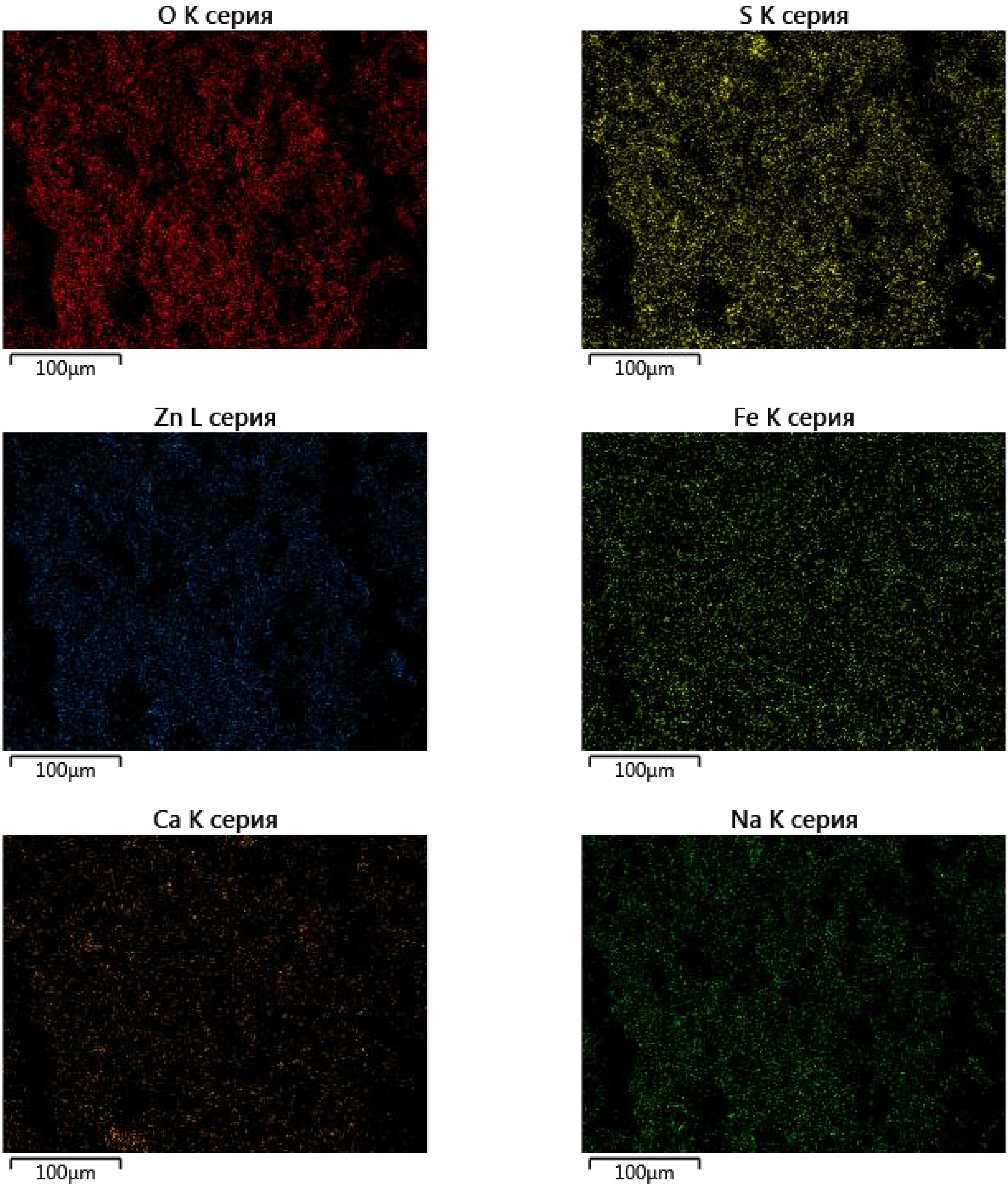

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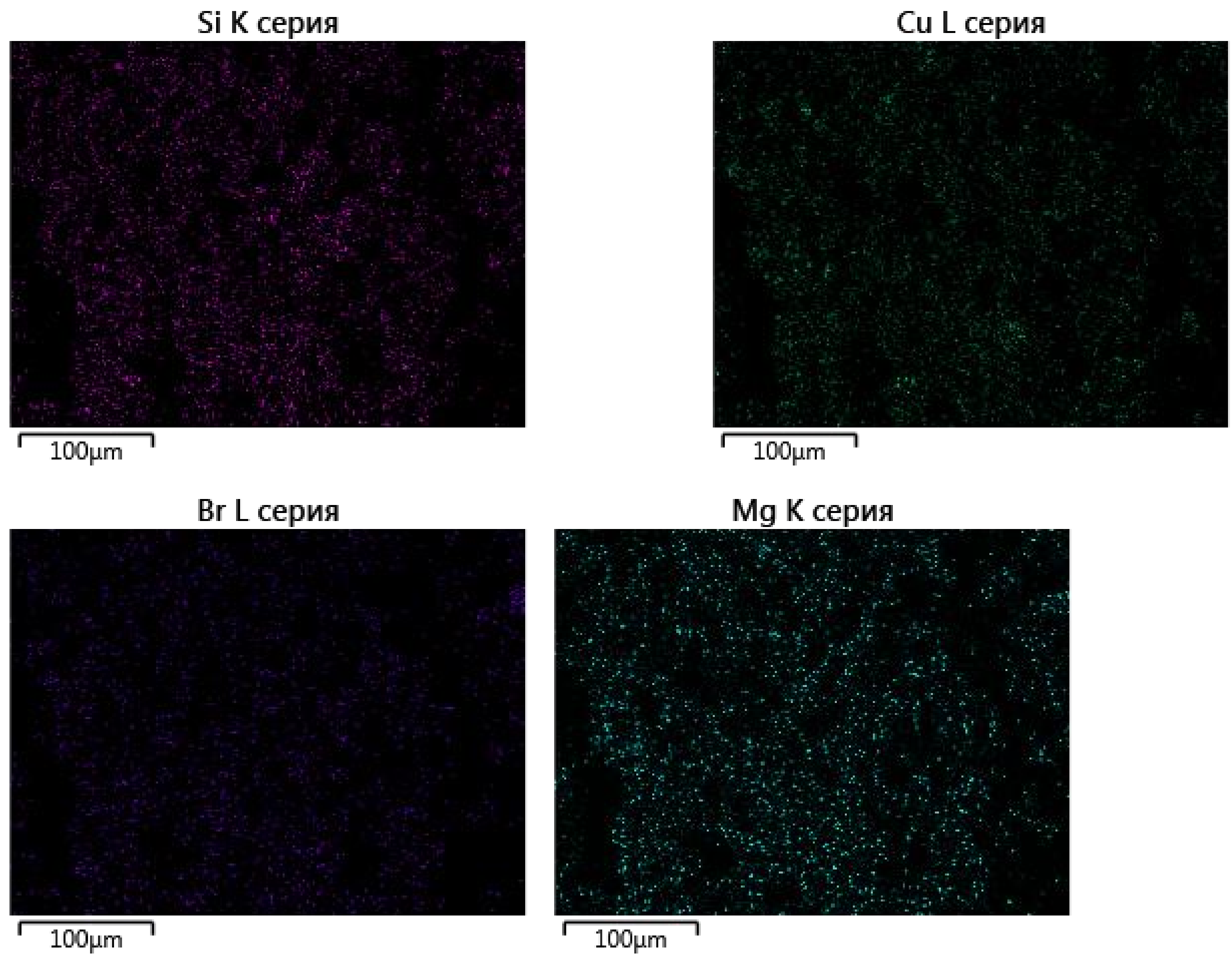

Fig. 3. SEM–EDS elemental mapping of the zinc production cake showing the spatial distribution of O, S, Zn, Fe, Ca, Na, Si, Cu, Br, and Mg across the analyzed surface.

The elemental maps show that oxygen and sulfur are widely distributed throughout the sample, indicating that oxide and sulfate phases are the dominant constituents of the cake. Zinc is also broadly dispersed over the analyzed area, confirming that Zn-bearing compounds are present across the porous matrix rather than being concentrated in isolated particles. The overlap between Zn, O, and S distributions suggests the occurrence of zinc oxides, hydroxides, and sulfate-related phases formed during hydrometallurgical processing.

Iron exhibits a relatively uniform but locally enriched distribution, implying the presence of finely dispersed iron-containing phases such as iron oxides or hydroxysulfates. The coexistence of Fe with oxygen-rich regions supports this interpretation. Copper appears in scattered microdomains rather than in a continuous phase, indicating that copper-bearing particles are entrained within the zinc residue and were not completely separated during processing.

Sodium and calcium are distributed heterogeneously, with localized concentrations that may correspond to residual process reagents, precipitates, or gangue-derived minerals. Silicon occurs in low abundance and is unevenly distributed, suggesting the presence of silicate impurities originating from the feed material. Bromine and magnesium are detected only in trace amounts and appear sparsely across the surface.

The elemental mapping results corroborate the EDS point analysis by demonstrating that the zinc production cake is chemically heterogeneous at the microscale. The simultaneous presence and partial co-localization of Zn, Fe, S, and O indicate a complex assemblage of oxide, hydroxide, and sulfate phases rather than a single homogeneous compound. Such heterogeneity is typical for residues generated during zinc hydrometallurgy, where multiple precipitation and filtration reactions occur.

From a process perspective, the broad distribution of zinc suggests that a considerable portion of the metal remains locked within fine porous aggregates. This morphology may enhance leaching kinetics due to the increased surface area, but it can also complicate selective separation because zinc is associated with iron- and sulfur-rich phases. The presence of dispersed copper-bearing regions further indicates the potential economic value of the residue for secondary metal recovery.

SEM–EDS mapping confirms that the studied zinc production cake consists of a porous heterogeneous matrix enriched in Zn, O, S, and Fe, with minor contributions from Cu, Na, Ca, Si, Br, and Mg. These findings provide important microstructural and compositional insights for the development of improved recovery and residue-treatment technologies.

Conclusion. The microstructural and elemental characteristics of zinc production cake samples were successfully investigated using SEM, EDS, and elemental mapping techniques. SEM analysis revealed that the residue possesses a highly heterogeneous, porous, and agglomerated structure, which is typical of materials formed during hydrometallurgical zinc production processes.

EDS analysis showed that oxygen, zinc, iron, and sulfur are the predominant elements in the investigated sample, indicating the presence of oxide-, hydroxide-, and sulfate-containing phases. The detection of significant amounts of zinc (25.3 wt.%) and copper (7.8 wt.%) demonstrates that the residue retains considerable quantities of valuable metals that could potentially be recovered through secondary processing.

Elemental mapping confirmed the widespread distribution of Zn, O, S, and Fe throughout the analyzed area and revealed a heterogeneous elemental arrangement at the microscale. The observed elemental associations suggest the coexistence of multiple mineral and reaction phases formed during zinc extraction and purification operations.

The results indicate that zinc production cake should be considered not only as a metallurgical waste but also as a promising secondary resource. The findings of this study provide a scientific basis for the development of advanced metal recovery technologies and sustainable residue management strategies in the zinc industry. Future investigations involving phase identification by X-ray diffraction (XRD) and quantitative mineralogical analysis are recommended to further clarify the composition and recovery potential of the studied material.

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