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MICROSTRUCTURAL CHARACTERIZATION OF LOW-GRADE RAW MATERIALS FOR CHROMIUM-MANGANESE LIGATURE SMELTING

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ABSTRACT

This article examines the mineralogical and structural features of chromite ore from the Kempirsai deposit and iron-manganese ore from the Kerege-Tas deposit. The relevance of the study is determined by the need to assess the suitability of these complex, multicomponent ores for metallurgical processing. The aim of the research is to conduct a comprehensive analysis of the morphology, mineral phases, and elemental composition of the ore samples using modern methods. Scanning electron microscopy (SEM) was applied to characterize the surface structure of the samples, while energy-dispersive spectroscopy (EDS) was used to determine the distribution and relative concentrations of elements. The results revealed the predominance of chromite in the chromite ore and Mn and Fe minerals in the iron-manganese ore, as well as their close association with silicate and oxide phases. The findings are fully consistent with the results of chemical analysis and X-ray diffraction (XRD) studies previously performed by the authors. The scientific novelty lies in the comprehensive characterization of the morphological and phase features of the ores, while the practical significance is in substantiating their suitability as an effective raw material for the production of Cr-Mn ligature.

INTRODUCTION

It is well known that the technological progress of modern civilization is closely linked to the development of new materials with predetermined operational properties (Backeberg, Bedder, & Sardain, 2021; Zhang et al., 2024). In this process, the ferrous metallurgy sector continues to play a leading role, as it forms the fundamental basis for the sustainable development of industrial production, energy, and transport infrastructure. In particular, the production of high-quality alloyed steels remains one of the key indicators of a country's industrial strength and scientific-technological potential (Akbari, Zamani, & Mostafaei, 2024).

In established industrial practice, the conventional method of steel alloying is applied, whereby standard ferroalloys such as FeCr and FeMn are introduced separately into the melt (Zhou, Tang & Wang, 2020). This approach makes it possible to comprehensively ensure the strength, plasticity, and corrosion resistance of steel (Wang, Liu, Li & Zheng, 2022; Cheng et al., 2019). However, this traditional technology also has inherent limitations. First of all, it imposes



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strict requirements on raw material quality. The stable chemical composition and high quality of ferroalloys used for alloying are based solely on the use of pre-enriched, high-grade ore concentrates. This, in turn, increases dependence on high-quality raw material resources and raises production costs. Moreover, the use of low-grade or poor-quality ores under conventional technological conditions is considered complex and economically inefficient.

In recent years, the observed depletion of high-grade ore reserves, combined with the abundant availability of low-grade and lean ores widespread across Kazakhstan, has posed new scientific and technological challenges for the metallurgical industry. At present, processing such ores using conventional methods requires expensive beneficiation stages, which significantly increase production costs and reduce overall economic efficiency. As a result, a significant contradiction has emerged between the existing technological paradigm and the actual capabilities of the available mineral resource base. This situation necessitates the search for innovative, resource-efficient, and environmentally sustainable solutions based on new technological approaches (Daver et al., 2016).

One of the most effective ways to overcome this fundamental contradiction is the development of modern technologies capable of directly processing low-grade raw materials. Such technologies would make it possible to bypass costly beneficiation stages and fully utilize the natural composition of the original ore.

In this regard, a particularly promising direction is the development of a technology for directly smelting a Cr–Mn ligature from low-grade local ores, specifically chromite ore from the Kempirsai deposit and iron-manganese ore from the Keregetas deposit. On the one hand, this approach offers an effective alternative under conditions of high-grade raw material scarcity, and on the other hand, it enables more complete and rational utilization of Kazakhstan's mineral resource potential.

Nevertheless, a mandatory and crucial prerequisite for developing such a new technology is a comprehensive and in-depth investigation of the physicochemical properties, mineralogical composition, and morphological and structural characteristics of the initial raw materials. Only the results of such scientifically grounded analysis can ensure the correct development of the future technological process and its effective implementation at an industrial scale.

In previous studies (Kabytkanov, Makhambetov, Abdullina, Zhakan, & Yucel, 2025), low-grade chromite ores and iron-manganese ores were comprehensively characterized using X-ray diffraction (XRD) and differential thermal analysis (DTA). XRD results demonstrated that chromite (FeCr_2O_4 , MgCr_2O_4) is the predominant mineral phase in the Kempirsai ore, along with associated phases such as hematite (Fe_2O_3), hercynite (FeAl_2O_4), quartz (SiO_2), and Cr_2O_3 . In samples from the Keregetas deposit, compounds such as MnO , Mn_3O_4 , and Fe_2O_3 were identified, as well as a stable spinel-type MnFe_2O_4 phase. In addition, SiO_2 and Al_2O_3 phases were also detected in the ore composition.

Derivatographic studies revealed that the ores undergo multi-stage physicochemical transformations upon heating. In the low-temperature range (100–400 °C), an endothermic effect was observed due to the release of moisture and volatile compounds. In the medium temperature range (500–750 °C), distinct exothermic peaks were recorded, associated with the oxidation of Cr and Fe and the formation of stable Cr_2O_3 and Fe_2O_3 phases. In the iron-manganese ore, the formation of intermediate phases such as MnO and Mn_2O_3 was identified in this temperature interval. At high temperatures (750–1400 °C), recrystallization of mixed oxide phases and the formation of spinel-type compounds were observed, while above 1400 °C the system reached relative stability (Kabytkanov, Abdullina, Makhambetov, & Yucel, 2025).

The results of the conducted analyses confirmed that the Kempirsai chromite ore and Keregetas iron-manganese ore possess a complex and multicomponent mineralogical nature. Their mineral composition is heterogeneous, characterized by the coexistence of various phases

and their intricate interrelationships. Furthermore, their thermal behavior is defined by multi-stage reactions involving different phase transformations, which increase process complexity. Based on these findings, the main objective of this study is to comprehensively characterize the morphological and microstructural features of these ores using modern scanning electron microscopy (SEM) and energy-dispersive spectroscopy (EDS) techniques.

The relevance of the present study is justified by several important aspects. From a scientific perspective, it focuses on the comprehensive characterization of low-grade chromite and manganese-containing raw materials. This includes not only determining their overall chemical composition but also analyzing mineral phase morphology, grain size parameters, structural features, and phase transformation behavior under thermal treatment. Such an integrated investigation provides a reliable scientific foundation for developing a direct Cr–Mn ligature smelting technology (Makhambetov et al., 2025).

From an industrial standpoint, the obtained results provide accurate and reliable baseline data for developing new technological regulations, conducting thermodynamic modeling, and performing engineering calculations. This helps reduce uncertainties in planning industrial trials and minimizes technical and economic risks associated with implementing new technologies. Moreover, these scientific data facilitate practical solutions related to efficient raw material utilization, energy optimization, slag formation control, and enhancement of process stability (Kabyllkanov et al., 2025).

Thus, the present work represents a comprehensive study of both scientific and applied significance. The research results not only demonstrate the feasibility of utilizing low-grade and complex ores but also lay the groundwork for developing new technological solutions aimed at improving metallurgical processing. This has strategic importance for expanding the domestic mineral resource base, reducing production costs, and decreasing dependence on imported raw materials in Kazakhstan’s metallurgical industry.

MATERIALS AND METHODS

In this study, natural raw material samples obtained from two major ore deposits in Kazakhstan were used as research objects. The first sample was chromite ore from the Kempirsai deposit located in the Khromtau district of Aktobe Region, while the second sample was iron–manganese ore from the Keregetas deposit situated in the Zhanaarka district of Karaganda Region. These ores are characterized by their low grade, multicomponent composition, and complex mineralogical nature; therefore, the development of scientifically grounded methods for their metallurgical processing represents a relevant and important task.

For the purpose of the study, several kilograms of samples were collected from each deposit. Each sample was initially crushed and ground, after which it was analyzed in the “Chemical Analysis” laboratory of the Zh. Abishev Institute of Chemical and Metallurgical Processes using wet chemical analysis methods. As a result, the overall chemical composition of the ores was determined, providing a basis for subsequent experimental and theoretical investigations. The main chemical characteristics of the studied ores are presented in Table 1, while the external appearance of the lump samples is shown in Figure 1.

Table 1. Chemical composition of the studied ores, wt.%.

Material	Composition, wt.%								
	Cr ₂ O ₃	Mn _κ	Fe ₂ O ₃	SiO ₂	Al ₂ O ₃	MgO	CaO	P ₂ O ₅	S
Chromite ore	40.49	–	12.03	11.10	8.99	28.29	0.65	0.006	0.013
Iron-manganese ore	–	26.62	21.78	16.74	2.57	0.15	2.34	0.43	0.17

Note – Compiled by the authors based on chemical analysis data

For morphological and microstructural investigations, selected ore lumps were placed into special molds and impregnated with epoxy resin. After complete curing of the resin, the samples were firmly fixed and subjected to multistage mechanical preparation. This process included stepwise grinding using abrasive materials of different grain sizes, followed by final polishing with diamond pastes to achieve a mirror-like surface finish. This sample preparation method ensured the preservation of the natural microstructure without distortion during processing, thereby enabling the acquisition of high-quality and well-defined SEM images.



Figure 1. Studied raw materials: (a) chromite ore; (b) iron–manganese ore

Note – Prepared by the authors

The study was conducted using a modern ZEPTOOLS–ZEM20 scanning electron microscope manufactured in China. This analytical system provides high technical performance, including magnification up to 360,000× and resolution down to 5 nm at an accelerating voltage of 20 kV (Nogayev et al., 2025). A key advantage of this microscope is its capability to examine non-conductive samples without conductive coating and under low-vacuum conditions. This is particularly important for metallurgical studies, as it allows the preservation of the sample's original natural structure.

The microscope is equipped with an Oxford Energy Dispersive Spectroscopy (EDS) system, which significantly enhances its analytical capabilities. This system enables rapid qualitative and quantitative determination of the elemental composition in selected micro-areas, as well as the construction of elemental distribution maps (mapping) on the sample surface (Jones, Roberts, Wuhrer, & Madsen, 2022). EDS analysis provides critical information for determining the stoichiometry of mineral phases, investigating phase boundaries, and evaluating the morphology of micro-inclusions (Kato, Sano, & Yamaguchi, 2025).

Thus, the synergistic application of SEM (for microstructure) and EDS (for microcomposition) ensures the acquisition of comprehensive and reliable data on the structural and morphological characteristics of the raw material samples (Hyde, McFadden, & Carter, 2024). The obtained dataset forms a microstructural basis for predicting the high-temperature interaction mechanisms between slag-forming and metal-forming components.

RESULTS AND DISCUSSION

The results obtained in this study enabled a comprehensive characterization of the mineralogical composition, morphological features, and chemical properties of the investigated ores. These data serve as a fundamental basis for assessing the technological potential of low-grade chromite and manganese-containing raw materials, as well as for evaluating their metallurgical behavior. Figure 2 presents the SEM image of the Kempirsai chromite ore together with the elemental distribution maps of its main constituents.

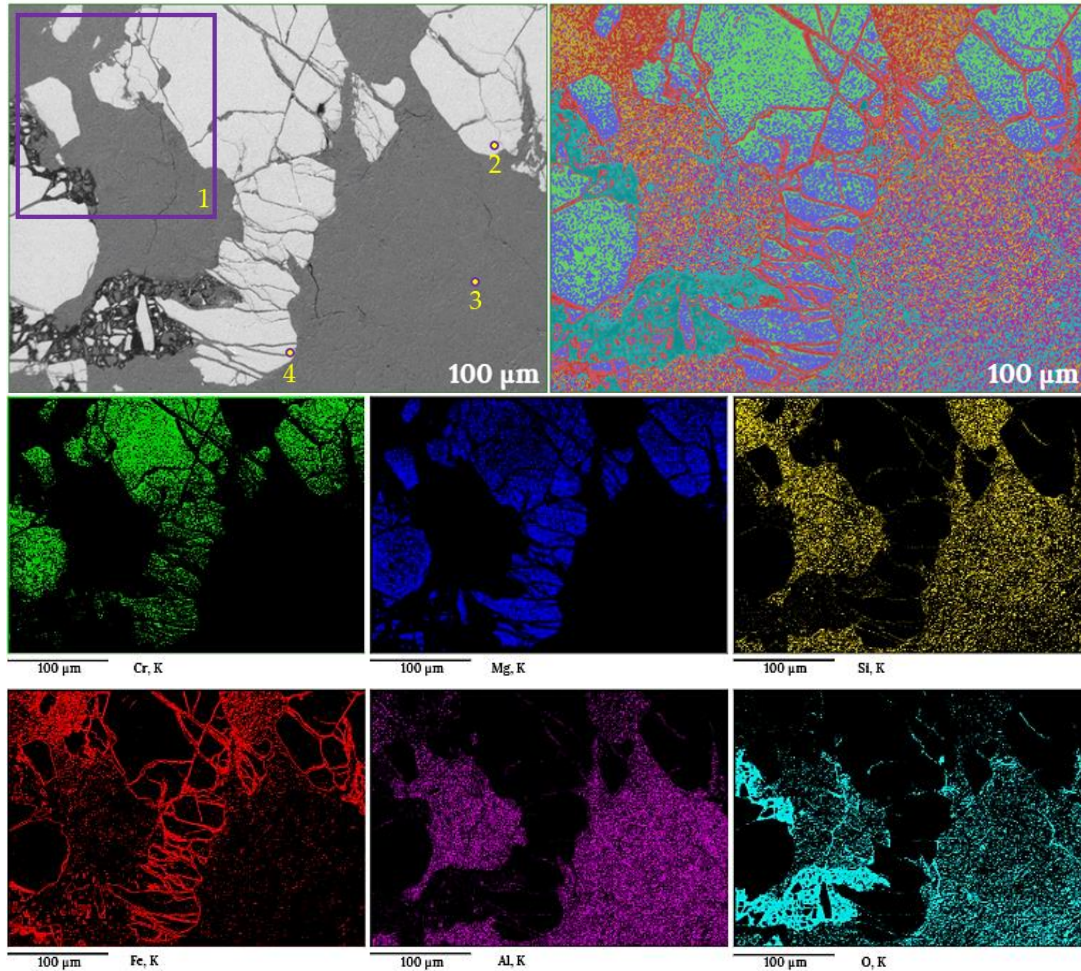


Figure 2. SEM image and elemental distribution maps of the Kempirsai chromite ore
Note – Prepared by the authors based on SEM analysis

Microstructural analysis clearly demonstrated that the mineral composition of the ore is heterogeneous. The bright regions were identified primarily as chromite grains. These grains exhibit a variety of shapes: some are close to regular geometric forms, while others are elongated or angular. The gray matrix corresponds to silicate minerals, which completely surround the chromite grains. Such a structure confirms the multicomponent mineralogical nature of the ore.

In addition, very fine dark particles were observed in certain areas. Their morphology is characteristic of iron-bearing or aluminosilicate inclusions, and they were identified as secondary phases. SEM analysis performed at magnifications up to 100 nm made it possible to clearly distinguish these fine structural features. As a result, the overall morphological characterization of the ore sample revealed a complex microstructural architecture.

To clarify the chemical elemental composition of the Kempirsai chromite ore, EDS analysis was performed. This method made it possible to determine the relative proportions of the main elements and to assess their distribution across different areas of the sample. The EDS results are presented in Figure 3 and Table 2; together with the morphological observations, they provide a deeper understanding of the phase relationships and elemental distribution patterns within the ore.

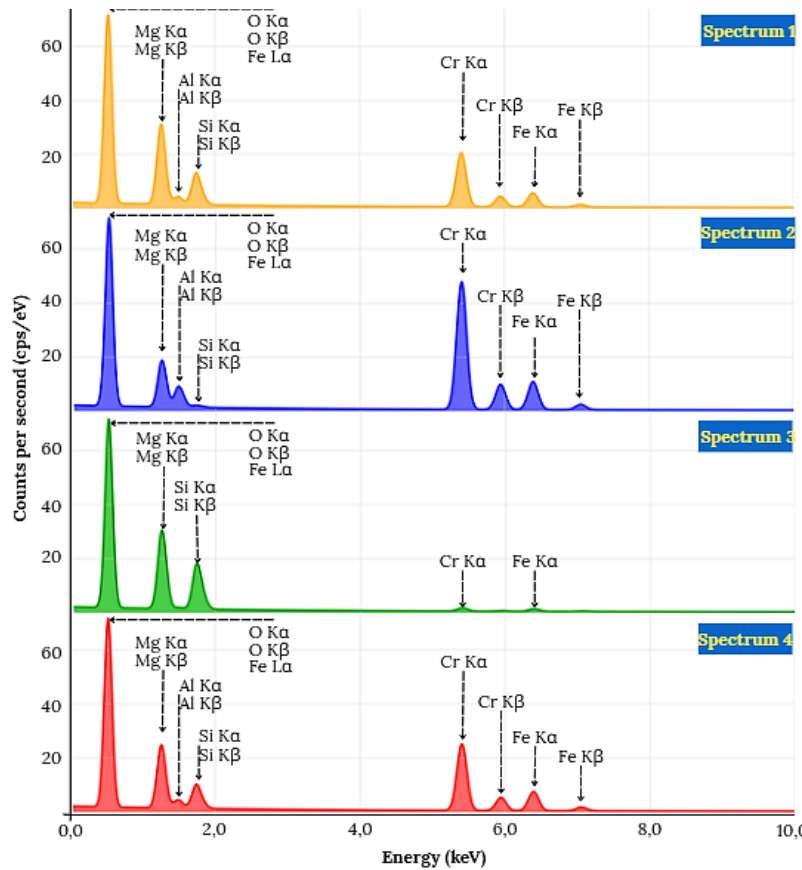


Figure 3. EDS spectra of the main elements identified in four selected areas of the chromite ore sample

Note – Prepared by the authors based on EDS analysis

Table 2. Mass fractions of the main elements in the chromite ore sample, wt. %

Spectrum No.	Composition, wt. %					
	O	Mg	Si	Cr	Al	Fe
1	43.99	17.55	7.91	16.50	1.82	4.42
2	38.16	8.85	0.46	33.85	4.20	7.62
3	53.59	20.95	13.73	1.28	0.37	1.06
4	48.80	15.30	6.62	22.45	2.15	6.68

Note – Compiled by the authors based on EDS analysis

Based on the spectra obtained from four selected areas of the ore sample (Figure 3 and Table 2), the mass fractions of the main elements were determined. Spectral analysis enabled the assessment of the mineralogical composition of the sample and the distribution patterns of elements across different regions.

The results showed that in the low-energy range (0.5–2.0 keV), distinct peaks of O, Mg, Al, and Si were recorded. These data confirm the predominance of silicate and oxide phases in the ore composition. In the high-energy range (5–7 keV), characteristic peaks of Cr and Fe were observed, indicating that these elements constitute the main mineral phases of the ore. The intensity variations of the spectral curves demonstrated that the elements are not uniformly distributed across the sample; in certain areas, specific elements were found to be dominant. Such heterogeneity may significantly influence the thermal and technological properties of the ore.

Analysis of individual spectra revealed the following:

- In Spectrum 1, a high concentration of Cr was detected alongside O and Mg, suggesting the predominance of a spinel-type MgCr_2O_4 phase.
- In Spectrum 2, the content of Cr reached 33.85 wt.%, Mg was 8.85 wt.%, and Fe was 7.62 wt.%, which is characteristic of chromite mineral composition.
- In Spectrum 3, elevated levels of Si (13.73 wt.%) and Mg (20.95 wt.%) were observed, while Cr was only 1.28 wt.%, indicating that this area corresponds to a silicate matrix.
- In Spectrum 4, Cr amounted to 22.45 wt.%, Mg to 15.30 wt.%, and Fe to 6.68 wt.%, characterizing a mixed region containing both chromite and iron-bearing inclusions.

In all spectra, Al was detected only in minor amounts (0.37–4.20 wt.%). Overall, the obtained data clearly reflect the complex mineralogical composition of the sample, revealing the distribution patterns of spinel phases, chromite-rich areas, and the surrounding silicate matrix.

At the next stage, in order to investigate the structural features of the iron–manganese ore from the Keregetas deposit in greater depth, its morphological characteristics were examined using SEM. The high magnification capability and excellent resolution of this method made it possible to conduct a detailed analysis of the sample's surface morphology, identify its textural and structural features, and evaluate the spatial arrangement, distribution patterns, and interrelationships of different mineral phases, particularly at phase boundaries. As a result, the obtained data confirmed the complex multiphase structure of the ore and demonstrated the heterogeneity of its mineralogical composition from a morphological perspective. The SEM image of the studied iron–manganese ore is presented in Figure 4.

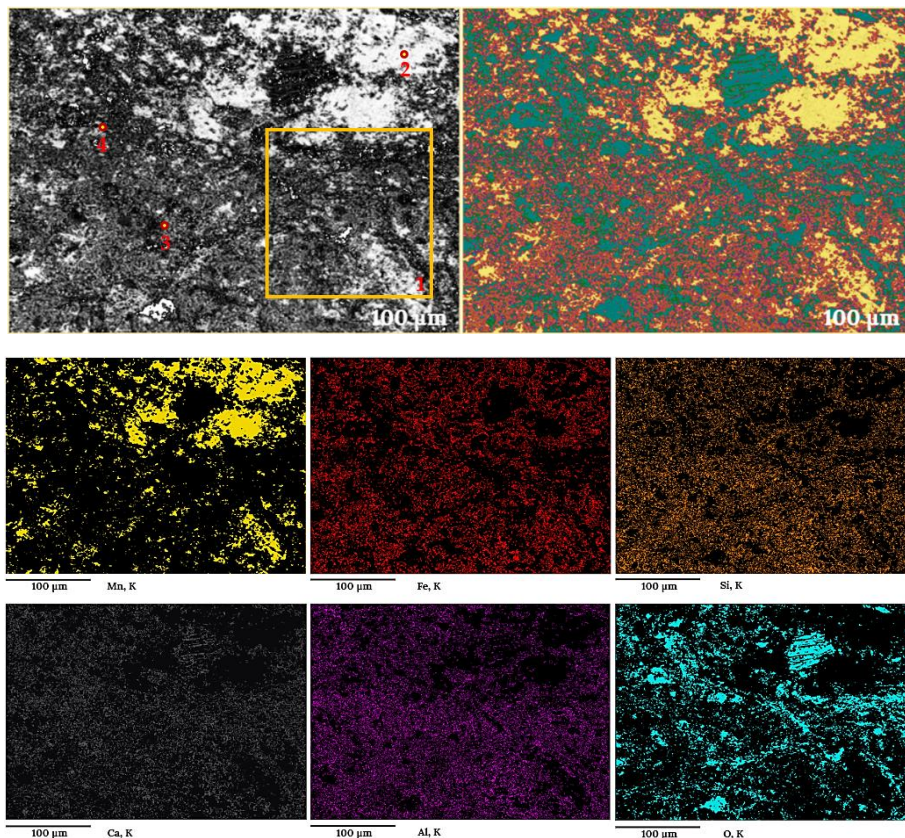


Figure 4. SEM image and elemental distribution maps of the Keregetas iron-manganese ore

Note – Prepared by the authors based on SEM analysis

In addition to SEM imaging, Energy Dispersive Spectroscopy (EDS) was employed to clarify the elemental composition of the sample and to obtain a deeper understanding of its chemical characteristics. This method not only enabled the identification of the distribution of major and minor elements within the studied ore but also allowed quantitative assessment of their relative proportions.

EDS analysis revealed that in different areas of the iron–manganese ore sample, key elements such as Fe, Mn, Si, and O are concentrated in varying amounts. At the same time, the presence of minor quantities of elements such as Al, Ca, and Cr indicates the existence of additional mineral phases. These data confirm the multiphase nature of the sample and highlight the complexity of its mineralogical composition.

The results of the conducted analysis are illustrated in Figure 5, while Table 3 summarizes the EDS spectral data of the main elements recorded in selected regions of the iron–manganese ore sample.

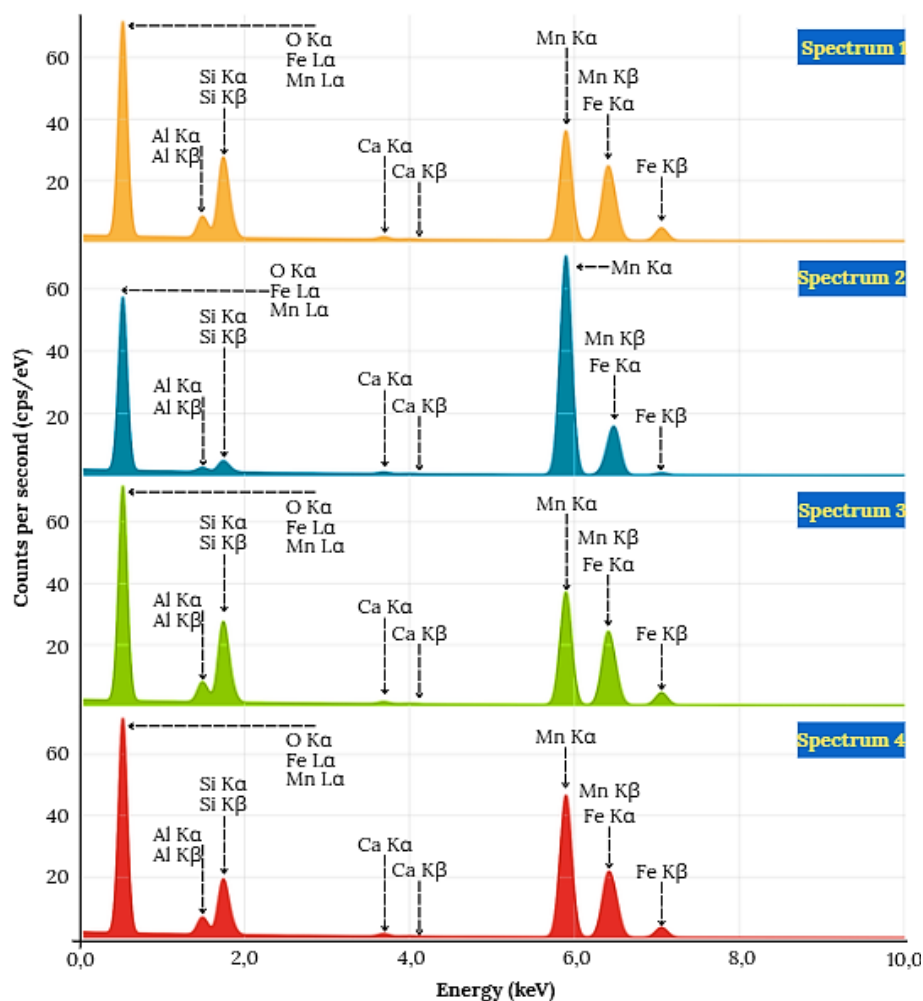


Figure 5. EDS spectra of the main elements identified in four selected areas of the iron–manganese ore sample

Note – Prepared by the authors based on EDS analysis

As part of the study, EDS spectra obtained from four selected areas of the iron–manganese ore sample were analyzed. Each spectrum exhibited clear characteristic peaks corresponding to the main elements comprising the mineral composition of the ore.

In the low-energy range (0.5–2.0 keV), intense peaks of O, Al, and Si, as well as Mn L α lines, were recorded, indicating their significant concentration in the silicate and oxide phases of the ore. In the mid-energy range (3–4 keV), weak Ca K α and K β lines were detected, suggesting the presence of minor accessory mineral phases. In the high-energy range (5–7 keV), distinct Mn and Fe K α and K β peaks were observed, confirming that these elements are the primary metal-forming components in the ore. The relative intensities of the spectral curves made it possible to assess the proportional distribution and spatial variability of elements in different regions of the sample.

Table 3. Mass fractions of the main elements in the iron–manganese ore sample, wt.%

Spectrum No.	Composition, wt.%					
	O	Ca	Si	Mn	Al	Fe
1	34.15	0.50	13.52	23.10	3.35	13.64
2	31.35	0.36	2.12	52.56	0.72	2.60
3	34.39	0.41	13.63	24.00	3.20	13.44
4	32.80	0.47	8.95	28.75	2.60	10.43

Note – Compiled by the authors based on EDS analysis

Based on the study of the iron–manganese ore, spectra obtained from four selected areas were analyzed. In Spectrum 1, significant amounts of O (34.15 wt.%) and Si (13.52 wt.%) were recorded, while Mn (23.10 wt.%) and Fe (13.64 wt.%) were also present in considerable proportions. Such a composition indicates the coexistence of iron–manganese minerals together with silicate phases.

In Spectrum 2, the Mn content increased sharply to 52.56 wt.%, while O reached 31.35 wt.%, Fe 2.60 wt.%, and Si 2.12 wt.%. These data demonstrate that this region is rich in manganese-bearing minerals.

In Spectrum 3, Mn amounted to 24.00 wt.%, Fe to 13.44 wt.%, Si to 13.63 wt.%, and O to 34.39 wt.%. This composition confirms the distribution of Fe–Mn minerals within a silicate–oxide matrix.

In Spectrum 4, Mn (28.75 wt.%) and Fe (10.43 wt.%) were present in relatively high proportions, while O reached 32.80 wt.%. This region corresponds to a complex mineral assemblage containing both iron–manganese inclusions and silicate phases.

Overall, in all four spectra Al was detected only in minor amounts (0.72–3.35 wt.%), while Ca content was very low (0.36–0.50 wt.%). The data presented in Table 3, together with the spectral analysis results, clearly characterize the complex mineralogical composition of the sample and reveal the distribution of Mn- and Fe-rich areas as well as silicate phases. Furthermore, morphological SEM investigations highlighted the textural features of the sample and confirmed the presence of localized concentrations of Mn and Fe minerals on its surface.

CONCLUSION

In this study, the mineralogical and structural features of the Kempirsai chromite ore and the Keregetas iron–manganese ore were comprehensively investigated using a complex of modern analytical methods. The results revealed the multicomponent and complex nature of these ores and enabled a metallurgical assessment of their morphological and phase behavior.

SEM analysis characterized the surface morphology of the samples and demonstrated that chromite (FeCr₂O₄) is the dominant mineral phase in the chromite ore from Kempirsai. In addition, the heterogeneous distribution of ferrosinels and silicate inclusions was identified. In

the iron–manganese ore, Mn- and Fe-bearing minerals were found to exhibit complex textural relationships and to coexist with silicate and oxide phases. These features confirm the heterogeneity and multiphase structure of the studied ores.

According to EDS results, the high Cr content in the Kempirsai chromite ore confirms that chromite is the main mineral-forming component. In the Keregetas iron–manganese ore, elevated concentrations of Mn and Fe indicate that these elements define the principal phase composition of the material. Minor elements such as Al, Ca, and Si were detected only in small amounts, confirming the presence of accessory mineral phases.

Overall, the obtained results demonstrate that the investigated ores possess a multiphase and structurally complex mineralogical nature. Such characteristics directly influence their thermal stability, susceptibility to phase transformations, and technological behavior during metallurgical processing.

The findings of this study are in full agreement with the results of chemical analysis and previously conducted XRD investigations. This confirms the complementarity of the applied methods and the reliability of the scientific conclusions drawn.

From a practical standpoint, the mineralogical composition of the Kempirsai chromite ore and the Keregetas iron–manganese ore indicates their suitability for metallurgical use. In particular, the obtained results demonstrate that these ores can serve as promising raw materials for the production of Cr–Mn ligatures.

CONFLICT OF INTEREST: The authors declare that they have no conflicts of interest.

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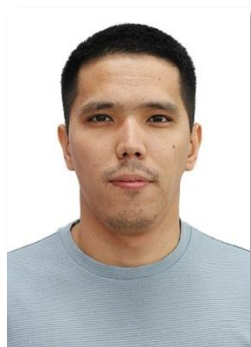
STATEMENT ON THE USE OF ARTIFICIAL INTELLIGENCE TECHNOLOGIES: The authors declare that no artificial intelligence (AI) tools were used in the preparation of this article.

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