



Characterization of Oolitic Iron Ore from Gara Djebilet, Algeria: Mineralogy, Chemistry, Morphology, and Thermal Properties

Messai Ali ^{a, #}, Lafdil Sana ^b, Boustila Amir ^a, Allem Habiba ^c, Bouzeriba Hadjer ^d,
Juan M. Menéndez-Aguado ^e, Mebarkia Mohammed ^{f, g}

^a National Polytechnic School of Algiers, Laboratory of Mining Engineering, Department of Mining Engineering, Algeria

^b National Higher School of Technology and Engineering, Department of Mining, Metallurgy and Materials Engineering, Annaba, Algeria

^c Badji Mokhtar Annaba University, Department of Geology, Annaba, Algeria

^d Abderrahmane Mira University, Department of Mining and Geology, Bejaia, Algeria

^e University of Oviedo, Polytechnic School of Mieres, Mineral Raw Materials Processing Research Group, Spain

^f Badji Mokhtar-Annaba University, Mining Department, Annaba, Algeria

^g University of Echahid Echikh Larbi Tebessi, Mining Institute, Environment Laboratory, Tebessa, Algeria

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ABSTRACT

The iron ore deposits of Gara Djebilet, located in the Tindouf region of Algeria, hold significant socio-economic importance among the indigenous iron ore deposits discovered in the country. To meet local steel production requirements, these ores require an effective enrichment process, necessitating detailed mineralogical, chemical, and morphological characterization. Techniques such as optical microscopy, scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDS), X-ray fluorescence (XRF), X-ray diffraction (XRD), and differential scanning calorimetry-thermogravimetric analysis (DSC-TG) were employed for this purpose. Mineralogical studies revealed that Gara Djebilet iron ore is primarily composed of hematite and goethite. XRF analysis indicated a composition of 61.95 % Fe, 4.27 % SiO₂, 2.22 % CaO, 4.72 % Al₂O₃, and 1.53 % P₂O₅. Data from the various analyses suggest that phosphorus is finely disseminated within the hematite phase, indicating that Gara Djebilet iron ore may not be amenable to upgrading using conventional techniques.

1. Introduction

Iron occurs in several minerals, but only a few serve as major primary sources, including hematite (Fe₂O₃),

magnetite (Fe₃O₄), pyrite (FeS₂), goethite/limonite (FeO(OH)), and siderite (FeCO₃). These iron-bearing minerals are often associated with gangue materials that contain impurities such as quartz, clays (e.g., kaolinite

[#] Corresponding author: ali.messai@g.enp.edu.dz

and gibbsite), and phosphorus (USEPA, 1994).

Phosphorus is often present in secondary iron oxide minerals, particularly in oolitic iron ores. The suitability of these ores for steel production depends on their phosphorus content, which typically must range between 0.08 % and 0.14 % (Cheng et al., 1999). Oolitic iron ores are classified into three categories: low-phosphorus ores contain less than 0.07 % P, medium-phosphorus ores range from 0.07 % to 0.10 % P, and high-phosphorus ores exceed 0.10 % P (Fisher-White et al., 2012).

High-phosphorus iron ores occur in many locations worldwide. In China, significant deposits are located in Hubei Province (Muhammed and Zhang, 1989; Tang et al., 2010; Wang et al., 2017; Zhu et al., 2017), Wushan Chongqing (Li et al., 2013), and Hunan Province (Xia et al., 2011; Li et al., 2013). Other notable occurrences include the Pilbara region in Australia (Fisher-White et al., 2012), Lisakovsk in Kazakhstan (Akatonov et al., 1969; Khassen et al., 2014), and the East Aswan region in Egypt (Omran, 2015). High-phosphorus iron ores are also found in Agbaja, Nigeria (Amadi et al., 1982; Adedeji and Sale, 1984; Uwadiae and Whewell, 1988; Uwadiae, 1991; Baba et al., 2005), Russia (Uwadiae, 1989), Portugal (Mammadov, 2016; Zhou et al., 2022), Minas Gerais in Brazil (Ranjbar, 2002; Anyakwo and Obot, 2011), and Algeria (Badjoudj et al., 2017).

In Algeria, iron ore is primarily sourced from the Boukhadra and Ouenza deposits, located in eastern Tebessa. These deposits are currently used by the El Hadjar Steel Complex in Annaba to produce steel for domestic consumption. However, because these reserves are expected to be depleted, efforts have been made to explore alternative ore sources to meet the growing demands of the steel industry. This has led to the establishment and development of two new steel plants: Algerian Qatar Steel in Jijel and Tussiali in Oran. These facilities aim to use oolitic iron ores from the Gara Djebilet deposit to support Algeria's steel production needs.

The Gara Djebilet deposits, which include Gara West, Gara Center, and Gara East, are of major importance to Algeria because of their substantial estimated reserves of approximately 3.5 billion tons. Despite this potential, the high phosphorus content of these ores presents a significant challenge for their direct use in industrial processes. To address this issue, enrichment methods are being investigated to reduce the phosphorus content to an acceptable level, thereby making the ores suitable for industrial applications.

Consequently, extensive research has focused on developing methods for phosphorus removal from high-phosphorus iron ores to reduce phosphorus content to acceptable levels. Various techniques have been employed for this purpose, including thermal treatment (Li et al., 2013; Omran, 2015; Gao et al., 2016; Tang et al., 2016; Roy et al., 2020; Han and Lu, 2022; Wu et al., 2023), microwave treatment (Nunna et al., 2021; Zhou et

al., 2022; Zhou et al., 2023), ultrasonic treatment (Donskoi et al., 2022), acid leaching (Wang et al., 2017; Cai et al., 2018; Silva et al., 2019; Pan et al., 2022), alkaline leaching (Gao et al., 2016; Tang et al., 2016; Zhang et al., 2019; Pan et al., 2022; Deng et al., 2023), bioleaching (Edwards et al., 2011; Kim et al., 2015; Xiao et al., 2015; Zhang et al., 2015; Mammadov, 2016; Abdel-Khalek et al., 2017; Kim et al., 2017; Selim and Rostom, 2018), and froth flotation (Abbaker et al., 2019; Batnasan et al., 2022).

A detailed investigation into the properties of Gara Djebilet iron ore is essential to identify the most suitable method for phosphorus removal. Optimizing this process will make it possible to produce high-grade iron ore, which could have a significant economic impact by meeting the demands of both national and international steel industries.

2. Study Area Description

2.1. Geographic situation

The Gara Djebilet deposit is located approximately 130 kilometers southeast of Tindouf city, near the Algerian-Mauritanian border. It lies 380 kilometers in a straight line from the Atlantic Ocean and approximately 1,600 kilometers south of the Algerian coastline. The geographic location of the deposit is shown in Figure 1.

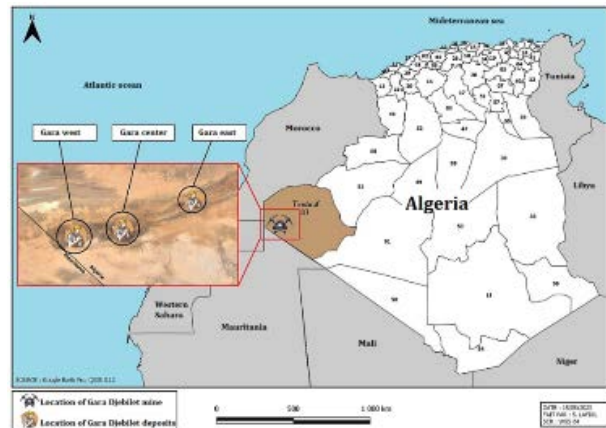


Figure 1. Location map of study area in Tindouf; source: own elaboration

2.2. Local geology

The Gara Djebilet iron deposit extends over a distance of 50 kilometers from west to east. It consists of three lenses of oolitic ores of sedimentary origin, all located at the same stratigraphic level, identified at the top of the Lower Devonian (Emsian). These lenses are elongated in a WSW-ENE direction, are subhorizontal, gently dipping at a consistent 2° to the north. They are distributed across kilometeric distances, spaced approximately 9 kilometers apart, and collectively cover an area of 130 square

kilometers. The iron formation varies in thickness from 15 to 30 meters and is characterized by mineralization occurring in three superimposed levels. The most abundant level is magnetic, with an average thickness of 8.5 meters and a total iron (FeT) content of 58 %. This magnetic layer extends over 34 square kilometers in Gara West and 72 square kilometers in Gara Center. For a detailed overview of the regional geology and the spatial distribution of the Gara Djebilet deposits, refer to Figure 2 (Bersi et al., 2016).

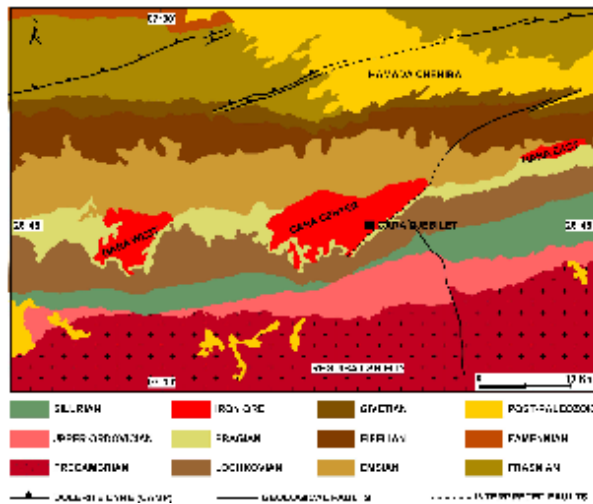


Figure 2. Regional geology and the location of Gara Djebilet deposits (Bersi et al. 2016)

3. Materials and Methods

3.1. Sample preparation

Samples were collected from various layers of the Gara Djebilet deposit over a 12-meter interval, with sampling every 2 meters. This strategy yielded a total of 60 kilograms of fragment and block samples.

At the Laboratory of Mining Engineering, large rock samples were selected to prepare cylinders for mechanical property tests, as well as thin polished sections and polished cubes. The remaining material was crushed using a single-toggle jaw crusher (Model 48-D0530/A) with a 5 mm setting. The crushed samples were then thoroughly mixed and prepared using the coning and quartering method to ensure representative sampling. These samples were subsequently used for characterization studies, including size analysis, physicochemical analysis, and mineralogical analysis. All sample preparation procedures were carried out at the Laboratory of Mining Engineering at ENSTI Annaba.

3.2. Characterization

3.2.1. Size analysis

A quantity of 2.000 grams of dried raw material was processed at the Laboratory of Mining Engineering at

ENSTI Annaba, Algeria. Sieving was performed using a CONTROLS shaking machine (Model 15-D0410) and a series of sieves with mesh sizes of 2 mm, 1 mm, 0.5 mm, 0.25 mm, 0.125 mm, and 0.063 mm. The sample was sieved for 30 minutes at an amplitude of 60 mm/g. After sieving, the material retained on each sieve was weighed using a precision scale with an accuracy of 0.01 grams.

3.2.2. X-ray fluorescence

The chemical composition of the solid materials was analyzed using X-ray fluorescence (XRF) at the Direction of Technology and Development (DTD) in Boumerdes, Algeria. Analyses were performed using a Bruker-Axs S8 TIGER spectrometer equipped with a rhodium anode. Loss on ignition (LOI) was determined by calcining the samples in an oven at 1050 °C for two hours. LOI represents the volatile fraction of the sample, including humidity, CO₂, and organic matter.

3.2.3. X-ray diffraction

The phase composition of the samples was analyzed using X-ray diffraction (XRD) at the Direction of Technology and Development (DTD) in Boumerdes, Algeria. Analyses were carried out using a PANalytical X'PERT-PRO diffractometer equipped with a copper anticathode ceramic X-ray tube. The instrument was operated at 40 mA and 45 kV.

3.2.4. Scanning Electron Microscopy

The microstructure of the solid products was examined using scanning electron microscopy (SEM) at the Laboratory of Metallurgy and Materials Sciences at ENSTI Annaba, Algeria. The analysis was performed using a FEI Quanta 250 SEM equipped with an energy-dispersive X-ray spectroscopy (EDS) detector for elemental analysis.

3.2.5. Differential Scanning Calorimetry - Thermogravimetry

Thermal analysis was carried out using differential scanning calorimetry and thermogravimetry (DSC-TG) at the Laboratory of Metallurgy and Materials Sciences at ENSTI Annaba, Algeria. The analysis was performed using a NETZSCH STA 449 F3 Jupiter instrument.

4. Results and Discussion

4.1. Size analysis

The results of the size analysis for the collected sample are presented in Table 1. The data show a predominant distribution of coarse particles, with the fraction larger than 2 mm constituting 60.35 % of the total mass. This indicates a significant presence of large mineral

fragments and confirms the high hardness of the studied ore. Moderately fine fractions account for 9.82 % (2 - 1 mm), 6.68 % (1 - 0.5 mm), and 8.27 % (0.5 - 0.2 mm) of the total mass, respectively. Very fine particles (< 0.2 mm) represent 14.88 % of the total mass. This particle size distribution suggests that the iron ore is primarily composed of coarse particles, which could affect subsequent processing steps, such as grinding and concentration.

4.2. X-Ray fluorescence

The chemical composition of Gara Djebilet iron ore, as determined by X-ray fluorescence (XRF) analysis, is presented in Table 2. The results indicate a high total iron (FeT) content of 61.95 % by mass, along with moderate proportions of silica (SiO₂) and alumina (Al₂O₃) signifying a high-quality iron resource.

However, particular attention must be given to the phosphorus content, which is 1.53 %. This level of phosphorus can significantly affect the ore's suitability for certain metallurgical applications, particularly in the steel industry, unless it is treated beforehand to reduce its concentration. Other trace elements, such as calcium, magnesium, sodium, titanium, and sulfur, are present in varying amounts but generally fall within acceptable limits.

4.3. X-Ray diffraction

Powder X-ray diffraction (XRD) analysis of Gara Djebilet crude ore, as shown in Figure 3, indicates that the ore is primarily composed of hematite and magnetite as valuable minerals, with chamosite present as the main gangue mineral.

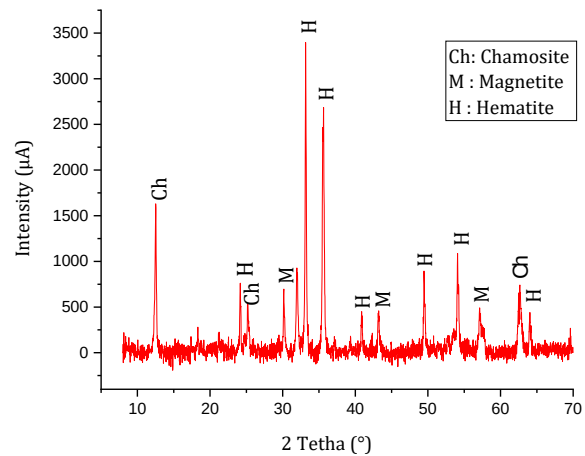


Figure 3. X-ray diffraction patterns of Gara Djebilet iron ore

4.4. Differential Scanning Calorimetry - Thermogravimetry

Differential scanning calorimetry (DSC) and thermogravimetric (TG) analyses of the iron ore samples, shown in Figure 4 and Table 3, provide crucial insights into the material's thermal transformations and phase transitions. As illustrated in Figure 4, the TG curve exhibits a steep change, while the DSC curve alternates between exothermic and endothermic peaks and valleys. This behavior indicates that the sample undergoes intense solid-phase reactions and thermal effects under high-temperature conditions. Specifically, the TG curve reveals a slight mass gain of 0.6 %, followed by a total mass loss of 6.6 %. This mass loss is likely due to the evaporation of free moisture, which is consistent with the low loss on ignition (LOI) value reported in Table 2.

Table 1
Results of particle size analysis of Gara Djebilet iron ore crushed to 5 mm

Aperture (mm)	Mass (g)	Yield (%)		
		Partial	Cumulative undersize $\sum Y_{\uparrow}$	Cumulative oversize $\sum Y_{\downarrow}$
+ 2	1014	60.35	100	0
- 2 + 1	165	9.82	39.65	60.35
- 1 + 0.5	112	6.68	29.83	70.17
- 0.5 + 0.2	139	8.27	23.15	76.85
- 0.2 + 0.125	74	4.40	14.88	85.12
- 0.125 + 0.063	96	5.72	10.48	89.52
- 0.063 + 0	80	4.76	4.76	95.24
Total	1680	100.0	0	100

Table 2
Chemical composition of Gara Djebilet raw ore

Elements	Fe	SiO ₂	Al ₂ O ₃	CaO	MgO	Na ₂ O	TiO ₂	MnO	P ₂ O ₅	SO ₃	LOI
Content	61.95	4.27	4.72	2.22	0.49	0.46	0.34	< 0.01	1.53	0.05	4.96

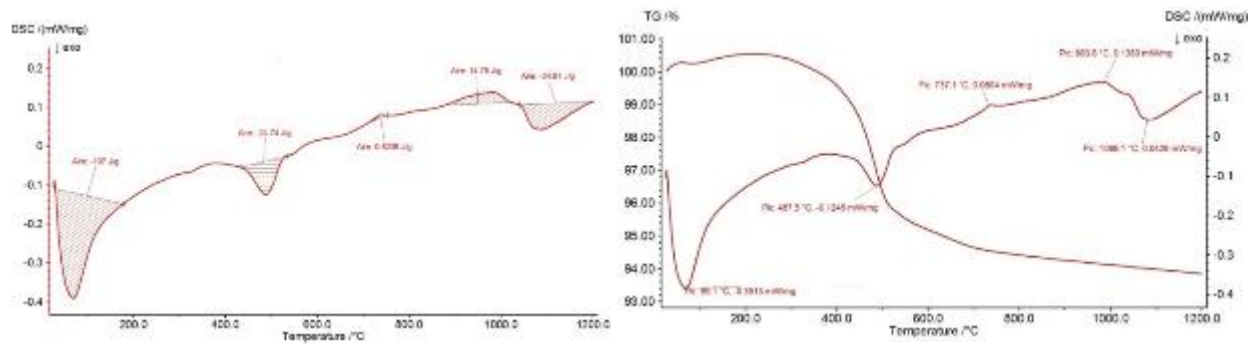


Figure 4. DSC-TG plots for Gara Djebilet iron ore

Table 3
Results from the analysis of observed peaks in DSC-TG measurements

Peaks	Peak type	Area (J/g)	Height mW/mg	Peak onset (°C)	Peak (°C)	Peak end (°C)
1	Endothermic	- 107	0.3913	16.6	69.1	156.7
2	Endothermic	- 24.74	0.1245	413.3	487.5	510
3	Exothermic	0.8239	0.0804	696.7	737.1	726.6
4	Exothermic	14.79	0.1389	850	983.8	933.3
5	Endothermic	- 34.91	0.0428	1010	1088.1	1166.6

The first endothermic peak observed in the DSC plot has an onset temperature of approximately 16.6 °C, an endpoint at 156.7 °C, and a maximum at about 69.1 °C. This peak likely corresponds to the dehydration process or the loss of physically adsorbed water from the ore surface.

The second endothermic peak, with an onset at approximately 413.3 °C and an endpoint at 510 °C, reaches maximum at 487.5 °C. This peak is attributed to the dehydroxylation of goethite (FeOOH), involving the loss of structural OH groups. This process leads to the transformation of goethite into hematite (Fe₂O₃) through the release of crystalline-bound water. In the thermogravimetric results, additional less intense peaks are observed, including exothermic peaks at 737.1 °C and 983.8 °C. These peaks are likely associated with the transformation of siderite into hematite and magnetite, as suggested by Kądziołka-Gaweł et al. (2023). Furthermore, an endothermic peak at 1088.1 °C is attributed to the recrystallization and transformation of dehydration products into metakaolinite, corundum, and silica, consistent with findings by Vaculikova et al. (2011).

4.5. Scanning Electron Microscopy

The results of scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDS) and SEM mapping are presented to corroborate the findings from chemical, mineralogical, and thermal analyses carried out using X-ray diffraction (XRD), X-ray

fluorescence (XRF), and differential scanning calorimetry-thermogravimetry (DSC-TG), respectively.

4.5.1. Polished sections

Examination of the polished- section morphology using scanning electron microscopy (SEM) (Figure 5a) shows that the predominant ore mineralization is mainly composed of iron. The associated gangue minerals consist primarily of quartz, with minor apatite. In Figure 5b, the detection of zirconium, oxygen, and silicon indicates the presence of zircon (ZrSiO₄), which occurs as small inclusions within the iron oxide matrix.

4.5.2. Polished cubes

Clays:

SEM-EDS analysis of clay cubes (Figure 6a) reveals significant amounts of titanium (Ti), oxygen (O), iron (Fe), silicon (Si), and aluminum (Al), along with variable quantities of manganese (Mn), phosphorus (P), molybdenum (Mo), and potassium (K). This composition strongly suggests the presence of ilmenite (FeTiO₃) and molybdenite (MoS₂) associated with clay minerals. Traces of manganese, commonly associated with ilmenite, were also detected.

The analysis in Figure 6b indicates the presence of iron oxide, likely pyrite, and molybdenite associated with clay minerals. Figure 6c shows a typical composition of clay minerals associated with ilmenite and/or rutile.

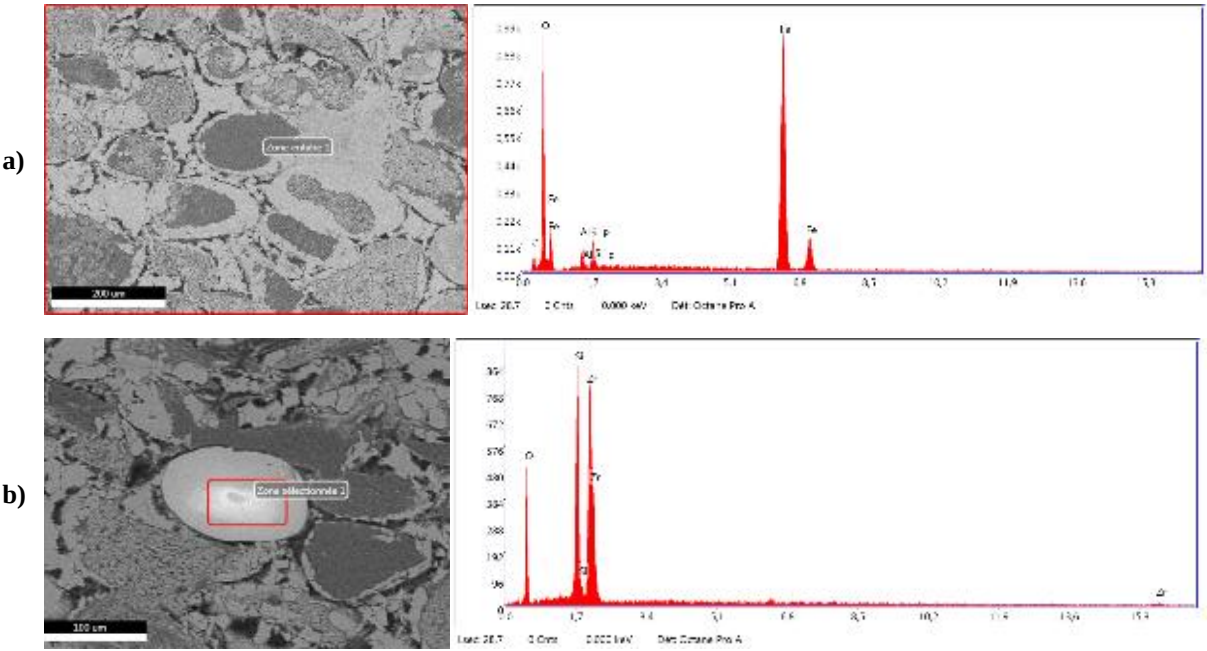


Figure 5. SEM-EDS results at different zones of polished section

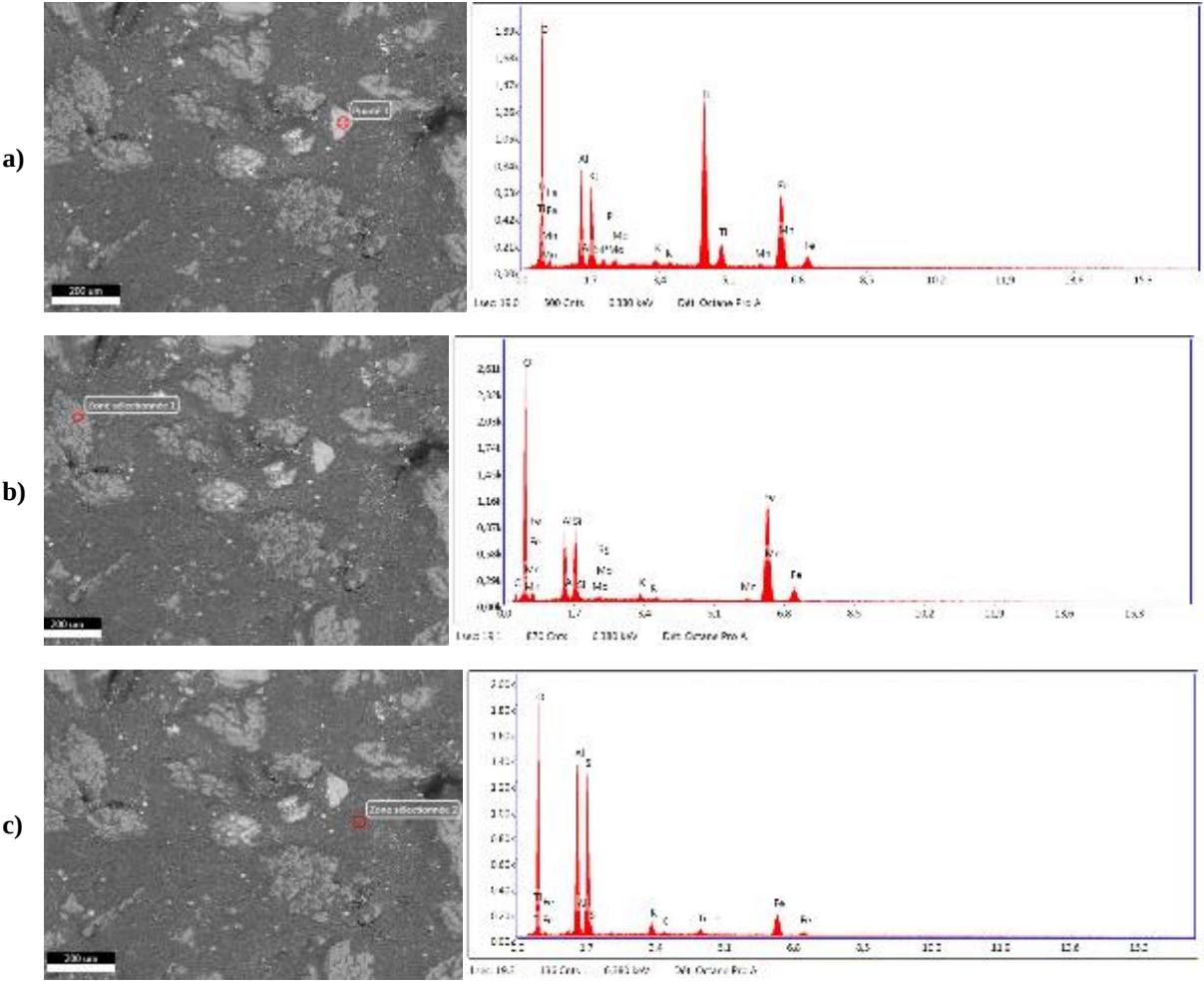


Figure 6. SEM-EDS results at different zones of clay polished cube

Hematite:

The ore mineralization is notably concentrated in grayish zones, as shown in Figures 7b and 7c,

with varying proportions. In contrast, the dark zones likely indicate clay minerals with high iron content, as illustrated in Figure 7a.

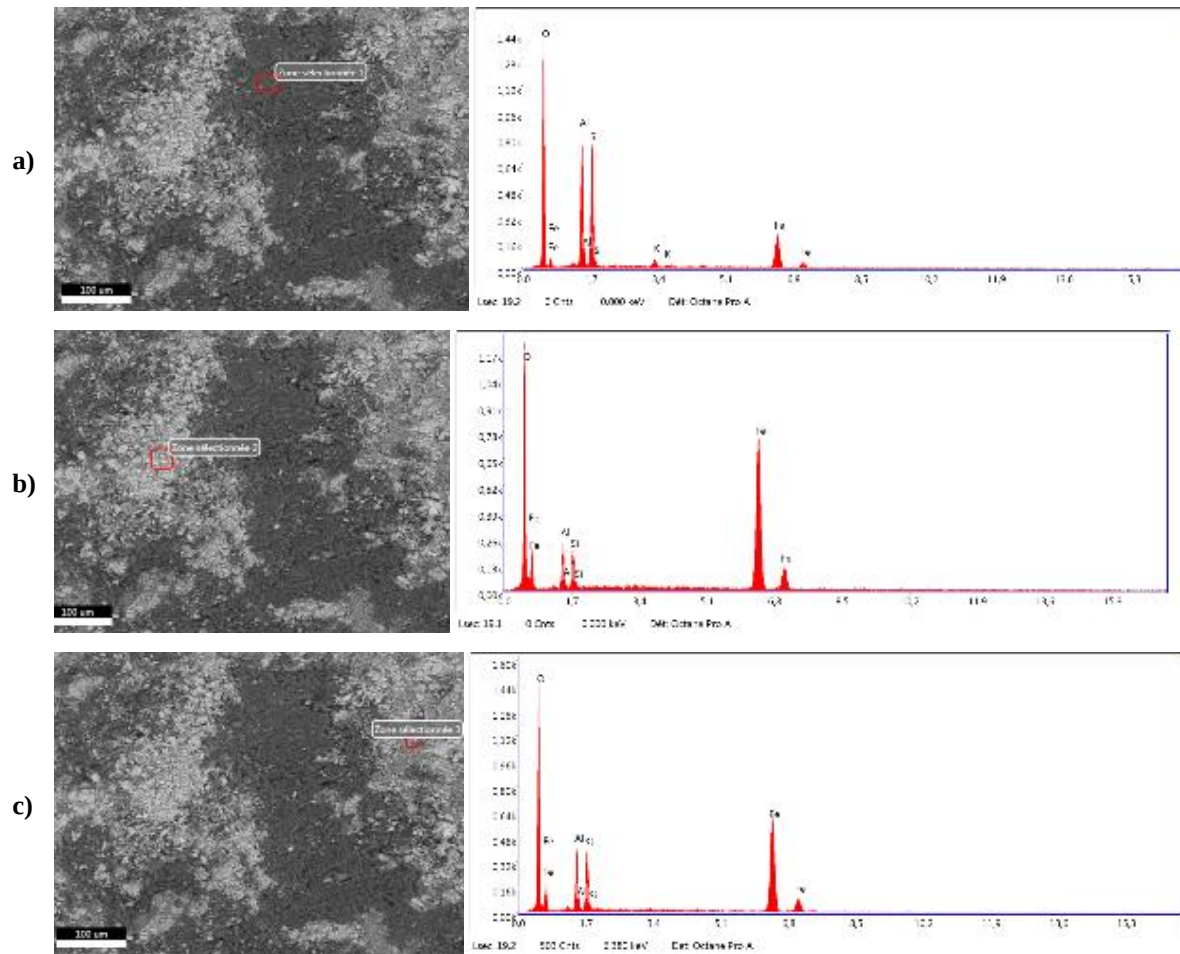
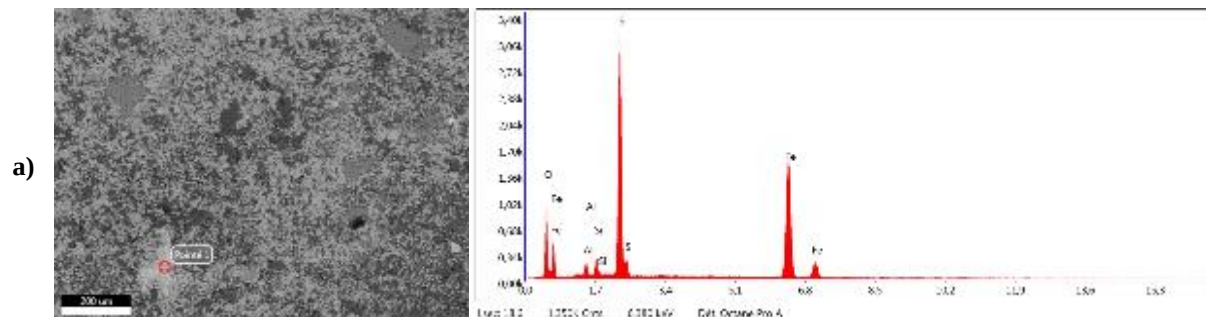


Figure 7. SEM-EDS results at different zones of hematite polished section

Magnetite:

MEB -EDS results reveal high levels of sulfur and iron, suggesting the presence of sulfide minerals, possibly pyrite or marcasite (Figure 8a). Figure 8b shows a clay mineral, likely kaolinite, with minor amounts of magnetite. High concentrations of oxygen, silicon, and

iron in Figure 8c indicate the presence of iron oxides. Additionally, enrichment in oxygen (O), silicon (Si), iron (Fe), titanium (Ti), aluminum (Al), phosphorus (P), and potassium (K) suggests the possible presence of ilmenite (FeTiO_3) and/or rutile (TiO_2), apatite ($\text{Ca}_5(\text{PO}_4)_3(\text{F,Cl,OH})_2$), and kaolinite ($\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$) (Figure 8d).



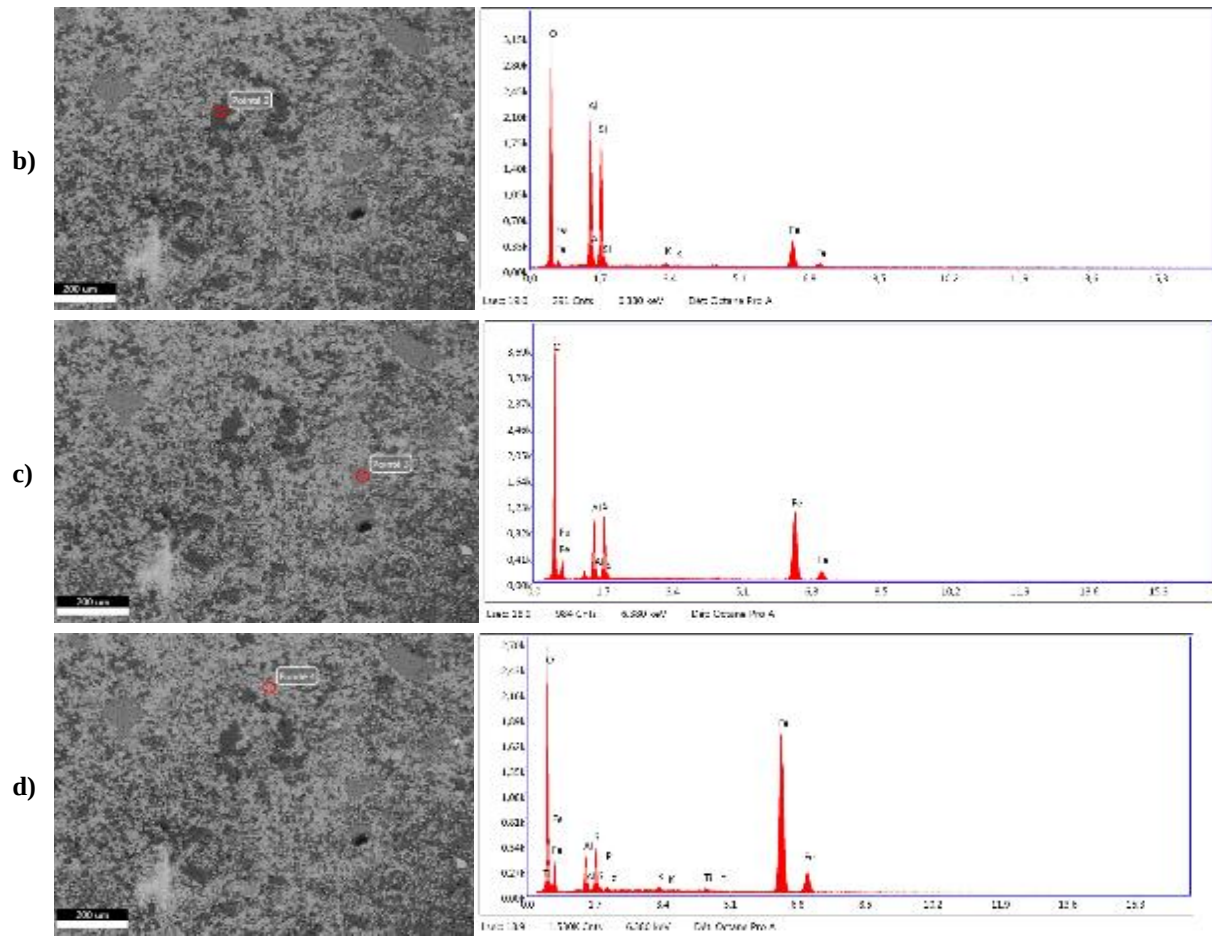


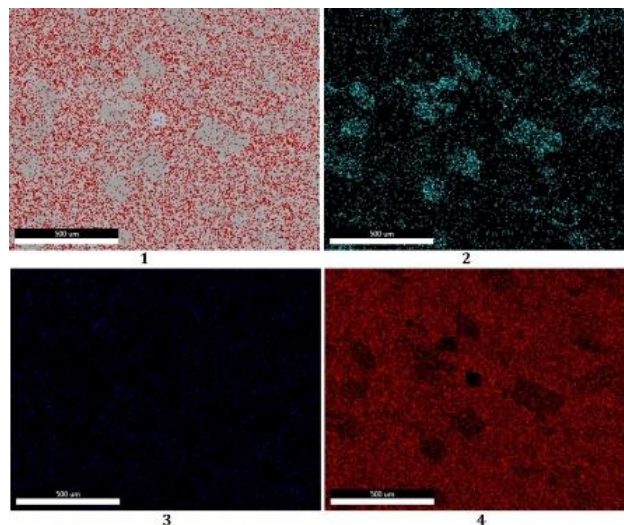
Figure 8. SEM-EDS results at different zones of magnetite polished section

4.5.3. Cartography (Mapping)

Polished section

Elemental mapping of the polished section, shown in Figure 9, reveals a heterogeneous composition with a varied distribution of elements. Iron and oxygen

dominate, reflecting the iron-rich nature of the ore. Silicon and aluminum are also present, indicating silicate minerals. Phosphorus, although present in low concentrations, is localized at specific points, indicating phosphate minerals such as apatite. Additionally, minor but notable amounts of zirconium are detected.



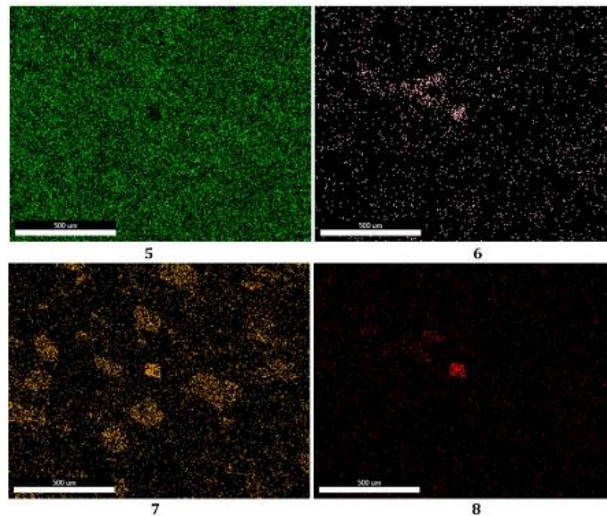


Figure 9. SEM cartography of polished section (1: global; 2: Al K; 3: C K; 4: Fe K; 5: O K; 6: P K; 7: Si K; 8: Zr L)

The sample exhibits overall homogeneity in the distribution of most elements. However, notable bright spots are observed for specific elements, including carbon, iron, zirconium, phosphorus, aluminum, and silica. Although phosphorus is present in small amounts, its localized distribution is particularly significant. These elements are distinctly highlighted in Figure 10.

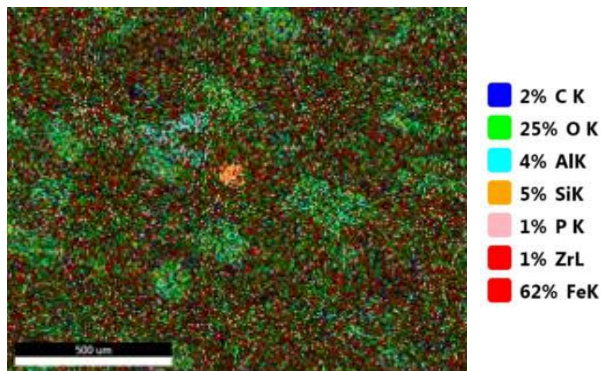


Figure 10. Distribution of different element contained in polished section of Gara Djebilet iron ore

The EDS results presented in Figure 11 confirm the presence of iron, aluminum, silicon, oxygen, phosphorus, and zirconium. It is important to note that hydrogen is not detected because of the inherent limitations of EDS, which is unsuitable for detecting elements with very low atomic masses, including hydrogen and the first four elements of the periodic table.

Ground sample (before and after DSC)

Figures 12a and 12b present elemental mapping of a ground sample before and after heating to 1.200 °C. A significant increase in total iron (Fe_T) content is observed, accompanied by a decrease in oxygen levels. These results are consistent with the DSC curves and confirm the hydroxylation of goethite and its transformation into hematite at elevated temperatures.

On the other hand, the phosphorus (P) content remains unchanged, suggesting that heat treatment may not be an effective pretreatment step for reducing phosphorus levels in the final concentrate.

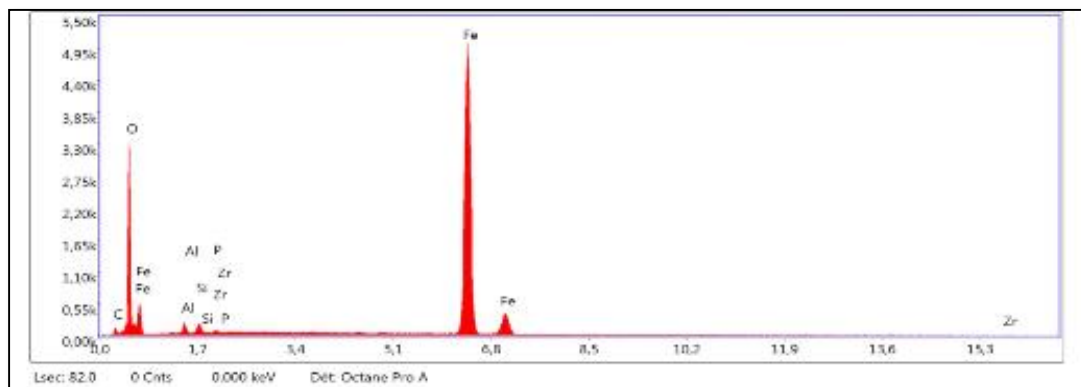


Figure 11. Energy spectrum scanning results of polished section

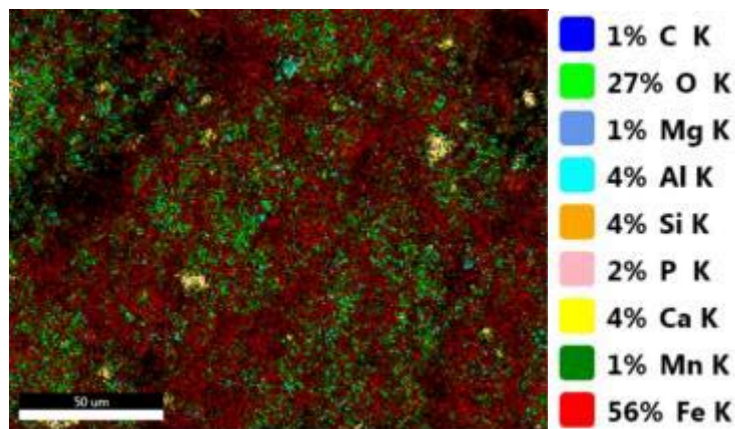


Figure 12. Mapping of ground sample before DSC

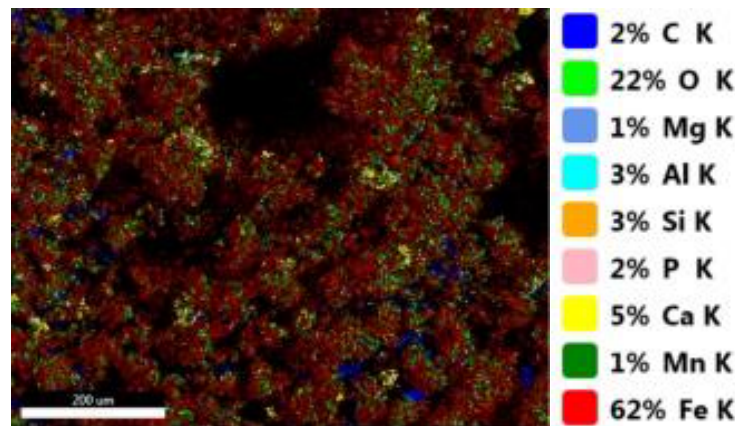


Figure 13. Mapping of ground sample after DSC

5. Conclusions

Gara Djebilet, a high-phosphorus iron ore, was analyzed using a range of characterization techniques, including X-ray fluorescence (XRF), X-ray diffraction (XRD), scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDS), and differential scanning calorimetry (DSC). These analyses were conducted to determine the distribution of phosphorus within the ore, which is critical for developing an effective beneficiation strategy for phosphorus removal. The results confirm a phosphorus concentration of 1.53 wt%, with its spatial distribution observed in both iron-rich phases and gangue minerals. However, XRD results did not reveal any distinct phosphorus-bearing phases, suggesting that phosphorus may be present in an amorphous form, as interstitial inclusions within the iron oxide/hydroxide structure, or within the structure of aluminosilicate gangue minerals.

6. Recommendations and perspectives

High-phosphorus oolitic iron ores pose significant challenges in mineral processing due to the detrimental effects of phosphorus on the metallurgical properties of

the final products. Conventional methods, such as magnetic separation, gravimetric separation, and flotation, are often ineffective or economically impractical for these ores. Therefore, it is essential to explore innovative strategies, including biological and chemical approaches, for the selective removal of phosphorus. In addition, the potential presence of rare earth elements (REEs) in these ores offers further opportunities for valorization. This article provides insights and recommendations for evaluating alternative processing methods, with an emphasis on emerging technologies and REE characterization.

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Karakterizacija oolitske rude gvožđa iz ležišta Gara Džebilet u Alžiru: mineralogija, hemijski sastav, morfologija i termička svojstva

Messai Ali ^{a, #}, Lafdil Sana ^b, Boustila Amir ^a, Allem Habiba ^c, Bouzeriba Hadjer ^d, Juan M. Menéndez-Aguado ^e, Mebarkia Mohammed ^{f, g}

^a Nacionalna politehnička škola u Alžiru, Laboratorija za rudarstvo, Departman za rudarstvo, Alžir, Alžir

^b Nacionalna visoka škola za tehnologiju i inženjerstvo, Departman za rudarstvo, metalurgiju i inženjerstvo materijala, Anaba, Alžir

^c Univerzitet Badži Muhtar u Anabi, Departman za geologiju, Anaba, Alžir

^d Univerzitet Abderrahmane Mira, Departman za rudarstvo i geologiju, Bežaja, Alžir

^e Univerzitet u Ovijedu, Politehnička škola u Mieresu, Istraživačka grupa za prerađu mineralnih sirovina, Španija

^f Univerzitet Badži Muhtar–Anaba, Departman za rudarstvo, Anaba, Alžir

^g University of Echahid Echikh Larbi Tebessi, Mining Institute, Environment Laboratory, Tebessa, Algeria

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I Z V O D

Ležišta rude gvožđa Gara Džebilet, koja se nalaze u regionu Tinduf u Alžiru, imaju značajan društveno-ekonomski značaj među domaćim ležištima rude gvožđa otkrivenim u ovoj zemlji. Da bi se zadovoljile potrebe domaće proizvodnje čelika, neophodno je efikasno obogaćivanje ovih ruda, što zahteva njihovu detaljnu mineralošku, hemijsku i morfološku karakterizaciju. U tu svrhu primenjene su optička mikroskopija, skenirajuća elektronska mikroskopija sa energetsko-disperzivnom rendgenskom spektroskopijom (SEM-EDS), rendgenska fluorescentna spektrometrija (XRF), rendgenska difrakcija (XRD), kao i diferencijalna skenirajuća kalorimetrija i termogravimetrijska analiza (DSC-TG). Mineraloška ispitivanja pokazala su da se ruda gvožđa iz ležišta Gara Džebilet pretežno sastoji od hematita i getita. XRF analizom utvrđeni su sadržaji od 61,95 % Fe, 4,27 % SiO₂, 2,22 % CaO, 4,72 % Al₂O₃ i 1,53 % P₂O₅. Rezultati različitih analiza ukazuju na to da je fosfor fino dispergovan u hematitnoj fazi, što ukazuje na to da ruda gvožđa iz ležišta Gara Džebilet možda nije pogodna za obogaćivanje konvencionalnim postupcima.