

Article

Selective Removal of Iron from Ferruginous Manganese Ore by Low-Temperature Magnetizing Roasting and Dry Magnetic Separation

Alibek Baisanov, Nina Vorobkalo * , Askhat Akuov, Yerulan Samuratov, Amir Makishev, Symbat Sharieva and Zhanna Ibrakhimova

Laboratory of Pyrometallurgical Processes, Zh. Abishev Chemical-Metallurgical Institute, Karaganda 100009, Kazakhstan; alibekbaisanov-chmi@mail.ru (A.B.); akuov.am@mail.ru (A.A.); yerulansamuratov2@gmail.com (Y.S.); amir.makishev.99.99@mail.ru (A.M.); shariyeva.symbat@mail.ru (S.S.); zhanna.ibrahimova@mail.ru (Z.I.)

* Correspondence: nina.timirbaeva23@gmail.com

Abstract

The beneficiation of ferruginous manganese ores is limited by the intimate intergrowth of manganese-, iron-, and silicate-bearing phases. This study evaluated coal-based magnetizing roasting followed by dry magnetic separation in an externally heated chamber furnace with a charge capacity of up to 100 kg. A 0–5 mm ore fraction with an initial Mn/Fe ratio of 2.9 was roasted with Shubarkol coal. The best separation was obtained at an actual ore–coal bed temperature of 550–600 °C and an ore-to-coal mass ratio of 1:0.4. Relative to the magnetic-separation feed, 80.0–83.4% of Fe was recovered in the magnetic fraction, while 69.9–72.6% of Mn remained in the non-magnetic product. Its Fe content decreased to 3.2–3.5%, increasing the Mn/Fe ratio to 7.30–7.84. X-ray diffraction showed preferential concentration of magnetite and jacobsite in the magnetic fraction, whereas hausmannite and braunite were concentrated mainly in the non-magnetic fraction together with the gangue phases. Multipoint measurements also demonstrated a substantial difference between the combustion-zone and actual bed temperatures. The results demonstrate that controlled low-temperature roasting can generate sufficient magnetic contrast for selective iron removal from ferruginous manganese ore.

Keywords: ferruginous manganese ore; magnetizing roasting; dry magnetic separation; iron removal; Mn/Fe ratio; phase transformation; temperature monitoring



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1. Introduction

Interest in the metallurgical processing of low-grade manganese ores, wastes, and other secondary manganese-bearing materials has increased in recent years. This trend is driven by the declining availability of high-grade raw materials and the need to reduce solid-waste generation, pressure on tailings storage facilities, and the risk of heavy-metal contamination [1–4]. The feasibility of integrated processing is further supported by the possibility of producing a range of useful metallurgical products from these materials [5,6].

Ferruginous manganese ores are refractory raw materials whose technological value is determined by their Mn and Fe contents, Mn/Fe ratio, mineral composition, and degree of mineral liberation. At low Mn/Fe ratios, their direct use in ferroalloy production is limited because excessive iron decreases the quality of the manganese product [7,8]. Therefore, pretreatment should promote selective iron removal and increase the Mn/Fe ratio [7,8].

Associated elements may also be redistributed during selective reduction and separation [9], while process efficiency depends strongly on the mineralogical and textural characteristics of the raw material [7,8,10–12].

Dry magnetic separation is attractive because it requires neither water nor complex reagent systems, although its efficiency strongly depends on the mineralogical forms of iron [12–14]. Separation selectivity can be improved by magnetizing roasting, during which weakly magnetic iron-bearing minerals are converted into magnetically susceptible phases and subsequently recovered in the magnetic fraction, while manganese-bearing components are predominantly retained in the non-magnetic product. The objective is therefore to create sufficient magnetic contrast between Fe- and Mn-bearing phases rather than to achieve deep reduction of all ore components [13–16]. Interpretation of this process requires consideration of the resulting phase assemblage and its redistribution during magnetic separation [17,18].

The efficiency of magnetizing roasting depends on temperature, duration, reductant consumption, particle size, reducing atmosphere, and magnetic-separation conditions. Reduction roasting followed by magnetic separation has been shown to substantially increase the Mn/Fe ratio through conversion of hematite and goethite to magnetite and subsequent iron removal [7,15]. Studies on suspension magnetizing roasting further demonstrate that Mn–Fe separation is highly sensitive to roasting conditions [19,20]. Liu et al. [19] reported efficient separation at an optimum temperature of 560 °C, whereas higher temperatures can promote the formation of mixed magnetic Mn–Fe spinels and decrease selectivity [16,21]. These findings support the use of moderate-temperature roasting for selective iron magnetization rather than deep reduction of both Fe- and Mn-bearing phases. The target temperature range in the present study was therefore selected from the literature evidence and further refined from the thermal behavior of the investigated ore–coal mixture.

However, increasing the roasting temperature does not necessarily improve Mn–Fe separation. Excessive heating can promote sintering and the formation of mixed Mn–Fe-containing phases, reducing mineral liberation and magnetic selectivity. At temperatures above 800 °C, increased iron recovery may be accompanied by lower manganese selectivity due to the formation of magnetic $\text{Mn}_x\text{Fe}_{3-x}\text{O}_4$ phases [16]. Composite Mn–Fe oxides and difficult-to-reduce aluminate phases can further limit separation [21]. These effects favor the use of a controlled low-temperature regime.

The use of coal as a reductant adds further complexity because reduction proceeds through both solid carbon and gaseous coal-pyrolysis products. Volatile components generate a reducing atmosphere that promotes transformation of iron-bearing minerals, while coal may simultaneously act as a reductant and fuel in externally heated furnaces. Consequently, coal consumption, ore-to-coal ratio, heating conditions, and fuel-feeding regime are interrelated process parameters [14,19,20].

Most studies of ferruginous manganese ore reduction roasting have been conducted with small batches under relatively homogeneous heat- and mass-transfer conditions. Scaling up to an enlarged laboratory furnace introduces additional thermal heterogeneity: the combustion-zone temperature may differ substantially from the actual material temperature, while different portions of the charge may experience different thermal histories. Therefore, evaluation of the roasting regime requires consideration of the actual material temperature field in addition to the final separation results. The influence of these factors on phase transformations and Mn–Fe separation at an enlarged laboratory scale remains insufficiently studied.

This study tested the hypothesis that an operating regime combining an actual ore–coal bed temperature of approximately 550–600 °C with an ore-to-coal mass ratio of 1:0.4 promotes the preferential conversion of weakly magnetic iron-bearing phases into magneti-

cally susceptible spinel phases while limiting manganese incorporation into the magnetic product. The novelty of this study lies in evaluating coal-based magnetizing roasting under enlarged laboratory conditions using an externally heated chamber furnace with a charge capacity of up to 100 kg. Unlike conventional small-batch roasting experiments, this approach enabled direct evaluation of the relationship between the measured ore–coal bed temperature, temperature variations across the monitored furnace zones and over time, furnace thermal inertia, phase transformations, and subsequent magnetic-separation performance. The obtained information is relevant to further scale-up and process control of magnetizing roasting.

The aim of this study was to identify an appropriate operating regime for coal-based magnetizing roasting of ferruginous manganese ore in a chamber furnace with a capacity of up to 100 kg and to evaluate its effect on phase transformations, Mn–Fe separation, component recoveries, and the Mn/Fe ratio of the resulting manganese-bearing product.

2. Materials and Methods

The experimental workflow comprised raw-material preparation and characterization, thermal characterization, large-scale magnetizing roasting with temperature monitoring, and subsequent dry magnetic separation and product characterization.

2.1. Raw Materials

The raw material was ferruginous manganese ore from the Ushkatyn III deposit (Zhanaarka District, Ulytau Region, Kazakhstan). The as-received ore was crushed and subsequently screened, and the 0–5 mm size fraction was collected for the roasting experiments. This particle-size range was selected to provide adequate contact between the ore particles and the carbonaceous reductant and to obtain a product suitable for subsequent dry magnetic separation.

The chemical compositions of the raw ore and magnetic-separation products were determined in the analytical laboratory of the Zh. Abishev Chemical-Metallurgical Institute. The major oxide components were determined by X-ray fluorescence analysis using a MAX-GVM spectrometer (Spektron SPA, St. Petersburg, Russia). The total manganese content was determined potentiometrically according to GOST 22772.2-96 [22], while total iron was determined using X-ray fluorescence analysis and additionally verified by chemical analysis according to GOST 22772.4-96 [23]. The carbon content of the roasting and magnetic-separation products was determined by the coulometric method after high-temperature combustion in an oxygen atmosphere using an AN-7529 carbon analyzer (Izmeritel Production Association, Gomel, Belarus), according to GOST 23581.9-79 [24]. Phosphorus and sulfur in the raw ore were determined according to GOST 22772.6-96 [25] and GOST 22772.7-96 [26], respectively. Loss on ignition was determined gravimetrically according to GOST 28077-89 [27].

The chemical composition of the raw ore is presented in Table 1.

Table 1. Chemical composition of raw ore.

Content, wt.%									Mn/Fe
Mn _{total}	Fe _{total}	SiO ₂	CaO	MgO	Al ₂ O ₃	P	S	Loss on Ignition	
17.81	6.16	41.3	1.46	1.25	5.92	0.034	0.025	8.79	2.9

The raw ore contained a moderate amount of manganese and a relatively high amount of silica. Its Mn/Fe ratio was approximately 2.9; such a relatively low Mn/Fe ratio limits

its direct use as a manganese raw material and motivates selective removal of iron prior to subsequent metallurgical processing [7,8,13].

Shubarkol coal (Karaganda region, Kazakhstan) was used as a carbonaceous reducing agent in low-temperature magnetizing roasting. Its use is due to its high yield of volatile components and its ability to form a reducing gas environment upon heating. Thermal decomposition of the coal releases gaseous reducing agents, including CO, H₂, and CH₄, which can participate in the reduction of iron oxides to magnetic phases. Furthermore, the solid carbon of the coal participates in the contact reduction of iron-bearing minerals.

The proximate analysis of Shubarkol coal was performed according to standard methods: total moisture according to GOST ISO 589-2012 [28], ash content according to GOST ISO 1171-2012 [29], volatile matter according to GOST 6382-2001 [30], and total sulfur according to GOST 2059-95 [31]. The chemical composition of the coal ash was determined according to GOST 10538-87 [32]. The gas released during thermal treatment of Shubarkol coal was analyzed using a Chromatec-Crystal 5000 gas chromatograph (Chromatec, Yoshkar-Ola, Russia). The main technical parameters of Shubarkol coal are presented in Table 2.

Table 2. Proximate analysis and calorific value of Shubarkol coal.

W_t^r , wt. %	A^d , wt. %	V^{daf} , wt. %	S_t^d , wt. %	Q , MJ/kg
4.8	9.85	36.0	0.5	31.9

Notation: W_t^r total moisture as received; A^d ash content on a dry basis; V^{daf} volatile matter on a dry ash-free basis; S_t^d total sulfur on a dry basis; Q , calorific value.

The chemical composition of the coal ash and the released gas are presented in Table 3.

Table 3. Chemical composition of ash and gas phase of Shubarkol coal.

Chemical composition of coal ash, wt. %							
SiO ₂ 56.97	Al ₂ O ₃ 21.3	Fe ₂ O ₃ 7.19	CaO 2.79	MgO 1.77	P ₂ O ₅ 0.46	K ₂ O 1.25	Na ₂ O 1.85
Gas composition, vol. %							
H ₂ S 1.54	CO ₂ 22.43	C _n H _m 0.5	CO 14.02	CH ₄ 28.32	H ₂ 29.76	O ₂ 0.5	N ₂ 2.91

2.2. Characterization Methods

To preliminarily characterize the source materials, X-ray diffraction analysis and differential thermal analysis were performed. X-ray diffraction analysis was used to identify the mineral phases of the ferruginous manganese ore. Differential thermal analysis was used to evaluate the thermal behavior of the ore upon heating, as well as to establish the temperature ranges for the physicochemical transformations occurring in the material under study.

X-ray diffraction analysis of the raw ore and magnetic separation products was performed on a Rigaku MiniFlex 600 diffractometer (Rigaku Corporation, Akishima, Tokyo, Japan) using Cu-K α radiation. The operating conditions were 40 kV and 15 mA. The diffraction pattern of the raw ore was recorded in the 2 θ range from 5 to 100° at a scan rate of 3°/min, while the magnetic and non-magnetic fractions were analyzed in the 2 θ range from 18 to 80° at a scan rate of 6°/min. In all cases, the scanning step was 0.01°. Crystalline phases were identified using Match! version 4 (Crystal Impact, Bonn, Germany) with the Crystallography Open Database (COD) as the reference diffraction database. Semi-quantitative phase analysis was performed using the reference intensity ratio (RIR)

method implemented in Match!. Corresponding JCPDS/PDF reference-card numbers are additionally provided in the diffraction figures for phase labeling.

Combined thermogravimetric and differential thermal analysis was performed on a Q-1500D (MOM, Budapest, Hungary) derivatograph in the range of 20–1150 °C in an oxidizing air atmosphere. The heating rate was 10 °C/min. The sample mass change (TG), the rate of mass change (DTG), and the differential thermal effect (DTA) were simultaneously recorded. The raw ore and the ore–coal mixture were studied, which made it possible to compare the thermal behavior of the mineral raw materials without a reducing agent and in the presence of coal. Each 600 mg sample was placed in a high-alumina corundum crucible (10 mm in diameter and 12 mm in height; approximately 99.8% Al₂O₃), suitable for derivatographic measurements up to 1200 °C [33].

Representative ore fragments were mounted, mechanically ground, and polished using progressively finer abrasives to obtain a smooth reflective surface suitable for reflected-light microscopy. Mineralogical and petrographic examination of the raw ore was conducted in reflected light using a Neophot-21 microscope (VEB Carl Zeiss Jena, Jena, Germany) at ×200 and ×500 magnifications. The study aimed to assess the textural and structural heterogeneity of the ore, the size of the ore grains, and the nature of the intergrowth of manganese-, iron-, and silicon-containing components.

2.3. Large-Scale Laboratory Magnetizing Roasting Tests and Temperature Monitoring

Large-scale laboratory tests of magnetizing roasting were conducted in a chamber furnace with an external heating system and a capacity of up to 100 kg. The design and appearance of the setup are shown in Figure 1.

The working chamber had dimensions of 1140 mm in height, 540 mm in length, and 250 mm in width. The overall dimensions of the furnace were approximately 1140 mm × 860 mm × 950 mm (height × length × width). The furnace consisted of a vertical working chamber designed for loading the ore–coal mixture and an adjacent firebox separated from the working chamber by a common wall. The furnace had a steel structural shell, while the thermally exposed sections of the working chamber and combustion zone were lined with refractory fireclay brick.

The working chamber was heated by the combustion of Shubarkol coal and gaseous pyrolysis products in the combustion zone. No external reducing gas was supplied to the working chamber. The reducing atmosphere was generated in situ within the ore–coal bed by gaseous coal-pyrolysis products, mainly CO, H₂, and CH₄, together with the action of residual solid carbon. The generated gases were removed through the furnace exhaust system. The furnace design allowed external heating of the ore–coal material, periodic fuel charging, additional loading of the feed mixture, and unloading of the roasted product during extended operation.

Before each experimental series, the furnace was preheated. The ore and Shubarkol coal were separately crushed and screened to the 0–5 mm fraction, weighed according to the required ore-to-coal mass ratio (1:0.2 or 1:0.4), mixed, and charged into the working chamber. The coal mixed directly with the ore acted as a reducing agent through both its gaseous pyrolysis products and residual solid carbon. During heating, CO, H₂, CH₄, and other volatile components contributed to the reducing atmosphere within the ore–coal bed, while the solid carbonaceous residue acted as an additional contact reductant. Additional Shubarkol coal of the same 0–5 mm particle-size fraction was used separately as furnace fuel. The specified ore-to-coal ratios refer only to the reductant mixed with the ore and exclude the fuel coal.

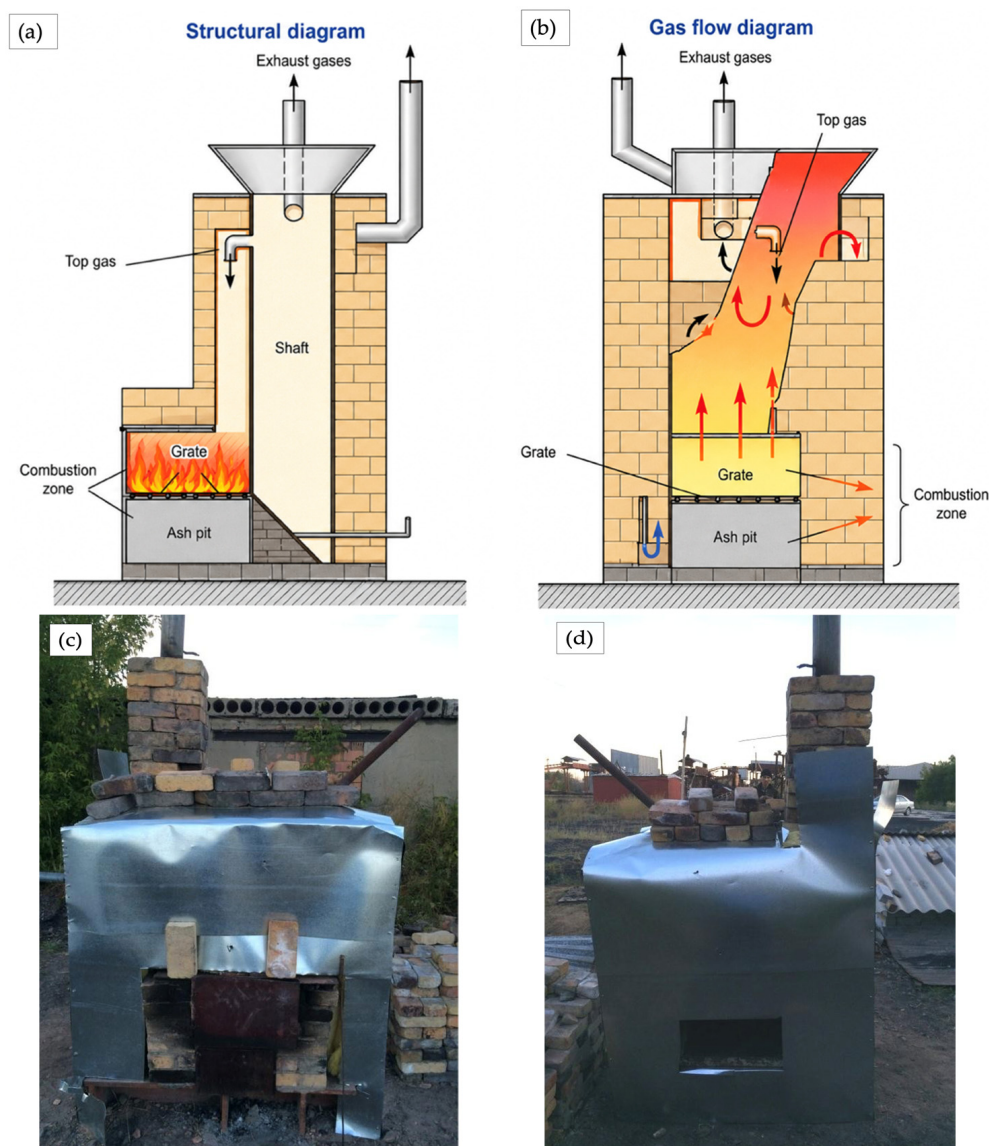


Figure 1. Large-scale laboratory chamber furnace with external heating and a capacity of up to 100 kg: (a) structural schematic; (b) gas-flow diagram; (c,d) photographs of the furnace.

The furnace was operated with periodic charging and discharge of material. In Series 1, 156 kg of ore and 31.3 kg of reductant coal were processed over 24 h, while approximately 120 kg of Shubarkol coal was consumed as furnace fuel. In Series 2, an initial 100.75 kg ore–coal mixture was loaded; fuel coal was subsequently supplied in approximately 30 kg portions at 4–5 h intervals, with a total fuel consumption of approximately 350 kg during the 48 h test. In Series 3, the initial ore–coal mixture and fuel charges were 70 and 30 kg, respectively; additional feed was introduced during the long-term test as the bed settled. Roasted material was periodically discharged from the lower part of the working chamber and sampled for subsequent screening and magnetic separation. For the approximately 400 °C condition, samples collected from different levels of the ore–coal bed were analyzed separately.

The temperature regime was monitored using TVR (A-1) thermocouples connected to a TRM-138 eight-channel measurement and control unit (OWEN Production Association LLC, Moscow, Russia). The specified measurement error of the TRM-138 for thermocouple inputs is $\pm 0.5\%$. During the long-term test series, temperatures were recorded at seven control points: D1, combustion zone; D2, ore–coal material in the working chamber; D3,

lower part of the rear wall; D4, left wall; D5, upper part of the front wall; D6, upper part of the rear wall; and D7, chimney. Thermocouple D2 was inserted directly into the central part of the ore–coal bed in the working chamber, with the measuring junction fully immersed in the material and separated from the chamber walls. In contrast, D3–D6 monitored different sections of the furnace walls and characterized heat transfer through the working chamber, while D1 and D7 characterized the combustion zone and chimney, respectively. Thus, the thermal regime was evaluated from the combined multipoint temperature response, while D2 provided the directly measured material temperature at the monitored central location. Because only one thermocouple was immersed directly in the ore–coal bed, these measurements do not establish spatial temperature uniformity throughout the entire bed.

Multipoint temperature recording made it possible to evaluate the temperature difference between the combustion and working zones, the thermal inertia of the ore–coal bed, temperature changes caused by additional material and fuel charging, and furnace stability during long-term operation.

Magnetizing roasting was conducted in three consecutive test series that differed in duration, reductant consumption, and temperature range. The first series provided a preliminary assessment of the feasibility of magnetizing the iron-bearing components at a lower temperature and lower coal consumption. In the second series, reductant consumption was increased, and the operating temperature range was determined. The third series was conducted over six days to evaluate furnace stability and the spatial and temporal temperature response across the monitored furnace zones. The main test conditions are presented in Table 4.

Table 4. Operating conditions for large-scale laboratory magnetizing roasting of ferruginous manganese ore.

Series	Temperature, °C	Duration	Ore:Coal Ratio	Target
1	250–400	24 h	1: 0.2	Assessment of magnetizing roasting at a lower temperature and lower reductant consumption
2	220–565	48 h	1: 0.4	Selection of the operating conditions for low-temperature magnetizing roasting
3	230–780	6 days	1: 0.4	Evaluation of process stability during long-term furnace operation

Because the furnace was operated with periodic discharge and recharging, the durations given in Table 4 represent the total duration of each test series rather than a fixed holding time for a single batch.

The reported temperature ranges represent the values recorded during the corresponding test series. The actual ore–coal material temperature was measured by thermocouple D2, whereas the combustion-zone temperature was recorded by thermocouple D1. The ore-to-coal ratios listed in Table 4 refer to the reductant mixed directly with the ore.

2.4. Dry Magnetic Separation and Performance Evaluation

After roasting, the product consisted of roasted ferruginous manganese ore and semi-coke formed during the thermal decomposition of Shubarkol coal. Before magnetic separation, coarse semicoke was removed by screening on a 5 mm sieve, and the resulting < 5 mm material was used as the separation feed. Each collected sample was processed separately in a single pass through the magnetic separator. The amount processed depended on the mass of the sample collected from the corresponding roasting condition; therefore,

separation was performed at a controlled throughput of 10–20 kg/h rather than at a fixed test mass.

Dry magnetic separation was performed using a 120-T-type large-scale laboratory magnetic separator (Yueyang, China) designed for the dry separation of weakly magnetic ores. The separator throughput was 10–20 kg/h. The magnetic field strength was maintained at 79.6–95.5 kA/m (1000–1200 Oe), corresponding to a transformer current of 10–12 A.

The performance of dry magnetic separation was evaluated from the product yields, major-component contents, Mn/Fe ratio, and distribution of Mn, Fe, SiO₂, and carbon between the magnetic and non-magnetic fractions. Because the mass and composition of the material changed during roasting and the subsequent removal of coarse semicoke, component recovery was calculated relative to the magnetic-separation feed using a closed two-product balance for the magnetic and non-magnetic fractions:

$$\varepsilon_{i,j} = \frac{\gamma_j \cdot \beta_{i,j}}{\gamma_m \cdot \beta_{i,m} + \gamma_{nm} \cdot \beta_{i,nm}} \cdot 100, \quad (1)$$

where $\varepsilon_{i,j}$ is the recovery of component i in fraction j , %; γ_j is the yield of fraction j , %; $\beta_{i,j}$ is the content of component i in fraction j , %; and the subscripts m and nm denote the magnetic and non-magnetic fractions, respectively.

For each experiment, the sum of the yields of the magnetic and non-magnetic fractions was taken to be 100%, and the sum of the recoveries of the corresponding component in both products was 100%.

Accordingly, the recovery values reported in this study refer to the material entering magnetic separation after roasting and removal of coarse semicoke. They characterize the distribution of Mn and Fe between the magnetic and non-magnetic products. An overall mass balance relative to the initial ore was not evaluated in the present study.

The manganese-to-iron ratio was used as an indicator of the selectivity of iron removal from the manganese-bearing product and was calculated as follows:

$$Mn/Fe = \frac{C_{Mn}}{C_{Fe}}, \quad (2)$$

where C_{Mn} and C_{Fe} are the mass contents of manganese and iron in the corresponding fraction, %.

3. Results and Discussion

3.1. Mineralogical and Microstructural Characteristics of the Raw Ore

X-ray diffraction analysis showed that the raw ferruginous manganese ore had a polymineral composition. Its X-ray diffraction pattern is shown in Figure 2.

The main mineral components were quartz SiO₂ and pyrolusite MnO₂. Kaolinite Al₂Si₂O₅(OH)₄, hematite Fe₂O₃, and calcite CaCO₃ have been identified in smaller quantities, while braunite Mn₇SiO₁₂ is present as a subordinate phase. Thus, the raw ore has a predominantly quartz–pyrolusite composition with the presence of aluminosilicate, iron oxide, carbonate, and manganese silicate components.

The presence of hematite is important for subsequent magnetizing roasting because its reduction to magnetically susceptible iron-bearing phases provides the basis for selective iron removal during magnetic separation.

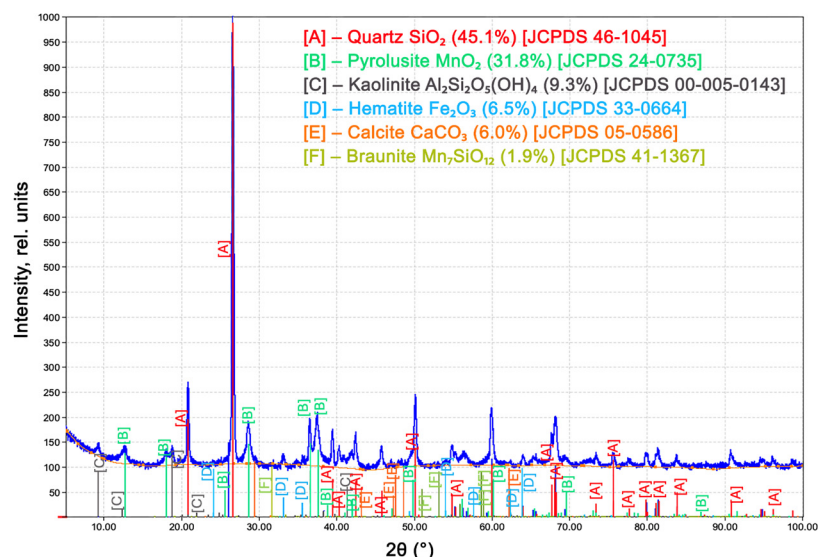


Figure 2. Diffraction pattern of the raw ore.

The reflected-light microstructure of the raw ore is shown in Figure 3. The samples exhibited a heterogeneous fine-grained structure with an uneven distribution of regions having different reflectance (Figure 3a). The lighter regions corresponded predominantly to oxide-rich areas, whereas the dark-gray regions were associated with the quartz–aluminosilicate gangue matrix. However, individual manganese- and iron-bearing phases could not be identified unambiguously from optical contrast alone because of their fine-grained occurrence and similar reflectance. The micrographs were therefore interpreted together with the X-ray diffraction results.

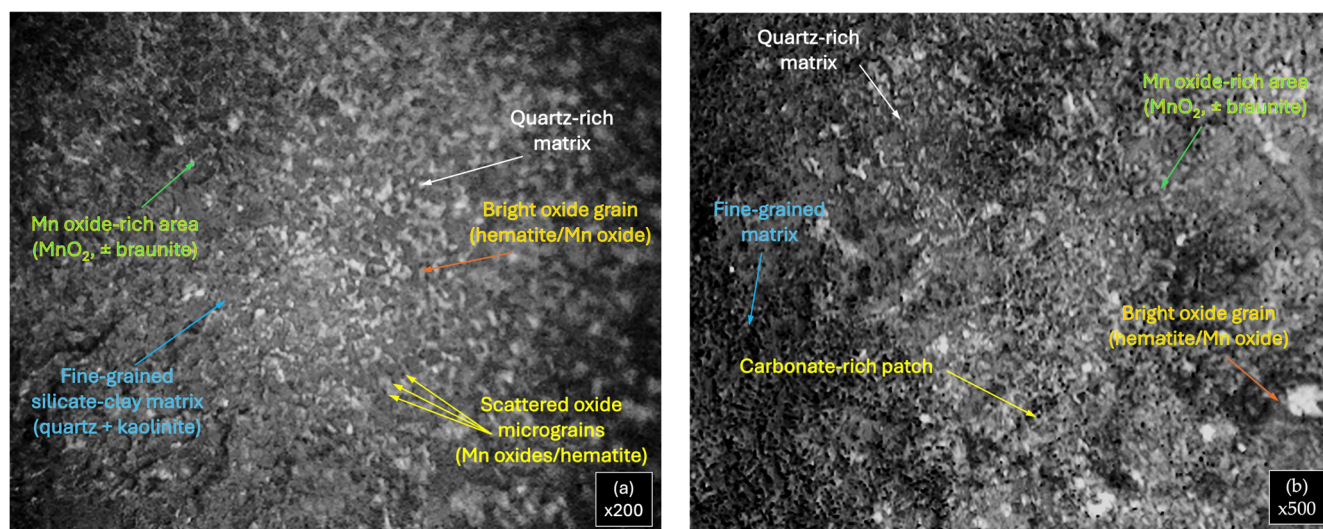


Figure 3. Microstructure of the raw ferruginous manganese ore in reflected light: (a) $\times 200$; (b) $\times 500$.

The oxide components occurred as small isometric and elongated grains and aggregates that were unevenly distributed within the quartz–aluminosilicate matrix (Figure 3b). Discrete coarse grains of ore minerals were rare, and a substantial proportion of the oxide components occurred in fine-grained, tightly intergrown aggregates. Braunite, which was present in small amounts according to the X-ray diffraction data, was likely associated with dispersed manganese-bearing regions and did not form clearly distinguishable coarse grains. Calcite occurred predominantly as fine intergranular and cementing material.

Thus, the combined phase and microstructural analyses showed that the raw ore was characterized by intimate intergrowths among manganese-, iron-, and silicate-bearing components. This structure limits the effectiveness of direct magnetic separation of the starting material, since iron-bearing phases are present not only in the form of individual grains, but also as part of fine aggregates with manganese minerals and a non-metallic matrix. The results obtained justify the use of preliminary magnetizing roasting, aimed at increasing the magnetic susceptibility of iron-bearing phases before dry magnetic separation.

3.2. Thermal Behavior of the Ore and Ore–Coal Mixture

The thermal behavior of the raw ferruginous manganese ore and the ore–coal mixture was evaluated using overlaid TG, DTG, and DTA curves. Comparison of the derivatograms allowed us to determine the temperature ranges for moisture removal, mineral phase transformations, and thermal transformation of the coal component (Figure 4).

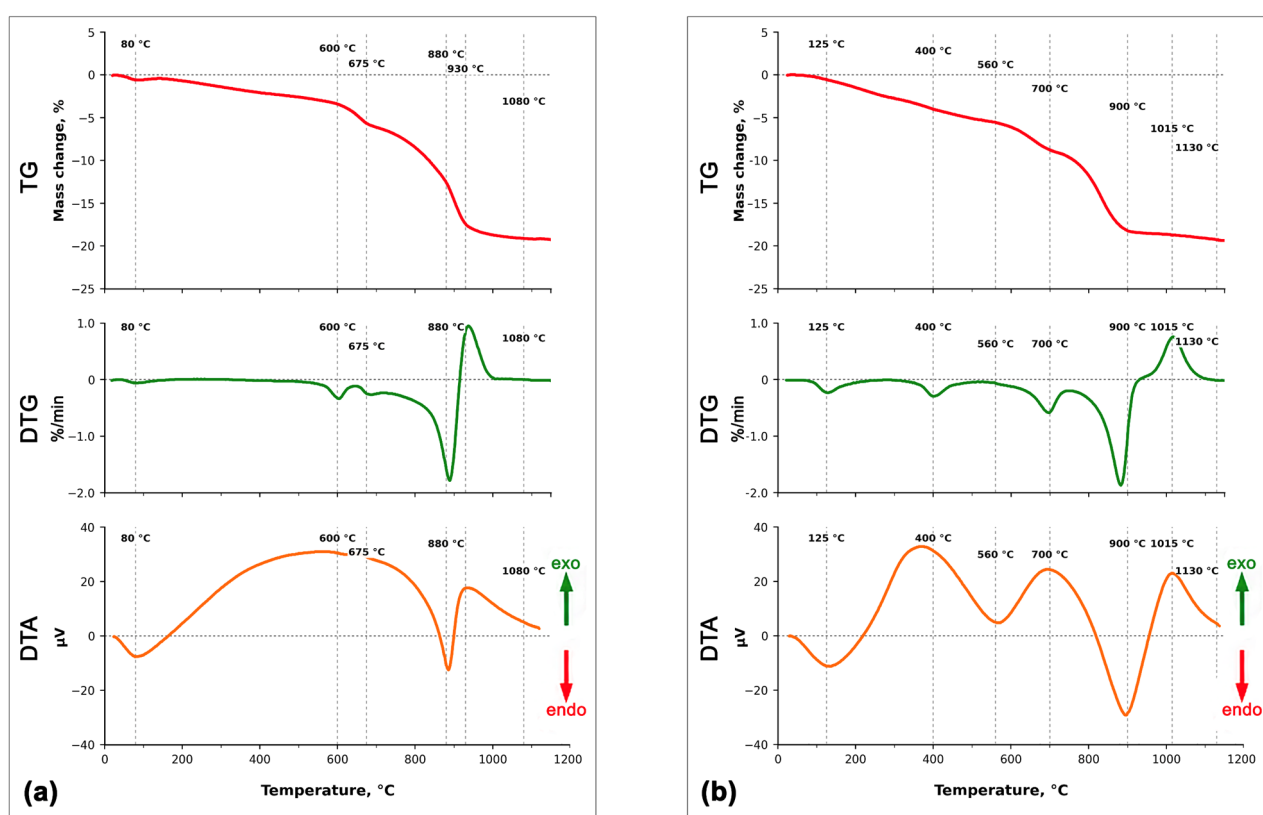
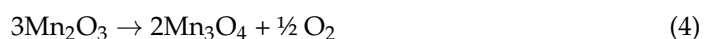


Figure 4. TG, DTG and DTA curves of the studied samples: (a) raw ferruginous manganese ore; (b) ore–coal mixture.

In the derivatogram of the raw ore, the first endothermic effect at 80–125 °C, accompanied by mass loss, corresponded to the removal of physically adsorbed and hygroscopic moisture. A broad region of variation in the TG, DTG, and DTA curves was observed between 400 and 700 °C, with the most pronounced effects occurring at 600–675 °C. Considering the XRD-determined phase composition of the ore, these changes can reasonably be associated with overlapping transformations of several mineral components. Kaolinite dehydroxylation occurs within this general temperature region and is accompanied by the removal of structurally bound water [34]. In parallel, higher manganese oxides undergo stepwise thermal decomposition to lower oxides, including the $\text{MnO}_2 \rightarrow \text{Mn}_2\text{O}_3$ and $\text{Mn}_2\text{O}_3 \rightarrow \text{Mn}_3\text{O}_4$ transformations [35]:





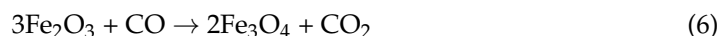
Because these transformations overlap within the temperature interval observed in the present TG–DTG–DTA curves, the individual thermal effects cannot be uniquely assigned to a single-phase reaction. Equations (3) and (4) therefore represent established transformation pathways of manganese oxides that are consistent with the phase composition of the studied material rather than individually resolved reactions in the recorded curves.

The pronounced mass change and thermal effect observed in the 880–930 °C region are consistent with the decomposition of calcite identified in the raw ore by XRD. The temperature of CaCO_3 decomposition is strongly affected by the heating conditions and surrounding CO_2 partial pressure [36]:



Simultaneously, the structural rearrangement of the oxide–silicate components and their solid-phase interactions intensify. The thermal behavior of the ore–coal mixture differed because of the presence of Shubarkol coal. The endothermic effect at 100–125 °C corresponded to moisture removal. Above 350–400 °C, changes in the curves were associated with thermal decomposition and oxidation of the organic coal component, volatile-matter release, formation of a carbonaceous residue, and transformations of the mineral component. The most intense changes occurred at 560–700 °C, where the thermal transformations of coal overlapped with those of the mineral phases. Since the thermal analysis was conducted in air, these effects characterize the overall thermal behavior of the mixture, while under the reducing conditions of chamber-furnace roasting the relative contribution of individual reactions may differ.

In the chamber furnace, the reducing environment within the ore–coal bed is formed by the combined action of gaseous coal-pyrolysis products and residual solid carbon. Under these conditions, CO and H_2 can promote the conversion of hematite into magnetite:



At the same time, it is possible to transform higher manganese oxides into hausmannite:



These reactions represent probable transformation pathways under the reducing conditions of chamber-furnace roasting, as reported for the reduction and magnetizing roasting of ferruginous manganese ores [13,16,19–21], and are consistent with the phase changes subsequently observed by X-ray diffraction.

Thus, the most technologically significant transformations of the ore–coal mixture occur in the range of 550–700 °C, while at higher temperatures, the likelihood of recrystallization and sintering of the material increases. Taking into account the thermal inertia of a large-scale laboratory furnace and the need to maintain product dispersion for subsequent separation, the target range of the actual temperature of the ore–coal material was set at 550–600 °C.

3.3. Temperature Behavior in the External-Heating Chamber Furnace

Based on the thermal-analysis results, the target actual temperature of the ore–coal material was set at 550–600 °C. To assess whether this range could be achieved under enlarged laboratory conditions, the temperature response of the combustion zone and

ore–coal bed was analyzed during charge loading, heating, and periodic fuel addition (Figure 5).

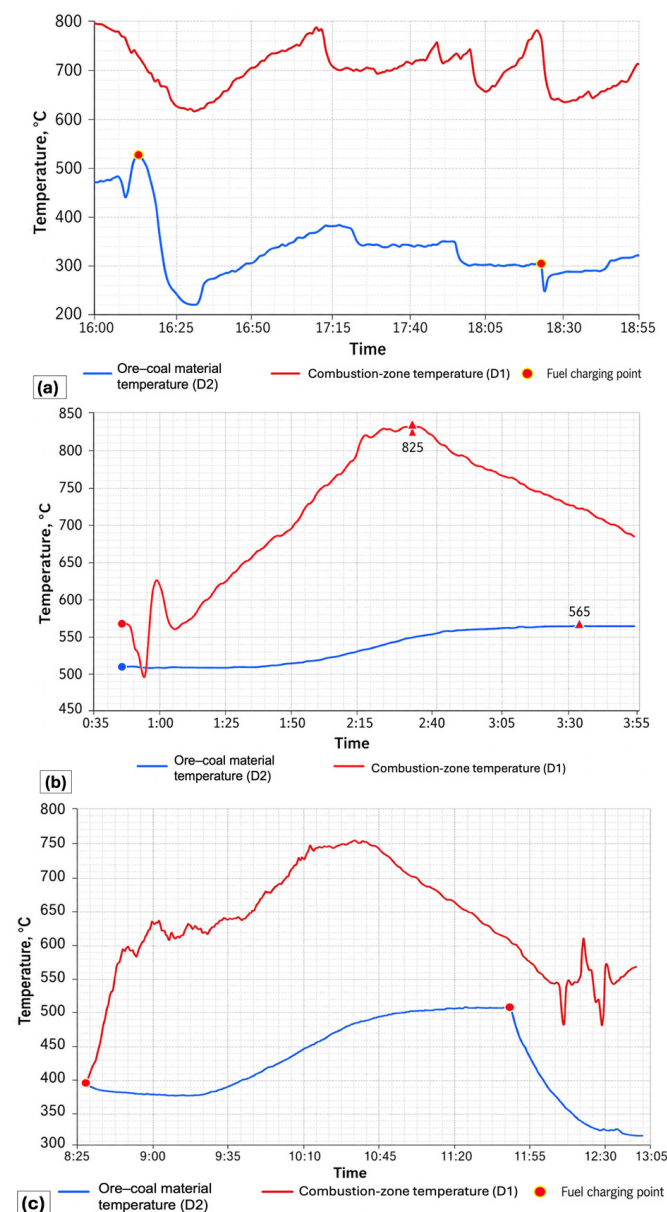


Figure 5. Change in temperature of ore–coal material and combustion zone during large-scale laboratory roasting: (a) temperature response after loading of charge; (b) reaching the maximum temperature of the material; (c) change in temperature between successive fuel loadings.

After loading, the material temperature decreased because of the introduction of the cold charge and subsequently increased as heat was transferred from the combustion zone and furnace walls to the ore–coal bed.

The temperature profiles revealed a substantial difference between the combustion zone and the processed material. In one experiment, D1 reached 825 °C, whereas the maximum temperature recorded directly in the ore–coal bed at D2 was approximately 565 °C, corresponding to a difference of about 260 °C. This difference reflects the external-heating configuration, thermal resistance between the combustion and working zones, and the heat absorbed during heating and transformation of the charge.

Charge and fuel additions produced different temporal responses in the combustion zone and ore–coal bed. Fuel charging caused a relatively rapid change in D1, reflecting

the successive heating, volatile release, and combustion of the added coal, whereas D2 responded more gradually because heat had to be transferred into the material bed. Similarly, after addition of a fresh ore–coal charge, D2 decreased and subsequently recovered more slowly than the combustion-zone temperature. The relative response of D1 and D2 therefore indicates that changes in combustion intensity precede the thermal response of the material, while sustained heating of the ore–coal bed, rather than short-term combustion-temperature peaks, governs the conditions for phase transformation.

To assess the temperature field during long-term furnace operation, multi-point temperature recording was used. The changes in thermocouple readings D1–D7 during selected periods of the six-day experiment are shown in Figure 6.

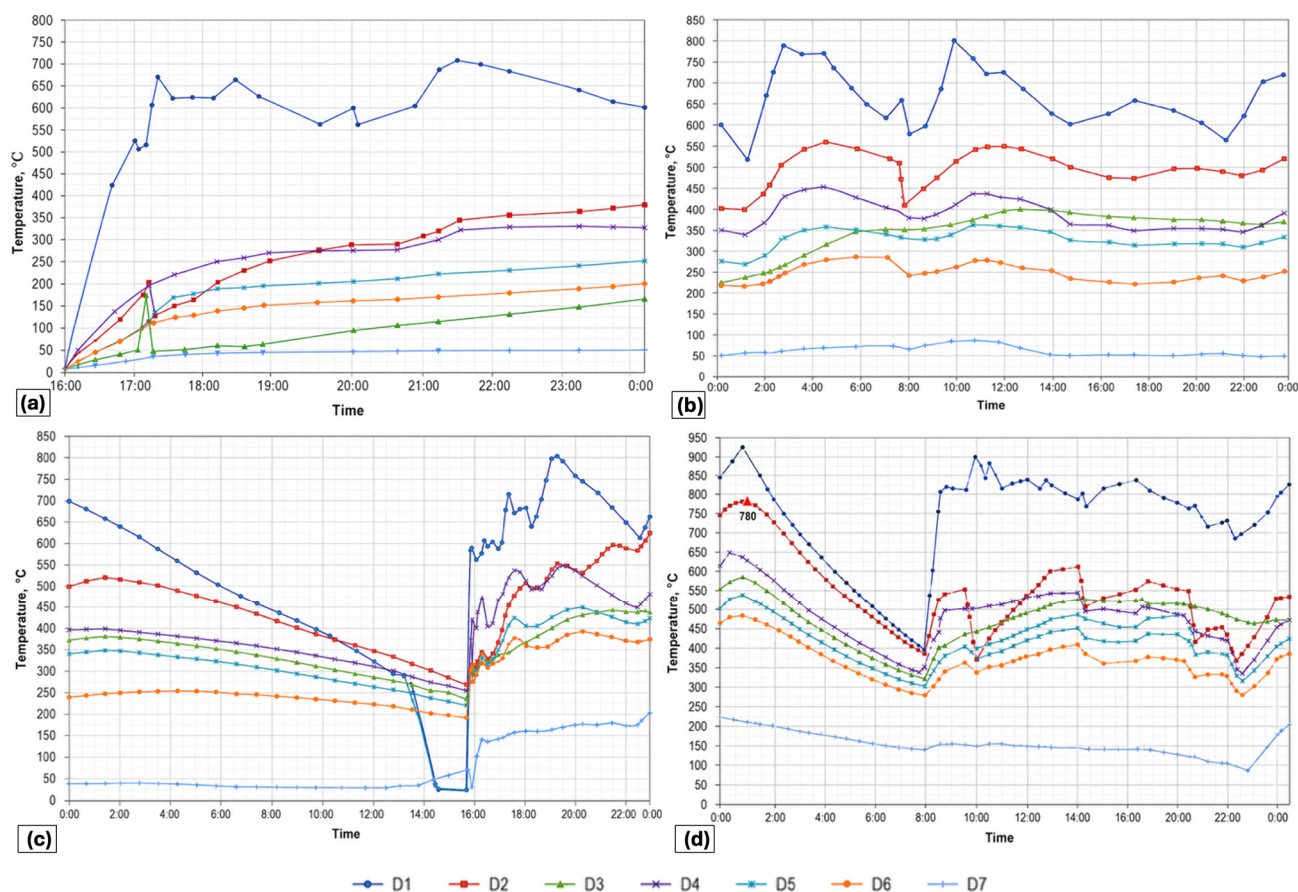


Figure 6. Temperature field dynamics during long-term magnetizing roasting: (a) first day; (b) second day; (c) third day; (d) fifth day. D1—combustion zone; D2—ore–coal material; D3—lower part of rear wall; D4—left wall; D5—upper part of front wall; D6—upper part of rear wall; D7—chimney.

The highest temperatures throughout the long-term test were recorded at D1, whereas D2 showed lower values and a slower response. Temperatures at D3–D6 generally occupied an intermediate range, reflecting heat transfer from the combustion zone through the furnace structure toward the working chamber, while D7 characterized the exhaust-gas region. During the initial heating period, D1 increased most rapidly, followed by the furnace-wall temperatures and D2. As operation stabilized, D2 and D3–D6 exhibited increasingly similar temporal trends. Periodic fuel charging, feed addition, and product discharge caused transient temperature changes, after which D1 recovered more rapidly than D2 and the wall zones.

The temperature profiles were interpreted together with the subsequent magnetic-separation and XRD results. The faster response of the combustion zone reflects changes in fuel combustion, whereas phase transformation of the ore is associated with the delayed

and more stable temperature response of the ore–coal bed. When D2 reached approximately 550–600 °C, subsequent separation showed substantially higher Fe recovery into the magnetic fraction, while XRD identified magnetite and jacobsite among the principal Fe-bearing phases. By contrast, products associated with the lower material-temperature regime showed less effective iron removal. This relationship indicates that formation of the magnetically susceptible product is governed primarily by the thermal history of the material bed rather than by the instantaneous maximum temperature in the combustion zone.

Based on the recorded material temperatures, the roasting products were grouped into those obtained at approximately 400 °C and those obtained within the target range of 550–600 °C. These groups were subsequently used to compare magnetic-separation performance under the tested combinations of material temperature and ore-to-coal ratio.

3.4. Effect of Magnetizing Roasting Conditions on Dry Magnetic Separation Performance

To evaluate the effects of magnetizing roasting conditions on the subsequent separation of manganese and iron, products obtained at an actual material temperature of approximately 400 °C and in the target range of 550–600 °C were subjected to dry magnetic separation. These conditions differed not only in temperature but also in the ore-to-coal ratio, so the obtained results characterize the combined effect of the degree of material heating and the consumption of reducing agent. The separation results are presented in Table 5.

Table 5. Results of dry magnetic separation of products obtained under different conditions of magnetizing roasting.

Temperature Range, °C	Fraction	Ore:Coal Ratio	Yield, %	Content, wt.%						Recovery, %		
				Mn	Fe _{total}	SiO ₂	C _{solid}	Mn/Fe	Mn	Fe _{total}	SiO ₂	C _{solid}
400	magnetic	1:0.2	47.10	18.00	18.30	30.60	1.90	1.00	33.50	65.90	51.70	59.90
	non-magnetic	1:0.2	52.90	31.72	8.40	25.40	1.10	3.80	66.40	34.00	48.20	40.00
	magnetic	1:0.2	37.62	18.43	14.66	32.90	2.20	1.30	29.80	45.90	41.50	49.10
	non-magnetic	1:0.2	62.38	26.15	10.38	28.00	1.40	2.50	70.10	54.00	58.50	50.90
550–600	magnetic	1:0.4	35.48	17.20	23.50	27.00	1.67	0.73	27.4	80.00	39.10	5.10
	non-magnetic	1:0.4	64.52	25.10	3.20	23.10	16.76	7.84	72.60	19.90	60.80	94.80
	magnetic	1:0.4	42.33	15.00	24.00	27.90	2.91	0.62	30.10	83.40	47.90	10.50
	non-magnetic	1:0.4	57.67	25.50	3.50	22.30	18.25	7.30	69.90	16.60	52.10	89.50

For the 400 °C condition, the first and second datasets correspond to samples collected from the upper and middle parts of the roasted bed, respectively. For the 550–600 °C condition, two separately collected samples are presented. The datasets are shown individually rather than treated as statistical replicates. Recovery values are calculated relative to the magnetic-separation feed.

After roasting at approximately 400 °C, most of the manganese was converted to the non-magnetic fraction, but iron removal remained insufficient. The total iron content in the non-magnetic product ranged from 8.4 to 10.4%, while iron recovery into the magnetic fraction ranged from 45.9 to 65.9%. The Mn/Fe ratio in the non-magnetic fraction ranged from 2.52–3.78. These values indicate that, under the lower-temperature regime with the lower reductant consumption, the conversion of the initial weakly magnetic iron-bearing components into magnetically susceptible phases was insufficiently complete.

Manganese recovery in the non-magnetic fraction reached 66.4–70.1%. Consequently, even under the lower-temperature, lower-reductant-consumption regime, preferential retention of manganese-bearing components in the non-magnetic product was observed. However, the achieved difference in the magnetic properties of the iron- and manganese-

containing phases was insufficient to consistently remove the majority of the iron and significantly increase the Mn/Fe ratio.

More pronounced separation of the components was achieved after magnetizing roasting at an actual material temperature of 550–600 °C and an ore:coal ratio of 1:0.4. The yield of the non-magnetic fraction was 57.7–64.5%. The total iron content in it decreased to 3.2–3.5%, while the manganese content was 25.1–25.5%. As a result, the Mn/Fe ratio increased to 7.30–7.84, which significantly exceeded the value of the raw ore.

On the basis of the magnetic-separation feed, 80.0–83.4% of Fe was recovered in the magnetic fraction, whereas 69.9–72.6% of Mn was retained in the non-magnetic product. Differences between the two separately collected samples may reflect heterogeneity of the periodically discharged material and variations in local thermal and reducing conditions. Although the datasets were not controlled replicates, both showed the same overall separation trend, confirming preferential Fe recovery into the magnetic fraction and Mn retention in the non-magnetic product.

The formation of the magnetic and non-magnetic products can be interpreted from the phase transformations occurring during roasting. At 550–600 °C, the reducing environment generated by coal promotes the conversion of hematite into magnetically susceptible magnetite, whereas higher manganese oxides are transformed predominantly into lower-valence Mn-bearing phases such as hausmannite [13,16,19,20]. The effectiveness of this moderate-temperature range is consistent with the optimum temperature of approximately 560 °C reported for suspension magnetization roasting [19]. During subsequent magnetic separation, magnetite-rich particles are preferentially recovered in the magnetic fraction, whereas hausmannite-, braunite-, and gangue-rich particles remain predominantly in the non-magnetic product. Partial formation of Mn–Fe spinels, particularly jacobsonite, provides a competing pathway for manganese transfer into the magnetic fraction [16,21]. In addition, the fine intergrowth observed in the raw ore may promote the recovery of composite Mn–Fe–silicate particles in the magnetic fraction, although this mechanism was not directly verified by microscopy of the roasted products.

A distinctive feature of the non-magnetic product obtained after roasting at 550–600 °C was its elevated solid-carbon content of 16.8–18.3%. Of the carbon accounted for in the two magnetic-separation products, 89.5–94.8% was recovered in the non-magnetic fraction. Because coarse semicoke particles were removed by screening before separation, the carbon in the non-magnetic fraction consisted predominantly of finely dispersed carbonaceous residue associated with the manganese-bearing mineral fraction.

The coexistence of manganese-bearing mineral phases and finely dispersed carbonaceous residue suggests that the non-magnetic fraction may be considered as a potentially suitable feed for subsequent metallurgical processing. Similar approaches based on reduction roasting followed by magnetic separation have produced Mn-enriched non-magnetic fractions suitable for further ferromanganese production [15,37]. In the present case, the residual carbon may additionally contribute to the reductant requirement during subsequent processing. This possibility will be evaluated in further reduction and smelting studies.

3.5. Phase Composition of the Magnetic and Non-Magnetic Products

X-ray diffraction analysis of the magnetic separation products confirmed a significant difference in the phase composition of the magnetic and non-magnetic fractions obtained after magnetizing roasting at an actual material temperature of 550–600 °C. The diffraction patterns and the results of a semi-quantitative assessment of the phase composition are presented in Figure 7.

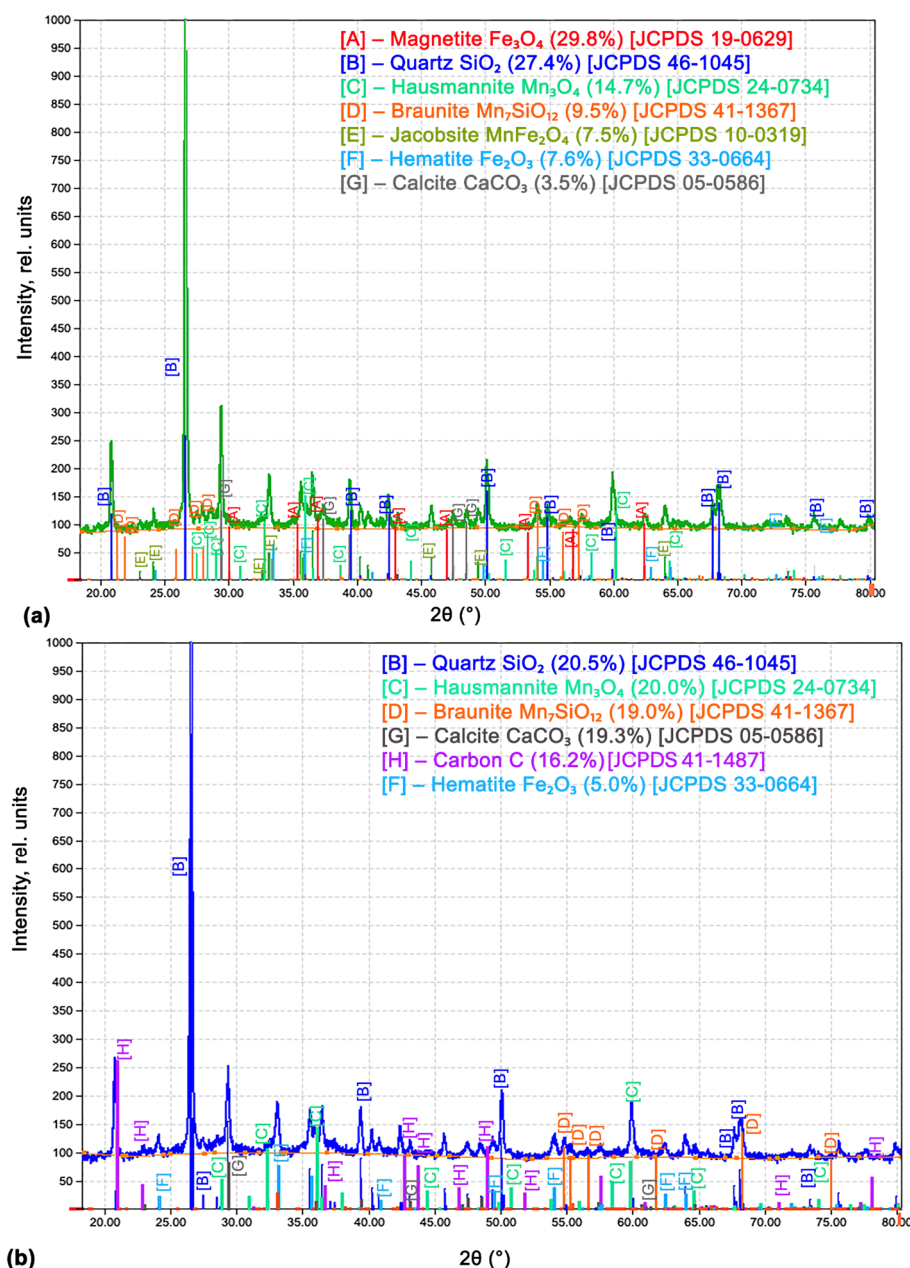


Figure 7. X-ray diffraction patterns and semi-quantitative phase composition of the products obtained after magnetizing roasting at 550–600 $^{\circ}\text{C}$ followed by dry magnetic separation: (a) magnetic fraction; (b) non-magnetic fraction.

The magnetic fraction was characterized by a predominance of magnetite Fe_3O_4 and the presence of jacobsonite MnFe_2O_4 . Magnetite formation is associated with the reduction of the initial hematite by the gaseous and solid reducing components generated from coal, whereas jacobsonite can form through solid-state interaction between closely contacting Fe- and Mn-bearing oxides during roasting. The coexistence of magnetite, jacobsonite, and residual hematite indicates that the phase transformation proceeded to different extents within individual mineral grains and composite particles. This behavior is consistent with the heterogeneous thermal and reducing conditions within the ore–coal bed and with the different accessibility of iron-bearing particles to reducing agents.

Hausmannite Mn_3O_4 , braunite $\text{Mn}_7\text{SiO}_{12}$, and quartz were also detected in the magnetic fraction. Hausmannite is consistent with the transformation of the initial higher manganese oxides toward lower-valence Mn oxides under the roasting conditions, whereas

braunite and quartz are largely inherited from the initial ore. Their occurrence in the magnetic product may be related to the intimate intergrowth of Mn-, Fe-, and silicate-bearing components observed in the raw ore, which can promote the recovery of composite particles together with magnetically susceptible Fe-bearing phases.

The non-magnetic fraction had a predominantly quartz–hausmannite–braunite–calcite composition with a substantially lower contribution from Fe-bearing phases. The disappearance of pyrolusite and the appearance of hausmannite indicate the reduction of higher manganese oxides during roasting, consistent with the transformation pathways discussed earlier. Braunite and quartz largely persisted from the initial ore, whereas calcite could remain under the selected low-temperature roasting regime because its intensive decomposition occurs at higher temperatures. The simultaneous presence of these phases therefore reflects a combination of newly formed reduction products and residual phases inherited from the original mineral assemblage. The carbon-bearing component corresponds to the finely dispersed carbonaceous residue remaining after coal pyrolysis and removal of coarse semicoke.

Overall, the multiphase character of the roasted products results from the simultaneous reduction of Fe- and Mn-bearing oxides, solid-state interaction between locally contacting Fe- and Mn-bearing components, and partial preservation of phases that are stable or only weakly transformed at 550–600 °C. The initial fine intergrowth observed in the raw ore may also contribute to the coexistence of these phases within composite particles. Consequently, magnetite and jacobsite become preferentially associated with the magnetic product, whereas hausmannite, braunite, quartz, calcite, and residual carbon are concentrated mainly in the non-magnetic fraction.

The selective removal of iron can therefore be interpreted as the result of the magnetic contrast generated during low-temperature roasting. Coal-pyrolysis gases and residual solid carbon create reducing conditions within the ore–coal bed, promoting the transformation of hematite into magnetically susceptible magnetite at 550–600 °C, while manganese remains predominantly in less magnetic phases such as hausmannite and braunite. During subsequent dry magnetic separation, magnetite-rich particles are preferentially recovered in the magnetic fraction, whereas Mn-rich and gangue-bearing particles remain mainly in the non-magnetic product, thereby increasing its Mn/Fe ratio. The formation of jacobsite and the fine intergrowth of Fe-, Mn-, and silicate-bearing phases limit complete selectivity because they promote partial manganese transfer into the magnetic fraction. Thus, efficient iron removal is achieved through controlled magnetization of the Fe-bearing component without extensive conversion of manganese into magnetic mixed phases.

4. Conclusions

Low-temperature magnetizing roasting followed by dry magnetic separation demonstrated that selective iron removal from ferruginous manganese ore is governed primarily by the development of magnetic contrast between Fe- and Mn-bearing phases within the actual ore–coal bed. Among the investigated conditions, an actual material temperature of 550–600 °C combined with an ore-to-coal mass ratio of 1:0.4 provided the most favorable balance between magnetization of the Fe-bearing component and retention of manganese in less magnetic phases. The principal transformation responsible for this behavior is the conversion of hematite into magnetically susceptible magnetite, whereas manganese remains predominantly associated with hausmannite- and braunite-bearing material. Formation of jacobsite and the fine intergrowth of Mn-, Fe-, and silicate-bearing phases are considered likely mineralogical factors limiting complete Mn–Fe separation.

The enlarged laboratory experiments further showed that process control should be based on the actual temperature of the ore–coal bed rather than on the combustion-zone

temperature alone, because external heating produces a delayed thermal response of the material relative to the combustion zone and furnace walls. This finding is important for subsequent scale-up, where stabilization of both the bed-temperature field and reducing conditions is expected to improve the reproducibility of phase transformations and separation performance. The Mn-enriched non-magnetic fraction obtained under the selected regime can be considered a potential intermediate feed for subsequent metallurgical processing. Further optimization should focus on enhancing selective magnetization of Fe-bearing phases while suppressing manganese incorporation into mixed magnetic Mn–Fe spinels.

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