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# Beneficiation of Ferruginous Manganese Ore of Um Bogma Locality, Sinai: Comparison Between Conventional Reduction Roasting Techniques and Microwave Technology

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Original scientific paper



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#### Abstract

The increasing demand for high-grade manganese ores in various industries has led to a growing necessity to enhance the quality of lower-grade manganese ores with time. A representative manganese ore was collected from Um Bogma, Sinai, Egypt, and subjected to various characterization methods including X-ray diffraction (XRD), X-ray fluorescence (XRF) and ore microscopy. XRF analysis revealed a low Mn/Fe ratio of 2.81 (as elements), indicating a low ore grade. For suitability in Mn alloy production at the Sinai Manganese Company (SMC) plant in Abu Zenimah, southeast of Sinai, Egypt, this ratio should ideally exceed 5. Additionally, XRD and ore microscopy identified the mineralogical composition of the ore as pyrolusite, hematite, and manganite, with manganese patches embedded in a ferruginous groundmass at the textural scale. Three different reduction roasting techniques were evaluated, involving conventional heating in a furnace utilizing either CO - CO<sub>2</sub> or charcoal as reducing agents, alongside reduction roasting with charcoal utilizing microwave technology. Following each technique, magnetic separation was employed, resulting in the production of high-grade manganese concentrates exhibiting high Mn/Fe ratios ranging from 19.8 to 27.45 and substantial Mn recovery rates ranging from 93.86% to 96.76%.

#### **Keywords**:

manganese ore; Um Bogma; beneficiation; microwave heating; reduction roasting

# 1. Introduction

Manganese ore has an essential role in many industrial applications, especially in ferro-manganese and silico-manganese alloy manufacturing, which are very important in steel production, which needs at least Mn > 30% and a Mn/Fe ratio = 5 (Tan et al, 2004). In addition, manganese is applied in dry-cell batteries and other chemical industries worldwide (Fahim et al., 2013; Yi et al., 2017; Liu et al., 2019; Gao et al., 2020). The manganese is essentially extracted from manganese oxide and manganese carbonate ores (Lei et al., 2021). According to the USGS report (USGS, 2023), the leading country in exporting manganese ores was: Gabon with 67%; participation. Australia is ranked as the first country to export ferromanganese alloys with 19% participation. However, Georgia has the top participation with 28% to produce and export silicomanganese alloys. The beneficiation of manganese ores became, in the last decades, an essential preliminary step prior to their use in industrial applications. This is due to the high consumption rate of higher grades of manganese ores in many

locations in the world (Wan et al., 2021; Singh et al., 2020; Sinha et al., 2020). Um Bogma Mn ore needs to be processed to meet the demand of the manganese consumptions in Egypt, Figure 1 (El-Shafei et al., 2022). Many worldwide research studies were conducted on the beneficiation of low-grade manganese ores. The beneficiation methods mainly depend on the impurities of the ore and the needed industrial application (Singh et al., 2020). Regarding ferruginous manganese ores, it is quite hard to separate Mn and Fe because of the similarity in mineralogical characteristics (Singh et al., 2021).

The beneficiation tests based on comminution, magnetic separation, gravity, and flotation techniques did not reach the needed specifications for the manganese concentrates required for industrial demands (Liu et al., 2019). The chemical leaching and chemical treatments of the ferruginous manganese ore is a better way (Tang et al., 2014; Zhang et al., 2015; Dan et al., 2016; Wang et al., 2017; Astuti et al., 2019) but is not economically recommended, especially when it is applied on the industrial scale, because of the high cost. The reduction roasting treatment is a very good choice to upgrade ferruginous manganese ores, because it gives direct way to separate manganese from its associated iron compo-

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Figure 1: Geological map of Um Bogma area (Sallam, 2018)

nents, however it needs to be commercialized to be feasible for the beneficiation of the Mn ore (Singh et al., 2021). Many reducing agents were used in previous literature such as CO/CO<sub>2</sub> (El-Geassy et al., 2000; Gao et al., 2012; Mpho et al., 2013; Yi et al., 2017; Larssen et al., 2021), charcoal (Zhang et al., 2015; Rath et al., 2018), coke (El-Hussiny et al., 2016; Singh et al., 2020), and anthracite (Yi et al., 2017). The application of microwave technology, as a green, cheap, and fast alternative way to the conventional electrical heating techniques, recently attracted many researchers who work in the ore dressing field (Amankwah & Pickles, 2005; Omran et al., 2014; Zhao et al., 2014; Elmahdy, et al., 2015; Omran, Fabritius, & Mattila, 2015; Celikdemir et al., 2016; Chen et al., 2017; Omran, Fabritius, Yoshikawa, 2018; Li et al., 2020; Lin et al., 2021; Zhou et al., 2021; Zhou et al, 2022).

Several Um Bogma manganese ore samples were studied using several characterization techniques (Moustafa et al., 2024). The obtained results assessed their potentiality for processing. The detailed investigation of Um Bogma manganese ore samples revealed that: sample (1) was unsuitable for upgrading due to its low manganese content and mineral interlocking, while sample (4) was similarly unviable because of its primary manganite content. Conversely, samples (2) and (3) showed promise, with sample (3) being particularly amenable to mineral processing due to its favourable textural characteristics and mineral associations. The present study underscores the importance of tailored beneficiation strategies to enhance the Mn/Fe ratio for sample 3, focusing on comparing conventional and microwave reduction roasting treatments for enhanced manganese recovery. In addition, to compare, at optimal conditions, between conventional reduction roasting treatments and that of microwave reduction roasting treatment. The different reduced Mn samples will be subjected to magnetic separation to separate the magnetic fraction (mainly magnetite) from the non-magnetic manganese concentrates.

# 2. Materials and methods

A representative 30 kg run of mine (ROM) manganese ore sample is collected from Um-Bogma locality, Sinai, Egypt, which belongs to Sinai Manganese Company (SMC) mines. In addition, a sample of charcoal (as a reducing agent) is supplied by a private company. It assayed as 84.31% fixed carbon, 8.93% volatile matter and 6.76% ash content.

# 2.1. Sample comminution and screening

A laboratory Jaw crusher (5x6 Denver) was used for primary crushing of the ROM manganese ore sample. This was followed by a secondary crushing step using a (Wedag) roll crusher. Sieve analysis was performed for the secondary crushed ore using sieves 590, 250, 125, 75, and 53  $\mu$ m mesh sizes. The crushed ore was directed to a grinding step in a closed circuit of screen (250  $\mu$ m) to yield a ground product (100%) beneath the estimated liberation size (lower than 250  $\mu$ m). This represents the feed to beneficiation experiments.

## 2.2. Sample characterization

The chemical analysis, of the ROM and concentrate samples, was performed using PANalytical Axios XRF spectrometer, Technical Institute, George Simon Ohm, Germany. A representative powder sample of 8 g was mixed with 2 g of Licowax C Micro-powder PM, then the mix was pressed to produce a disc that was inserted into the XRF machine to be measured. The XRF data errors of major and minor oxides in Um Bogma manganese ore samples are  $\pm 0.10\%$  and  $\pm 0.01\%$  respectively. The identification of the mineralogical phases (ROM and Mn samples) was carried out using Panalytical X'Pert PRO XRD device, Technical Institute, George Simon Ohm, Germany. About 2 g of each powder sample was pressed and measured as disc in the XRD machine with Cu-radiation (1 = 1.542 Å) within a 2g range between  $2q = 5^{\circ}$  and  $80^{\circ}$ . The results were interpreted using PC "Match" software to calculate the semi-quantitative percentages of the presented mineral phases. The XRD data errors of Um Bogma manganese ore samples are ranged from  $\pm 0.10\%$  to  $\pm 0.5\%$ . A rock-cutting machine was used to get a hand specimen of the manganese ore sample, and then the specimen was mounted and polished. Then, the mounted polished sample was subjected to reflected-light microscope (Olympus. Portugal), with an attached camera and PC Olympus workspace software to take images and study the mineralogical and textural characteristics of the samples.

### 2.3. Liberation size assessment

In the beneficiation of low-grade ores, it is very important to determine the best liberation size that produces a higher degree of liberation between manganese minerals and their associated gangue constituents. This was achieved through the microscopic-textural examination of the ROM manganese ore sample. A screen size analysis was performed to support the estimation of liberation size, as a representative one-kilogram sample of the crushed manganese ore sample (30 kg) was sieved using various sieve mesh sizes (590, 250, 125, and 75  $\mu$ m). Then, each size fraction was subjected to chemical analysis to estimate the best size fraction of maximum liberation.

#### 2.4. Reduction roasting techniques

The conventional reduction roasting treatment of the manganese ore sample was carried out using two different reducing agents during reduction processes. The first one is a gas system (CO/CO<sub>2</sub>) and the second one is a solid system (charcoal). The reduction using gas system CO/CO<sub>2</sub> was conducted using a prepared representative manganese ore sample of about 120 g. The sample is sieved beneath 250 µm, then adjusted in a horizontal tube electrical furnace with the optimal conditions of temperature =  $700^{\circ}$ C and (100-CO / (CO-CO<sub>2</sub>)) gaseous condition ratio = 20 for 2.0 hr (El-Geassy et al., 2000). Then, the product was subjected to low-intensity magnetic separation to separate the magnetite phase from the manganese minerals. The second technique for reduction roasting was to use charcoal as a reducing agent (Sharma, 1992). This was performed using 'Nabertherm' electrical chamber-muffle, with the optimal conditions of a mix of -250 µm sized-sample of manganese ore (80 wt.%) and charcoal (20 wt.%). The mixture was subjected to thermal treatment at temperature 800°C for 2.0 hr. In recent years, the application of microwave muffle, as a novel technology, for thermal treatment of difficult to treat ores was studied to upgrade iron ores (Omran, 2014). So, it was decided to test this recent technology for thermal reduction of Um Bogma manganese ore. The experiment was performed using a «Sirem» microwave muffle at a power of 1.5 kW. The feed to microwave muffle was composed of a blend containing manganese ore (80 wt.%) of below 250 µm in size and charcoal (20 wt.%). The experiment was performed at a temperature of 800°C for 0.13 hr.

# 2.5. Magnetic separation of different reduced samples

Application of wet magnetic separation, after reduction roasting, for different samples was established using a 'Davis tube' wet magnetic separator device. This process aimed to separate the paramagnetic (non-magnetic) fraction, that would not be attracted to the low intensity magnet, that represented the manganese concentrate fraction, from the ferro-magnetic (magnetic) fraction represented by the iron bearing mineral phase "magnetite Fe<sub>3</sub>O<sub>4</sub>". The optimal conditions were established (**Ahmed, 1977; Galal, 2002**), as 20 g of the treated sample was subjected to a magnetic field of 300 gauss, a tube axis inclination equal to 220° from horizontal, a wash water rate of 400 cm<sup>3</sup>/min for 0.25 hr, and an oscillation rate equal to 64 cycle/min.

## 3. Results and discussion

# 3.1. Characterization of Um Bogma manganese sample

Table 1 shows the results of the X-ray fluorescence analysis (XRF) of the ROM sample. In the context of Um Bogma manganese ore, XRF analysis could provide valuable information on the concentrations of not only manganese but also other elements that may be present as impurities. Common elements of interest in manganese ore analysis include iron, silicon, aluminium, calcium, magnesium. The major oxides are MnO (55.40%) and Fe<sub>2</sub>O<sub>2</sub> (21.86%). This means that the Mn/Fe ratio of 2.81 (as elements) is low. This ratio should be greater than 6 to be suitable for Mn alloys production. Also, SiO<sub>2</sub> (2.52%) and CaO (1.98%) represent minor oxides exceeding 1%. Other minor oxides (such as Al<sub>2</sub>O<sub>2</sub>, MgO, P<sub>2</sub>O<sub>5</sub>, SO<sub>2</sub>, K<sub>2</sub>O, CuO, ZnO, and BaO) vary between 0.38 and 0.67%. The values of oxides (such as Na<sub>2</sub>O, Cl, NiO, and SrO) are trace amounts. In addition, the loss-on-ignition (LOI) is about 12%.

According to the mineralogical study of the manganese ore sample (see **Figure 2**), the XRD diffraction patterns show that pyrolusite (MnO<sub>2</sub>) (85.9 wt.%, PDF No.



**Figure 2:** X-ray diffraction pattern of Um Bogma Manganese ore. (Py: pyrolusite, Hm: Hematite, Mng: Manganite)

Oxide (%)	Um Bogma manganese ore sample (ROM)	-590 +250 μm	-250 +125 μm	-125 +75 μm	-75 +53 μm
Na <sub>2</sub> O	0.16	0.18	0.20	0.15	0.18
MgO	0.47	0.88	0.93	0.97	0.95
Al <sub>2</sub> O <sub>3</sub>	0.67	1.10	1.14	1.22	1.57
SiO <sub>2</sub>	2.52	2.69	2.93	3.22	3.88
$P_2O_5$	0.52	0.61	0.36	0.32	0.27
SO <sub>3</sub>	0.38	0.42	0.43	0.45	0.51
Cl	0.11	0.04	0.03	0.03	0.04
K <sub>2</sub> O	0.35	0.69	0.77	0.72	0.67
CaO	1.98	1.30	1.21	1.27	1.37
TiO <sub>2</sub>	0.02				0.05
Cr <sub>2</sub> O <sub>3</sub>	0.02	0.03	0.03	0.03	
MnO	55.40	55.45	56.31	55.54	54.42
Fe <sub>2</sub> O <sub>3</sub>	21.86	21.98	21.66	21.64	21.52
NiO	0.04	0.05	0.05	0.05	0.06
CuO	0.47	0.11	0.09	0.09	0.11
ZnO	0.64	0.47	0.50	0.45	0.45
As <sub>2</sub> O <sub>3</sub>	0.03	0.04			0.05
SrO	0.19	0.11	0.11	0.11	0.10
Y <sub>2</sub> O <sub>3</sub>	0.02	0.02	0.02	0.02	0.02
BaO	0.52	1.26	1.15	1.21	1.10
PbO	0.04	0.04	0.04	0.06	0.03
LOI	12.25	12.56	12.04	12.43	12.70

Table 1: X-ray fluorescence results for Um Bogma manganese ore sample and the different ore size fractions



Figure 3: Photomicrograph of polished section of Um Bogma manganese ore sample under reflected light microscopy

96-900-9083) represents the major Mn mineral phase, in addition to hematite ( $Fe_2O_3$ ) (10 wt.%, PDF No. 96-900-0140) with the low existence of manganite (MnO(OH)) (4.1 wt.%, PDF No. 96-901-6547). Pyrolusite and hematite minerals are paramagnetic and therefore direct magnetic separation is not convenient for efficient sepa-

ration of these minerals from each other. A reduction roasting process would convert hematite into magnetite and consequently the separation of paramagnetic pyrolusite from the ferromagnetic magnetite could be achieved with a Davis tube as a low intensity magnetic separator.

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The reflected-light ore microscopy examination (see **Figure 3**) shows that the manganese ore is composed mainly of light-coloured phases, which represent manganese minerals (pyrolusite  $MnO_2$  and manganite MnO(OH)). On the other hand, the darker phases represent iron bearing mineral (hematite  $Fe_2O_3$ ). The texture displays semi-equant polygonal particles of manganese minerals immersed in the ferruginous groundmass.

## 3.2. Liberation size assessment

Based on the microscopical study of the manganese ore's polished section (see Figure 4a, b and c), it is clear that the predominant texture observed in the manganese ore sample is granular manganese patches, distinguishable by their (yellowish white) appearance, dispersed within a groundmass primarily composed of iron mineral phases with (faint and dark brown). This granular texture suggests a heterogeneous distribution of manganese within the ore matrix, indicating that liberation processes may be necessary to extract the manganese content efficiently. The described textural pattern implies that the manganese granules are encapsulated within the surrounding iron mineral phases, potentially hindering their liberation during subsequent processing stages. Liberation, in this context, refers to the process of breaking down the ore particles to release valuable minerals from their host matrix, thereby making them accessible for further beneficiation. The estimation of the average radius of the manganese granules, believed to be below 250 µm, is crucial for determining the optimal size range for comminution processes. Comminution involves crushing and grinding operations aimed at reducing the particle size of the ore to facilitate the liberation of valuable minerals. The identified liberation size provides a target for the comminution circuit, guiding the selection of appropriate crushing and grinding equipment to achieve efficient liberation of manganese from the ore matrix. Achieving liberation sizes below the estimated average radius of the manganese granules is essential to maximize the recovery of manganese and optimize the overall processing efficiency. However, it is important to note that liberation size assessment is influenced by various factors, including ore mineralogy, texture, and the desired end product specifications.

**Figure 5** presented the size and cumulative analysis of the studied manganese ore sample. Most of the weight (about 81 wt.%) of sample lie between -125  $\mu$ m +75  $\mu$ m in size. About 90% by weight (d<sub>90</sub>) lies in sizes less than about 400  $\mu$ m while the d<sub>50</sub> of the sample at about 125  $\mu$ m. These results depict that a such ground sample has a reasonable degree of liberation. In general, the existence of a high percentage of iron in Um Bogma manganese ore is expected, as it is a ferruginous ore type (**Kordi et al., 2017; Araffa et al., 2020; and El-Shafei et al., 2022**). The concentrations of Fe<sub>2</sub>O<sub>3</sub> are in the reverse order which was explained by **Fahim et al. (2013)**.



**Figure 4:** The photomicrographs show the estimation of manganese grains liberation size (average 250μ) through textural examination of manganese ore sample polished section (a) in the range 250-400 μm (b) in the range 520-700 μm (c) in the range 200-600 μm.



**Figure 5:** Distribution of MnO and Fe<sub>2</sub>O<sub>3</sub> in different size fractions of manganese sample

The XRF analysis of different size fractions are illustrated (see **Table 1**). The results highlight the distribution of various oxides across different particle sizes. Manganese oxide content is highest in the  $-250 + 125 \mu m$  fraction (56.31%) and lowest in the finest fraction (54.42%). This shows manganese concentration slightly decreases with finer particles but remains relatively high across all sizes, indicating that Um Bogma manganese ore has a consistently high MnO content. Iron oxide content remains relatively stable across all fractions, indi-

Oxide %	ROM	$Conv + CO/CO_2$	Conv + charcoal	Microwave + charcoal
MnO	55.40	82.18	89.04	89.26
Fe <sub>2</sub> O <sub>3</sub>	21.86	3.32	3.84	4.99
MgO	0.47	0.53	0.53	0.59
CaO	1.98	0.85	1.26	1.48
Al <sub>2</sub> O <sub>3</sub>	0.67	1.13	0.90	0.91
SiO <sub>2</sub>	2.52	1.74	0.99	1.35
SO <sub>3</sub>	0.38	2.77	0.09	0.13
Cl	0.11	0.02	0.00	0.00
K <sub>2</sub> O	0.35	0.21	0.26	0.17
NiO	0.04	0.00	0.04	0.03
CuO	0.47	0.1	0.06	0.06
ZnO	0.64	0.21	0.61	0.13
% as Metal				
Mn%	42.95	63.6	68.92	69.08
Fe%	15.29	2.32	2.68	3.49
Mn/Fe	2.81	27.45	25.71	19.82
Recovery%	-	93.89	96.43	96.76

Table 2: X-ray fluorescence of different manganese concentrates in comparison to ROM samples

cating an even distribution of iron oxide in the ore. These results supported the estimated liberation size where the values of MnO (54.42 - 56.3%) and Fe<sub>2</sub>O<sub>3</sub> % (21.52 - 21.98%) are, more or less, evenly distributed in different size fractions (see **Figure 5**). The variations in other oxides, particularly SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, and P<sub>2</sub>O<sub>5</sub>, suggest differential distribution of these elements, which could impact the ore beneficiation and processing. The presence of high SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> in finer fractions might require additional treatment steps to achieve optimal manganese extraction. Titanium, chromium, arsenic, yttrium, strontium and lead oxides, along with chlorine are present in very low concentrations across all fractions.

## 3.3. Results of reduction roasting followed by magnetic separation

Beneficiation of ferruginous manganese ores by physical methods, such as gravity separation or direct application of magnetic separation techniques, did not give satisfactory results (Sharma, 1992; Youssef & Abdel-Khalek, 2011). This is due to the similarities between manganese and its associated iron bearing minerals. For this reason, the best way to beneficiate such ferruginous manganese ore is to conduct a reduction roasting process under certain conditions. This leads to transforming iron oxides (mainly hematite,  $Fe_2O_2$ ) into the magnetite  $(Fe_2O_4)$  phase. It is known that magnetite has high magnetic susceptibility followed by hematite and goethite (Ravisankar et. al., 2017). Hence, magnetite, as a ferromagnetic mineral, can be easily attracted to a low-intensity magnetic field while manganese oxides will be separated as non-magnetic (Sharma, 1992).

Proper reducing agents such as carbon (e.g. charcoal) and CO gas are conducted during this study as reducing agents for hematite to be converted into magnetite at a specific temperature and duration of reduction. Any iron oxide can undergo reduction through carbon monoxide in three distinct stages above 570°C (Chartterjee, 2010):

Stage: Stage 1  $\rightarrow$  Stage 2  $\rightarrow$  Stage 3

Phase:  $Fe_2O_3 \rightarrow Fe_3O_4 \rightarrow Fe$ 

This reduction roasting or magnetizing roasting process transforms a particle or substance from having nonweak magnetic properties to exhibiting high magnetic content within a critical temperature range of 500 to 900°C, employing a reducing agent over a specific time frame. Below this critical temperature, the conversion and reduction processes remain incomplete, while above it, fusion leads to the formation of wustite, a paramagnetic material. The particle transformation is described by the equations provided below (Jang et al., 2014).

$$2 \operatorname{FeO(OH)} \rightarrow \operatorname{Fe_2O_3} + \operatorname{H_2O}$$
(1)

$$3 \operatorname{Fe}_2 O_3 + \operatorname{CO} \rightarrow 2 \operatorname{Fe}_3 O_4 + \operatorname{CO}_2$$
 (2)

$$Fe_3O_4 + CO \rightarrow 3 FeO + CO_2$$
 (3)

The reduction of iron oxide occurs through the CO mechanism, as illustrated in Boudouard diagram (Jang et al., 2014; Dawei et al., 2014). The Boudouard diagram displays that below the temperature range of 600-800°C, and CO/CO<sub>2</sub> gaseous environment of  $[100*CO/(CO*CO_2)]$  equals a value in the range of 5-30, (see Figure 8) the iron bearing minerals including hematite would be converted into a magnetite phase (Jang et al., 2014; Dawei et al., 2014).

Charcoal, serving as a solid carbon source, is also employed as a substitute for CO/CO<sub>2</sub> gas systems. In such instances, the reduction roasting process advances in two phases. Initially, carbon undergoes transformation into CO gas at elevated temperatures, wherein carbon atoms combine with oxygen atoms to yield  $CO_2$  and CO gases. This initial phase sets the stage for reduction in the subsequent step for hematite (**Sharma, 1992**), depicted as:

$$2 C + O_2 \rightarrow CO_2$$
 (4)

$$CO_2 + C \rightarrow 2 CO$$
 (5)

Reduction roasting process for manganese samples has been conducted by three different techniques as follows:

- 1 Reduction roasting in a muffle furnace using a (CO/CO<sub>2</sub>) gas system as a reducing agent,
- 2 Reduction roasting in a muffle furnace using charcoal as a solid reducing agent, and
- 3 Reduction roasting using a microwave muffle in the presence of charcoal as a solid reducing agent.

The reduction roasting processes are carefully controlled in such a way that the associated 'hematite' phase will be reduced to a 'magnetite' phase which can be easily attracted to a magnetic separator. The reduced samples, produced from the application of such different reduction roasting techniques, are subjected to a magnetic separation process using a 'Davis Tube' magnetic separator unit. The latter separates the reduced samples into two fractions. A magnetic (magnetite  $Fe_3O_4$ ) fraction from the non-magnetic (manganese) product. The different non-magnetic products (manganese concentrates) are subjected to evaluation using XRF and mineralogical (XRD) analyses as shown below.

# 3.3.1. X-ray fluorescence (XRF) analysis of manganese concentrates

The non-magnetic products (manganese concentrates) are subjected to XRF analysis, the results of which are shown in Table 2. These results for Mn concentrates are compared with that of a ROM sample. The content of MnO% is increased significantly in the three Mn concentrates. For example, MnO% is increased from 55.40% in ROM to about 82.18% MnO in non-magnetic concentrate as a result of reduction roasting in a furnace equipped with a CO - CO<sub>2</sub> gas system as a reducing agent. Also, MnO% is increased to about 89.04% in the non-magnetic concentrates after reduction roasting in a muffle furnace using charcoal as a reducing agent. On the other hand, the application of the reduction process in a microwave in the presence of charcoal as a reducing agent, followed by magnetic separation, gives a Mn concentrate with the highest MnO% (89.26%) in comparison to the other two techniques for reduction roasting.

While the  $Fe_2O_3\%$  content is decreased significantly from 21.86% in the ROM sample to only 3.32% in the

non-magnetic concentrate reduced in a muffle furnace using a CO-CO<sub>2</sub> gas system, and to  $Fe_2O_3 3.84\%$  in the sample reduced in a conventional furnace with charcoal as a reducing agent. Also, reduction in a microwave with charcoal gives a non-magnetic concentrate containing 4.99% Fe<sub>2</sub>O<sub>3</sub>

Regarding the Mn/Fe ratio, it raised from only 2.81 in the ROM sample to 27 in the non-magnetic concentrate with a recovery of 93.90% while using the conventional reduction technique with a CO-CO<sub>2</sub> gas system as a source for reducing gas. Conventional reduction in a muffle furnace with charcoal as a reducing agent gives a non-magnetic concentrate with a Mn/Fe ratio of 25.70 and a recovery of 96.43%. On the other hand, microwave reduction of a sample with charcoal yields a nonmagnetic concentrate with a Mn/Fe ratio of 19.82 and a recovery of 96.76 %.

These results clearly indicate that the three techniques for reduction roasting of such low-grade manganese ore (with Mn/Fe 2.81) are capable for producing high grade Mn concentrates containing both a high Mn/Fe ratio (19.82 - 27.45) and a high Mn recovery (93.89 -96.76%). Moreover, it seems that the selectivity of magnetic separation (after the reduction process) between MnO and Fe<sub>3</sub>O<sub>4</sub> (magnetite) phases is much better in comparison to that between MnO and Fe<sub>2</sub>O<sub>3</sub> (hematite) phases. This is expected since, as mentioned above, magnetite has high magnetic susceptibility in comparison to hematite (Chatterjee, 2010; Jang et al., 2014). Also, the best reducing agent used is the gas (CO-CO<sub>2</sub>) system where it gave better reduction results than using solid reducing agents (such as charcoal or coal). This is in agreement with the work of Larssen, et al. (2021).

However, the results clearly indicate that the reduction of Mn ore in a microwave (even while using a solid reducing agent) gave superior results in terms of grade (highest MnO% 89.26) and high recovery (96.76%). This could be related to the fact that conventional thermal treatment depends on the use of electrical powerbased furnaces with an outside-inside heating process. The microwave thermal treatment depends on vibrating inner molecules and produces heat from the inside to the outside of a sample (**Omran, et al. 2014; Li et al, 2020**).

# 3.3.2. X-ray diffraction (XRD) analysis of manganese concentrates

XRD results (see **Figure 6**) of manganese concentrates show that the peak patterns and semi-quantitative percentages of the presented minerals are changed in comparison to the ROM sample, as a result of the reduction roasting process, as follows (see **Table 3**):

a) XRD of manganese concentrate obtained after reduction roasting in a muffle with a  $CO/CO_2$  system (see **Figure 6a**) shows that the majority of manganese oxides reduced into manganosite (~91.2% MnO) (PDF No. 96-600-6661) while ~ 8.8% remains as pyrolusite (MnO<sub>2</sub>).



**Figure 6:** X-ray diffraction of the three manganese concentrate samples. (a) conventional + CO/CO<sub>2</sub>. (b) conventional + charcoal. (c) microwave + charcoal.

 Table 3: X-ray diffraction's semi-quantitative analysis of identified phases % in concentrates after reduction-roasting and magnetic separation

Sample	Manganosite (wt. %) (PDF No. 96-600-6661)	Hausmannite (wt. %) (PDF No. 96-101-1263)	Pyrolusite (wt. %) (PDF No. 96-900-9083)	Magnetite (wt. %) (PDF No. 96-901-3536)
Conventional + CO/CO <sub>2</sub>	91.20		8.80	
Conventional + charcoal		98.00		2.00
Microwave + charcoal	23.10	76.90		

On the other hand, the reduction roasting in a microwave with charcoal (see **Figure 6c**) produces manganosite with a relatively lower Mn percentage (~23.10% MnO).

b) Hausmannite ( $Mn_3O_4$ ) (PDF No. 96-101-1263) is the essential manganese mineral identified in XRD patterns after reduction roasting using charcoal, as a reducing agent, both in a conventional muffle (see **Figure 6b**) (~ 98%  $Mn_3O_4$ ) or in a microwave (see **Figure 6c**) (~ 76.9%  $Mn_3O_4$ ).

c) Magnetic separation after reduction roasting is a successful process for separating (as magnetic fractions) the majority of magnetite ( $Fe_3O_4$ ) (PDF No. 96-901-3536) phase formed during the reduction with the three

different reduction roasting techniques, where traces of  $Fe_3O_4$  (~2%) are detected only in the concentrate obtained from reduction in a muffle with charcoal (see **Figure 6b**).

# 3.4. Flowsheet for beneficiation of low-grade manganese ore

A suggested tentative flowsheet was designed (see **Figure 7**), to manage the beneficiation process for such a ferruginous manganese ore. As the first step, the ROM sample is subjected to comminution (primary crushing and secondary crushing). Then, the crushed ore is directed to the grinding step in a closed circuit of screen (250)



Figure 7: Flowchart for the beneficiation of Um Bogma manganese ore

	Conventional heating + CO/CO <sub>2</sub> gas system	Conventional heating + charcoal	Microwave heating + charcoal
Type of energy	Electrical	Electrical	Microwave (green energy)
Energy consumption	High	High	Low
Time [hr]	2.0	2.0	0.13
Cost	Very high	High	Cheap
Mn [%]	63.60	68.92	69.08
Fe [%]	2.32	2.68	3.49
Mn/Fe ratio	27.45	25.70	19.80
Recovery [%]	93.86%	96.43%	96.76%

Table 4: Comparison between the three reduction roasting techniques

 $\mu$ m) to yield a ground product (100%) beneath the estimated liberation size (-250  $\mu$ m). This represents the feed to beneficiation tests (reduction roasting followed by magnetic separation) using one of the following routes:

- 1 The conventional reduction roasting in a muffle using either a gaseous reducing agent (CO/CO<sub>2</sub> gas system) or a solid reducing agent (charcoal), both reduction techniques should be followed by the application of low-intensity magnetic separation for the reduced samples.
- 2 Reduction roasting in a microwave with charcoal (as a solid reducing agent) followed by the application of low-intensity magnetic separation for the reduced ore.

**Table 4** represents a comparison between applying conventional reduction techniques (in a muffle) and using a microwave for reduction roasting. These results illustrate that the conventional methods led to high Mn/Fe ratios up to 27.45 but this was achieved after 2.0 hr. While the microwave technology achieves good results

as it led to a Mn/Fe ratio of 19.80 with very high recovery of 96.76% after only 0.13 hr. In addition, it represents a green and novel technology (Amankwah & Pickles, 2005; Omran et al., 2014; Zhao et al., 2014; Elmahdy et al., 2015; Omran et al., 2015b; Celikdemir et al., 2016; Chen et al., 2017; Omran et al., 2018; Li et al., 2020; Lin et al., 2021; Zhou et al., 2021), since it caused a reduction in energy consumption as it needs a very short time for the reduction roasting process (0.13 hr. instead of 2.0 hr.) which reduced cost as well (Zhou et al., 2022).

# 4. Conclusions

Um Bogma manganese ore, characterized as ferruginous with a low Mn/Fe ratio of 2.81, is not directly suitable for industrial use, which requires a higher Mn/Fe ratio above 5. This study investigated methods to improve the suitability of the ore through beneficiation processes. The primary mineralogical components of Um

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Bogma manganese ore include pyrolusite (MnO<sub>2</sub>), hematite (Fe<sub>2</sub>O<sub>2</sub>), and manganite (MnO(OH)). The research focused on reduction roasting techniques aimed at transforming ferruginous mineral phases into magnetite (Fe<sub>2</sub>O<sub>4</sub>), a ferromagnetic phase that can be efficiently separated from paramagnetic manganese phases via low-intensity magnetic separation. The study evaluated the efficacy of both conventional and microwave heating methods, identifying optimal conditions at 700-800°C. Conventional heating required 2 hours, whereas microwave heating needed only 0.13 hours at 1.5 kW power. The optimal proportions of reducing agents were determined to be 20% for both CO/CO<sub>2</sub> and charcoal. The results revealed that all reduction roasting techniques followed by magnetic separation produced high-grade manganese concentrates, with Mn/Fe ratios between 19.8 and 27.0 and Mn recovery rates ranging from 93.89% to 96.76%. The CO-CO<sub>2</sub> gas system yielded a higher Mn/Fe ratio of 27.45 compared to the 25.70 ratio achieved with charcoal, though the latter is more economically viable for industrial applications. Additionally, reduction roasting with CO/CO<sub>2</sub> in a muffle converted most of the manganese into manganosite (~91.2% MnO). In contrast, using charcoal as a reducing agent in a conventional muffle produced mainly hausmannite  $(\sim 98\% \text{ Mn}_3\text{O}_4)$ , while microwave reduction resulted in a mix of hausmannite (~77%  $Mn_3O_4$ ) and manganosite (~23% MnO). Microwave reduction, even with solid reducing agents, demonstrated significant results, achieving high MnO content (89%) and high recovery rates  $(\sim 97\%)$ . This method also presents benefits in terms of energy efficiency, cost savings, and reduced processing time. However, further research into optimizing microwave heating parameters to enhance efficiency is a promising avenue.

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# SAŽETAK

# Oplemenjivanje željezne rude mangana s lokaliteta Um Bogma, Sinaj: usporedba konvencionalnih tehnika redukcijskoga prženja i mikrovalne tehnologije

Sve veća potražnja za visokokvalitetnim manganskim rudama u raznim industrijama dovela je do rastuće potrebe za poboljšanjem kvalitete niskokvalitetnih manganskih ruda. Reprezentativni uzorak manganske rude prikupljen je iz Um Bogme, Sinaj, u Egiptu, i podvrgnut je različitim metodama karakterizacije uključujući rendgensku difrakciju (XRD), rendgensku fluorescenciju (XRF) te rudnu mikroskopiju. XRF analiza uputila je nizak omjer Mn/Fe od 2,81 (kao elementi), što upućuje na nizak sadržaj rude. Za prikladnost u proizvodnji Mn legura u tvornici Sinai Manganese Company (SMC) u Abu Zenimahu, jugoistočno od Sinaja u Egiptu, ovaj bi omjer idealno trebao biti iznad 5. Dodatno, XRD analiza i rudna mikroskopija determinirali su mineraloški sastav rude kao piroluzit, hematit, i manganit, s manganskim masama ugrađenim u željezni matriks na teksturalnoj razini. Korištene su tri različite tehnike redukcijskoga prženja, koje uključuju konvencionalno zagrijavanje u peći uz korištenje ili CO – CO<sub>2</sub> ili drvenoga ugljena kao redukcijskoga sredstva, uz redukcijsko prženje drvenim ugljenom korištenjem mikrovalne tehnologije. Nakon svake tehnike primijenjena je magnetska separacija, što je rezultiralo proizvodnjom visokokvalitetnih koncentrata mangana koji pokazuju visoke omjere Mn/Fe u rasponu od 19,80 do 27,45 i znatne stope iskorištenja Mn u rasponu od 93,86 % do 96,76 %.

#### Ključne riječi:

manganska ruda, Um Bogma, oplemenjivanje, mikrovalno grijanje, redukcijsko prženje

# Author's contribution

Ahmed Moustafa (1) (Demonstrator at Geology department, Faculty of Science, Ain Shams University) gathered rock samples, characterized samples and contributed in crushing, grinding, magnetic separation, and reduction roasting processes. Nagui Abdel-Khalek (2) (Emeritus Professor of Mineral Processing at CMRDI) evaluated the results, reviewed the draft manuscript, and provided technical suggestions. Ahmed Sharaf-Eldin (3) (Associate professor at Geology department, Faculty of Science, Ain Shams University) described the characterization and the evaluation of the final products, and evaluated the results, reviewed the draft manuscript, and provided technical suggestions. Abdel-monem Soltan (4) (Professor at Geology department, Faculty of Science, Ain Shams University) gathered rock samples, characterized samples, evaluated the results, reviewed the draft manuscript, and provided technical suggestions. El-Sayed Hassan (5) (Associate professor of Mineral Processing at CMRDI) contributed in crushing, grinding, magnetic separation, reduction roasting processes, and described the characterization and the evaluation of the final products.