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CHARACTERIZATION AND BENEFICIATION OF IRANIAN LOW-GRADE MANGANESE ORE

Akbar MEHDILO, Mehdi IRANNAJAD, Mohammad Reza HOJJATI-RAD

* Department of Mining and Metallurgical Eng., Amirkabir University of Technology, Tehran, Iran iranajad@aut.ac.ir

Abstract: The mineralogical studies indicated that the Charagah ore deposit contains approximately 17% pyrolusite, 78% calcite and 3–4% quartz. Pyrolusite as a main valuable mineral is found in the forms of coarse and fine pyrolusites. The coarse grains pyrolusite with simple texture is liberated at 180 micrometers. Another kind of pyrolusite with particle size finer than 10 μ m is disseminated inside gangue phases. This kind of pyrolusite has important effect in beneficiation processes and can affect the manganese grade of the concentrate and its recovery negatively. By jigging machine a pre-concentrate with 20% MnO and a final tailing with about 13% manganese loss are obtained. Using tabling technique or wet high intensity magnetic separation (WHIMS) and also their combination with jigging machine, production of a final pyrolusite concentrate with suitable grade but average recovery is possible. By jigging-tabling a concentrate with – 500+45 μ m size fraction, 44.3% MnO and 61.3% recovery is obtained while jigging-WHIMS produces a concentrate containing 52.6% MnO with a recovery up to 56.6% and d_{80} = 180 μ m.

Keywords: pyrolusite, manganese ore, ore characterization, gravity separation, magnetic separation

Introduction

Manganese is used mainly in steel production, directly in pig iron manufacture and indirectly through upgrading ore to ferroalloys. Globally, the most of (90 to 95%) Mn is used in the metallurgical industry as a requisite deoxidizer and desulfurizer in steel making and as an important alloy component. Various amounts of Mn commonly are added to the steel for industrial use, making low or high Mn alloys. High grade ores (Mn content more than 42%) are usually used in metallurgical application. The remainder of the Mn (5 to 10%) is used in the chemical industry, light industry, production of dry cell batteries, in plant fertilizers and animal feed, and as a brick colorant (Fan and Yang, 1999; Lasheen et al., 2009). The world annual production of the total manganese alloys passed 10.3 million MT and it will increase (Lasheen et al., 2009).

The main manganese minerals are the oxide types, such as pyrolusite MnO_2 , hausmannite Mn_3O_4 and manganite MnO(OH). Manganese is also found in several minerals, such as pink rhodochrosite (MnCO₃), rhodonite (MnSiO₃), wad and alabandite (MnS) (Fuerstenau et al., 1986; Fan and Yang, 1999; Corathers and Machamer, 2006). Minerals such as rhodochrosite, rhodonite and hausmannite are often replaced by pyrolusite. Pyrolusite containing 63.2% Mn is the most common manganese mineral) (Zhang and Cheng, 2007). Manganese ores may accumulate in metamorphic rocks or as sedimentary deposits, frequently forming nodules on the sea floor (Jessica et al., 2006).

The main sources of manganese come from the former U.S.S.R, Brazil, South Africa, Australia, Gabon and India. Russia and South Africa produce about 85% of the world's pyrolusite. Manganese nodules or ferro-manganese concretions, usually containing 30–36% Mn, have been found on ocean floors (Jessica et al., 2006; Mohapatra et al. 2009) and could provide another source of manganese. These nodules are found in both the Atlantic and Pacific Oceans, but principally in the Pacific Ocean. Although the primary interests in deep sea nodules are nickel, copper, and cobalt values, the large quantities of manganese could also be of future importance. As a result, much research work has been devoted to recovering not only nickel, copper and cobalt but also manganese as well (Zhang and Cheng, 2007).

Each type of a manganese deposit is a problem by itself in the matter of selection of a proper method of concentration, depending on the manganese minerals and their gangue constituents. Beneficiation technology as applied to manganese ores is similar to that for iron ores. Most ores are crushed and screened, with the lump product (+6 mm) generally being smelted and the fine product (-6 mm) used as feed for chemical and/or electrolytic processing. Sink-float, jigging, tabling, flotation and high-intensity magnetic separation are usually used to upgrade fine manganese ore. Physical separation technologies such as flotation and roasting, and chemical separation process such as leaching have been developed for beneficiation lower grade and more refractory resources (Abeidu, 1972; Fuerstenau et al., 1986; Rao et al., 1988; Mohapatra et al., 1995; Jessica et al., 2006; Zhang and Cheng, 2007; Ito et al., 2008; Mishra et al., 2009).

With increasing the steel production in Iran, exploration and processing of the manganese ores in the country is necessary. There are some manganese mines (venarch mine) and deposits in Iran. The Charagah ore deposit locating in the 82 km at the North-West of Tabriz, is one of the new indexes (Mehdilo et al., 2010; Hojjati-rad and Irannajad, 2011). The characterization of Charagah ore from process mineralogical viewpoint and its upgrading are the main purposes of this article.

Materials and methods

Around one megagram of representative sample, collected from different trenches of deposit, was prepared according Fig. 1 and used for characterization and upgradation

experiments. The detailed characteristics of the sample are presented in section 3-1. The corresponding polished thin sections were studied for ore and rock-forming minerals and their textural relationships by reflected and transmitted light microscopy. The chemical and mineralogical composition of different samples carried out by X-ray fluorescence (XRF) and X-ray diffraction (XRD). The Philips scanning electron microscopy (model: XL30) equipped with WDX (wave length dispersive X-ray) was used for description of texture and determination of valuable mineral. The microspec WDX (model: 3pc) was used for X-ray mapping analysis.

ASTM standard screens were used in sieve analysis. The gravity separation tests were performed using the laboratory jigging machine, shaking table and some heavy liquids. Methylene iodide (density 3.32 g/cm^3) and bromoform (density 2.89 g/cm^3) were used in heavy liquid experiments. The wet high intensity magnetic separation tests were carried out using a Boxmag separator.



Fig. 1. The procedure of sample preparation

Results

Ore characterization

Chemical and mineralogical composition

According to the X-Ray diffractography (Fig. 2), the ore consist of pyrolusite as the main valuable mineral of manganese and calcite as well as quartz as gangue minerals.

The chemical composition of a representative sample analyzed by XRF is shown in Table 1. The studied ore sample by average grading of 13.8% MnO which implies about 17% pyrolusite (by considering 81.6% theoretical MnO for pyrolusite), is one of the low grade deposits in the world. Based on XRD and XRF analysis, the amounts of calcite and quartz of the ore are about 79% and 2–3%, respectively.

Microscopical studies

Based on the results obtained from transmitted-light microscopy studies, the coarse and fine grains of ore minerals are surrounded by gangue minerals containing calcite and quartz. The interlocking of ore and gangue minerals is shown in Fig. 3. The study of polished sections by reflected-light microscopy showed that pyrolusite (about 12–15 vol %) is the main ore mineral (Fig. 4). The small amount of hematite is also found in the samples.

The studies by scanning electron microscopy indicated that the mineralization of pyrolusite is occurring in two forms. The coarse grains pyrolusites with size about 200 µm have simple interlocking with gangue minerals (Fig. 5a and 5b). Another kind of pyrolusites with complicated texture and interlocking are even finer than 10 micrometers sizes (Fig. 5g). In fact, these very fine pyrolusites are disseminated inside gangue minerals. The distribution or X-Ray mapping of manganese (Mn) as demonstrator of pyrolusite is shown in Figs 5c, 5e and 5h. The small amount of barite particles is also observed in this part of study which is evidenced by analysis using WDX (X-Ray mapping of Ba in Fig. 5f).



Fig. 2. X-ray diffraction pattern of representative sample



Table 1 the chemical composition of representative sample





Fig. 4. Study by reflected light microscopy (interlocking of pyrolusite and gangue minerals)



a)





Fig. 5. BSE (Backscattered Electron) images taken by Scanning Electron Microscopy (SEM) and WDX X-Ray mapping: (a) Interlocking of coarse grains of pyrolusite and gangue minerals containing calcite and quartz (-1190+600 µm size fraction); (b) Interlocking of coarse grains of pyrolusite and gangue minerals containing calcite and quartz (-180+150 µm size fraction); (c) X-Ray mapping of manganese (Mn) as demonstrator of pyrolusite in Fig. 5b; (d) Interlocking of coarse grain pyrolusite and barite with gangue minerals containing calcite and quartz (-1190+600 µm size fraction); (e) X-Ray mapping of manganese (Mn) as demonstrator of pyrolusite in Fig. 5d; (f) X-Ray mapping of barium (Ba) as demonstrator of barite in Fig. 5d; (g) Interlocking of fine grains of pyrolusite and gangue minerals containing calcite and quartz; (h) X-Ray mapping of manganese (Mn) as demonstrator of pyrolusite in Fig. 5g

Liberation degree

One of the 1 kg samples was sieved and a representative polished section was prepared from each of eight sieve fractions. BSE (Backscattered Electron) images were taken from different parts of each polished section. In all images of each section the surface area of liberated and locked pyrolusites were measured by using JMicroVison software. Then, the percent of liberation degree is calculated by using Eq. 1 for different fractions. The results, as percent of liberation degree versus size fraction, are shown in Fig. 5. Based on these results the liberation degree of pyrolusite

$$LD(\%) = \frac{S_{LP}}{S_{LP} + S_{IP}} \cdot 100$$
(1)

is determined as 180 micrometers, where S_{LP} is the total surface area of liberated pyrolusites in all images of each polished section and S_{IP} is the surface area of interlocked pyrolusites in all images of each polished section.



Fig. 5. Liberation degree of pyrolusite, determined using JMicroVison

The laboratory grinding of 1 kg samples with size distribution according Table 2 showed that about 90% of the sample is entered to $-180 \ \mu m$ size fraction after grinding for 15 and 9.5 minutes by rod and ball mill, respectively.

Sieve and chemical analysis

After crushing the sample under 2 mm according Fig. 1, a 1 kg representative sample was subjected to sieve analysis using ASTM standard screens and each fraction is

chemically analyzed by XRF. The results of size and chemical analysis of fractionated materials are shown in Table 2. By decreasing the size fraction, the MnO content is increased but the CaO content is decreased gradually. The size and chemical analysis of a sample (-2000 μ m) ground by the rod mill for 15 min is presented in Table 3. The results show that the distribution of MnO and CaO in all the size fractions of mill product (Table 3) is almost more uniform than mill feed materials (Table 2). Based on the obtained results, it is clear that pyrolusite is ground more easily than calcite and quartz and also its distribution in fine size fractions is more than coarse size fractions. These fine particles could have negative effects on physical separation processes

0.	XX · 1 /	t Cumulative	MnO				С	aO	SiO ₂			
Size (um)	Weight		0/_	Distribution		0/	Distribution		0/	Distribution		
(p)	(,,,,,	pubbilig (70)	/0	(%)	Cumulative	/0	(%) Cumulative (%		(%)	Cumulative		
-2000+1680	10.4	100	10.4	7.9	100	48.1	10.7	100	4.4	15.2	100	
-1680+1190	22.8	89.6	12.2	20.3	92.1	47.9	23.4	89.3	3.3	25.1	84.8	
-1190+600	33.3	66.8	12.4	30.1	71.8	48.1	34.4	65.9	2.7	30.0	59.7	
-600+425	7.2	33.5	12.8	6.7	41.7	47.8	7.4	31.5	2.6	6.2	29.7	
-425+300	4.4	26.3	14.1	4.5	35.0	47.7	4.5	24.1	1.7	2.5	23.5	
-300+180	5.0	21.9	16.2	5.9	30.5	44.5	4.8	19.6	2.8	4.7	21.0	
-180+75	3.5	16.9	20.7	5.3	24.6	41.0	3.0	14.8	4.6	5.3	16.3	
-75	13.4	13.4	19.8	19.3	19.3	40.8	11.8	11.8	2.4	11.0	11.0	
Bulk	100	-	13.7	100	-	46.6	100	_	3.0	100	_	

Table 2. The size and chemical analysis of a sample crushed under 2000 μ m

Table 3. Size and chemical analysis of a sample ground by rod mill for 15 min

Size	Weight Cumulative		MnO				С	aO	SiO ₂			
Size			0/2	Distribution		0/2	Distribution		0/2	Distribution		
(µ)	(-)	, pubbing (, v)	/0	(%)	Cumulative	e	(%)	Cumulative	70	(%)	Cumulative	
-300+210	4.1	100	7.5	2.2	100	45.9	4.2	100	12.2	8.3	100	
-210+150	15.1	95.9	9.9	10.6	97.8	47.5	16.1	95.8	6.55	16.5	91.7	
-150+75	24.5	80.8	13.0	22.6	87.2	45.5	25.1	79.7	6.96	28.4	75.2	
-75+38	17.5	56.3	15.6	19.3	64.6	43.1	17.0	54.6	6.05	17.6	46.8	
-38	38.8	38.8	16.4	45.3	45.3	43.0	37.6	37.6	4.45	29.2	29.2	
Bulk	100	-	14.1	100	-	44.4	100	-	6.0	100	-	

Beneficiation tests

Jigging tests

Some jigging tests were performed on different size fractions including -9500+4750, -4750+2000 and -2000+1180 µm to remove some gangue minerals as a clean tailing with minimum content of MnO and a suitable pre-concentrate. The results shown in Table 4 indicated that the tailing with the minimum grade and recovery is obtained using the feed material by sizing of -9500+4750 µm the produced pre-concentrate in this size fraction with 20% MnO is favorable for other separation techniques such as tabling, magnetic separation and flotation.

Size fraction (µm)	product	Weight (%)	MnO (%)	Recovery (%)
0500+4750	Concentrate	50.3	20.0	77.4
-)500+4750	Tailing	49.7	6.2	22.6
4750+2000	Concentrate	40.2	23.7	65.2
-4750+2000	Tailing	59.8	8.5	34.8
2000 ± 1180	Concentrate	36.4	22.4	56.7
-2000+1180	Tailing	63.6	10.3	43.3

Heavy media separation studies

The $-180+75 \ \mu\text{m}$ size fraction sample was subjected to sink and float studies using methylene iodide (density, γ , 3.32 g/cm³) and bromoform ($\gamma = 2.89 \ \text{g/cm}^3$) as medium. The results of the studies are presented in Fig. 6 and the chemical analysis of the products is shown in Table 5. The results show that using a medium with $\gamma=3.32 \ \text{g/cm}^3$, a pyrolusite concentrate containing 71.7% MnO and about 88% purity is produced. This product contains 76% of the manganese. By separating the float product of medium with $\gamma = 3.32 \ \text{g/cm}^3$ in bromoform solution another concentrate (sink in $\gamma 2.89 \ \text{g/cm}^3$) with 32% MnO and 14.5% recovery is obtained. The SiO₂ content in the sink product of bromoform is relatively high and this can be due to unliberated hard quartz. The float product of the medium with $\gamma = 2.89 \ \text{g/cm}^3$ is a final tailing with 68.6% weight percent which contains only 2.86% MnO. This indicates that most of the gangue minerals are present in the liberated form. It is also observed (Table 6) that BaO content is increased in sink products which related to minor amount of barite mineral in the ore sample.

Tabling tests

The representative sample was ground to below 1190 μ m size and classified into four fractions viz. -1190+425 μ m, -425+180 μ m, -180+75 μ m and -75 μ m. These fractions were subjected to tabling studies and results are shown in Table 6 and Fig. 7.



Fig. 6. Procedure and results of heavy liquid tests

Table 5. Chemical analysis of the sink floats products

Composition (%) Product	MnO	CaO	SiO ₂	Fe ₂ O ₃	Al_2O_3	MgO	BaO	SO ₃	L.O.I	Others
Sink in 3.32	71.7	6.6	1.8	0.81	0.0	0.81	2.46	0.6	13.2	2.02
Sink in 2.89	32.1	28.1	11.6	1.05	0.36	0.71	1.06	0.46	22.96	1.6
Float in 2.89	2.98	52.5	5.1	0.52	0.66	0.74	0.083	0.087	36.84	0.51

Size fraction (µm)	product	Weight (%)	MnO (%)	Recovery (%)
	Concentrate	27.4	29.2	59.6
-1180+425	Middling	36.4	11.2	30.4
	Tailing	36.2	3.7	10.0
	Concentrate	19.4	42.8	52.1
-425+180	Middling	34.8	13.1	28.6
	Tailing	45.8	6.7	19.3
	Concentrate	15.3	47.8	41.1
-180+75	Middling	42.3	15.6	37.0
	Tailing	42.4	9.2	21.9
	Concentrate	10.4	57.6	32.2
-75	Middling	Weight (%)MnO (%)ate 27.4 29.2 g 36.4 11.2 g 36.2 3.7 ate 19.4 42.8 g 34.8 13.1 ate 15.3 47.8 g 42.3 15.6 ate 10.4 57.6 g 42.3 16.9 ate 10.4 57.6 g 47.3 11.6	38.4	
	Tailing	47.3	ight (%) MnO (%) Rec 27.4 29.2 36.4 11.2 36.2 3.7 19.4 42.8 34.8 13.1 45.8 6.7 15.3 47.8 42.3 15.6 42.4 9.2 10.4 57.6 42.3 16.9 47.3 11.6	29.4

Table 6. Results of tabling tests on different size fractions

These results indicate that by decreasing the particle size a concentrate with high MnO content is obtained but the recovery is also decreased. The $-425+75 \mu m$ size fraction (mixture of $-425+180 \mu m$ and $-180+75 \mu m$ fractions) is selected as suitable feed for

separation by tabling which could result in a concentrate containing more than 44% MnO with about 50% recovery.

After removing $-30 \ \mu\text{m}$ particles as slime, the tabling test was performed on $-180+30 \ \mu\text{m}$ size fraction and a concentrate having 66% MnO with 40.4% recovery and a tailing with 10.1% MnO are obtained. The size distribution of the concentrate and tailing according Fig. 8 showed that the average particle size of the concentrate $(d_{80} = 134 \ \mu\text{m})$ is finer than tailing $(d_{80} = 163 \ \mu\text{m})$. These results confirm that it is more easily to grind pyrolusite than gangue minerals and its distribution in fine size fractions.

Magnetic separation

The representative sample for wet high intensity magnetic separation (WHIMS) tests was prepared according Fig. 9. The feed material ($d_{80} = 0.18$ mm) was subjected to magnetic separation at different magnetic intensities (1.2, 1.5 and 1.77 tesla). The results are presented in Table 7. The results show that the weight percent of magnetic product and recovery of MnO increase with increasing of magnetic intensity while the MnO grade is decreased slightly. As seen from Halbich upgrading curves presented in Fig. 10, the better separation is achieved at higher intensity (1.77 Tesla) which results in more selectivity index $f(f = \beta \cdot \sum \varepsilon/100$ (Drzymala, 2007)) value in comparing with that's of two other options. It is possible to get a product with 52.6% MnO by recovering 64.6% MnO. The loss of MnO in non-magnetic fractions has also been reduced at higher intensity. About 90% of CaO and 84% of SiO₂ are concentrated in the non-magnetic product or tailing fraction.



Fig. 7. MnO grade and recovery as a function of size fraction in tabling tests



Fig. 8. The size distribution of concentrate and tailing of tabling test on $-180+30 \mu m$ size fraction



Fig. 9. The procedure of sample preparation for wet high intensity magnetic separation tests

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Intensity	Product	Weight (%)		Grade (%)		Recovery (%)			
(tesla)	Tiouuci	weight (70)-	MnO	CaO	SiO ₂	MnO	CaO	SiO_2	
1.2	Concentrate	18.1	53.5	15.9	5.8	52.3	7.9	13.8	
	Tailing	81.9	10.8	40.9	8.0	47.7	92.1	86.2	
	Feed	100	18.5	36.4	7.6	100	100	100	
	Concentrate	21.6	52.1	16.6	6.5	60.4	9.5	17.1	
1.5	Tailing	78.4	9.4	43.5	8.7	39.6	90.5	82.9	
	Feed	100	18.6	37.7	8.2	100	100	100	
	Concentrate	22.6	52.6	16.2	6.5	64.6	9.8	16.1	
1.77	Tailing	77.4	8.4	43.5	9.9	35.4	90.2	83.9	
	Feed	100	18.4	37.3	9.1	100	100	100	



Fig. 10. The Halbich upgrading curves for magnetic separation at different intensities



Fig. 11. Procedure and results of jigging-WHIMS combination

Combination of gravity and magnetic separation methods

Based on the results obtained from gravity and magnetic separation tests, different combinations of them were also examined. The procedure and results of two suitable

combination including jigging-WHIMS and jigging-tabling are shown in Fig. 11 and Fig. 12, respectively. By combination of jigging-WHIMS a concentrate (magnetic product) with 52.6% MnO and 56.6% recovery is obtained. But, the combination of jigging-tabling produces a concentrate containing 44.3% MnO with 61.3% recovery. The separation efficiency (SE) was calculated 49.83% and 48.5% for jigging-WHIMS and jigging-tabling, respectively. The Halbich upgrading curves were also displayed for both combinations in Fig. 13. These curves showed that the selectivity index (f) for jigging-WHIMS combination is always higher than jigging-tabling one. However, the selection of the best flowsheet depends on the technical and economical condition but from separation efficiency (SE) and selectivity index (f) point of view, the Jigging-WHIMS combination is a little better than jigging-tabling alternative.

Discussion

The Charagah ore by average grading of 13.8% MnO which implies about 17% pyrolusite (by considering 81.6% MnO for pyrolusite) as main valuable is one of the low grade deposits in the world. Mineralogical studies indicated that the ore contains about 79% calcite and 3–4% quartz as gangue minerals. The amount of other minerals such as barite and hematite which were observed in microscopical studies is negligible. So, pyrolusite and calcite are two major phases and the ore mineralization is mainly occurred in limestone. Pyrolusite is found in the forms of coarse and fine pyrolusites. The coarse grains pyrolusite with simple texture is liberated in 180 micrometers. Another kind of pyrolusite is very fine particles which have been disseminated inside the gangue phase.

Two major phases, pyrolusite and calcite, differ significantly in respect of their densities (4.7 and 2.7 g/cm³, respectively). With efficient density of 2.2 g/cm³, it is expected that the upgradation of the ore will be possible easily. This matter is well demonstrated in the heavy liquid separation studies using bromoform (2.89 g/cm³) as the separating media. The results of this study clearly demonstrate upgrading of Mnvalue up to 59% MnO with 90.5% recovery at -180+75 µm size fraction. Using jigging separation, considerable amount of gangue minerals is removed as tailing with about 13% manganese loss. This tailing is very important in decreasing energy consumption in the later milling process. The results of tabling experiments revealed that a concentrate containing 44% MnO with about 52% recovery is obtained in the size fraction of -425+180 µm. With reduction of the size, the MnO content of the concentrate is increased (47.8% for -180+75 µm and 57.6% for -75 µm) but the recovery is decreased significantly. The lower grade of the manganese in the coarse fractions is due to finely disseminated pyrolusite, which takes the locked gangue minerals into the concentrate. The decreasing recovery in the fine fractions is related to entrance of very fine liberated pyrolusites into the tailing. As an alternative, wet high intensity magnetic technique was employed on -180 µm size fraction to find out



Fig. 12. Procedure and results of jigging-tabling combination

the possibility of pyrolusite separation from gangue minerals. Pyrolusite is a paramagnetic mineral and can easily be separated from calcite and quartz as diamagnetic minerals using high intensity magnetic separators. By increasing magnetic intensity, the recovery of manganese is increased significantly while the MnO grade is decreased slightly. Using WHIMS the fine particles of pyrolusite are recovered more efficiently than the tabling technique.



Fig. 13. The Halbich upgrading curve for the two examined combinations

Conclusion

The ore mainly contains pyrolusite and calcite with significant difference in their densities and magnetic susceptibilities. It is expected that the concentration of the ore can be done easily. Some mineralogical features of pyrolusite can affect the manganese grade and recovery in the concentrate of the physical separation techniques negatively. These features are dissemination of very fine pyrolusites inside gangue minerals and distribution of the most of pyrolusites in fine size fractions (-75 μ m) in the grinding process to achieve the determined liberation degree (180 μ m). Nevertheless, using jigging-tabling or jigging-WHIMS, production of a concentrate with suitable grade (44–53% MnO) and average recovery (56–62%) is possible.

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