

Phase, Microstructural Characterization and Beneficiation of Iron Ore by Shaking Table

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Abstract. To know about the nature of gangue associated with the ores, characterization has become an integral part in mineral processing and beneficiation, therefore, the as-mined iron ore collected from Karak region of KP has been characterized for its phase, microstructure and chemical composition *via* XRD, SEM and EDS respectively. Beneficiation of the iron ore has been carried out by shaking table and magnetic separator. XRD analysis confirmed the presence of iron oxide (Fe_2O_3) as the major phase along with quartz (SiO_2) as the minor phase. Finely grinded iron ore powder of 100 (149 μm) and 200 (74 μm) mesh sizes were passed via shaking table and magnetic separator subsequently. The iron ore was successfully upgraded from 28.27 wt.% to 36.51 wt.% at 100 mesh and 38.70 wt.% at 200 mesh *via* shaking table, thus achieving a maximum of 10% upgraded iron ore. The magnetic separator did not become so effective due to non-magnetic nature of hematite.

Keywords: iron ore, shaking table, microstructure, phase, minor magnetic separator

Introduction

With increasing global demand of steel and rapid depletion of high grade iron ores, the utilization of low-grade iron ores acquired much attention in recent years. Complex mineralogy, high content of silica and other gangue minerals are the major difficulties in the processing and use of low grade iron ores. Thus, up-gradation of low grade iron ores to remove the gangue minerals and enhance their quality is a major area of research today (Ali 2004; Ali *et al.*, 2003; Abu-zeid 1967). In most of the known iron ore deposits in Pakistan, many deposits are low grade and needs proper beneficiation, so as to produce and fulfill the requirement of the steel making.

Iron ore deposit of Karak (KP) Pakistan is of low grade and the problem in up-grading this ore deposit is twofold. Firstly, it occur as a mixture of hematite and silica, and secondly, the fine dissemination of iron mineral in silica and silicates, which requires fine grinding to obtain adequate degree of liberation of the desired iron mineral and the gangue constituents.

Gravity and magnetic separation are the economical and most commonly used beneficiation techniques recovery of valuables from natural ores (Burt, 1999). Although other techniques i.e. hydrocyclon, jigging, spirals, flotation and magnetic separation partially

replaced the gravity separation method but they have not made it obsolete. Due to the lowcost, ease of operation, and eco-friendly nature, the gravity separation techniques is widely used in mineral beneficiation practices, since it is based on the principle of settling velocities of the constituents of particles in ore (Mukherjee *et al.*, 2006).

The influential factors in the shaking table and jigging techniques that govern the settling velocity of ore particles include the weight (volume and density) of particles, buoyancy and drag forces (Roy, 2009). Being a powerful technique to recover fine particles of the valuable minerals, many researchers investigated the theoretical and experimental performance of shaking table (Mansar *et al.*, 1991; Gaudin, 1987; Shivamohan 1985). Shaking table efficiency has been reported to be high if the difference in specific gravity is high between valuable and gangue minerals (Samykin, 2005). For the beneficiation of iron ore, a single step magnetic separation can be performed, if the nature of ore is magnetic and the gangue is nonmagnetic. However, if the ore is non-magnetic then a combination of magnetic separation along with gravity separation can be applied (Svoboda, 1994; Svoboda *et al.*, 1989).

Guney studied the beneficiation of Camdag hematite ore that is composed of limonite-hematite interlocked in each other. Using a combination of gravity separation and high intensity magnetic separation techniques he

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successfully increased the ore grade from 5 - 7% in concentrates with ~ 60% recovery by both methods (Guney, 2000).

Abouzeid (Abu-zeid, 1967) worked on the beneficiation of El-Gedida iron ore using high intensity magnetic separator. An ore sample containing 51.6% Fe, 8.76% SiO₂ and 3.06% BaO was studied and successfully point out that high intensity magnetic separator was limited to ore size of 2 and 0.125 mm. The final magnetic product assayed 61% Fe at an iron recovery of 90.3%. However, 5% by weight was obtained as middling assaying 31.4% Fe. These middling products, together with the fine fraction minus 0.125mm which constitute 35% by weight of the whole sample, were not possible to be upgraded by magnetic separation.

Rowayshed (Rowayshed, 1983) investigated the beneficiation of El-Gedida iron ores using high intensity magnetic separator with limit up to 2 mm and concluded that neither the coarse nor the very fine sizes of ores can be treated using this technique. The dominant iron minerals present in this ore are the hydrated minerals, goethite and hydro-goethite, which are difficult to treat through high intensity magnetic separator. (Fatma *et al.*, 1999) studied the beneficiation of iron ore sample containing 44% Fe, 1.59% SiO₂ and 20% BaO using magnetic separation. After cleaning several times an iron concentrate assaying 58.48% Fe, and 0.73% BaO is obtained. Faraghaly in 2002 studied an iron ore containing 23.5% Fe and 34% BaO. The beneficiation of iron ore was carried out using a dry high intensity magnetic separator and found a concentrate containing 56.78% Fe and 1.61% BaO at a recovery of 82.76%.

According to the Geological Survey of Pakistan (GSP) the total estimated reserves of different grades of iron ore in Pakistan are > 1.5 billion tons; however, there is only ~ 30,000 tons/year capacity for commercial mining. With further exploration, in depth geological study and appropriate mining techniques a medium scale steel plant can be installed using the local iron ore to fulfill the steel demand of the country (Shah *et al.*, 2004; Kazmi *et al.*, 2001; Siddiqui *et al.*, 2000).

The major aim of this study was to investigate phase, microstructure and possible up-gradation of low-grade iron ore of Karak region via gravity and magnetic separation. The sieve parameters of 100 and 200 mesh sizes of iron ore were investigated through shaking table (exploiting the difference in specific gravity of the constituents in ore) and magnetic separator

(exploiting the differences in magnetic susceptibility of the constituents in ore) were investigated.

Materials and Methods

The representative sample was collected from active mines of iron of Karak region, Khyber Pakhtunkhwa (KP), Pakistan. The sample was washed, dried and ground using a stainless steel mortar and pestle in Materials Research Laboratory (MRL), University of Peshawar. The powdered sample was sieved through 100 (149 μ m) and 200 mesh (74 μ m) size according to the US standards in MRL. A JEOL 3532, X-ray diffractometer with Cu K α (λ ~ 1.54Å) radiations operating at 40 KV and 30 mA at Centralized Resource Laboratory (CRL) was used for phase analysis. For microstructural analysis, ~ 4×4×4 mm³ piece was cut from iron ore sample with a TeckCut 4TM precision low speed diamond saw (Allied High Tech. Products, USA) in MRL. The sample was finely polished with a TwinPrep 3TM grinding/polishing machine (Allied High Tech Product, USA) using various grades of sandpaper (silicon carbide) and diamond paste on imperial adhesive back polishing cloth using water as a lubricant in MRL. The smooth polished surfaces were chemically etched in 40% hydrofluoric acid (HF) fumes for one minute. For SEM, the polished sample was mounted onto aluminum stub with silver paint and gold-coated to avoid charging in the SEM. The surface morphology and approximate size of the grains and micro-regions were examined using a JEOL JSM 5910 SEM. Elemental analysis were carried out using energy dispersive X-ray electron spectroscopy (EDS) coupled with SEM. A PMG-3 microscope coupled with a DP-12 CCD camera, Olympus (Japan) was used for recording optical images in the present study at CRL. For physical beneficiation via shaking table and magnetic separator, ~ 100 Kg iron ore was crushed in jaw and roller crushers and grinded in a ball mill to get fine powder up to 100 and 200 mesh at the Multipurpose Mineral and Metallic Ore Up-gradation Plant (MPMMOUP), Department of Physics, University of Peshawar.

The shaking table surface is made of 2 mm thick fibre glass with dimensions of chassis length (2450 mm) and width (1200 mm) connected with an adjustable variable speed induction motor (0.75 KW / 1 Hp) to generate back and forth movement and shake the table surface. Height of shaking table from floor surface depends on the angle of the table. The magnetic separator used in present study consists of a permanent cylindrical magnet

of 300 mm diameters and 460 mm length driven by a 0.75 KW / 1 Hp induction motor with a speed reducer with a ratio of 1:30. Both the shaking table and magnetic separators were locally manufactured and supplied by Mobashir Engineers, Peshawar.

Results and Discussion

Figure 1 shows room temperature XRD pattern of the as-mined iron ore. The inter planer spacing (d-values) and relative intensities corresponding to the major XRD peak recorded for iron ore matched PDF # 5 – 490 for quartz of silicon oxide SiO_2 indicating to be major phase in the examined sample.

Additionally some peaks matched PDF # 6 – 502 for Iron Oxide Fe_2O_3 (Hematite) with main peaks appearing at 2θ of 33° , 36° , 56° and 68° as shown in Figure. Some peaks appeared to overlap with each other at 2θ of 36° , 56° and 68° . The experimental d-values and intensities of the as-mined iron ore is given in Table 1.

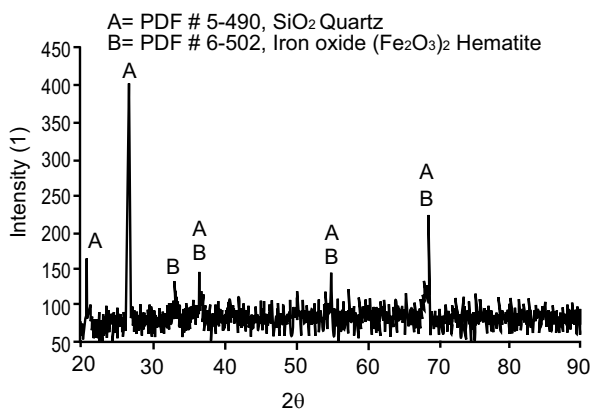


Fig. 1. XRD pattern of as-mined iron ore showing presence of Quartz (labeled as A) as the major phase along with iron oxide (Hematite) as minor phase (B).

The microstructure and the distribution of the impurities within the matrix of iron ore was investigated and analysed using reflected light optical microscopy and scanning electron microscopy (SEM). Figure 2 shows the reflected light micrograph of chemically etched (40 % HF fumes for 50 sec) iron ore sample showing the presence of three contrasted microregions with different texture and appearance. The micro region with dark grey texture comprising quartz, appearing as a plane and smooth surface with crystalline plates of irregular shapes (marked as “A”) with size ranging from 10 – 70 μm . The rough microregion with whitish appearance is Hematite matrix (marked as “B”) and is very finely immersed along the grain boundary and cracks of the large platy type silica grains. The hematite grains are very finely immersed though out the matrix and the

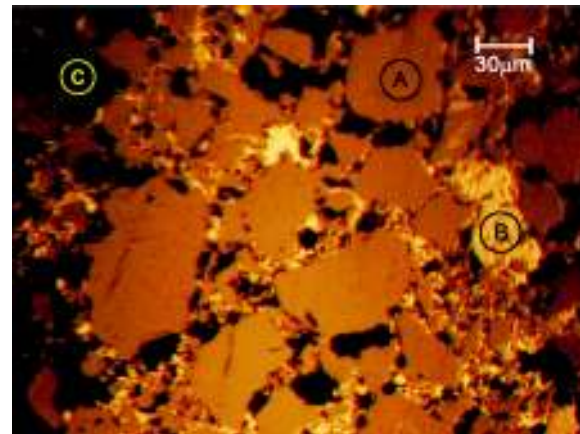


Fig. 2. Reflected light micrograph of iron ore showing dark grey quartz micro-region marked as “A”, the rough micro-region with whitish contrast of hematite marked as “B” and black opaque contrast of magnetite marked as “C”.

Table 1. comparison of d-value and relative intensity for observed XRD pattern of as-mined iron ore

Iron ore sample		PDF # 5- 490, SiO_2 Quartz			PDF # 6- 502, Iron oxide (Fe_2O_3), Hematite		
d-value	I/Io	d-value	I/Io	(hkl)	d-value	I/Io	(hkl)
3.34	100	3.34	100	(101)	-----	-----	
4.28	25	4.26	35	(100)	-----	-----	
2.70	23	-----	-----		2.69	100	(104)
2.46	27	2.45	12	(110)	2.51	80	(113)
1.67	26	1.67	7	(202)	1.69	80	(110)
1.36	49	1.37	11	(203)	1.38	20	(100)

grain size ranges from 5 – 25 μm . The blackish and opaque grains of roughly rectangular and rounded shape may appear to be magnetite (marked as “C”), with an average grain size to be $\sim 15 \mu\text{m}$ (Miaha *et al.*, 2009).

All the three type morphologies are non-uniformly immersed among each other with no clear boundaries among them. Since the average size of the identified hematite grain is $\sim 50 \mu\text{m}$ as clear in the Fig. which showed that the representative iron ore must be crushed to a mesh size of $\sim 300 (47\mu\text{m})$ in order to unlock the hematite grains from the gangue.

Figure 3 shows the secondary electron SEM image (SEI) of as-mined iron ore sample demonstrated the presence of two distinct microregions i.e. plane microregion with grey contrast and rough microregion with whitish contrast. The grey plane microregion with platy type morphology of large irregular shape labeled as “A” is quartz. The average size of the quartz is $\sim 30 \mu\text{m}$, with grains ranges in size from 10 – 90 μm . The rough and granular type white microregion/grains labeled as “B” is hematite. The hematite grains are observed to be irregular in shape with an average size of $\sim 50 \mu\text{m}$. They appeared to be along the grain boundary of the quartz grains and seemed to interlock these grains. The alternated layers of hematite and quartz with microfolds and faulted features dissecting each other can be clearly viewed in the SEM micrograph. The concentration of iron is more or less uniform in hematite layer. From SEM image, the ore shows a complex sort of interlocking between quartz and hematite. Nirlipta *et al.*, 2013 observed that in such type of iron ores the hematite phase is of secondary origin and form a Martite phase due to the oxidation of magnetite.

A well crystalline formation of the three phases i.e. martite, hematite and quartz occur in such ores. However, hematite which occurs as Martite is pseudomorphic magnetite and it retains the shape of an original magnetite. From geological point of view the transformation of one mineral phase to another occurs due to variation in heat, temperature and pressure that causes the homogeneous crystallization and eventually small crystals are formed by re-crystallization in a large number. Bhattacharya *et al.*, (2007) during their investigation confirmed that hydrothermal fluids caused the re-crystallization of hematite and due to oxidation magnetite gets converted to hematite and then appeared in the form of martitized product. SEM pattern shows presence of acicular hematite with interstitial spaces

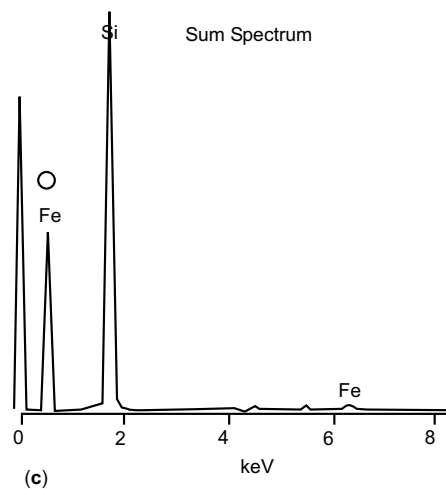
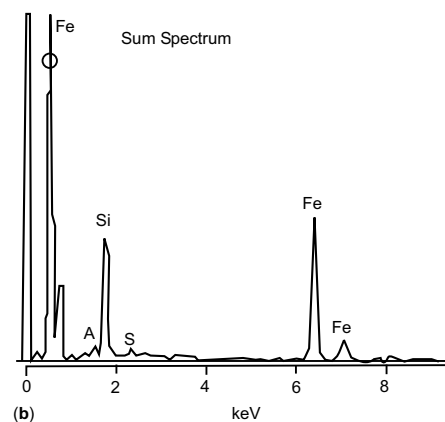
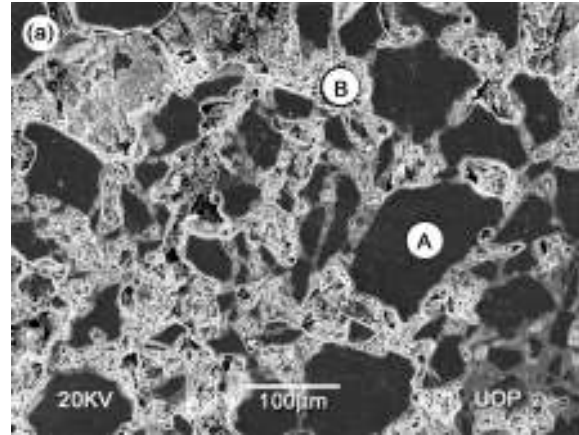


Fig. 3. (a) SEI of the as-mined iron ore sample showing the presence of Quartz with blackish contrast and plan morphology marked as “A” along with Hematite with white contrast and rough granular morphology marked as “B”, (b) EDS spectra of the marked regions B (c) EDS spectrum of the marked region A.

occupied by quartz. EDX analysis shows a high percentage of Si (71 wt.%) and complex interlocking of hematite and silica which made it very difficult for beneficiation. The detailed elemental and relevant oxide composition of the gross and marked micro-regions shown in Fig. 3 is given in Table 2.

EDS analysis showed the presence of high concentration of iron in rough granular micro-regions marked as 'B' and high concentration of silicon in the microregion with black contrast marked as 'A'.

Beneficiation of iron ore via shaking table. EDS spectra of the concentrate obtained from shaking table for iron ore at 100 and 200 mesh sizes are shown in Fig. 4 (a, b). The gross elemental and oxide compositions for 100 and 200 meshes are given in Table 3.

Table 2. Elemental and oxide composition of different micro-regions shown in Fig. 3.

Micro-region/ grain	Elemental composition (wt.%)		Oxide composition (wt.%)		
	Fe	Si	O	Fe ₂ O ₃	SiO ₂
Gross	23.37	39.64	36.99	28.27	71.73
A	99.99	---	---	100
B	71.12	8.35	20.53	85.06	14.94

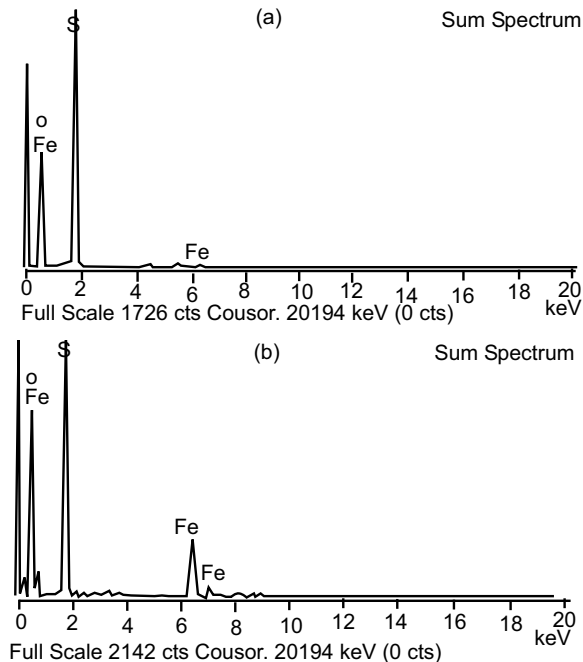


Fig. 4. (a, b): EDS spectra of iron ore after passing via shaking table at 100 and 200 meshes.

EDS study confirmed the concentration of iron in the as-mined ore sample to be 23.37 wt.% and after beneficiation through shaking table at 100 and 200 mesh it was 36.51 and 38.70 wt.% respectively indicating an increase in the concentration after beneficiation. Similarly the content of silica decreased from 39 to 27 wt. %. A comparison of oxide concentration of iron and silica before and after beneficiation is shown in Table 4. Figure 5 shows an increase in concentration of iron and decrease of silica by increasing mesh size from 100 to 200 mesh.

From these results it can be concluded that by reducing particle size of ore the concentration of iron increases and that of silica decreases and more beneficiation will take place. It indicates that minerals begin to separate into iron and silica after 100 mesh particle size due to differences in density. But economically it is not fruitful due to high energy consumption for getting fine particle size. Isnugroho *et.al.* (2011) observed that the process of iron mineral separation from gangue will be higher with increasing level of fine particle size. Passing the ore through magnetic separator brings no up-gradation because of non-magnetic nature of hematite

Table 3. Elemental and oxide composition of iron ore concentrate obtained from shaking table at 100 and 200 mesh sizes.

Mesh size	Gross elemental and oxide composition (wt. %)				
	Elements		Oxide		
	Fe	Si	O	Fe ₂ O ₃	SiO ₂
100	23.50	27.30	49.20	36.51	63.49
200	25.90	25.06	49.04	38.70	61.30

Table 4. Comparison of iron oxide and silica content in the as-mined ore and after beneficiation via shaking table at 100 and 200 mesh sizes.

Gross oxide composition of as-mined iron ore	Oxide composition of iron ore at 100 mesh (149 μm) after beneficiation via shaking table	Oxide composition of iron ore at 200 mesh (74 μm) after beneficiation via shaking table
Oxides	wt%	Oxide wt%
Fe ₂ O ₃	28.26	Fe ₂ O ₃ 36.51
SiO ₂	71.73	SiO ₂ 63.49
		Fe ₂ O ₃ 38.70
		SiO ₂ 61.30

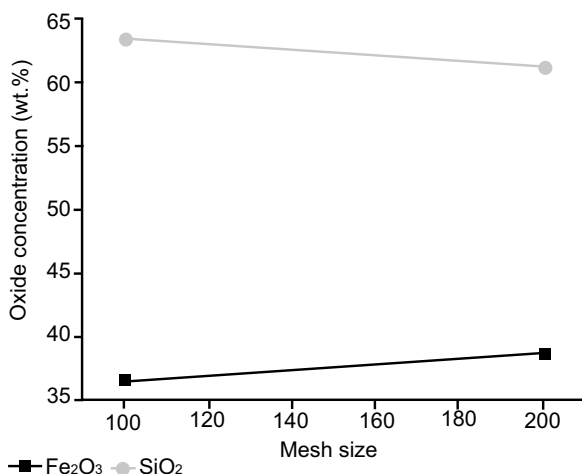


Fig. 5. Variation of the oxide concentration of iron and silica with mesh size, indicating an increase in the iron oxide content and decrease in silica content.

ore. The nonmagnetic iron ore can be changed into a magnetic iron ore through roasting.

Conclusion

In this research work iron ore from Karak, KP, Pakistan was characterized using XRD, SEM and RLM. Karak iron ore is a low grade siliceous iron ore. The major elements detected by EDS in this ore are Iron and silicon. From XRD the presence of iron oxide (Hematite) and silica phases were identified. The up-gradation/beneficiation of iron ore was performed using shaking table and magnetic separator. Through shaking table the iron ore was up-graded from 28.27 wt.% to 38.70 wt.% at 200 mesh, thus achieving a maximum of 10% upgraded ore. Due to non-magnetic nature of the ore the technique of magnetic separation was proved to be ineffective.

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Conflict of Interest. The authors declare no conflict of interest

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