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Micaceous iron oxide production by application of magnetic separation

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Abstract: In this study, different flowsheet options were evaluated to achieve the best upgrading conditions for a micaceous iron oxide ore. The first option included the recovery of micaceous iron oxide particles using a double stage magnetic separation circuit after the grinding and classifying of the ore into coarse (-1000+106 µm) and fine (-106 µm) size fractions. A belt type dry high gradient magnetic separator (BHGMS) was used to beneficiate the coarse fraction. The concentrate of the BHGMS was ground to -106 µm, and combined with the fine fraction produced at screening stage, and subjected to high gradient magnetic separation (HGMS) at 1.2 T. A concentrate grading about 61.33% Fe with 60.3% recovery was obtained applying the separation process incorporating BHGMS and HGMS. A single stage separation circuit considering the use of HGMS after the grinding the ore below 106 µm was employed as the second concentration option. A concentrate having 63.80% Fe with 37.1% weight recovery was obtained in this test. As the highest Fe grade and the lowest S concentration was obtained by application of HGMS after the grinding the ore below 106 µm, and it was decided to conduct a pilot scale study using pulsating HGMS. A concentrate assaying 69.45% Fe with 60.1% weight recovery was produced by operating the pulsating HGMS at 0.6 T. The results showed that it was possible to obtain a micaceous iron oxide concentrate to be used in pigment production using magnetic separation method.

Keywords: *micaceous iron oxide (MIO), magnetic separation, belt type high gradient magnetic separator, high gradient magnetic separator, pulsating high gradient magnetic separator*

1. Introduction

Natural iron oxides occur widely, and are exploited from a variety of deposit types. World iron oxide pigment mine production in 2014 is given in Table 1. Micaceous iron oxide (also known as specular hematite, specularite) is a variety of hematite mineral with a silvery metallic luster. It is mainly used in pigment industry and in welding electrodes as a substitute for iron. Micaceous iron oxide is an excellent pigment in paints providing physical protection for use in protective coatings for steel constructions. The lamellar pigment particles in the paint orientate themselves as overlapping array of parallel plates on the surface which offers greatly enhanced barrier protection against the corrosive substances and water (Cornell and Schwertmann, 2003; Kalenda et al., 2004; Baena, et al., 2009; Ravi et al., 2015). International Standard ISO 10601 requires >85% Fe_2O_3 content in pigment quality micaceous iron oxide concentrates. It also defines the lamellar grade of the concentrates according to the amount of thin flake particles (ISO 10601).

As for the other iron ore types, gravity concentration, magnetic separation, and flotation processes are promising methods for the beneficiation of micaceous iron oxide. Wang et al. (2011) conducted a research study on a micaceous iron oxide ore containing 35% Fe using high intensity magnetic separation and reverse flotation in sequence. A concentrate grading about 66.62% Fe with a weight recovery of 30.65% was obtained in this study. Ravi et al. (2015) studied on a micaceous iron oxide ore

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containing 52.44% Fe applying shaking table and high intensity magnetic separation (HGMS) techniques. A concentrate assaying 67.80% Fe was obtained when HGMS employed at >0.85 T. Vapur and Top (2016) obtained a concentrate containing 92.47% Fe_2O_3 grade and 69.91% recovery yield from the micaceous iron oxide ore containing 62.94% Fe_2O_3 by high intensity wet magnetic separation at 0.6 T.

Table 1. World natural iron oxide mine production in 2014 (Tanner, 2016)

Country	Mine Production
United States	W
Austria (micaceous iron oxide pigment)	3500
Cyprus (umber)	4000
France	1000
India (ochre)	1600000
Pakistan (ochre)	33000
Spain (ochre and red iron oxide)	16000
Germany*	200000
World total	NA

W: Withheld to avoid disclosing company proprietary data, NA: Not available, a significant number of other countries are thought to produce IOPs, but there is no available data. *Includes natural and synthetic iron oxide pigment

High gradient magnetic separation is an effective method for the recovery of fine weakly magnetic particles from the ores (Zheng et al., 2016). On the other hand, pulsating high gradient magnetic separators show better performance to recover fine hematite particles compared to conventional high gradient magnetic separators (Chen et al., 2009). These separators also show high performance in the removal of trace amounts of iron impurities from similar non-metallic ores (Haozi et al., 2016).

There are only limited studies about beneficiation of micaceous oxides in literature (Cornell and Schwertmann, 2003; Kalenda et al., 2004; Baena, et al., 2009; Wang et al., 2011; Ravi et al., 2015; Vapur and Top, 2016). It is a very important raw material, but there are only limited reserves containing different gangue materials. Researchers have mostly worked on removal of gangue materials by laboratory scale traditional magnetic separation or gravity separation techniques. However, it is very difficult to produce clean micaceous iron oxide concentrate when the ore contains pyrite. Pyrite particles are recovered together with micaceous iron oxide particles in gravity concentration process and, therefore specularite grade of concentrate decreases. The traditional magnetic separation equipment are also not sufficient due to mechanical entrainment of pyrite particles into the magnetic product. For this reason, different flowsheet arrangements were applied in this study. A pilot scale pulsating high gradient magnetic separator was also employed to reduce sulfur content of the ore. In the present work, the concentration of a micaceous iron oxide ore using laboratory scale belt type high gradient magnetic separator (BHGMS) and wet high gradient magnetic separator (HGMS) was studied in order to obtain a high-quality product assaying over 85% Fe_2O_3 suitable to be used in pigment industry.

2. Materials and methods

A micaceous iron oxide ore obtained from Manisa, Turkey was used in the study. The sample, as received, was crushed to below 3 mm (d_{80}) using a jaw crusher in two stages. A laboratory size impact crusher was used for crushing the -3 mm material to below 1 mm. The crusher was run with a 1 mm screen in closed circuit. The product of the crusher was screened at 1 mm and oversize material was returned to the impact crusher for further size reduction. The aim of using impact crusher in the size reduction circuit was to increase the amount of flake shaped micaceous iron oxide particles in the feed material before beneficiation tests. The crushed material (-1 mm) was mixed and quartered to small samples of about 1 kg each.

The representative samples were taken for chemical analysis and mineralogical studies after grinding 1 kg of crushed material to below 106 μm using a rod mill. The elemental composition of the samples and products of the experiments were determined by wet chemical analysis methods and AnayltikJenaAG novAA 330 model atomic absorption spectrometry. Powder X-ray diffraction (XRD) analysis was done using RIGAKU-Dmax-2200 PC equipment, operating at 20 kV and 40 mA with Cu–Kα radiation. The elemental composition of the ore sample is presented in Table 2. The result of X-ray diffraction analysis is shown in Fig. 1.

The major phases observed in the XRD scan of the ore were Fe_2O_3, FeS_2, Fe_3O_4, and SiO_2. According to obtained results of X-ray diffraction analysis, it can be suggested that the ore sample from Manisa deposit were observed to be mainly of a hematitic nature. It also contained magnetite as another iron mineral. The main gangue minerals were found as SiO_2 and FeS_2.

Table 2. Chemical composition of micaceous iron oxide ore

Components	(%)
Total Fe	52.49
SiO_2	8.80
Al_2O_3	1.58
CaO	1.05
MgO	2.32
Na_2O	0.06
K_2O	0.09
S	7.23
FeS_2 (calculated)	13.64

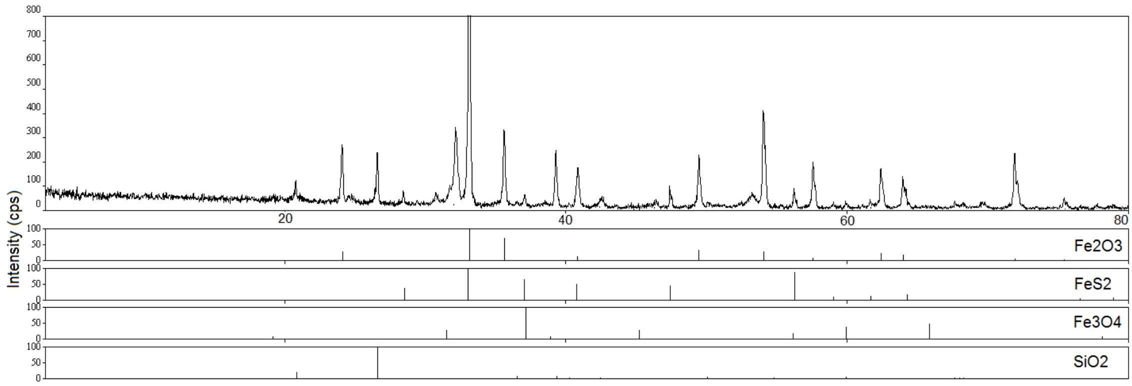

Fig. 1. XRD diffractogram of micaceous iron oxide ore

An optical microscope was used to determine the liberation conditions for micaceous iron oxide particles. The material was sieved into different size fractions for this purpose. As can be seen from Fig. 2, the number of flake-shaped micaceous iron oxide particles increased by decreasing particle size. It was found that the number of flake shaped micaceous iron oxide particles considerably increased in -106 µm size fraction.

A dry high gradient belt magnetic separator was used for BHGMS tests which was mainly consisted of a permanent magnet drum, rotating drum, and a conveyor belt (Fig. 3a). The separator has a fixed magnetic induction of 0.4 T. The wet high gradient magnetic separator of Boxmag Rapid limited was used for the HGMS tests (Fig. 3b). The separator consisted of 2 energizing coils and a removable steel wedge-wire grid pack sitting between coils. The magnetic field intensity could be varied between 0-2 T. The batch type laboratory scale HGMS has a limited space for magnetic product in between its matrix material. Therefore, HGMS tests were conducted using 10 g of samples to prevent from clogging. These small samples were produced from main materials using a Retsch rotary sample divider to provide sufficient accuracy in the sampling process. The tests were also repeated 5 times to increase the reliability of the experiments.

A cyclic pilot-scale pulsating high gradient magnetic separator with 1000 mm ring diameter, 20 mm pulse stroke, 0-300 r/min pulse frequency and up to 7 Mg/h capacity was used in the pilot tests.

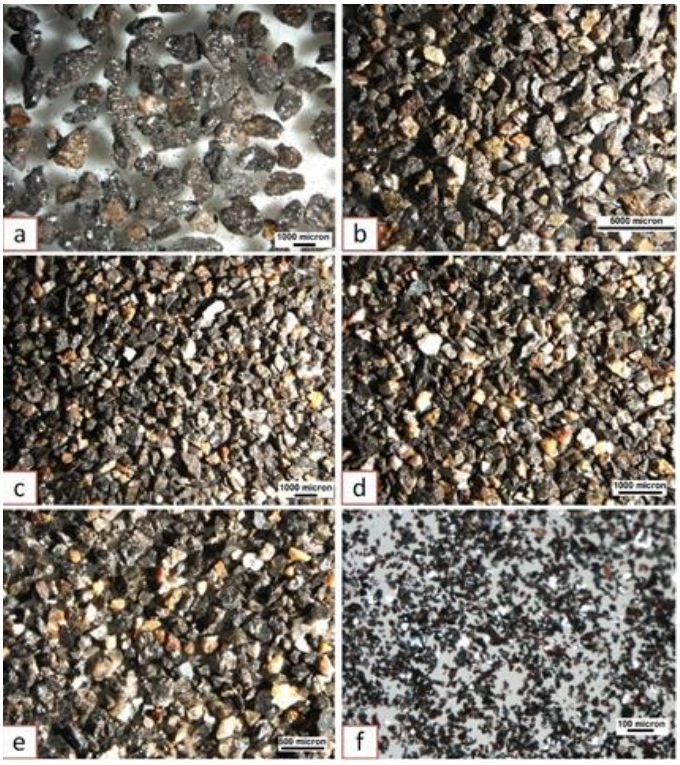

Fig. 2. Pictures of different size fractions (a) 2-1 mm (b) 1000-600 µm (c) 600-400 µm (d) 0.4-0.212 mm € 212-106 µm (f) -106 µm

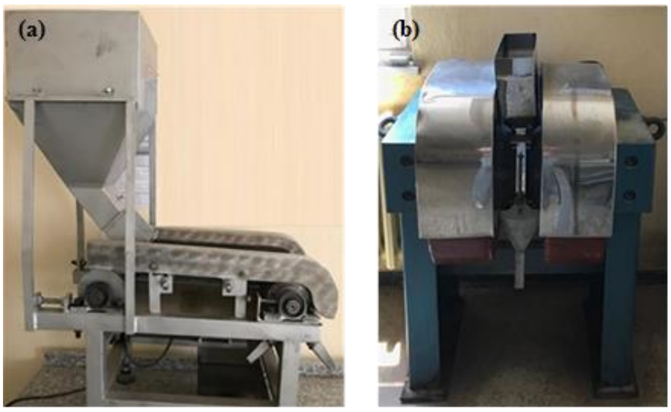

Fig. 3. Laboratory scale (a) dry high gradient belt magnetic separator (b) wet high gradient magnetic separator

3. Results and discussion

3.1 Efficiency of the flowsheet incorporating BHGMS and HGMS

The first experimental flowsheet applied in the study involved the use of BHGMS and HGMS methods in combination to recover iron oxide minerals. The ore sample was classified into coarse (1000-106 µm) and fine (-106 µm) size fractions before the beneficiation tests. The weight and assay distribution of size fractions are presented in Table 3. It can be seen from Table 3 that 1000-106 µm size fraction contains about 83% of the sulfur content of the ore.

The coarse fraction was fed to BHGMS separator at a feed solids rate around 100 g/min for a consecutive 10 min. Beneficiation of coarse fraction by BHGMS produced a magnetic concentrate containing 55% Fe with 39.5% weight and 41.39% Fe recovery. About 76% of sulfur was rejected to tailing by BHGMS at this stage. The concentrate of BHGMS was ground using a rod mill to below 106 µm and combined with the fine fraction from screening (-106 µm).

Table 3. Particle size distribution and size-wise chemical analysis of coarse and fine ore fractions

Product	Weight (%)	Fe (%)	S (%)	Fe Distribution (%)	S Distribution (%)
Coarse fraction (1000-106 µm)	58.7	49.64	10.26	55.51	83.30
Fine fraction (-106 µm)	41.3	56.54	2.92	44.51	16.70
Feed material	100	52.49	7.23	100	100

A 10 g of the produced bulk material was mixed with water to produce a slurry containing 20% of solids. The slurry was subjected to HGMS at 1.2 T (Fig. 4). The grid pack was rinsed with low pressure water after each test to remove attached non-magnetic particles. After the cleaning with low pressure water, the concentrate accumulated in the grid pack was pulled out rinsing with higher pressure water. The test was repeated 5 times to increase the reliability of the experiments. A final concentrate assaying 61.33% Fe was obtained with 60.3% weight and 70.46% Fe recovery by HGMS. Sulfur content of the final concentrate was reduced to 0.67%.

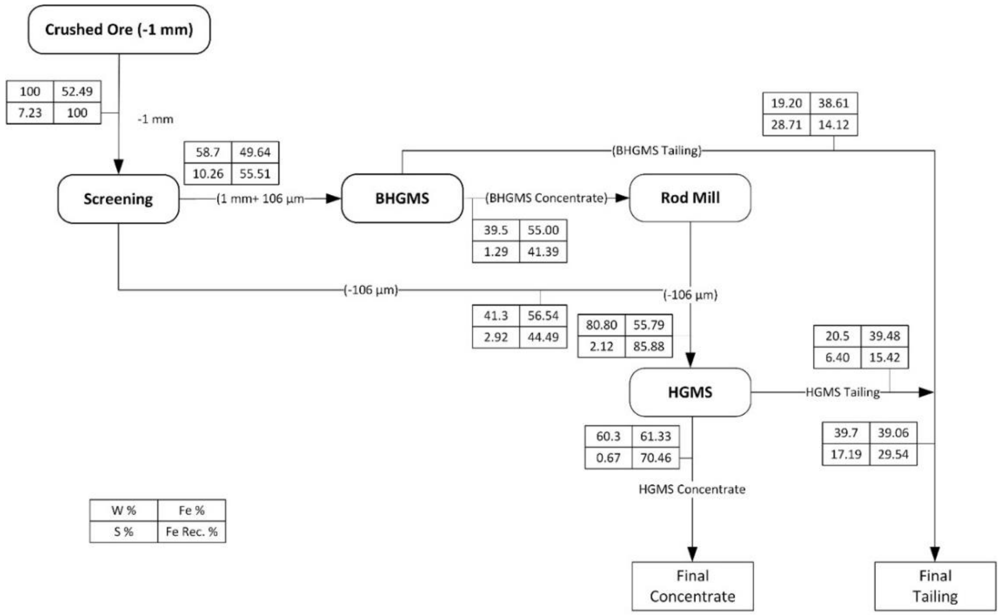

Fig. 4. Beneficiation circuit incorporating BHGMS and HGMS

3.2 Efficiency of the flowsheet with HGMS for the recovery of iron oxides

Based on the experimental results obtained, HGMS was found to be efficient for producing high grade Fe concentrate. In this part of the study, it was decided to simplify the flowsheet by enhancing the liberation degree of the feed material by grinding and using HGMS for separation.

The feed material was ground to below 106 µm using a rod mill prior to separation by HGMS. 10 g of the ground material was mixed with water to produce a slurry containing 20% of solids and subjected to HGMS at 1.2 T (Fig. 5). The concentrate was subjected to cleaner magnetic separation to increase the purity of the concentrate. The test was repeated 5 times to increase the reliability of the experiments.

The concentration of the ground feed material by HGMS allowed increasing the Fe content in the concentrate up to 63.80%. The weight and Fe recovery values of the concentrate were 37.1% and 45.09% respectively. The sulfur content of the concentrate was higher compared to the previous test due to the presence of interlocked particles of pyrite. It seemed removing the coarse pyrite particles by BHGMS before grinding helped to improve the performance of HGMS in pyrite rejection.

As can be seen from Fig. 6, a flake-rich high grade micaceous iron oxide product assaying 67.28% Fe was obtained with 21.5% weight and 27.56% Fe recovery by sieving the final concentrate at 45 µm. The sulfur content was reduced to 0.53% in this fraction (Fig. 5). Higher content of sulfur in +45 µm fraction of the concentrate indicates the presence of unliberated pyrite particles.

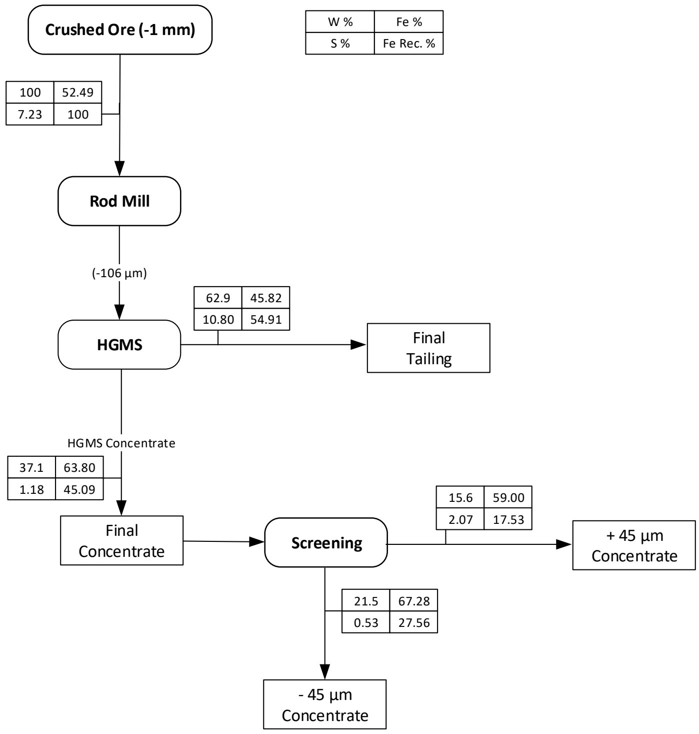

Fig. 5. Single stage beneficiation by HGMS

3.3 Results of the pilot scale pulsating high gradient magnetic separator test

The previous tests showed that the flake shaped micaceous iron oxide particles can be successfully obtained from the magnetic separation concentrates by sieving at 45 µm. Considering the market specifications and the presence of unliberated pyrite particles in the +45 µm fraction of the HGMS concentrate in the previous test, it was decided to grind the feed material below 45 µm particle size. The feed material was subjected to hand picking to remove coarse pyrite particles before comminution. The sulfur content of the feed was reduced to about 2% by hand picking.

A 1000 kg of -15 mm feed material was ground below 45 µm (d_{90}) using a pendulum ball mill and subjected to magnetic separation using a cyclic pilot-scale pulsating high gradient magnetic separator. The particle size distribution of the ground feed is seen in Fig. 7.

The tests were conducted at 0.9 T and 0.6 T under the operational conditions of 2 t/h feed rate and 20% of solid ratio. The results are presented in Table 4.

Table 4. Results of the pilot scale pulsating high gradient magnetic separator test

Magnetic field intensity (T)	Product	Weight (%)	Fe (%)	S (%)	Fe Recovery (%)	S Recovery (%)
0.6	Concentrate	60.1	69.45	0.19	75.10	5.58
	Tailing	39.9	34.69	4.84	24.90	94.42
	Feed material	100	55.58	2.05	100	100
0.9	Concentrate	79.8	61.54	0.45	90.08	17.52
	Tailing	20.2	26.79	8.37	9.92	82.48
	Feed material	100	54.52	2.05	100	100

Pulsating high gradient magnetic separator played an important role as a concentrating device in improving the quality of the iron concentrate, especially for the reduction of the sulfur content. A concentrate containing 69.45% Fe grade and 0.19% S with 60.1% weight recovery was obtained by operating the equipment at 0.6 T. The concentrate is suitable for production of micaceous iron oxide pigments for paints (ISO 10601) according to Fe grade, particle size and lamellar structure (Fig. 8). Iron concentrate weight recovery of 79.8% was achieved with 61.54% Fe grade when the separator was operated at 0.9 T. However, the sulfur content of the concentrate increased to 0.45%.

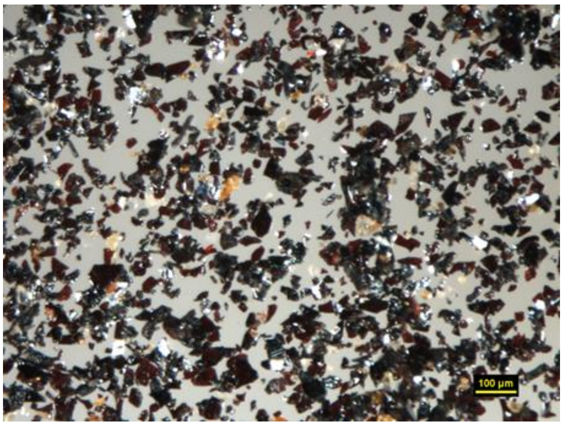

Fig. 6. Picture of the -45 μm fraction of concentrate produced from single stage beneficiation process by HGMS

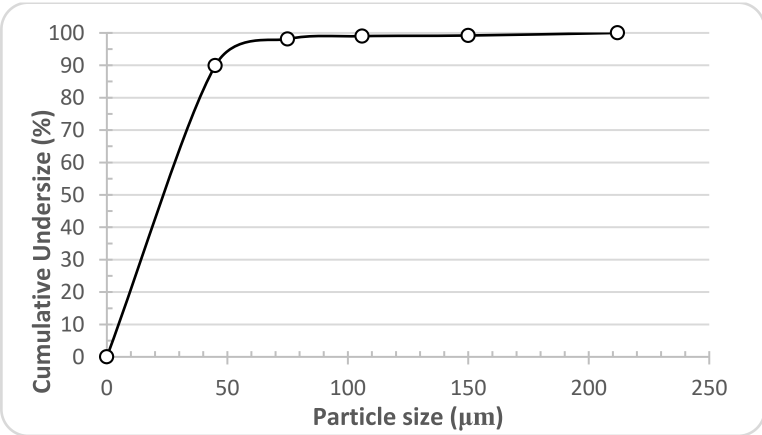

Fig. 7. Particle size distribution of the feed after grinding in pendulum ball mill

Fig. 8. Picture of the pulsating high gradient magnetic separator concentrate produced at 0.6 T

4. Conclusions

The results of this study clearly showed that the effective separation of micaceous iron oxide particles from the specular hematite ore sample by magnetic separation was possible as it was stated by earlier studies for different micaceous iron oxide ores. A final iron concentrate assaying 61.33% Fe with 60.3% recovery was obtained by the application of an experimental flowsheet incorporating BHGMS and HGMS. A higher grade concentrate (63.80% Fe) was obtained by the grinding of the ore below 106 µm and applying HGMS, however the recovery decreased to 37.1%. Both the flowsheet options were failed to reduce the sulfur content of the iron concentrate below 0.2%. The separation performance was found better when the pulsating high gradient magnetic separator was employed in the pilot scale test at fine particle size. The equipment successfully removed pyrite particles which were mechanically entrained into the magnetic product. A concentrate assaying 69.45% Fe with 60.1% recovery and 0.19% S content was produced by operating the pulsating high gradient magnetic separator at 0.6 T. The product of pilot plant pulsating high gradient magnetic separator test at 0.6 T meet the requirements described in ISO 10601 "Micaceous iron oxide pigments for paints- Specifications and test methods". The economic value of the product is relatively high (>1000 USD/t) compared to the other iron ore concentrates (about 100 USD/t). Overall, it can be concluded that the suggested process will be economically viable in future.

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