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Comparative Methods on The Upgrading of Fe Goethite Iron Ore

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ABSTRACT

Iron ore is one of the most vital supplies in the world after oil, and its demand has increased intensely in the last two decades. Goethite (α-FeO(OH)) is an iron oxyhydroxide containing ferric iron ore that exists abundantly in the earth's crust however, it consists of Fe with a low grade which makes it an unfavourable resource. Thus, Fe upgrading and economic consideration of such ore are crucial. The analysis result of the iron ore rock sample taken from Kelantan comprises 51.98% Fe, 4.01% Si, 1.99% Al, 0.0053% Mn, 0.0023% P, 0.6103% C and 0.0460% S. Yet, based on XRF and XRD results, the raw sample consists of 50.79% Fe and 60% of goethite minerals, respectively. The Fe upgrading processes used were the combination of two or three methods processes namely roasting, gravity, magnetic, and electrostatic separations. The methods were (1) spiral – double disc magnetic separator (DDMS), (2) shaking table (ST) – wet highintensity magnetic separator (WHIMS), (3) ST – high tension separator (HTS), (4) roasting – WHIMS – ST, and (5) roasting – ST. The fifth method appeared to be the most effective and economical. The Fe was successfully upgraded to 60.52% (medium grade) with a recovery of 59.76%. Other elements were also slightly reduced such as 1.36% Si, 1.77% Al, 0.0050% Mn, 0.0021% P, 0.4809% C, and 0.0386% S.

Keywords: *Fe content, Goethite, Iron ore, Roasting, Shaking table*

1. INTRODUCTION

Iron ore is known to be one of the most vital commodities in the world after oil. As the main backbone of a global infrastructure for crude steel production, iron ore demand has elevated intensely in the last two decades. As of 2022, Australia is the largest iron ore mining country in the world with a total production of 8.00 x 10⁸ metric tonnes followed by Brazil (4.10 x $10⁷$ metric tonnes) and China (3.80 x $10⁷$ metric tonnes) [1]. Meanwhile, 9 out of 15 of the world's largest crude steel producers in 2022 were dominated by China. China's Baowu Group ranked first place and produced 1.32 x 10⁸ metric tonnes of crude steel in the same year [2].

In the meantime, the production of iron ore in Malaysia $(4.98 \times 10^6 \text{ metric tonnes})$ was found to be a slight decline compared to 2020 [3]. This can be attributed to the postimpact of the COVID-19 pandemic, the Russia-Ukraine war, and the high inflation which led to the slow production of iron ore. Most iron ores mined in Malaysia contain low grades of iron ore and their grades can be categorised as high-grade, medium-grade, and low-grade ores corresponding to their Fe content > 65%, 62 – 64%, and < 58%, respectively [4]. To that concern, the diminishing of high-grade iron ore reserves is one of the major issues faced by steel industries and therefore the low-grade iron ores are mined as an alternative feedstock [5].

Several studies have been conducted, by utilising a lowgrade iron deposit such as goethite minerals for the highgrade iron ore replacement. Nevertheless, goethite-iron ore is another challenging issue for Fe upgrading which is using a direct physical separation such as gravity or magnetic separations seems to be a less effective approach. This is because goethite consists of oxygen and hydroxyl anions (OH group) with a formula molecule of α -FeO(OH) and bonded via hydrogen bonding that is tightly packed in hexagonal arrays [6]. Thus, this mineral involves an endothermic reaction (200 – 300 °C) [7] so that magnetic materials can be formed as well as removing the H2O (e.g., water or vapour) from the gauge for better separation. Due to this complexity, instead of using direct physical separation, some researchers used pyrolysis prior to magnetic separation to upgrade the Fe.

Recent studies by Nunna and co-workers [8] used microwave-assisted reduction roasting to upgrade Fe from the rejected waste of goethite-rich iron ore from Western Australia. They employed roasting temperature between 370 – 1000 °C under the gas atmosphere of 30:70 and 40:60 CO/CO² for 20 mins. The results revealed that the best condition for the formation of synthetic magnetite from goethite was at 600 °C in a gas atmosphere of 40:60 CO/CO² for 20 mins. Roy *et al*. also implemented microwaveassisted reduction roasting followed by magnetic separation via a low-intensity magnetic separator (LIMS) [9]. The final Fe content can be increased to 61.57% from 49% with a Fe recovery of 64.47%.

Jaiswal *et al*. [10] from India have performed magnetising roasting using coal as the reducing agent (700 – 900 °C) and followed by magnetic separation via wet high-intensity magnetic separator (WHIMS) of low-grade goethite iron ore. The study shows that the Fe content can be improved from 40.21% to 59.20% with Fe recovery of 68.23% using the optimal condition roasting at 900 °C for 50 mins and magnetic field at 2000 Gauss. Ravisankar and team investigated the beneficiation of goethite-rich iron ore using reduction roasting using coal followed by magnetic separation through LIMS [11]. They had successfully upgraded the Fe content to 66.6% from 58% with a recovery of 90.4%. Another study conducted by Umadevi and team developed a beneficiation process including crushing, grinding, jigging, spiral, size reduction, desliming, and magnetic separations of goethite-iron ore in India [12]. The Fe content can be upgraded as high as 63.5% from 49.2%.

Therefore, the main aim of the study is to investigate the most effective method for Fe upgrading from low-grade iron ore enriched with goethite minerals. The methods used were direct physical separations namely gravity and magnetic separations or a combination of roasting followed by gravity or magnetic separations.

2. MATERIAL AND METHODS

2.1. Raw Material

The Iron ore sample used in this study was from Iron ore rock from an undisclosed area in Kelantan, Malaysia as shown in Figure 1. All the experiments were conducted at the Mineral Research Centre, Department of Mineral and Geoscience (JMG) *Ipoh*, Perak.

Figure 1. Raw sample of iron ore rocks.

2.2. Sampling Method

Approximately 45 kg of an iron ore rock sample was dried under the dryer for 3 to 4 hours. The sample was crushed using jaw and cone crushers. The sample was mixed 16 times using the rolling and matting method and then sampled according to the cone and quartering method. All samples were weighed and recorded.

2.3. Grinding Process

Approximately 1 kg of sample was ground using a grinding mill at 11 minutes at 78 rpm. Spherical mill balls made of steel with three different sizes were used to grind the sample [13]. The ground sample was then sieved using a 600 μ m sieve and a sample below 600 μ m (-600 μ m) was collected as shown in Figure 2. All samples used were -600 µm unless stated.

Figure 2. Ground sample after sieved.

2.4. Fe Upgrading Methods

2.4.1. Method 1: Spiral and Double Disc Magnetic Separator (DDMS)

The sample was separated using gravity separation by means of the spiral, then followed by magnetic separation via DDMS as shown in Figure 3a and Figure 3b, respectively. The feed flow rate of the spiral was fixed at 80 L/min using 16% of feed solid. After the spiral, all the products were further separated using the DDMS. The current intensity applied at disks 1 and 2 were fixed at 0.4 Amp and 0.6 Amp, respectively.

Figure 3. (a) Gravity separation via spiral and (b) magnetic separation via DDMS.

2.4.2Method 2: Shaking Table (ST) and Wet High-Intensity Magnetic Separator (WHIMS)

The sample was separated using gravity separation via ST, then it underwent magnetic separation via WHIMS as shown in Figure 4(a) and Figure 4(b), respectively. The inclined angle, water flow rate, and shaking frequency of the ST were fixed at 3°, 120 L/min, and 29 Hz, respectively and the sample size used was $-106 \mu m$. After ST, all the products were further separated using the WHIMS. The current intensity applied was at 0.5 Amp with no repetition.

Figure 4. (a) Gravity separation via ST and (b) magnetic separation via WHIMS.

2.4.3Method 3: Shaking Table (ST) and High-Tension Separator (HTS)

The sample was separated using gravity separation via ST followed by conductivity separation through HTS (Figure 5). All the ST parameters were the same as in section 3.4.2 however, the sample size used was -300+45 µm. After ST, all the products were further separated using the HTS. The voltage applied was at 30 kV with no repetition.

Figure 5. Conductivity separation via HTS.

2.4.4Method 4: Roasting, Wet High-Intensity Magnetic Separator (WHIMS), and Shaking Table (ST)

Approximately 260 g of samples were roasted at 400 °C, 600 °C, and 800 °C using a furnace (CWF1200, CARBOLITE GERO, Germany) for 1 hour as shown in Figure 6. After roasting, all samples underwent magnetic separation twice via WHIMS at 1.0 Amp. The samples were further separated using ST and the parameters were the same as in section 3.4.2, however, the separation was repeated in three stages.

Figure 6. (a) Roasting via furnace, (b) raw sample, roasted sample at (c) 400 °C, (d) 600 °C, and (e) 800 °C.

2.4.5 Method 5: Roasting and Shaking Table (ST)

The sample was roasted at 800 °C using a furnace for 1 hour. Then the sample underwent gravity separation via ST. The parameters were the same as in section 2.4.2, however, the separation was repeated in four stages.

2.5. Determination of Oxide Elements by X-ray Fluorescence (XRF)

The oxide elements of the sample were determined using X-Ray Fluorescence Spectrometer (XRF-1700, Shimadzu, Japan).

2.6. Determination of Mineral Phases by X-ray Diffraction (XRD)

The mineral phases of the sample were determined using Xray Diffraction (XRD-D8, Bruker, Germany). The XRD analysis was performed at a scanning rate of 2˚ per minute and 2θ range of 4° - 70 $^{\circ}$.

2.7. Determination of Iron (Fe) Content

Approximately 45 kg of an iron ore rock sample was dried under the dryer for 3 to 4 hours. The sample was crushed using jaw and cone crushers. The Fe content was determined based on the titration method using Potassium dichromate (VI) $(K_2Cr_2O_7)$ solution and barium diphenylamine sulfonate $(C_{24}H_{20}BaN_2O_6S_2)$ solution used as the Fe indicator [13].

2.8. Determination of Silicon (Si) Content

The Si content was determined using an Ultraviolet-Visible (UV-Vis) Spectrophotometer (Lamda 25, Perkin Elmer, UK).

2.9. Determination of Aluminium (Al) Content

The Al content was determined using Atomic Absorption Spectroscopy (AA6800, Shimadzu, Japan).

2.10. Determination of Carbon (C) and Sulfur (S) Contents

The C and S contents were determined using a carbon and sulfur analyser (G4 ICARUS HF, Bruker, Germany).

2.11. Determination of Manganese (Mn) and Phosphorus (P) Contents

The Mn and P Contents were determined using an Inductively Coupled Plasma – Optical Emission Spectrometry (ICP-OES) (Optima 5300 DV, Perkin Elmer, USA).

2.12. Microscopic Observation

The sample was viewed using a light microscope (U-PMTVC 3K00462, Olympus, Japan).

3. RESULTS AND DISCUSSION

3.1. Characterisation of Raw Sample

3.1.1. Chemical Assay

The chemical assay results of the iron ore rock raw sample are presented in Table 1. The sample consists of 51.98% Fe which can be categorised under low-grade iron ore.

3.1.2 X-Ray Fluorescence (XRF) Analysis Result

The chemical compositions of the iron ore rock sample are shown in Table 2. The XRF analysis shows that the sample mainly contains 72.6286% Fe₂O₃ and is followed by compounds such as SiO² (9.6196%), Al2O3 (9.6196%), MnO (9.6196%) and P_2O_5 (9.6196%). Other compounds were also detected but less than 1%.

3.1.3 X-Ray Diffraction (XRD) Analysis Result

The result of the XRD analysis of the iron ore rock sample is illustrated in Figure 7. From the result, the sample mainly consists of goethite, α-FeO(OH) mineral. Other minerals such as hematite, magnetite, quartz, leucite, and manganese hydrogen phosphate hydrate were also detected on the sample.

Figure 7. XRD diffractogram of iron ore rock from an undisclosed area of *Kelantan*, Malaysia.

3.2. Fe Content and Recovery Results

3.2.1. Method 1: Spiral and Double Disc Magnetic Separator (DDMS)

Tables 3(a) and 3(b) show the Fe content and recovery after the sample was processed using spiral and DDMS, respectively. Via spiral, the Fe content from the spiral concentrate sample can be upgraded up to 52.49% with a

Fe recovery of 61.54%. Via DDMS, the Fe content from Spiral Midd/Mag 0.4 A sample was slightly increased to 53.19% with a Fe recovery of 90.40%. Method 1 was found to be less effective for Fe upgrading that consists of goethite minerals.

Product	wt. (%)	Fe $(%$	Fe Recovery (%)
Spiral Concentrate	24.22	52.44	24.44
Spiral Middling	60.91	52.49	61.54
Spiral Tailing	14.87	48.99	14.02
Total	100.00		100.00

Table 3(a) Fe content and recovery after spiral

Table 3(b) Fe content and recovery after DDMS

Product	wt. (%)	Fe (%)	Fe Recovery (%)
Spiral Conc/DDMS 0.6 A	14.81	52.70	15.01
Spiral Conc/DDMS 0.4 A	73.46	52.70	74.45
Spiral Conc/ DDMS Non-Mag	11.73	46.73	10.54
Total	100.00		100.00
Spiral Midd/DDMS 0.6 A	4.25	52.19	4.21
Spiral Midd/DDMS 0.4 A	89.56	53.19	90.40
Spiral Midd/ DDMS Non-Mag	6.19	45.85	5.38
Total	100.00		100.00
Spiral Tail/DDMS 0.6 A	80.53	50.76	82.93
Spiral Tail/DDMS 0.4 A	3.49	50.12	3.55
Spiral Tail/ DDMS Non-Mag	15.98	41.73	13.53
Total	100.00		100.00

3.2.2. Method 2: Shaking Table (ST) and Wet High-Intensity Magnetic Separator (WHIMS)

Tables 4(a) and 4(b) show the Fe content and recovery after the sample was processed using ST and WHIMS, respectively. Via ST the Fe content from the ST Middling sample can be upgraded up to 54.06% with the Fe recovery of 0.85%. Via WHIMS, the Fe content from ST Conc/WHIMS DDMS 0.5 A was slightly increased to 54.20% with a Fe recovery of 3.11%. Unfortunately, the ST Midd/WHIMS DDMS 0.5 A sample was not analysed due to the amount of sample attained from the ST Middling sample being too low. Thus, method 2 was also found to be less effective for Fe upgrading that consists of goethite minerals.

*NA- sample not analysed due to sample amount < 0.5g; *NC- cannot be calculated as one of the Fe was not analysed.

3.2.3Method 3: Shaking Table (ST) and High-Tension Separator (HTS)

Tables 5(a) and 5(b) show the Fe content and recovery after the sample was processed using ST and HTS, respectively. Via ST the Fe content from the ST Concentrate sample can be upgraded up to 54.67% with the Fe recovery of 32.37%. However, via HTS all the Fe contents were lower than in Table 5(a). Therefore, method 3 was an ineffective method for Fe upgrading that consisted of goethite minerals.

Product	wt. (%)	Fe (%)	Fe Recovery (%)
ST Conc/HTS Conductive	40.58	54.41	40.64
ST Conc/HTS Middling	30.74	54.55	30.86
ST Conc/HTS Non- Conductive	28.68	53.98	28.50
Total	100.00		100.00
ST Midd/HTS Conductive	31.03	52.89	30.92
ST Midd/HTS Middling	68.06	53.21	68.22
ST Midd/HTS Non- Conductive	0.91	50.13	0.86
Total	100.00		100.00
ST Tail/HTS Conductive	14.38	51.58	14.19
ST Tail/HTS Middling	84.03	52.44	84.29
ST Tail/HTS Non- Conductive	1.59	50.03	1.52
Total	100.00		100.00

Table 5(b) Fe content and recovery after HTS

3.2.4Method 4: Roasting, Wet High-Intensity Magnetic Separator (WHIMS), and Shaking Table (ST)

Tables 6(a), 6(b), and 6(c) show the Fe content and recovery after the sample was roasted and processed using WHIMS and ST, respectively. Via roasting all the Fe contents have significant improvement. The highest was from the 800 °C samples with 58.98% Fe. However, via WHIMS all the Fe contents were observed to have no improvement. Moreover, all the Fe from magnetic samples were lower than non-magnetic samples. Meanwhile, via ST x 3 both samples from 800 °C/ WHIMS Non-Mag /ST Conc x 3 and 800 °C/ WHIMS Non-Mag /ST Midd x 3 can be upgraded up to 59.06% and 59.15% with the Fe recovery of 57.56% and 31.06%, respectively. Method 4 was observed to have a significant improvement in Fe content however the Fe grade is still under low-grade quality. Perhaps slightly modifying by skipping the WHIMS might improve the Fe quality.

Table 6(b) Fe content and recovery after WHIMS

Product	wt. (%)	Fe (%)	Fe Recovery (%)
400 °C/WHIMS 1.0 A x 2	1.54	53.58	1.52
400 °C/WHIMS Non-Mag	98.46	54.26	98.48
Total	100.00		100.00
600 °C/WHIMS 1.0 A x 2	1.26	56.64	1.26
600 °C/WHIMS Non-Mag	98.74	56.63	98.74
Total	100.00		100.00
800 °C/WHIMS 1.0 A x 2	1.50	55.35	1.43
800 °C/WHIMS Non-Mag	98.50	58.26	98.57
Total	100.00		100.00

Table 6(c) Fe content and recovery after ST

The comparison of the XRD diffractogram for the raw sample and roasted samples is illustrated in Figure 8. The increase in Fe % is likely due to the removal of the hydroxy group (-OH) and the conversion of wustite (FeO) from the goethite mineral (α -FeO(OH)). Moreover, the LOI value was increased when the roasting temperature was increased indicating high hydroxy-compound goethite [9]. Wustite mineral was detected when the roasting temperature increased to 400 °C and so on. By increasing the temperature to 600 °C and 800 °C, it was shown that some of the wustite phases were also converted to hematite $(Fe₂O₃)$ however not much of magnetite $(Fe₃O₄)$ was detected in 600 °C and 800 °C samples. Hence, in method 5 roasting temperature applied was 800 °C.

3.2.5 Method 5: Roasting and Shaking Table (ST)

Table 7 shows the Fe content and recovery after the sample was roasted and separated using ST. The Fe content of the roasted sample has shown an improvement with 55.72%. Via ST x 4 all samples were shown to have a significant improvement in Fe. The 800 °C/ST Conc x 4 and 800 °C/ST Midd x 4 samples provide Fe content up to 60.52% and 60.46% and Fe recovery of 59.76% and 29.57%, respectively. Both Fe grades have achieved medium-grade iron. Hence in this study, method 5 was the most effective method for Fe upgrading consisting of goethite minerals.

Figure 8. Comparison of XRD diffractogram of the raw iron ore, 400 °C, 600 °C, and 800 °C samples.

Table 7 Fe content and recovery after roasting and ST

Figure 9 shows the raw and roasted samples under a light microscope. From these samples, it can be clearly observed that after being roasted at 800 °C the samples (Figure 9(d) and 9(e)) were intact and agglomerated with irregular shapes compared to the raw sample (Figure 9(b) and 9(c)) which more liberated form. In fact, based on colour observation the raw and roasted samples have dark reddish and light brown colours, respectively.

Figure 9. (a) Raw and roasted samples at 800 °C under naked eyes, (b) and (c) raw sample viewed under a light microscope at a magnification of 10x, roasted sample viewed under a light microscope at a magnification of (d) 10x and (e) 20x.

3.3. Overall Comparison Method

Table 8 and Figure 10 show the comparison table and illustrated graph for each method based on the highest Fe content, respectively. This study elucidated that, using the combination of gravity followed by dry or wet magnetic separation does not improve the Fe grade. This is due to the presence of a major mineral which is goethite (α-FeO(OH)). Goethite is a non-magnetic mineral thus using magnetic separation is not compatible. Similarly, using gravity followed by conductivity separation also does not improve the Fe grade as goethite also has poor electrical conductivity. However, poor magnetic susceptibility does not necessarily represent poor electrical conductivity. Thus, changing the chemical structure of goethite perhaps can enhance the magnetic separation.

Figure 10. Comparison of Fe content and recovery from each method.

Applying high temperatures (400 – 800 °C) on the sample proves the conversion of goethite to other minerals such as wustite, hematite, and magnetite. However, the number of magnetic minerals formed was slight. In addition, by removing the -OH group or water from the sample the specific gravity of certain minerals is changed. Thus, performing gravity separation in multi-stages is observed to be effective. In comparison with other works of literature studies [8 – 10], that used a reducing agent (e.g., coal and CO² gas) during the roasting process, in this study, no additional chemicals or gas were added. Although, without a reducing agent, the temperature required for roasting could be high. The overall process flowchart of the study is presented in Figures 9(a) and 9(b).

4. CONCLUSION

The study was performed to upgrade Fe from low-grade goethite-iron ore. Five methods were employed which are a combination of physical methods namely roasting, gravity, magnetic, or conductivity separations. The importance of the pyrolysis process for goethite-iron ore is crucial due to the hydroxyl group that contributes to the low magnetic properties of the iron ore. However, after pyrolysis, not all goethite minerals can be fully converted to magnetic minerals, thus some researchers have introduced reducing roasting to overcome this issue.

In this present study, a conventional via high-temperature roasting approach was used. Among the methods being implemented, method 5 was found to be the most effective method as it can upgrade the Fe from 51.89% up to 60.52% which reached medium-grade (60 – 64% Fe). Besides, the Fe from the tailing product also improved which is provided through method 5, the % of Fe increment was around 7.67 – 8.54%. Method 5 provides an alternative method that skips the magnetic separation, but instead from roasting to gravity separation which is more economical and feasible to be used on an industrial scale. Table 9 shows the

comparison of overall assay results among the raw sample with the products attained from method 5 and the specification of standard primary steel making. From this Table, it can be concluded that most of the product quality does not meet the specification standard for primary steel making. Hence, further separation can be done to achieve the target range.

Assay of Element	Percentage (%)				
	Raw sample	800°C/ST Conc x 4	800°C/ST Midd x 4	800°C/ST Tail x 4	Standard*
Fe	51.98	60.52	60.46	59.65	>60
Si	4.01	1.36	1.63	2.97	$0.4 - 1.2$
Al	1.99	1.77	1.77	2.89	NS
Mn	0.0053	0.0050	0.0075	0.0075	$0.6 - 1.2$
P	0.0023	0.0023	0.0017	0.0017	${}_{0.2}$
C	0.6103	0.4809	0.5476	0.5637	$3.8 - 4.5$
S	0.0460	0.0386	0.0523	0.0769	0.04

Table 9 Comparison of the assaying result of the raw and processed samples from Method 5 with the standard for primary steel-making

*Standard for primary steel-making [14];

*NS – Not stated.

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