DEVELOPMENT OF IMPURITIES REMOVAL PROCESS FOR LOW-GRADE SANJE IRON ORE USING MINERAL PROCESSING TECHNOLOGIES

Moses Charles Siame[a], Julius Kaoma[b], Atsushi Shibayama[c]

Abstract

In this study, the impurities removal process for low-grade Sanje iron ore was developed using Wet High-intensity magnetic separation (WHIMS) and Reverse flotation (RF). Sanje Iron Ore is the low-grade hematite ores found in Nampundwe area of Zambia from which Iron is to be extracted and used as the feed in the steelmaking process. The ore contains 34.18 mass per cent Iron grade, 31.10 mass% of Silica (SiO_2) and 7.65 mass per cent Alumina (Al_2O_3). Magnetic Separation experiments were done using Series L Model 4 laboratory magnetic Separator (L-4 Machine) as the first stage impurity removal process and the effect of various magnetic separation parameters such as magnetic flux density, particle size density and pulp density of the feed were studied. The results showed that 10 T was optimal magnetic flux density which enhanced the recovery of 93 per cent of iron with 53.22 mass per cent grade. The iron concentrate produced from magnetic separation contained 12.04 massper cent Silica and 3.94 massper cent Alumna and therefore, it was further treated using Reverse flotation. In reverse flotation, various parameters such as pH, collector dosage, Iron depressant dosage and quartz activator dosage were investigated. The results showed that 81.94 per cent was recovered at the concentrate's pH of 6.8 using 200 g/T of 0.1 per cent calcium oxide (CaO) as silica activator and one kg/T of 0.1 per cent causticised starch as Iron depressant. Sodium Oleate (NaOL) and Dodceylamine Acetate (DAA) each with discrete dosage, were used as Anionic and Cationic collectors respectively. Alumina was consequently reduced to 1.04 mass per cent and Silica to 2.04 mass per cent at optimum respective collector's dosage of 0.250 kg/T using 0.02 kg/T of Methyl Isobutyl Carbinol (MIBC) frother. Additionally, phosphorous was also observed to be reduced from 0.05 mass per cent to 0.01 mass per cent. The designed multi-stage process involving feeding the concentrate from WHIMS into RF process therefore produced a high-grade iron concentrate iron with 67.27 mass per cent grade, 2.04 mass per cent silica, 1.04 mass per cent alumina.

Keywords: *Sanje Iron Ore, Magnetic Separation, Reverse floatation, Silica, Alumina.*

[a,c] Graduate School of International Resource Sciences, Akita University, 1-1 Gakuen-cho, Tegata Akita-shi 010-8502, Japan

[b] Universal Mining and Chemical Industries Limited, Kafue steel plant, plot 3944, Chibuku road, Lusaka, Zambia
Email: mosescharlessiame@gmail.com)

INTRODUCTION

Extraction of iron from low-grade ores has recently become an alternative iron beneficiation process due to decreasing high-grade Iron Ores and increasingly high demand of iron concentrate for steelmaking process, especially in the developing countries, Ravelomanana *et al.* (2014). Sanje Iron Ore is the hematite ore deposit found in Nampundwe area of Zambia from which iron is to be used as the feed in the steelmaking process. The ore contains 48.90 mass per cent of hematite (Fe_2O_3) with 34.18 mass% iron grade. The ore also contains very high silica (SiO_2) and alumina (Al_2O_3) of 31.10 mass per cent and 7.65 mass per cent respectively. The presence of high alumina and silica in the iron ores adversely affects productivity in the blast furnace and the rate of impurity reduction in sponge iron/DRI plants, Umadevi (2014). In blast furnaces, high alumina and silica create high viscous slag with greater volume which requires more flux and hence fuel consumption. On the other hand, thick scaling is produced in sponge iron or direct reduced iron (DRI) plants when higher high alumina and silica containing concentrates are fed. This hampers the required heat transfer process during Iron oxide reduction in sponge Iron production, Prithviraj *et al.* (2016). This study aimed at removing silica and alumina from low-grade Sanje ore to produce high-grade iron concentrate (Fe > 60 mass per cent) with minimum impurities (SiO_2+Al_2O_3 < 7 mass per cent), using mineral processing technologies. Removal of silica and alumina from low-grade iron ores is considerably difficult due to the aggregation of fine silica-alumina particulates with iron oxide particles. Magnetic separation technique has therefore been known as one of the most effective iron extraction processes which are commonly used in the treatment of low-grade ore high amounts of impurities, Rath *et al.* (2016). Another commonly used method of iron beneficiation is flotation, which is done either direct or reversed, Araujo *et al.*(2005). Flotation processes using either cationic or anionic collectors is further considered to be one of the more effective methods to separate Iron-gauge minerals which are aggregated together and are weakly magnetic particle, Arol (2004). The choice of the type of flotation method used depends on the chemical and mineralogical composition of the ore to be treated, Kumar (2015).

MATERIAL AND METHODS

Sample Preparation

Lumps of the ore samples were crushed and reduced to 2mm using the P-1 Jaw crusher (Fritsch Co, Ltd) and then milled to 200 µm using P-13-disc mill (Fritsch Co, Ltd). The milled sample were harmonised by thorough mixing of cone and quartering then analysed for chemical and mineralogical composition. The X-ray fluorescence spectrometer (XRF, ZSX Primus II Rigaku) was used to analyse the chemical composition of the ore while the mineralogy and mineral distribution of the ore was also done using RINT Rigaku X-ray diffractometer and Hitachi SU-70 SEM-EDS respectively. Further, the milled ore was screened to 180 µm, 150 µm, 100 µm, 60 µm and 32 µm using 5 JIS Z 8801 Sieves (IIDA Manufacturing Co, Ltd) mounted on the AS200 sieve shaker (Retsch Japan Co., Ltd).

Magnetic separation of Sanje ore

The Series L Model 4 (L-4 machine) (Eriez Manufacturing Co.) was used for magnetic separation of the ore samples. The L-4 machine uses wire mesh or pipe matrix fixed inside the casing as magnetic particle collectors, Shao *et al.* (1996). The effect of magnetic field intensity on silica and alumina removal was first investigated by adjusting for 3, 5, 7.5 and 10 Tesla while pulp density of the feed and its pulp flow were kept constant at 2 per cent and 7 L/min respectively. The effect of feed pulp density on iron recovery was investigated by adjusting the density of slurry fed into the machine to 2 per cent, 2.5 per cent, 5 per cent, 7.5 per cent and 10 per cent at the optimum magnetic field intensity obtained from the first stage while keeping the pulp flow of 7 L/min. The efficacy of the wire mesh and the pipe matrix was also studied using optimum magnetic flux density and suitable feed pulp density.

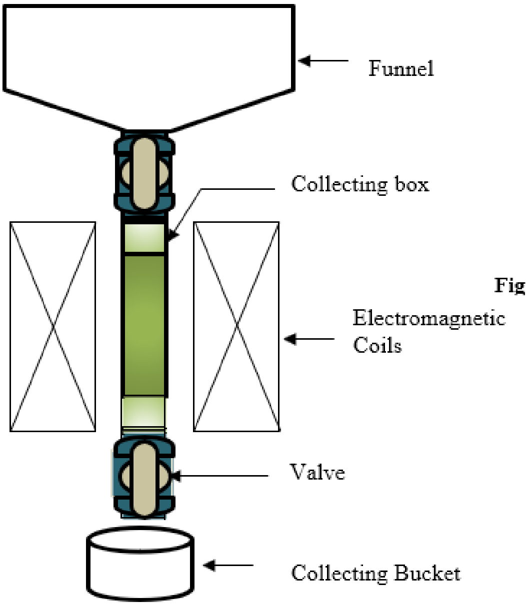


Figure 1 (a): Series L model 4 wet high-Intensity magnetic separator

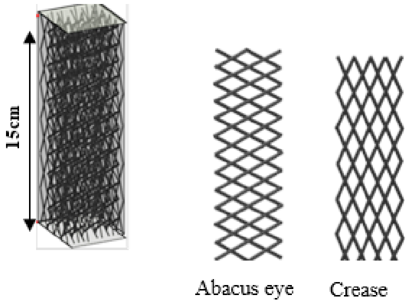


Figure 1 (b): Wire Mesh collector

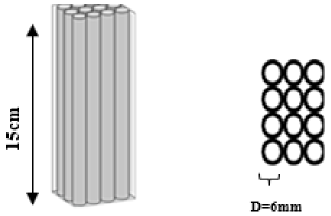


Figure 1 (c): Pipe matrix Collector

Reverse flotation of Sanje Ore

To ascertain the best pH range needed for effective impurity removal in flotation, zeta potential analysis was first determined using zeta-potential and particle size analyser ELSZ-1000 (Otsuka Electronics co., Ltd). The slurry was prepared from

Sanje iron ore samples for 10 per cent pulp density and using 0.1 per cent of Sodium Hydroxide (NaOH) (supplied by Kanto Chemical co., Inc.) to adjust pH from 6, 8, 10 to 12. Flotation experiments were then done using 500mL 237FL Flotation machine (Mekhar-Tekhnika corp.), mixing the slurry at 900 rpm and 0.5L/min of air injected. The effect of iron depressant dosage and activator dosage were studied to establish best flotation parameters. Sodium Oleate (NaOL) of 0.1 per cent solution was used as an anionic collector and 0.1 per cent Dodecyl amine acetate (DAA) as a cationic collector. Iron depressant was prepared by mixing 1% Dextrin starch ($C_{18}H_{32}O_{16}$) and 0.1 per cent sodium hydroxide solution to 1:1 molar ratio. CaO of 0.1 per cent was used as quartz activator while Methyl Isobutyl Carbinol (MIBC) was as silica-alumina Frother.

Two Stage Separation processes

The concentrate from the first stage magnetic separation was fed to the L-4 Machine using the same optimum conditions as from the first stage i.e. magnetic flux density, pulp density and the suitable collecting matrix while pulp flow was kept constant at 7L/m. The concentrate from the first stage of froth flotation was also fed for the second stage flotation using optimum conditions obtained from the first stage. On the other hand, the concentrate from the first stage magnetic separation was used as feed of the reverse flotation process. The efficacy of the three processes were compared to establish the best method of greater impurity removal and minimum metal loss.

RESULTS AND DISCUSSION

Chemical and Mineralogical Analysis

The chemical composition analysis using X-ray Florence spectrometer showed that Sanje Ore contains 48.90 per cent mass of hematite (Fe_2O_3) with 34.18 per cent mass iron grade. As shown in table 1, the ore also contains silica and alumina of 31.10 per cent mass and 7.65 per cent mass respectively.

Table 1: Chemical Composition of Sanje Iron ore

% Fe_2O_3	% Fe	% Al_2O_3	% SiO_2	% P	% MgO
48.90	34.18	7.65	31.10	0.05	1.33

The mineralogical analysis results in Figure 2 show that the major mineral components of the ore are hematite and quartz indicated by the major mineral phases while magnetite and aluminates are the minor minerals as shown by fewer mineral phases. This confirms the prominence of hematite in the Sanje Iron ore. The mineral particle distribution analysis done using Electron Microscope-Energy Dispersion Spectrometry (SEM-EDS) shows that iron mineral particles in the ore exists as free particles and aggregated with silica mineral particles. The picture in Figure 3 also indicates that the mineral arrangement of alumina-silicate gauge exists mainly as iron-bearing interlocked particles.

Figure 2: XRD patterns for Sanje Iron ore

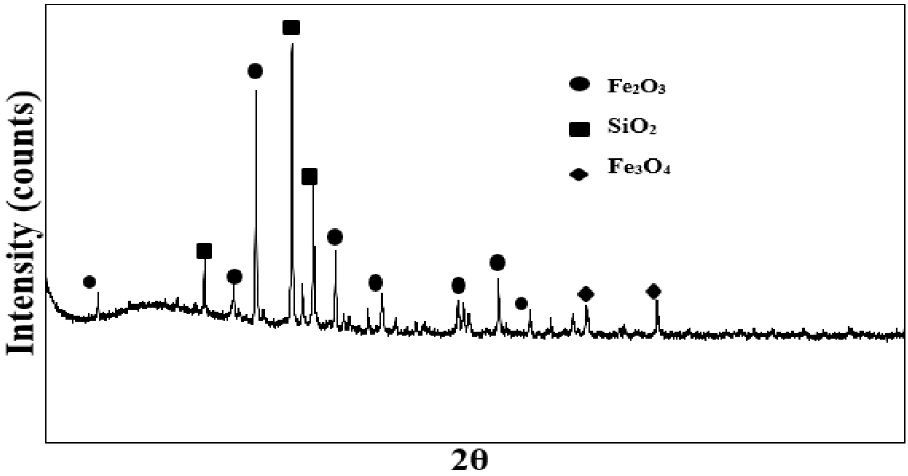


Figure 3: SEM-EDS Pictures of Sanje ore

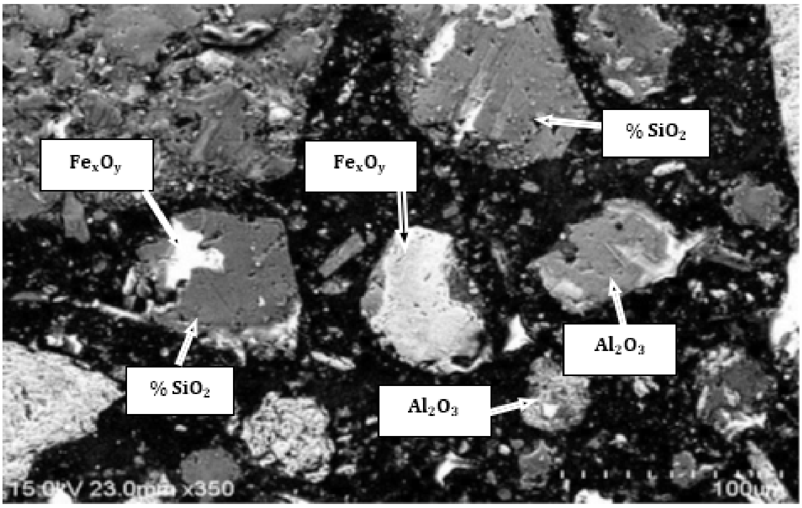
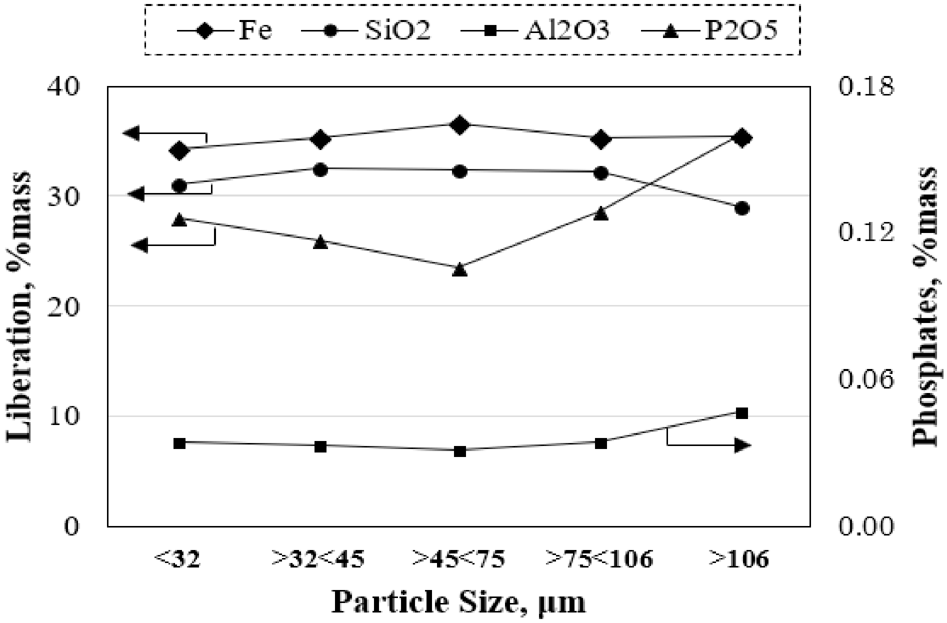


Figure 4: SEM-EDS Pictures of Sanje ore



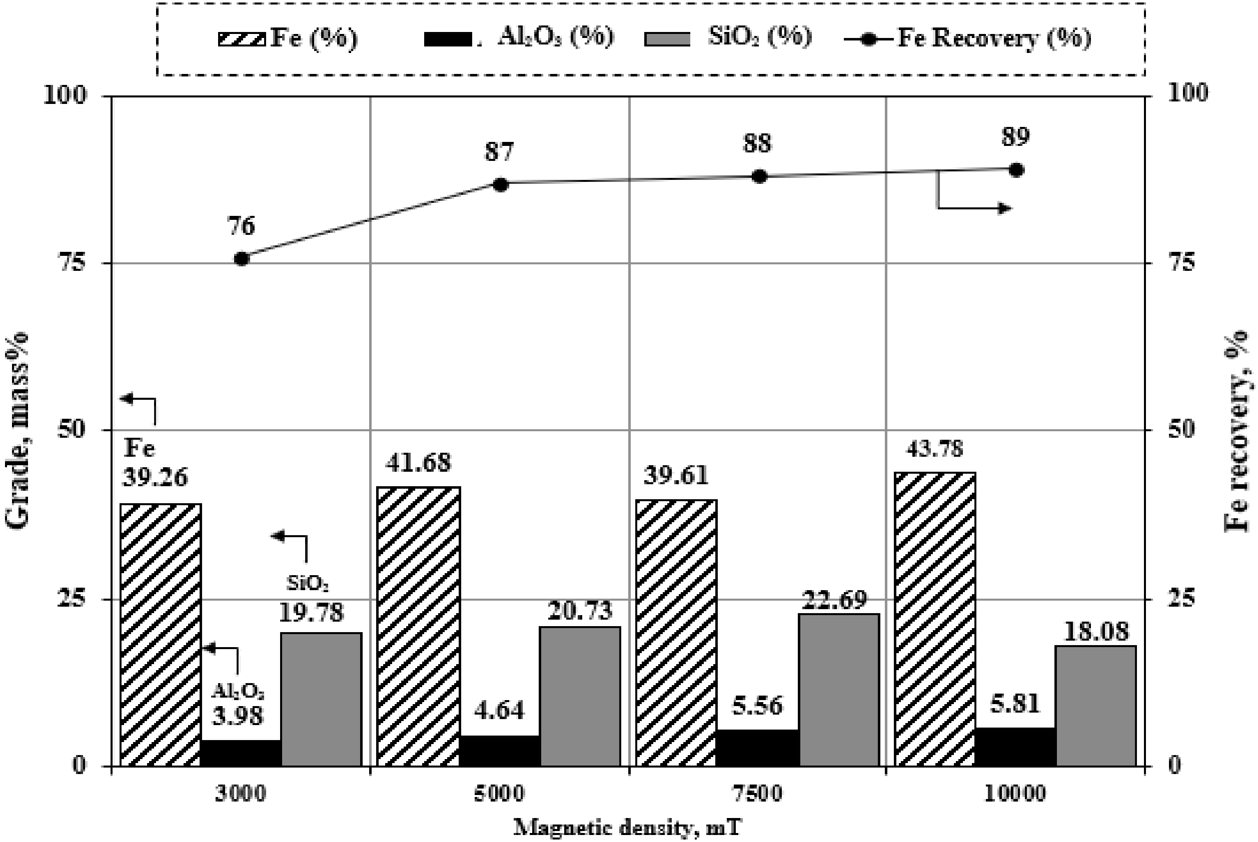
In Figure 4, the grade of Iron and liberation of Silica shows a relatively increasing trend with a decrease in particle size until optimal liberation at particle size fraction of -75 + 45µm. Alumina and Phosphorous shows decrease in liberation with a decrease in particle size which indicates that alumina-phosphorous gauge minerals are locked up more in coarse particles. Iron, silica and alumina shows better content in a particle size fraction of less than 32 µm and further milling of allowed further liberation. The sample was further milled to obtain average particle size range of 24 µm with 80 per cent of the particles.

Magnetic Separation

Effect of Magnetic density on Iron Recovery

Recovery of Iron and its grade increased with increase in magnetic intensity. Evidently, Iron recovery improved from 76 mass percentage to 89 mass percentage with an increase in magnetic field density from 3,000mT to 10,000mT. Silica content in the concentrate was reduced to 18.08 mass percentage at 10,000 mT as compared to 19.78 mass per cent at 3,000mT. Increase in the magnetic field beyond 10,000 mT resulted in decreased recovery due to the buildup of magnetic particles on matrix or pipe walls Chen *et al.,* (2015). However, this was not a case for Alumina which was reduced to 3.98 mass percentage at a lower magnetic flux density of 3,000mT and to 5.81mass per cent at a higher magnetic flux density of 10,000mT. The Increase in magnetic field density had no effect on alumina removal which indicates that magnetic field density had less effect on the degree of magnetisation for alumina than for silica and Iron.

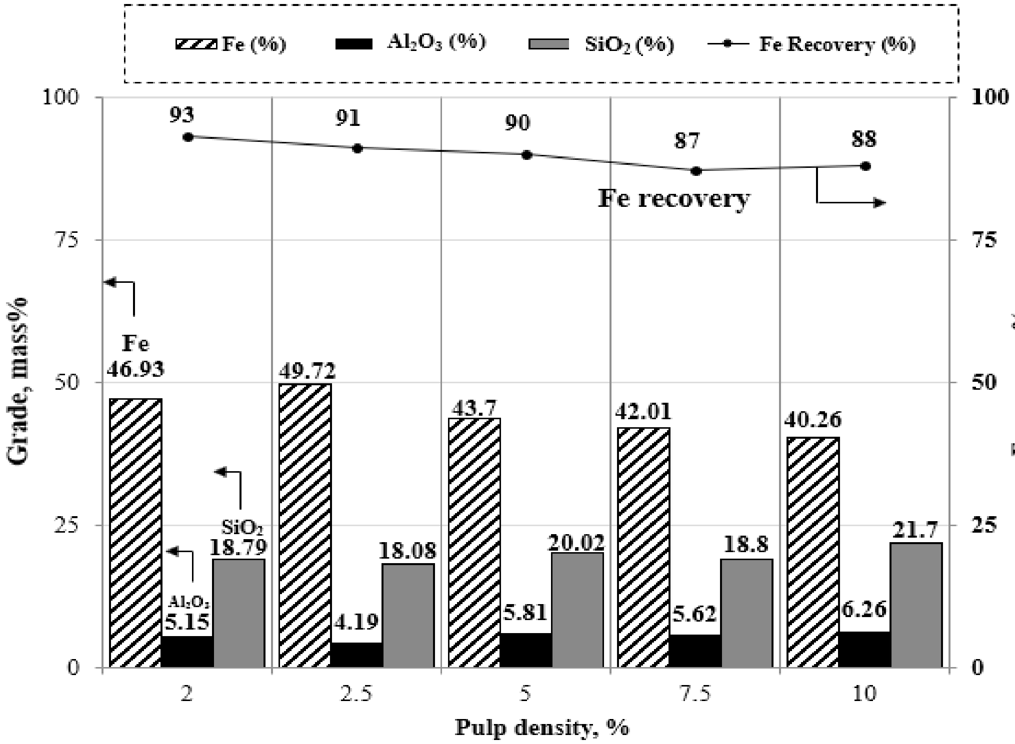
Figure 5: The effect of magnetic density of iron recovery and silica/alumina removal



Effect of feed pulp density on Iron Recovery

Figure 5 shows that recovery of iron decreased from 93 per cent to 91 per cent for pulp density of 2 per cent to 2.5 per cent at constant magnetic field density of 10,000mT. Further increase in pulp density caused a relatively decreasing trend in iron recovery and lower silica-alumina gangue removal. The optimum pulp density was obtained at 2.5 per cent at which 89 per cent of iron was recovered with the grade of 49.72 mass per cent 4.19 mass per cent alumina and 18.08 mass per cent of silica. At 2.5 per cent the solid-liquid ratio at which the magnetic forces dominated over the liquid drag force. Lower pulp density increased pulp flow reduced the probability at which the particles were induced and captured by the matrix. Alternatively, higher pulp flow caused overcrowding on the matrix and reduced separation efficiency.

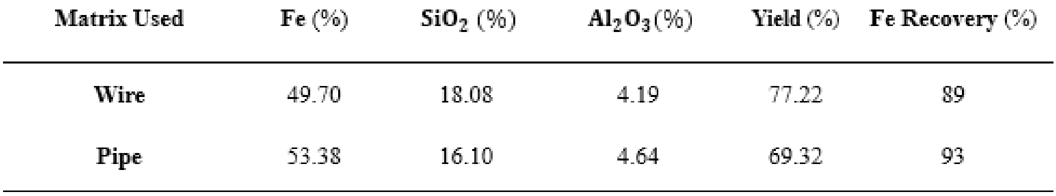
Figure 6: The efffect of pulp density of iron recovery and silica/alumina removal



The effect of matrix equipment on silica and alumina removal

The optimum magnetic field density of 10,000mT and feed pulp 2.5 per cent was used to investigate the effect of matrix equipment on silica and alumina removal. As shown in Table 2, Iron with a grade of 49.70 mass per cent and recovery of 89 per cent was obtained for the wire mesh while 93 per cent was recovered using the pipe matrix.

Table 2: The effect of matrix used on Iron recovery and silica/alumina removal



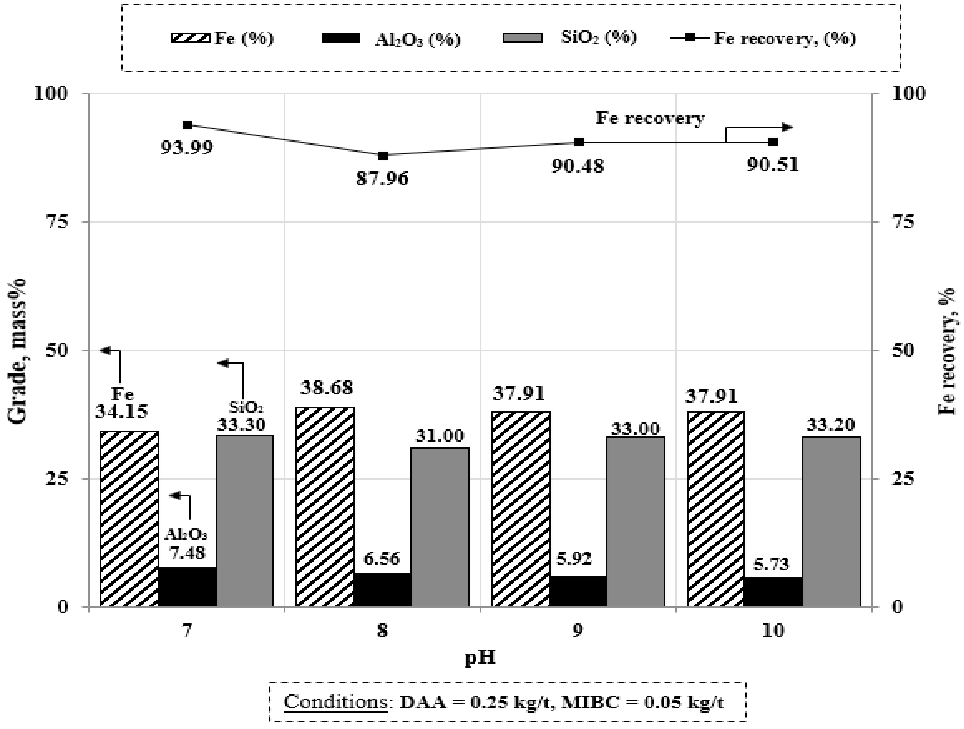
Matrix Used	Fe (%)	SiO_2 (%)	Al_2O_3 (%)	Yield (%)	Fe Recovery (%)
Wire	49.70	18.08	4.19	77.22	89
Pipe	53.38	16.10	4.64	69.32	93

The iron grade also showed the higher content of 53.38 mass per cent when the pipe was used as compared to the wire mesh. The lower yield of 77.22 per cent was obtained when the pipe was used as compared to 69.32 per cent for the wire mesh. The low yield, indicating mass loss can be explained that the wire mesh had more magnetic field gradient created on the abacus and crease planes than it was created in the cylindrical walls of the pipes matrix

Reverse Flotation

Effect of pH on flotation of silica and alumina

Figure 7: The efffect of pH on flotation of silica/alumina removal



The effect of pH on silica and alumina flotation was investigated, and the lowest silica content of 31 mass per cent in concentrate was obtained at pH 8. Iron recovery reduced from 93.87 per cent to 87.90 per cent. Further adjustment of pH from 8 to pH 10 increased both the grade and recovery with an iron grade of 37.85 mass per cent obtained at pH 9. Iron recovery showed a constant trend with an increase in pH, this indicated that there was no direct dependence on pH for the flotation of silica and alumina using DAA collector alone.

Effect of Collector dosage on flotation of Silica and Alumina Floatation

Table 3 shows the results for the effect of collector dosage of flotation on silica and alumina floatation. Iron recovery increased from 76.24 per cent when DAA was used alone to 77.01 per cent when the collector mixture of DAA and NaOL was used. The suitable molar ratio was obtained at 1:1 which corresponds to the lowest silica content of 28.30 mass per cent in the concentrate produced. The effect of mixed collector dosage on the iron recovery showed a decrease from 76.24 per cent at 1:1 ratio to 73.36 per cent at 1:10 ratio.

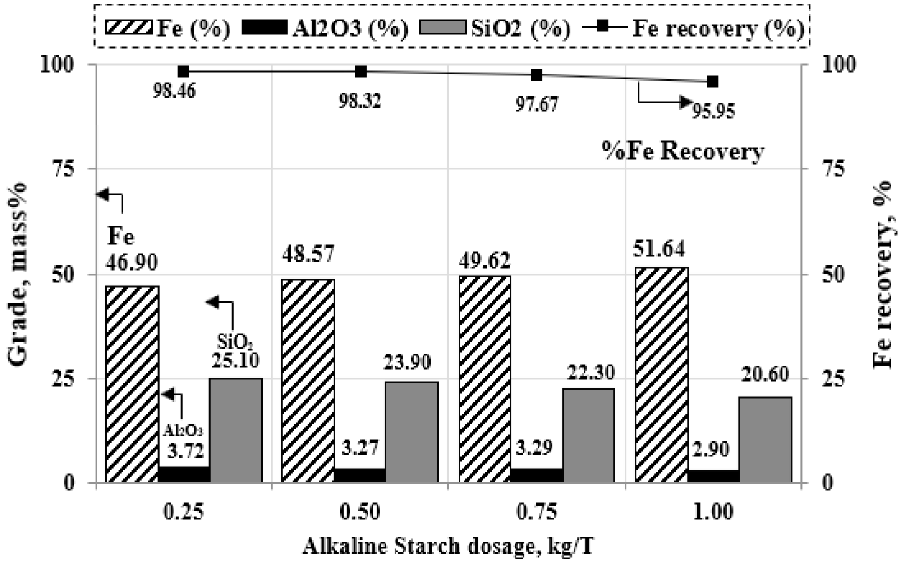
Table 3: Results for the effect of collector dosage of flotation on silica and alumina

Collector ratio DAA: NaOL	Fe Grade (%)	SiO_2 (%)	Al_2O_3 (%)	Recovery (%)
1:0	39.44	31.10	4.81	76.24
1:1	42.09	28.30	4.93	77.01
1:2	41.74	28.00	5.26	76.20
1:5	38.47	31.90	5.33	71.46
1:10	39.17	30.90	5.20	73.36

Effect of starch on flotation behaviour of hematite, silica and alumina

Figure 8 shows that iron grade increased dosage from 46.90 mass per cent to 51.64 mass per cent with increase in starch dosage from 0.25 kg/T to 1 kg/T respectively.

Figure 8: The effect of Iron depresant (alkaline starch) on Iron recovery

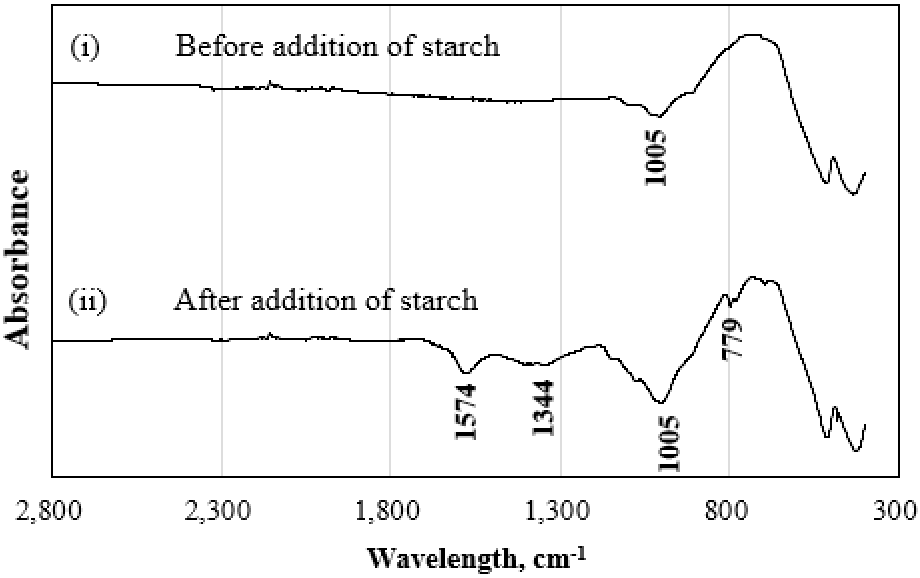


Conditions: **CaO = 0.20 kg/T, NaOL/DAA = 0.75 kg/T, MIBC = 2 mL, pH = 8**

More alumina was reduced at 1 kg/T consumption with 2.90 mass per cent of alumina remaining in the concentrate. Silica also showed an improved reduction with an increase in starch dosage with 20.60 mass per cent in the concentrate at a considerably optimum starch dosage of 1kg/T.

During flotation, CaO was added to the pulp and its purpose was to change the surface charge of silica-alumina bearing particles from negative to positive to allow adsorption of sodium oleate (NaOL) to take place. In the presence of DAA and CaO, the addition of NaOL as an anionic surfactant enhanced the attachment of silica-alumina bearing minerals to the froth bubbles. Conversely, alkaline starch was used to create the cationic polymer to mask the adsorption of the amine on iron particles and form a polymer-surfactant layer on silica-alumina agglomerated particles which in the presence of anionic surfactant (NaOL) enhanced flotation, Vieira *et al.* (2013) and Ana *et al.* (2007). Iron depression was enhanced by the Adsorption of starch and DAA on the iron oxide surface**.** Figure 9 shows the adsorption of starch on iron particles during flotation as indicated by the infra-red spectrum.

Figure 9: Infra-red adsorption spectrum of starch on Iron



After contact with starch, weak CH_2 bonds were created and characterized by the small rocking vibration at 779cm^{-1}, a bending asymmetric peak of C-H bond at 1344cm^{-1} and a small stretch at 1574cm^{-1} characterizing the CH_3 bond, Kou *et al.* (2010). The figure proves the adsorption of starch on Iron mineral particles which enhanced Iron depression. When zeta potential of the hematite is more negative, the hydrophobicity of quartz increases. Increase in hydrophobicity of quartz during hematite flotation is attributed to more silicates attracted to cationic collectors and consequently increased silica-gauge recovery in the froth.

Two stage separation Process

Effect of multi-stage separation process on iron recovery

Two-stage magnetic separation (M-M process) recovered 83.41 per cent of iron with a grade of 67.07 mass per cent containing 1.30 mass per cent of silica and 2.14 mass per cent alumina. The two stage reverse flotation (2RF process) showed a relatively lower iron grade of 55.53 per cent with the lowest mass yield of 72.95 per cent with 2.60 mass per cent of alumina and 15.80 mass per cent of silica containing in the concentrate. On the other hand, the combination process involving magnetic separation followed by reverse flotation (M-RF process) recovered 89.50 per cent of iron having a higher grade of 67.28 mass per cent. The M-RF process as shown in Figure 5 was designed and proposed as the multi-stage process for the production of high-grade iron concentrate with minimal alumina and silica content.

Effect of two-stage separation process on %Fe recovery

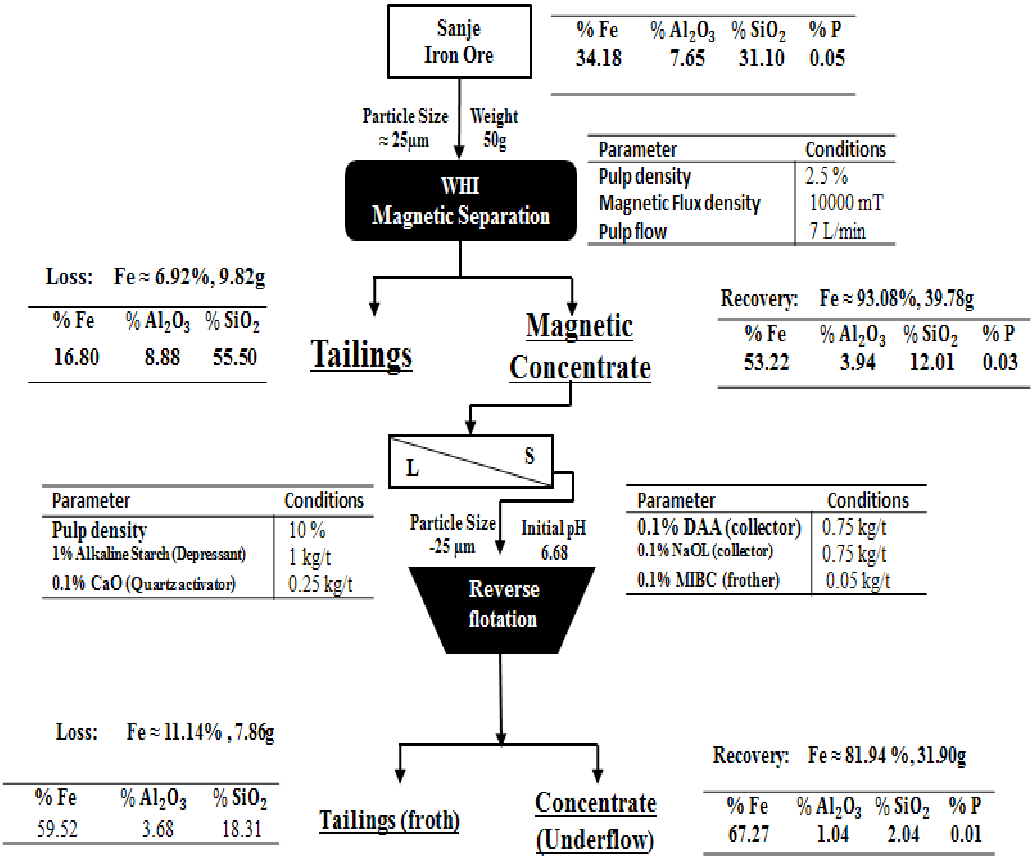
Two-stage processes were investigated to ascertain the best way to recover height grade Iron with minimum impurities. Two-stage magnetic separation recovered 92.87 per cent of Iron with a grade of 67.07 per cent, slightly less than the Magnetic-Floatation process which retained iron grade of 67.28 per cent. Less iron metal was lost during Magnetic-Flotation separation as indicated by 78.65 per cent mass yield.

Table 7: Composition of Concentrates two stage for Separation processes.

Separation Process	% Fe	% Al_2O_3	% SiO_2	% P	% MgO	% Recovery	% Yield
Two Stage Magnetic	67.07	1.30	2.14	0.02	0.35	92.87	**73.23**
Two Stage Flotation	55.53	2.60	15.80	-	0.74	87.65	**72.95**
Magnetic and Flotation	67.28	1.04	2.02	0.01	0.33	89.50	**78.65**

Two-stage floatation shows relatively lower Iron grade of 55.53 per cent with the lowest mass yield of 72.95 per cent, however almost all the phosphorous was removed with a better iron recovery of 87.65 per cent as compared to two-stage flotation. It can be inferred that Magnetic-Flotation process is suitable for removal of impurities from Sanje Iron ore because of less metal loss, higher Iron grade, more impurity removed, and a better recovery as compared to two-stage magnetic separation and flotation processes.

Figure 10: Proposed two stage Process flow for impurity removal.



CONCLUSION

To establish the best mineral processing technique for effective removal of impurities from Sanje Iron ore, this research concluded that;

1. Sanje Iron ore is a low-grade hematite ore which contains Iron-silica aggregates and their liberation requires adequate milling to fine particle fraction of less than 32µm.
2. Effective removal of impurities from Sanje ore using Wet High-Intensity magnetic separation requires lower feed pulp density of 2.5 per cent and higher magnetic field intensity of 10,000mT. However, higher Iron grade with minimum impurities is not attained by single Magnetic separation process with just up to 53.22 per cent mass Iron grade attained with 12.04 per cent mass silica and 3.94 %mass of alumina. Phosphorous is also observed to be removed with only 0.02 per cent mass remaining in the concentrate.

3. Reverse flotation using a mixture of cationic and anionic collectors' i.e. Dodceylamine Acetate (DAA) and Sodium Oleate (NaOL) shows a better silica-alumina gauge removal than when only DAA is used. However, it requires the use of small amounts of CaO as quartz activator and Alkaline starch as Iron depressant. Iron recovery increased with increase in alkaline starch dosage and decreased when more CaO dosage is added.

4. Evidently, 1 kg/T of 1 per cent Alkaline starch is required for effective Iron depressing, 0.2 kg/T of 0.1 per cent CaO required for quartz surface activation and a 1:1 ratio mixture of 0.75kg/T DAA and NaOL required for silica and alumina flotation which reduces silica by more than 27 per cent as shown in figure 19 and recovers 95.95 per cent of iron with 51.64 mass per cent grade.

5. A combined process of Magnetic separation followed by reverse flotation is effective for removal of silica and alumina from Sanje Iron ore. As proposed in figure 21, the combined process retained iron grade of 67.28 per cent with only 7.44 per cent Iron metal loss to the tailings. Much of silica is reduced to 2.04 per cent and alumina 1.04 per cent with phosphorus just a percentage fraction of 0.01per cent remaining in the concentrate. The flow sheet was therefore proposed as shown in figure 10.

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