RESEARCH PAPER

Beneficiation of an Oolitic-Iron Ore by Magnetization Roasting and Magnetic Sepration

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Abstract: Low-grade iron ores contain many impurities and are difficult to upgrade to make appropriate concentrates for the blast furnace (BF) or direct reduction (DR) technologies. In this study, the beneficiation of an Oolitic-iron ore (containing 45.46wt% Fe_2O_3) with magnetization roasting by non-coking coal (containing 62.1wt% fixed carbon) under a stream of argon gas was investigated. Then, a 2500 Gaussian magnet was used for dry magnetic separation method. The effects of roasting time, ore particle size and reaction temperature on the amount of separated part and grade of the product were examined. It was found out that the hematite inside the ore could almost be completely converted into magnetite with stoichiometric ratio of coal to ore at the roasting temperature of 625 °C for 25 min. Under the optimum condition, a high amount of magnetic part of the product (72.22 wt.%) with a grade of 92.7% was separated. The most important point in this process was the prevention of reduced ore from re-oxidation reaction by controlling roasting atmosphere, time and temperature. In addition, different analytical methods such as X-ray fluorescence (XRF), X-ray diffraction (XRD), differential thermal analysis (DTA), thermogravimetric analysis (TG) and scanning electron microscopy (SEM) were applied to investigate and expound the results.

Keywords: Low-grade iron ore, Non-coking coal, Magnetization roasting, Magnetic separation.

1. INTRODUCTION

In conventional blast furnace (BF) or direct reduction (DR) technologies for producing iron, conversion of the ore to high-grade iron oxide is vital [1]. This is essential for the appropriate operation of both blast furnaces and direct reduction process. Otherwise, producers won't be able to tackle the problem of producing a high amount of slag [2]. Besides that, purification of the ore has numerous benefits, such as reducing the cost of energy to melt additional impurities and decreasing loss of metal due to dissolving in the slag and increasing the furnace efficiency[3]. Hence, enhancing the ore concentration restricts the industrial use to those ores which are economically separable from gangues [4]. Hematite is the most abundant type of iron oxide that is used in processes of producing iron[5]. Due to demand and consumption of industries, the concentration of the majority of hematite deposits have decreased and the volume of the waste or lowgrade ores have increased significantly in recent years[6].

Researchers have investigated different ways for recovery of iron from low-grade iron-ore such as flotation process[7], anionic flotation of quartz [8], hydrometallurgical method by using different solvents[9] and magnetite separation. It is well known that from the economic and environmental points of view the most efficient method to increase the grade of iron is magnetite separation[10]. This method separates and captures magnetic particles by applying a controllable magnetic force. Hence, hematite should be reduced to magnetite or wustite which have magnetic properties by using different reductants like H₂[11], CO/CO₂[12 - 14] and CH₄ [15]. Production and use of reductant gases need more advanced and costly equipment. Furthermore, in most countries such as China, Russia, Iran and the United States which have many coal resources use of coal as a reductant for reducing hematite seems to be more economically efficient [16,17].

Magnetite is highly magnetic and can therefore be easily separated from the other non-magnetite particles such as quartz and hematite[18]. More-







over, magnetite production needs less temperature and time compared to making wustite. Therefore, magnetizing roasting followed by magnetic separation has been proven as an efficient solution of using low-grade iron ore by decreasing the cost of purification and increasing the production performance[19].

Alavi Fard et al. studied the recovery of lowgrade hematite ore from Neyshabor using coalbased direct reduction and magnetic separation. They found that the best conditions for recovery was temperature of 1050 °C, reaction time of 1 h, and 125% of stoichiometry ratio of coal to hematite [20].

Yu et al.[21] studied hematite to magnetite reduction by using iron ore fine mixed with 8 wt% coal at roasting temperature of 800°C for 8 min. They achieved 92.7% iron recovery under optimized condition. Liu et al.[22] found that Fe₂O₂ is reduced to Fe₂O₄ between 500 and 670°C by using coal as the reductant. Also, they mentioned that by increasing temperature, magnetite reduced to wustite between 740 and 870°C. Ponomar et al.[23] used 4% carbohydrates as the reductant and heated up the mixture until 650°C with the heating rate of 65 °C/min to produce magnetite. Ravisankar et al.[24] investigated reductive roasting with coal followed by low-intensity magnetic separation to upgrade specific iron ore. They found that the best results can be obtained at 800°C for 30 min using 10% coal in the charge. Using these conditions a product containing 66.6% Fe was produced at 90.4% recovery.

This paper aims to develop a new economic process for upgrading low concentration iron ores from an Iranian mine by converting hematite to magnetite instead of producing wustite. Different studies have been done in various countries with different iron ore and coal mixtures in order to reduce hematite to magnetite or wustite. Reduction of hematite has also been investigated in fluidized bed using reductive gases. However, the results of these investigations cannot be generalized to all iron ores. Because the analysis and specification of raw materials in various mines and countries are different. Therefore, each iron ore and coal mixture should be studied separately to obtain the best condition for reduction of hematite to magnetite. This study for the first time investigates the best conditions for reducing iron ore from Fars mine by non-coking coal. To achieve that goal, the factors that may influence magnetite production during the reduction of low-grade Oolitic-iron ore by non-coking coal were explored under a stream of argon gas. The main reason of using protective atmosphere is to prevent oxidation. The results of this research can be used for further investigation to utilize excessive amount of non-coking coal instead of argon atmosphere. Then, similar to industry conditions, a 2500 Gaussian magnet was used for magnetic separation of produced magnetite. The ore and coal were characterized by X-ray fluorescence (XRF) and other chemical analysis. Also, X-ray diffraction (XRD), differential thermal analysis (DTA), thermogravimetric analysis (TG) and scanning electron microscopy (SEM) were applied to investigate the results. Besides that, titration method was used to find the percentage of Fe+2 that represented the amount of Fe₃O₄ in the final product.

2. EXPERIMENTAL PROCEDURE

2.1. Materials and Characterization

The commercial Oolitic-iron powder used in this research was obtained from Fars Iron Ore Mine located in Fars province, Iran. The iron ore was characterized by using X-ray fluoresce (XRF, Philips PW1480 analyzer) and X-ray diffraction (XRD, Philips X'Pert Pro diffractometer) with CuK_a radiation, at the wavelength of $\lambda = 0.154187$ nm, scanning angle of 5 to 100°, and the step size of 0.02°. The X'Pert HighScore Plus v2.2b software of PANalytical Company was applied to identify the phases. Meanwhile, titration test with potassium dichromate and biphenyl amine was used to identify the percentage of Fe⁺² in the iron ore. Also, the non-coking coal was used as reductant. The coal was characterized according to the following standards: ASTM D3172-13 for fixed carbon content, ASTM E1915-13 for sulfur content, ASTM D3174-12 for ash content, INSO 11098-1290 for L.O.I content.

2.2. Thermogravimetric and Differential Thermal Analysis (TG-DTA)

TG - DTA analysis (TGA Q2000 Autosampler) were used to record weight change as a function of







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temperature and differential temperature as a function of temperature (to detect transitions). The crucible in this experiment was made of platinum with a height of $\approx 0.1 \, \mathrm{cm}$ and a diameter of $\approx 1 \, \mathrm{cm}$. Firstly, some ore and coal were mixed with stoichiometric ratio between hematite content of ore and fixed carbon of coal according to reaction 1:

$$3Fe_{2}O_{3} + C = 2Fe_{3}O_{4} + CO$$
 (1)

Then, the sample was heated up from room temperature to 1000°C under a stream of 0.5L/min of argon gas (99.99% purity) with the controlled heating rate of 20 °C/min to find the temperature range of the reactions that can happen. For the in-depth analysis first derivative of the TGA curve (the DTG curve) was plotted to indicate inflection points and reaction temperatures.

2.3. Magnetization Roasting and Magnetic Separation

The non-coking coal was mixed with the ore with a ratio similar to the TG-DTA experiment and well-distributed in silicon crucible for the magnetization roasting in the tube furnace under a stream of argon gas. Argon gas stream was being used continuously to prevent entering oxygen in the furnace atmosphere. Consequently, carbon would react with hematite in the absence of oxygen. After roasting, the roasted samples were cooled in the furnace to room temperature under a stream of argon gas to avoid re-oxidation.

For each one of the circumstances, the weight loss of sample was calculated based on the difference between the initial and final weight. But, the extent of reduction and weight change percentage were calculated based on the following equations:

Extent of reduction
$$(W_{ch}) = (W_{mixed sample} - W_{ore} - W_{coal})$$
 (2)

Weight change percentage=
$$(W_{ch}/W_{initial}) *100$$
 (3)

where, $W_{\rm mixed}$ sample represents the weight loss of mixture of ore and coal after reduction and $W_{\rm ore}$ and $W_{\rm coal}$ are the weight loss of ore and coal individually in a similar circumstance. Also, $W_{\rm initial}$ is the weight of mixture before roasting. The effect of different parameters such as particle size of the ore, temperature and reduction time on sample weight change and magnetite production were investigated.

In the magnetic separation test, a magnetic sloping surface was performed. The magnetic field intensity was set to 0.25 T. After the magnetic separation process, titration test with potassium dichromate ($K_2Cr_2O_7$) was used to detect the exact amount of Fe⁺² in the product according to the equation 4:

$$6Fe^{+2} + Cr_2O_7^{-2} + 14H^+ \rightarrow 6Fe^{+3} + 2Cr^{+3} + 7H_2O$$
 (4)

3. RESULTS AND DISCUSSION

3.1. Chemical Composition

Chemical composition of the iron ore as determined by XRF and the XRD trace for initial ore is indicated in Table1 and Fig.1, respectively. The main phase structures of the ore are hematite (Fe₂O₃) and quartz (SiO₂). Meanwhile, iron ore composition contains less than 0.8% FeO. As it can be seen in Table1, the iron ore contains high amounts of gangue minerals and more than 10% L.O.I. The main gangue minerals are manganese dioxide (Mn₂O₃) and barium oxide (BaO) and MgAl₂O₄(MgO+Al₂O₃). It is important to notice that, there is goethite (FeOOH) peak in XRD pattern, which means there will be weight loss due to the dehydroxylation of goethite. The chemical analysis of the coal is given in Table 2, which shows that the fixed carbon content of coal was 62.1%.

Table 1. Chemical composition of initial iron ore

Composition	Fe ₂ O ₃	Al ₂ O ₃	SiO ₂	Mn ₂ O ₃	ВаО	SO ₃	MgO	FeO	L.O.I	Others
wt%	45.46	5.01	27.69	3.85	1.89	0.96	0.39	0.71	10.54	3.5

Table 2. Analysis results of non-coking coal

Fixed carbon (%)	Volatile matter (%)	Ash content (%)	Sulfur (%)	
62.1	23.8	11.7	2.4	







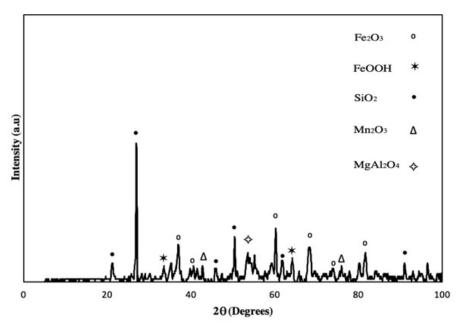


Fig.1. XRD pattern of initial iron ore.

3.2. Thermogravimetric and Differential Thermal Analysis (TG-DTA)

The weight loss curve and differential thermal analysis results for the mixture of iron ore and coal as determined by TG-DTA is shown in Fig.2. As it can be seen, the weight decreased gradually in at least two stages from 70 to 1000°C, so that exact temperature regions for various reactions can be determined by DTA and DTG results. In DTG curve, three temperature regions were detected where weight loss in the sample occurred. Besides that, two endothermic peaks were identified in the DTA curve which match the related peaks observed in DTG.

The first region in DTG curve occurred between 200 and 360°C which has been attributed to the removal of moisture from sample and dehydroxylation of goethite which is reported that typically occurs between 210 and 370°C [1, 24]. The second identified region was observed between 400 and 580°C. This has been attributed to the decomposim tion of compound materials and the release of gases from ore or coal. Due to the small amount of heat required for this reaction, it is not detected as a peak

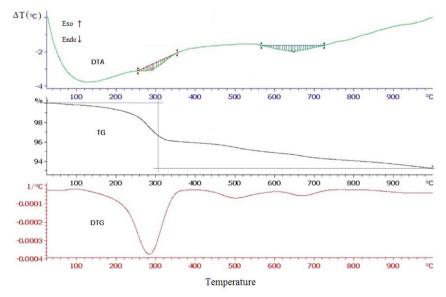


Fig.2. The results of TG-DTA analysis of iron ore mixed by carbon under the argon atmosphere.









in DTA curve. The final region occurred in the temperature range of 600 -730°C (centred on 650°C approximately). Also, there is a peak in DTA curve in that temperature period which shows the occurrence of an endothermic reaction. This is obviously due to the reduction of hematite to the magnetite. It is important to notice that, the observed weight loss was more than the theoretical calculation for reduction of hematite to magnetite due to the high amount of L.O.I in initial iron ore. It should be noted that most of the L.O.I material evaporates under 340°C during the test. However, the remaining L.O.I content may be evaporated after that temperature.

3.2. Magnetization Roasting

In the magnetization roasting process, the essential factors affecting the reaction were roasting temperature, particle size, time of reduction, atmosphere of reaction and coal to ore ratio. Firstly, the effect of temperature on the weight loss was studied at two different particle size under argon atmosphere in the tube furnace. The results are given in Fig.3. To explore the effect of ore particle size, the first size of the ore was varied between 180 to 250µm and the other one from 250 to 750µm named small and big particle size, respectively. Meanwhile, roasting time and coal particle size was 25min and less than 150µm for all tests, respectively. These values were selected according to the primary experiments.

In this study, the amount of coal mixed with ore was equal to stoichiometric ratio of reduction reaction of hematite to magnetite according to reaction 1. A flow of 500 mL min⁻¹ of argon was continuously supplied to maintain an inert atmosphere and to purge out the produced gases.

Theoretically, there can be a 5.69% weight loss due to the production of CO in equation 1. But, as mentioned above, there was only 45.46% hematite in the ore used in this study. Therefore, by considering the amount of hematite in the ore, maximum weight change in theoretical calculation is equal to 2.58%. It should be noted that due to the very low amount of coal in the powder mixture the amount of volatile material in the coal was negligible in comparison with whole weight change. It is important to notice that, due to fact that wustite cannot be produced under 750°C [1], the weight change only rephresents magnetite production.

As it can be seen in Fig.3, by increasing temperature the weight loss for two particle sizes increased gradually until reaching the maximum value at 600 and 625°C for small and big particle sizes, respectively and then started to decrease slowly. Although argon gas was fed inside the furnace continuously, there can be a small amount of oxygen or moisture in the argon atmosphere as impurity for reaction with carbon (reaction 5). Meanwhile, at higher temperatures, oxygen can be absorbed by solid carbon and the reduction of hematite to magnetite is speeded up due to the oxidation of solid carbon and the production of CO according to equation 6. Also, it is important to mention that, by increasing temperature, solid carbon reacts with CO, as Boudouard reaction (reaction 7) and produces more reductant gas for reduction of hematite (reaction 8).

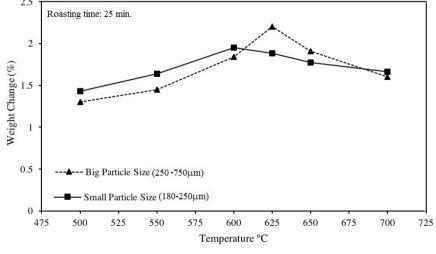


Fig.3. Effect of temperature on the weight change in two particle size of iron ore.







$$C+O_2 = CO_2 \tag{5}$$

$$C+0.5O_2 = CO$$
 (6)

$$CO_2 + C = 2CO \tag{7}$$

$$Fe_2O_3 + CO = Fe_3O_4 + CO_2 \tag{8}$$

Moreover, due to the fact that the mixture of ore and coal was heated up in the furnace from room temperature, by increasing temperature, carbon has reacted completely and after reaching the maximum value of weight change, the formed magnetite could be re-oxidized to hematite by reacting with oxygen (exists as impurity in purged gas) because the solid carbon and reductant gas in the furnace are run out [20]. It is obvious that smaller particle size ores have more surface area and reacts faster with

coal at low temperatures in comparison with a bigger particle size. Thus, the maximum weight change occurred in the lower temperature in ores with smaller particle size. Meanwhile, agglomeration was observed in smaller particle size by increasing temperature and it resulted in decreasing magnetite production rate and re-oxidation of produced magnetite.

The effect of time on the reduction of hematite to magnetite was explored with 4 different times varied between 10 and 30 min for two particle sizes, separately. In these tests, the powders were initially heated (20 °C/min) under a flow of Argon (500 mL/min) to the predetermined temperature. The roasting time was calculated after reaching the defined temperature. The effect of roasting time has been studied for different temperatures around the temperature which maximum weight loss happened during the preliminary tests for each particle size

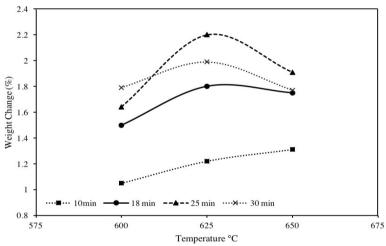


Fig.4. Effect of time on the weight change for ore with big particle size (250 -750μm).

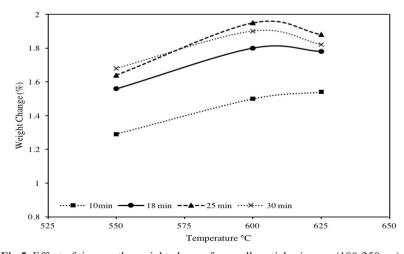


Fig.5. Effect of time on the weight change for small particle size ore (180-250μm).









separately which means, temperatures 550-600-625°C and 600-625-650°C were studied for the small and big particle sizes, respectively.

As it can be seen in Fig.4 and Fig.5, increasing time have a positive effect on the hematite to magnetite reduction under the above mentioned roasting condition. By increasing the roasting time the percentage of weight loss showed an increase in each temperature individually. Meanwhile, time has a more significant effect on reduction in bigger particle size because agglomeration could occur by increasing time in small particle size. This issue will be discussed later. It is important to notice that, by increasing temperature, the trend of changing weight altered because the amount of solid carbon reduced and subsequently the amount of reduction decreased. Also, some re-oxidation happened at higher temperatures which resulted in decreasing the weight change percentage. The highest weight change was 1.95% for small particle size at temperature of 600°C in 25 min remaining time and for big particle size was about 2.2% at temperature of 625°C and 25 min remaining time which is close to its theoretical value.

Phase identification analysis and microscopic investigations were carried out on the powders

obtained from testing in the best conditions at two different particle sizes (temperature 600 and 625°C for small and big particle size respectively) by using XRD and SEM as given in Figs 6 and 7, respectively.

In the XRD pattern of initial iron ore sample (Fig.1), it is apparent that iron exists mainly as hematite. For ore roasted at 600 and 625°C in 25min remaining time (Figs.6a and b), XRD patterns illustrate that hematite peaks decreased whilst magnetite peaks appeared in comparison to initial ore. As it can be seen, the presence of hematite peaks shows that conditions are not sufficiency reducing for complete conversion. It is important to notice that, wustite (FeO) peaks can not be observed in the XRD pattern of roasted powders. It is reported that wustite is not formed at roasting temperature less than 800°C which is in agreement with the obtained results[24]. Furthermore, FeOOH peak disappeared in the pattern of roasted powder which means that the compound has been decomposed by increasing temperature. Meanwhile, other compounds like MgAl₂O₄ and Mn₂O₃ remained stable at temperatures around 600°C. It can be concluded that there wasn't any decrease in weight due to the decomposition of those components.

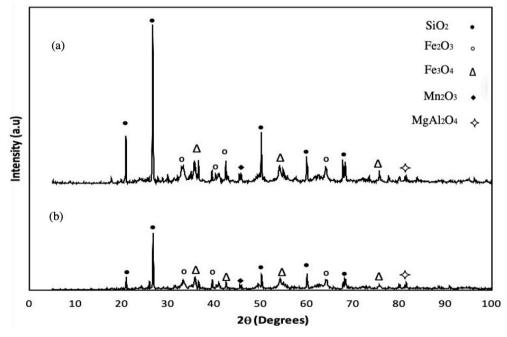


Fig.6. XRD patterns obtained from powders tested in the best condition in two different particle sizes, (a) small particle size iron ore in temperature 600°C and 25min roasting time (b) big particle size iron ore (250-750μm) in temperature 625°C and 25min roasting time.



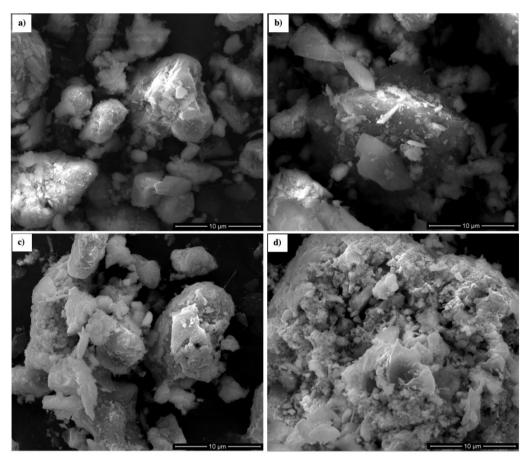


Fig.7. SEM images obtained from powders in two different particle sizes, (a) small particle size initial iron ore(180-250μm) (b) big particle size initial iron ore(250-750μm) (c) small particle size iron ore roasted at 600°C and 25min (d) big particle size iron ore roasted at 625°C and 25min.

Fig.7. a and b illustrate SEM images for the initial iron ore with small and big particles sizes, respectively. Also, it shows the results of samples, which had the highest weight change at 25min roasting time in two different temperatures, 600°C (Fig.7c) and 625°C (Fig.7d) for small and big ore particle sizes, respectively. It should be noted that because of high particle size in these samples, the powders was crushed slightly for improving the images quality. Therefore, the size of particles in these Figs are lower than their real size. As can be seen, the product that belongs to bigger particle size ore (Fig.7d) has lots of pores due to the reduction of hematite to magnetite and producing gas (reaction 8). The hematite particle starts to be reduced from the surface, and the magnetite appears on the outer layer of the particle. Thus, the produced gas due to the reduction of the inner layers comes out and makes lots of pores on the surface of the particle. Meanwhile, Fig.7c shows less porosity in comparison with Fig.7d, due to the lower amount of reduction. Also, it can be seen that agglomeration happened in comparison with initial ore during the roasting process, due to the evaporation of moisture and high surface contact of particles.

3.3. Magnetic Separation and Titration

The weight loss of hematite which was used as an indication of magnetite production is based on the oxygen removal by producing CO in the system. It seems that other methods are required for calculating the efficiency of roasting process. In this regard, dry magnetite separation method was used to separate magnetic part of product. It is supposed that the magnetic part was the product of reduction reaction in the ore. The percentage of magnetic part that was separated versus reduction temperature has been shown in Fig. 8. Also, this part was analyzed by titration test to identify its Fe⁺² amount according to equation 4 and calculating the separated amount of









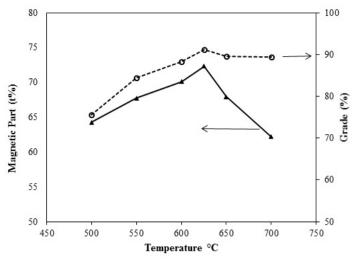


Fig.8. Effect of temperature on grade of product and the amount of magnetic part (roasting time: 25min, big particle size ore (250-750μm)).

 ${\rm Fe_3O_4}$, i.e. grade of product. Then, the grade of product was computed through the ratio of molar mass of ${\rm Fe^{+2}}$ (55.8 g/mol) and ${\rm Fe_3O_4}$ (231.5 g/mol). This value represents the amount of ${\rm Fe_3O_4}$ in the separated part of product.

As shown in Fig.8, for the big particle size of iron ore, the grade and the amount of magnetic part increased with the increase of roasting temperature until 625°C, gradually. However, after 625°C the grade of product decreased slightly and the amount of separated magnetic part dropped sharply with increment of temperature. It can be concluded that the reduction of hematite to magnetite increased with increment of temperature. Then, the magnetite could not be kept stable by increasing temperature and re-oxidation happened as mentioned above [20]. Therefore, the grade of product decreased slightly.

Another important aspect is that a single magnetite particle might be re-oxidized partly to the hematite and the rest of that remained in the form of magnetite. Thus, it could not be separated completely which led to sudden drop in the fraction of separated magnetic part. It is important to mention that, the highest grade and separated magnetic part was required at the same time. Therefore, the highest grade and separated magnetic part which was equal to 91.22 and 72.32%, respectively at temperature of 625°C can be selected as suitable conditions.

Figs. 9 and 10 exhibit the effect of roasting time in 3 different temperatures on the amount of separated magnetic part and the grade of product (the amount of magnetite in separated part) for big particle size ore, respectively. Fig.9 shows that, by increasing the time until 25 min, the amount of separated part was

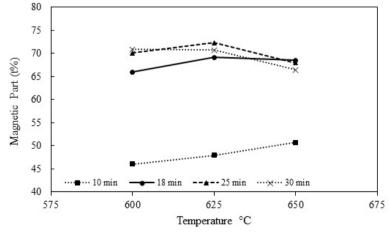


Fig.9. Effect of roasting time in 3 different temperatures (600-625-650°C) on the amount of separated magnetic part (big particle size iron ore (250-750μm), roasting time: 25min).







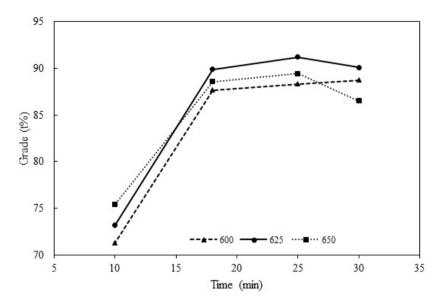


Fig.10. Effect of roasting time in 3 different temperatures (600-625-650°C) on grade of product (big particle size iron ore (250-750μm), roasting time: 25min).

enhanced in each temperature. It can also be seen that the amount of the separated part of the product remains constant with increasing temperature from 625 to 650°C in 18 min roasting time. While, in 10 min roasting time, the amount of separated part was elevated by increasing temperature due to the inadequate time for reducing hematite, i.e. carbon still remains for the reducing reaction. Meanwhile, re-oxidation reaction was observed for 25 and 30min roasting time at 625 and 650°C, respectively. It is observed that in these conditions by increasing temperature and time together the amount of magnetite is decreased.

As it can be seen in Fig.10, the grade of product increased continuously with the prolonging of roasting time for all temperatures until reaching the maximum value (91.22%) around 25 min at temperature 625°C and then decreased slightly. These results might be due to the shortage of magnetic production due to the reduction reaction if the roasting time was too short (e.g. 10 min), and re-oxidation of the produced magnetite if the roasting time was over long (such as 30 min), even at proper temperatures.

It is seen in Fig.11 that the general trend of results for small particle size is similar to the big one. But,

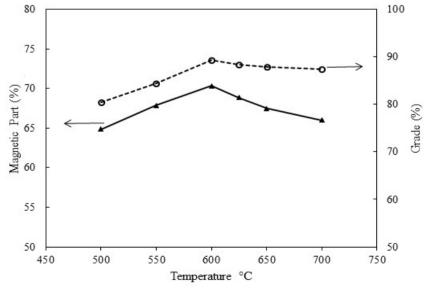


Fig.11. Effect of temperature on grade of product and the amount of magnetic part in different temperatures (roasting time: 25min, small particle size ore (180-250µm)).









due to the more ore surface area, the highest amount of separation (70.33%) appears in the lower temperature compare with the big particle size ore. But, by increasing temperature agglomeration happened and the porosity of the ore decreased. Thus, the reducing agent could not contact with the ore properly and the rate of both reduction and re-oxidation reactions declined in comparison with bigger particle size. Consequently, the amount of decrease in grade of product is lower in comparison with big particle size. For this particle size of ore, with the increase of roasting temperature until 600°C, the amount of separated magnetic part and production grade rose slightly to 70.33 and 89.32%, respectively. Then, the grade became approximately constant but the percentage of separated part decreased. As mentioned earlier, this result might be due to the fact that by increasing temperature the reducing agent was consumed faster and in the absence of reducing agent the formed magnetite part could be re-oxidized.

Figs.12 and 13 exhibit the effect of roasting time at three different temperatures on the amount of separated magnetic part and the grade of product for small particle size ore, respectively. As shown in Fig.12, by increasing temperature from 550 to 600°C the amount of separated magnetic part was increased for all remaining times. However, the rate of that increase is less at higher roasting time (such as 30 min). That might be due to the long roasting time and consumption of carbon content of the mixture. The highest amount of separated part equal to 70.33% was obtained at temperature of 600°C and 25min roasting time. By increasing temperature to higher than 600°C for 25 and 30 min roasting time, the magnetic part of product was decreased sharply due to the re-oxidation problem. In lower remaining times the amount of separated part enhanced slightly by increasing temperature due to the insufficient reaction time between carbon and hematite for complete reaction.

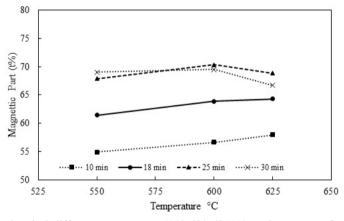


Fig.12. Effect of roasting time in 3 different temperatures (550-600-625°C) on the amount of separated magnetic part (small particle size iron ore (180-250μm), roasting time: 25min).

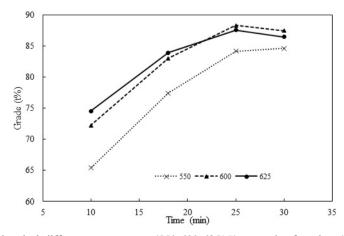


Fig.13. Effect of roasting time in 3 different temperatures (550-600-625°C) on grade of product (small particle size iron ore (180-250μm), roasting time: 25min).







As is shown in Fig.13, the general trend of grade of product for temperatures 550 and 600°C is simu ilar. But the percentage of grade for temperature 600°C in all roasting times is higher in comparison with temperature 550°C. The highest grade percenth age was equal to 88.32% for 25min roasting time at temperature 600°C. At higher temperatures like 625°C, agglomeration occurred in lower roasting time. Therefore, the grade of product would have increased slightly as long as there was some carbon in the mixture and then re-oxidation reaction happened. Oxygen can hardly diffuse into the ore because of the agglomeration, thus the grade starts to decline slowly after 25 minutes.

4. CONCLUSIONS

In this study, the suitable condition for beneficiation of an Oolitic-iron ore (containing 45.46wt% Fe_2O_3) from Fars Mine in Iran with magnetization roasting by non-coking coal (62.7% fixed carbon) and dry magnetic separation for ore particle sizes of $180 - 250 \mu m$ (small particle size) and $250-750 \mu m$ (big particle size) has been proposed and the following results were obtained:

- 1. Results of thermal analysis experiments showed that weight loss of the samples occurred in three different temperature regions. Two endothermic peaks were also identified in DTA curve. The weight losses belonged to removal of moisture from sample and dehydroxylation of goethite, decomposition of compound materials and the release of gases from ore or coal and the reduction of hematite to magnetite.
- 2. By increasing temperature the weight loss for two particle sizes increased gradually until reaching the maximum value at 600°C and 625°C for small and big particle sizes, respectively and then started to decrease slowly.
- 3. Reduction experiments in furnace clearly showed that by increasing the roasting time the percentage of weight loss increased in each temperature individually. Meanwhile, time had a more significant effect on reduction in bigger particle size because agglomeration could occurred by increasing time in small particle size which resulted in decreasing magnetite production.

- 4. The highest weight change was 1.95% for small particle size at temperature of 600°C in 25 min remaining time in furnace and for big particle size was about 2.2% at temperature of 625°C and 25 min remaining time which is close to its theoretical value. After these optimum temperatures, the reduction percentage was dropped due to the re-oxidation of magnetite.
- 5. The presence of hematite peaks in XRD patterns of the ores roasted at optimum conditions illustrated that the conditions were not sufficient for complete conversion. The wustite (FeO) peaks was not observed in the XRD pattern of roasted powders. Furthermore, FeOOH peak disappeared in the pattern of roasted powder. Meanwhile, other compounds like MgAl₂O₄ and Mn₂O₃ remained stable at temperatures around 600°C and there wasn't any decrease in weight due to the decomposition of those components.
- 6. According to the measurement of the amount of Fe⁺² by titration of roasted powder the highest amount of percentage of magnetic part that was separated and the grade of product (amount of Fe₃O₄ in the separated part) equal to 72.32 and 91.22%, respectively were achieved at magnetization roasting temperature of 625°C and remaining time of 25 min in furnace with stoichiometric ratio of non-coking coal to iron ore.

REFERENCES

- Rashid, R. Z. A., Salleh, H. M., Ani, M. H., Yunus, N. A., Akiyama, T., and Purwanto, H., "Reduction of low grade iron ore pellet using palm kernel shell, Renew". Energ., 2014, 63, 617-623.
- 2. Man, Y., Feng, J., Li F., Ge, Q., Chen, Y. and Zhou, J., "Influence of temperature and time on reduction behavior in iron ore—coal composite pellets", Powder Technol., 2014, 256, 361-366.
- 3. Feinman, J., "Excerpt from: The making, shaping and treating of steel: direct reduction and smelting processes", Iron Steel Eng., 1999, 76, 75-77.
- 4. Anameric, B., and Kawatra, S. K., "Laboratory study related to the production and properties of pig iron nuggets Min". Metall. Explor., 2006, 23, 52-56
- 5. Peacey, J. G., and Davenport, W. G., "The iron blast furnace: theory and practice", Elsevier, 2016.
- 6. Srivastava, U., and Kawatra, S. K., "Strategies for





- processing low-grade iron ore minerals, Miner". Proc. Ext. Met. Rev., 2009, 30, 361-371.
- 7. Rao, K. H., and Narasimhan, K., "Selective flocculation applied to Barsuan iron ore tailings", Int. J. Miner. Process., 1985,14, 67-75.
- 8. Yuhua, W., and Jianwei, R., "The flotation of quartz from iron minerals with a combined quaternary ammonium salt", Int. J. Miner. Process., 2005, 77, 116-122.
- 9. Mishra, R., Rout, P., Sarangi K., and Nathsarma, K., "Solvent extraction of Fe (III) from the chloride leach liquor of low grade iron ore tailings using Aliquat 336", Hydrometallurgy, 2011, 108, 93-99.
- Li, C., Sun, H., Bai, J., and Li, L., "Innovative methodology for comprehensive utilization of iron ore tailings: Part 1. The recovery of iron from iron ore tailings using magnetic separation after magnetizing roasting", J. Hazard. Mater., 2010, 174, 71-77.
- 11. Pineau, A., Kanari, N., and Gaballah, I., "Kinetics of reduction of iron oxides by H2: part I: low temperature reduction of hematite", Thermochim. acta., 2006, 447, 89-100.
- 12. Et-Tabirou, M., Dupre, B., and Gleitzer, C., "Hematite single crystal reduction into magnetite with CO-CO₂, Metall". Trans. B., 1988, 19, 311-317.
- Ponomar, V., P., Brik, A., B., Cherevko, Yu., I., Ovsienko, V.,V., "Influence of Particle Size on Magnetizing Roasting of the Hematite Quartzite in an Atmosphere of Carbon Monoxide", Mineral. J. 2017, 39, 19-28.
- 14. Yu, J., Han, Y., Li, Y., Gao, P., "Recovery and separation of iron from iron ore using innovative fluidized magnetization, roasting and magnetic separation", 2018, J. Min. Metall. Sect. B-Metall. 54B, 21 27.
- Monazam, E. R., Breault, R. W., Siriwardane, R., Richards, G., and Carpenter, S., "Kinetics of the reduction of hematite (Fe₂O₃) by methane (CH₄) during chemical looping combustion: a global mechanism", Chem. Eng. J., 2013, 232, 478-487.
- Chun, T., Zhu, D., and Pan, J., "Simultaneously roasting and magnetic separation to treat low grade siderite and hematite ores", Miner. Proc. Ext. Met. Rev., 2015, 36, 223-226.
- Sahu, S., N., Sharma, K., Barma, S., D., Pradhan, P., Nayak B. K., Biswal, S., K., "Utilization of lowgrade BHQ iron ore by reduction roasting followed by magnetic separation for the production of magnetite-based pellet feed", Metall. Res. Technol., 2019, 116, 611.
- Lan, Y. P., Liu, Q. C., Meng, F., Niu, D. L., and Zhao, H., "Optimization of magnetic separation process for iron recovery from steel slag", J. Iron

- Steel Res. Int., 2017, 24, 165-170.
- Yu, J., Han, Y., Li, Y., Gao, P., and Sun, Y., "Separation and recovery of iron from a low-grade carbonate-bearing iron ore using magnetizing roasting followed by magnetic separation", Separ. Sci. Technol, 2017, 52, 1768-1774.
- Alavi Fard, N., Schalchian, H., Rafsanjani-Abassi, A., Vahdati Khaki, J., Babakhani, A., "Recovery iron from low-grade hematite ore using coal-based direct reduction followed by magnetic separation", Iran. J. Mater. Sci. Eng., 2016, 13, 60-71.
- 21. Yu, J., Han, Y., Li, Y., and Gao, P., "Beneficiation of an iron ore fines by magnetization roasting and magnetic separation", Int. J. Miner. Process., 2017, 168, 102-108.
- Liu, G.S., Strezov, V., Lucas, J. A., and Wibberley, L. J., "Thermal investigations of direct iron ore reduction with coal", Thermochim. Acta., 2004, 410, 133-140.
- Ponomar, V., Dudchenko, N., and Brik, A., "Reduction roasting of hematite to magnetite using carbohydrates", Inter. J. Miner. Process., 2017, 164, 21-25.
- Ravisankar, V., Venugopal, R., Bhat, H., "Investigation on beneficiation of goethite-rich iron ores using reduction roasting followed by magnetic separation", Trans. Inst. Min. Metall., Sect. C, 2019, 128, 175-182.
- Faris, N., Tardio, J., Ram, R., Bhargava, S., and Pownceby, M. I., "Investigation into coal-based magnetizing roasting of an iron-rich rare earth ore and the associated mineralogical transformations", Miner. Eng., 2017, 114, 37-49.



