Analysis effect of temperature on the results of titanomagnetite (Fe_2TiO_4-Fe_3O_4) formation in iron sand reduction using coal and sodium sulphate as an additive

Devara Clarissa[1], *Bambang* Priyono[1], *Ahmad* Maksum[1,2], *Memed* Sumantono[2], and *Johny* Wahyuadi Soedarsono[1,*]

[1]Center of Minerals Processing and Corrosion Research, Department of Metallurgical and Materials Engineering, Universitas Indonesia, Depok 16424, Indonesia
[2]Department of Mechanical Engineering, Politeknik Negeri Jakarta, Depok 16425, Indonesia

Abstract. Indonesia is a developing country that has rich deposit of natural resources particularly in mineral ores. Many of iron sand found in coastal Indonesia that contains some valuable minerals such as hematite (Fe_2O_3), ilmenite ($FeTiO_3$) sand rutile (TiO_2). Research study related to iron sand has developed that aims to determine the effect of temperature against the form of titanomagnetite in reduction process using coal as reductor and sodium sulphate (Na_2SO_4) as an additive. The results of XRD characterization and semi- quantitative with temperature variable 700^{O}C, 800^{O}C and 900^{o}C obtained that 900^{o}C is an optimum temperature with amount of titanomagnenite (Fe_2TiO_4-Fe_3O_4) as much as 48.5.

1. Introduction

Iron sand is one of the many minerals found on the coast of Indonesia. It contains some valuable minerals of oxide compounds such as hematite (Fe_2O_3), magnetite (Fe_3O_4), ilmenite ($FeTiO_3$) and rutile (TiO_2) [1]. Titanium has an excellent physical properties because its high melting point and resistance to corrosion. Applications of titanium are also very wide, covering aerospace and biomedical[2]. Seeing the high potential of precious minerals found in iron sand, this becomes the background of the authors examine it.

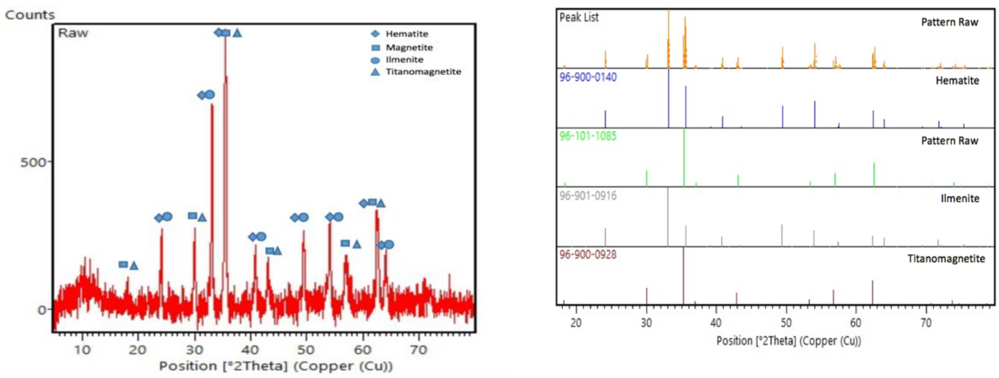
Study about iron sand has been done by pyrometallurgy method using various reductant[3]–[7]. The iron sand reduction process is generally carried out at high temperatures referring to the Boudard diagram where the CO gas is stable at a temperature of 1000^{o}C. Further research of extraction iron sand with lower temperature are needed, considering the energy and cost effectiveness.

In this experiment, the iron sand reduction process are expected to form magnetite and titanomagnetite compounds using the variables of temperature 700^{o}C, 800^{o}C and 900^{O}C within 30 minutes.

2. Materials and method

2.1. Materials

Samples of iron sand used in this experiment were tested by X-Ray Diffracrion test to characterize the compounds contained in there. Figure 1 is an XRD result of iron sand samples. Meanwhile, Table 1 is a semi-quantitative data of iron sand samples showing the composition of the compound on XRD results



(a) (b)

Fig. 1. (a) XRD result and (b) XRD patterns of iron sand initial samples

* Corresponding author: jwsono@metal.ui.ac.id

Tabel 1. Semi-quantitative analysis of intial samples

Compound Name	Chemical Formula	Semi-Quant (%)
Hematite	Fe_2O_3	30
Magnetite	Fe_3O_4	24.2
Ilmenite	$FeTiO_3$	29.3
Titanomagnetite	$xFe_2TiO_4(1-x)Fe_3O_4$	16.4

Proximate and ultimate tests were performed to determine the composition contained in coal. Proximate and ultimate test can be seen in Table 2 and Tabel 3.

Tabel 2. Proximate analysis of coal

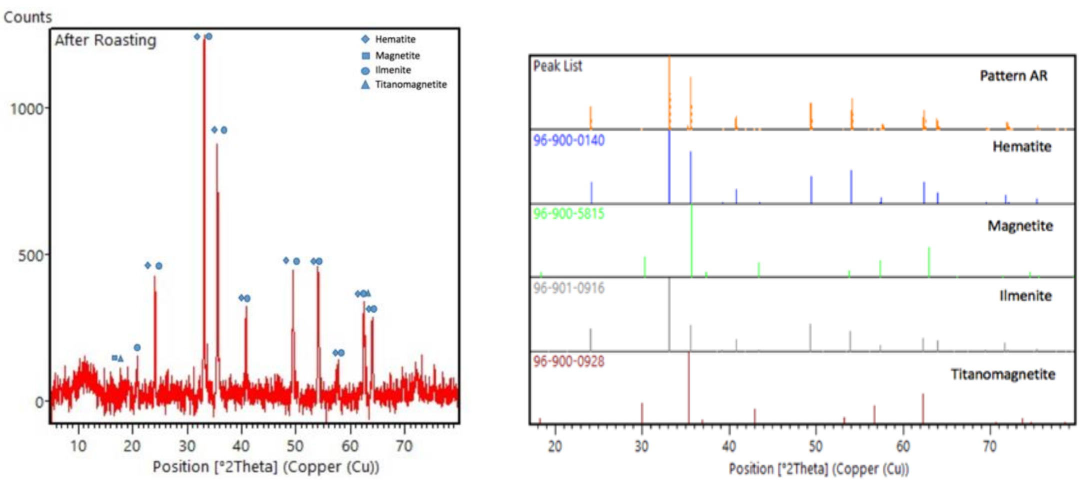
Parameter	Result
Moisture	7,26%
Ash	15,87%
Volatile metter	16,44%
Fixed Carbon	60,43%

Tabel 3. Ultimate analysis of coal

Parameter	Result
Carbon (C)	64,02%
Hydrogen (H)	3,11%
Nitrogen (N)	0,60%
Oxygen (O)	16,32%

2.2. Experiment

Iron sand samples are milled by using a ball mill for 10 minutes. After that, the sieving process is done to make the sample become powder shaped 200 mesh. Then, the sample was mixed with Na_2CO_3 with a ratio of 1: 0.4 for roasting process. Sample were roasted for 2 hours at 800°C temperature, then quenched with aquadest and dried into oven for 20 hours. After it dries, 10 grams of the sample was mixed with 5% coal and 15% Na_2SO_4 with variation of temperature 700 C, 800C and 900C for 30 minutes. Quenching with water and drying process are done same as after roasting sample. After roasting and reduction process, XRD testing was done to determine the compound and semi-quantitative in the sample and analyzed using Xpert High Score Plus Software.



(a) (b)

Fig. 2. (a) XRD result and (b) XRD pattern of after roasting samples

Table 4. Semi-quantitative analysis of after roasting samples

Compound Name	Chemical Formula	Semi-Quant(%)
Hematite	Fe_2O_3	48
Magnetite	Fe_3O_4	0
Ilmenite	$FeTiO_3$	50.8
Titanomagnetite	$xFe_2TiO_4(1-x)Fe_3O_4$	1.2

* Corresponding author: jwsono@metal.ui.ac.id

3. Result and discussion

3.1. Roasting process

The oxidation process in iron oxide aims to make the hematite compound become the dominant compound[8], [9]. This following reactions are the direct reduction equation on iron:

$$3Fe_2O_3 + CO \rightarrow 2Fe_3O_4 + CO_2 \quad (1)$$

$$3Fe_3O_4 + CO \rightarrow 3FeO + 2CO_2 \quad (2)$$

$$FeO + CO \rightarrow Fe + CO_2 \quad (3)$$

Hematite and ilmenite compounds are dominant compounds in iron sand samples that have been done roasting process. In some peaks, magnetite and titanomagnetite compounds appear to be transformed into hematite and ilmenite. The change of ulvospinel to ilmenite is an oxidation process referring to the equation:

$$6Fe_2TiO_4 + O_2 \rightarrow 6FeTiO_3 + 2Fe_3O_4 \quad (4)$$

While in the form of iron oxide, the compound formed is magnetite that oxidized from hematite, referring to equation (1).

The loss of the alumunium oxide (Al_2O_3) compound is due to binding to sodium carbonate (Na_2CO_3) forming the $NaAlO_2$ compound by the following reaction :

$$Na_2CO_3 \rightarrow Na_2O + CO_2 \quad (5)$$

$$Na_2O + Al_2O_3 \rightarrow 2NaAlO_2 \quad (6)$$

A highly soluble $NaAlO_2$ compound in water causes it to dissolve during the quenching process using aquadest.

3.2. Reduction process

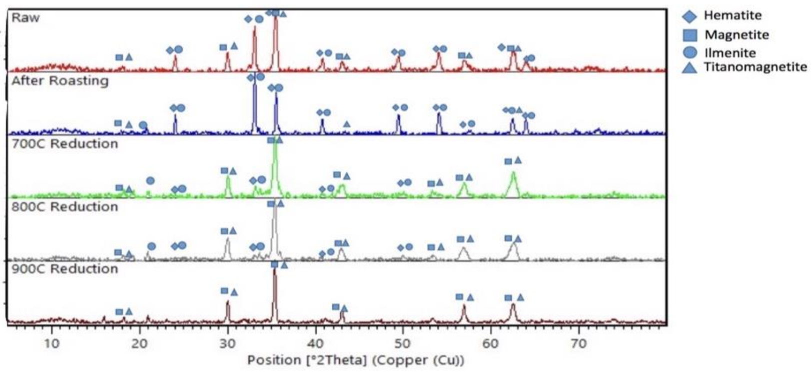
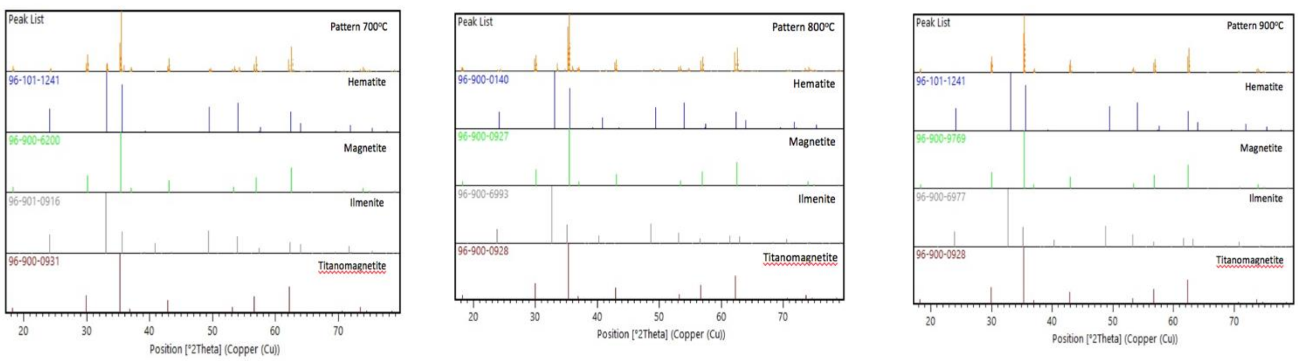


Fig. 3. XRD results of after reduction samples at 700°C,800°C, and 900°C



(a) (b) (c)

Fig. 4. XRD pattern of after reduction samples. (a) 700°C, (b) 800°C, (c) 900°C

As can be seen from Figure 3, the initial sample diffraction peak removal can be observed at the positions of 2θ, 21°, 24°, 33.5°, 41.2° and 50°, respectively, which are hematite and ilmenite compounds along with increasing temperatures. This shows that there has been a decomposition of all hematite and ilmenite compounds that become magnetite and titanomagnetite compounds at

* Corresponding author: jwsono@metal.ui.ac.id

temperatures of 900°C.

The hematite reduction process becomes magnetite occurs by adding FeO reffered to the equation 1. While the process of reduction of ilmenite to ulvospinel can be seen from following equation:

$$FeTiO_3 + FeO \rightarrow Fe_2TiO_4 \quad (7)$$

Magnetite and ulvospinel are transformed into a solid solution that called titanomagnetite.

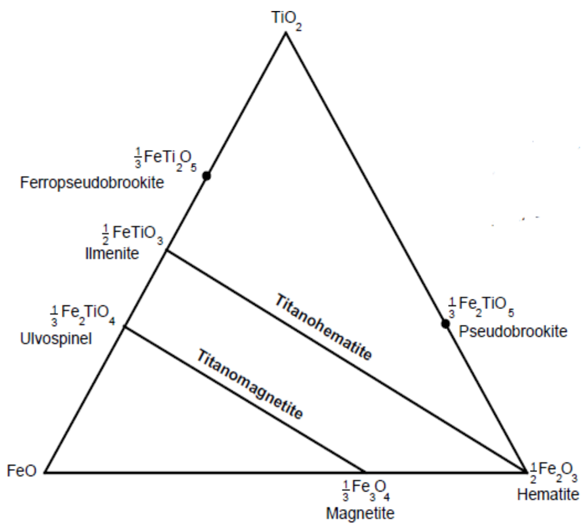


Fig. 6. Ternary diagram TiO_2-FeO-Fe_2O_3 [10]

Table 5. Semi-quantitative analysis of after reduction sample with 700°C, 800°C and 900°C

Compound Name	Formula	Semi-quantitative (%)		
		700°C	800°C	900°C
Hematite	Fe_2O_3	13.1	18.6	0
Magnetite	Fe_3O_4	51.8	34.7	51.5
Ilmenite	$FeTiO_3$	3.9	10.5	0
Titano-magnetite	$xFe_2TiO_4(1\text{-}x)Fe_3O_4$	31.3	36.2	48.5

Semi-quantitative results at temperatures of 900°C showed that magnetite and titanomagnetite compounds were the dominant compounds of 51.5% and 48.5%. The semi-quantitative results of magnetite and titanomagnetite compounds in 700 were 51.8% and 31.3% and at temperatures of 800 by 47.2% and 30.1%. This indicates that the reduction process occurs maximally at a temperature of 900°C.

On the other hand, Na_2SO_4 acts as a catalyst in order to optimize the reduction process. This was stated in the study [11] which proved that with the addition of Na_2SO_4 of 2-6%, showed an increase in concentrate on Fe and Ti in the TTM sample.

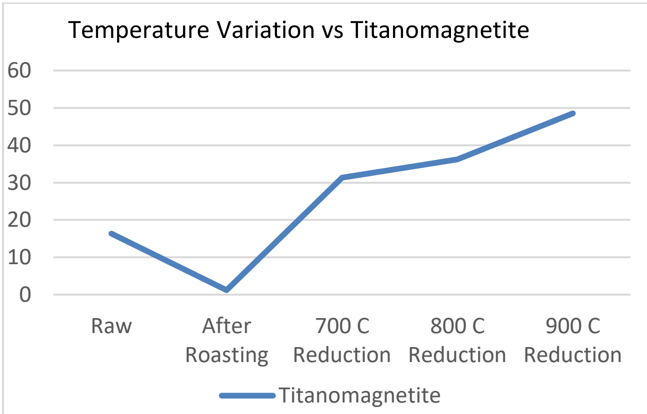


Fig. 7. Comparison graph of temperatures and titano-magnetite content

Temperature has an important influence on the formation of titanomagnetit. The higher the temperature, the titanomagnetite formed becomes maximum.

Based on this experiment that has been done related to the effect of temperature on iron sand reduction process by using coal reducer and Na_2SO_4 additive, temperature of 900°C is the optimum temperature in the

4. Conclusion

Roasting process with Na_2CO_3 additive aims to decompose elements of Al and other impurities. The result of XRD testing of reduced iron sand at temperature 700°C, 800°C and 900°C showed that hematite (Fe_2O_3) reduced to magnetite (Fe_3O_4) while ilmenite ($FeTiO_3$) decomposed into titanomagnetite ($xFe_2TiO_4(1\text{-}x)Fe_3O_4$).

* Corresponding author: jwsono@metal.ui.ac.id

process of reduction with the highest titanomagnetite, as much as 48.5%.

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* Corresponding author: jwsono@metal.ui.ac.id