

Analysis effect of temperature on the results of titanomagnetite ($\text{Fe}_2\text{TiO}_4\text{-Fe}_3\text{O}_4$) formation in iron sand reduction using coal and sodium sulphate as an additive

Devara Clarissa¹, Bambang Priyono¹, Ahmad Maksum^{1,2}, Memed Sumantono², and Johnny Wahyuadi Soedarsono^{1,*}

¹Center of Minerals Processing and Corrosion Research, Department of Metallurgical and Materials Engineering, Universitas Indonesia, Depok 16424, Indonesia

²Department 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°C , 800°C and 900°C obtained that 900°C is an optimum temperature with amount of titanomagnetite ($\text{Fe}_2\text{TiO}_4\text{-Fe}_3\text{O}_4$) 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°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°C , 800°C and 900°C within 30 minutes.

2. Materials and method

2.1. Materials

Samples of iron sand used in this experiment were tested by X-Ray Diffraction 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

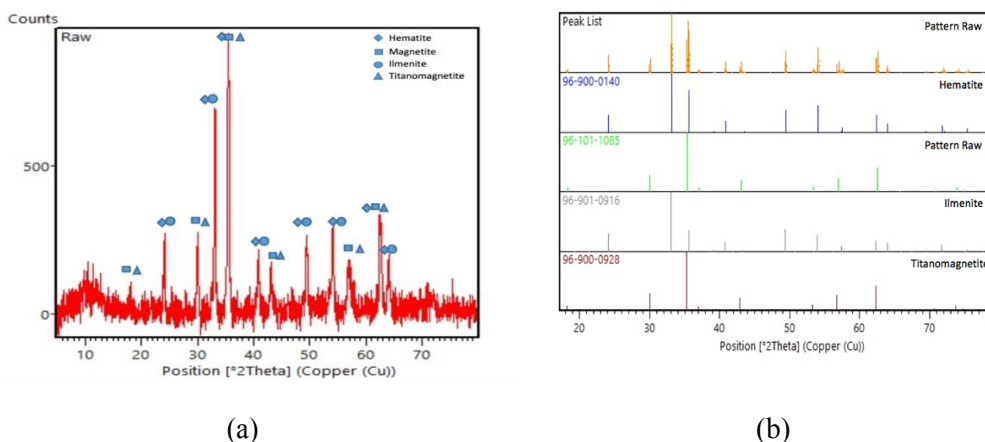


Fig. 1. (a) XRD result and (b) XRD patterns of iron sand initial samples

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

Tabel 1. Semi-quantitative analysis of intial samples

Compound Name	Chemical Formula	Semi-Quant (%)
Hematite	Fe ₂ O ₃	30
Magnetite	Fe ₃ O ₄	24.2
Ilmenite	FeTiO ₃	29.3
Titanomagnetite	$x\text{Fe}_2\text{TiO}_4(1-x)\text{Fe}_3\text{O}_4$	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 Na₂CO₃ 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% Na₂SO₄ 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.

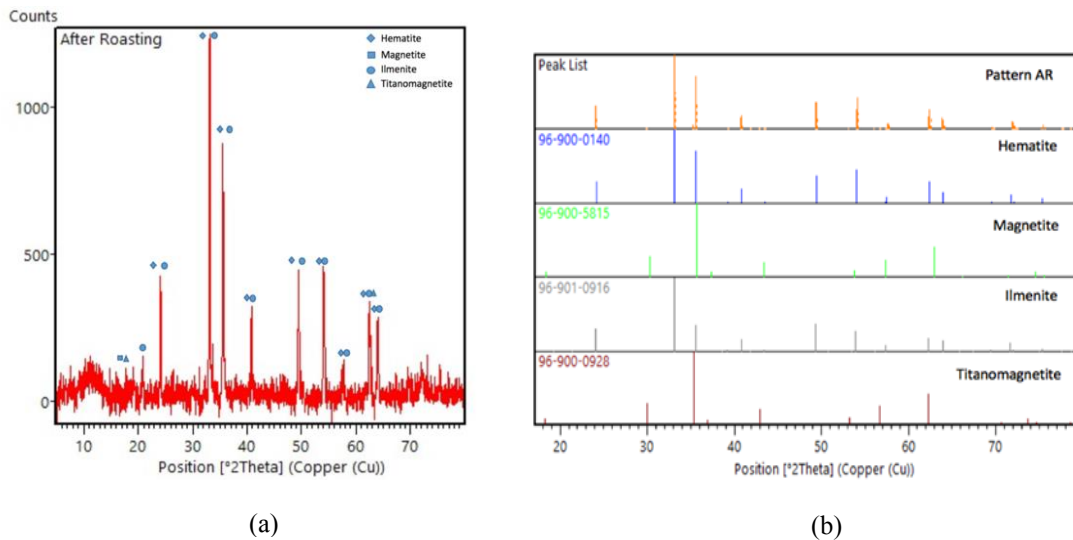


Fig. 2. (a) XRD result and (b) XRD pattern of after roasting samples

Table 4. Semi-quantitative analysis of after roasting samples

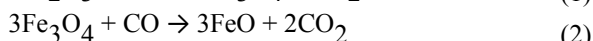
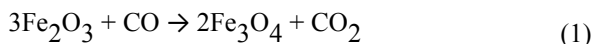
Compound Name	Chemical Formula	Semi-Quant(%)
Hematite	Fe ₂ O ₃	48
Magnetite	Fe ₃ O ₄	0
Ilmenite	FeTiO ₃	50.8
Titanomagnetite	$x\text{Fe}_2\text{TiO}_4(1-x)\text{Fe}_3\text{O}_4$	1.2

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

3. Result and discussion

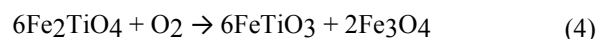
3.1. Roasting process

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:



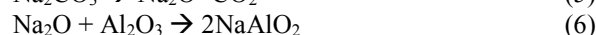
Hematite and ilmenite compounds are dominant compounds in iron sand samples that have been done roasting process. In some peaks, magnetite and titanomagnetite compounds appear to be transformed into hematite and ilmenite. The change of ulvospinel to

ilmenite is an oxidation process referring to the equation:



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

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



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

3.2. Reduction process

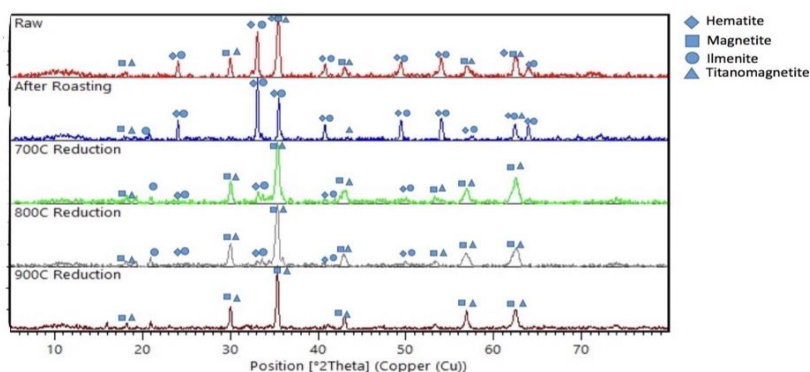


Fig. 3. XRD results of after reduction samples at 700°C, 800°C, and 900°C

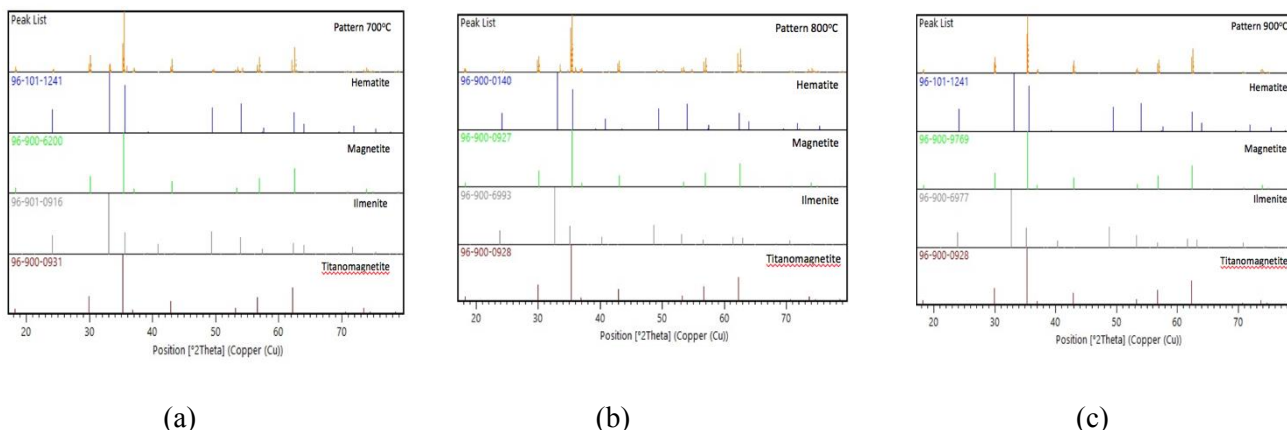


Fig. 4. XRD pattern of after reduction samples. (a) 700°C, (b) 800°C, (c) 900°C

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 referred to the equation 1. While the process of reduction of ilmenite to ulvospinel can be seen from following equation:



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

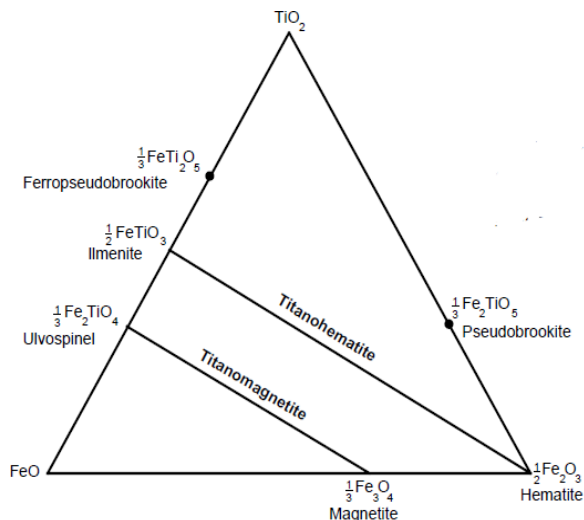


Fig. 6. Ternary diagram TiO₂-FeO-Fe₂O₃ [10]

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 ₂ O ₃		13.1	18.6	0
Magnetite	Fe ₃ O ₄		51.8	34.7	51.5
Ilmenite	FeTiO ₃		3.9	10.5	0
Titano-magnetite	$x\text{Fe}_2\text{TiO}_4(I-x)\text{Fe}_3\text{O}_4$		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, Na₂SO₄ 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 Na₂SO₄ of 2-6%, showed an increase in concentrate on Fe and Ti in the TTM sample.

4. Conclusion

Roasting process with Na₂CO₃ 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 (Fe₂O₃) reduced to magnetite (Fe₃O₄) while ilmenite (FeTiO₃) decomposed into titanomagnetite ($x\text{Fe}_2\text{TiO}_4(I-x)\text{Fe}_3\text{O}_4$).

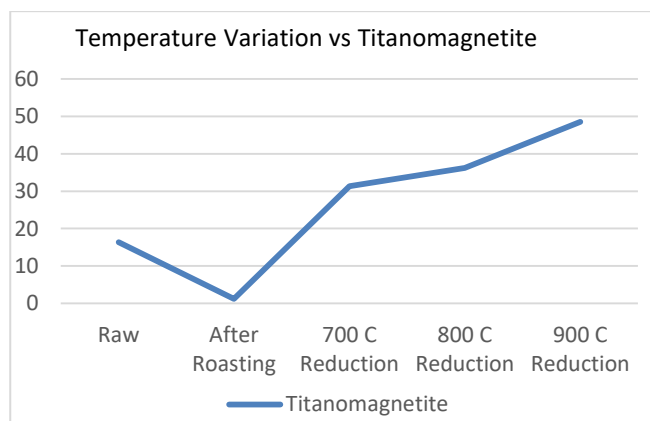


Fig. 7. Comparison graph of temperatures and titanomagnetite content

Temperature has an important influence on the formation of titanomagnetite. 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 Na₂SO₄ additive, temperature of 900°C is the optimum temperature in the

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

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

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References

1. P. A. Nugraha, S. P. Sari, W. N. Hidayati, R. Dewi, and D. Y. Kusuma, "The Origin and Composition of Iron Sand Deposit in the Southern Coast of Yogyakarta," in *AIP Conference Proceedings*, 2016, vol. 20028, pp. 1–5.
2. Zulfalina and A. Manaf, "Identifikasi Senyawa Mineral Dan Ekstraksi Titanium Dioksida Dari Pasir Mineral," *Indonesia Journal of materials Science*, vol. 5, no. 2, pp. 46–50, 2004.
3. Y. ran Liu, J. liang Zhang, Z. jian Liu, and X. dong Xing, "Phase transformation behavior of titanium during carbothermic reduction of titanomagnetite ironsand," *Int. J. Miner. Metall. Mater.*, vol. 23, no. 7, pp. 760–768, 2016.
4. J. Tang, M. S. Chu, C. Feng, Z. G. Liu, Y. T. Tang, and X. X. Xue, "A New Process of Oxidation Roasting-Gas-Based Direct Reduction/Electric Furnace Smelting Separation for High-Chromium Vanadium-Titanium Magnetite," in *7th International Congress on the Science and Technology of Ironmaking*, 2015, no. 11, pp. 1123–1134.
5. S. Samanta, S. Mukherjee, and R. Dey, "Upgrading Metals Via Direct Reduction from Poly-metallic Titaniferous Magnetite Ore," *Jom*, vol. 67, no. 2, pp. 467–476, 2015.
6. B. K. Sarkar, S. Samanta, R. Dey, and G. C. Das, "A study on reduction kinetics of titaniferous magnetite ore using lean grade coal," *Int. J. Miner. Process.*, vol. 152, pp. 36–45, 2016.
7. T. Hu, X. W. Lü, C. G. Bai, and G. B. Qiu, "Isothermal reduction of titanomagnetite concentrates containing coal," *Int. J. Miner. Metall. Mater.*, vol. 21, no. 2, pp. 131–137, 2014.
8. J. W. Soedarsono, A. Rustandi, Y. Pratesa, R. D. Sulamet-ariobimo, B. H. Prabowo, and J. S. Exsaudy, "The Effects of Reduction Parameter to Composite Pelet of Iron Ore and Coal Using Single Conveyor Belt Hearth Furnace," *Adv. Mater. Res.*, vol. 842, pp. 115–119, 2016.
9. R. Hidayanti, S. Permana, A. Maksum, and J. Wahyuadi, "Study on the effects of temperature in the carbothermic reduction laterite ore using palm kernel shell as reducing agent," *IOP Conf. Ser. Earth Environ. Sci.*, vol. 105, 2017.
10. M. W. McElhinny and P. L. McFadden, *Paleomagnetism*. International Geophysics Series.
11. C. Geng, T. C. Sun, H. F. Yang, Y. W. Ma, E. X. Gao, and C. Y. Xu, "Effect of Na₂SO₄ on the Embedding Direct Reduction of Beach Titanomagnetite and the Separation of Titanium and Iron by Magnetic Separation," *Isij Int.*, vol. 55, no. 12, pp. 2543–2549, 2015.

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