REDUCTION OF IRON ORE BY CHARCOAL UNDER MICROWAVE IRRADIATION

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Abstract— Iron sand is a source of iron ore containing hematite, magnetite and other impurities which is raw materials for ferrous materials production. Recently, there are so many extractive metallurgy process that was developed. One of these processes was using microwaves irradiation as power sources in iron ore reduction process . The concept of iron ore reduction by microwave heating is proposed of this paper. A microwave would be operated in various of power supply and irradiation time to get the optimum reduction process indicated by large amount of Fe content. Then it was characterized using X-Ray Diffraction (XRD) to identify chemical compound and phase transformation that occurs after reduction process, X-Ray Fluorescence (XRF) to determine amount of element and increasing level of Fe content, Scanning Electrone Microscophy (SEM) to observe morphology of the sample. this study concluded that the highest Fe content is 81.67% yield by 60 minutes microwaves irradiation in power supply as high as 3000 watt.

Index Terms—Iron ore, Microwaves Irradiation, Reduction, Fe Content

I. INTRODUCTION

Iron is believed to be the fourth most abundant in the earth's crust up to 4.5%. it is the most used of all the metals, comprising 95% of all the metal tonnage produced worldwide. Moreover, in India, the National Steel Policy (revised 2008), envisages domestic steel production of that country to be 180 million tones per annum by 2019-2020. Industrially iron is produced from iron ores, principally hematite (Fe_2O_3), magnetite (Fe_3O_4), and wustite (FeO) that have been reduced in the solid state in a variety of reactors such as retorts, shaft furnaces, rotary kilns and fluidized beds to produce Direct Reduced Iron (DRI). The reducing agent used is either a reducing gas or coal based such as charcoal [5]. Fe content in iron ore is one of many factors that determine quality of product. Fe content in many ores are different each others. Generally, iron deposites are limited to 25 – 70% Fe content, or roughly 5 to 15 times the average iron content of the earth's crust. So increasing level of Fe content in iron ore to the higher level is one of many techniques to get high quality production in iron making industry. Most of heavy metals oxides and carbon, as charcoal or coke, respond to microwave heating. Therefore, the microwave assisted carbothermic reduction of metal oxides is possible. Various researchers have demonstrated that iron oxides hematite (Fe_2O_3) and magnetite (Fe_3O_4) mixed with carbon charcoal or coke, could be reduced to metallic iron [2]. Recently, microwave heating has become a new method of supplying energy, instead of heating with fire. Microwave heating has been studied extensively to apply several chemical reductions and heating processes. This is because microwave heating is more advantages than conventional methods. Firstly, microwave heating is a new energy supply method that rapidly heats the objects to high temperatures without high speed blasting and a thermal driving force. Secondly, microwave heating is a form of internal heating. A material can be heated to a high temperature uniformly in the field. So, the material can be uniformly-heated in a microscopical sense [1]. Lastly, As the function of carbon was limited to the reducing agent, almost half amount of CO_2 gas emissions can be reduced in comparison to the existing blast furnace, assuming the microwave is excited from greenable electric power by solar, hydro and nuclear plants [3]. Production cost in microwave heating is also extremely cheaper than cupola furnace process. In this paper, the concept of iron ore reduction by microwave heating is proposed. A microwave would be operated in various of power supply and irradiation time to get the optimum reduction process indicated by large amount of Fe content.

II. EXPERIMENTAL PROCEDURE

A. Materials and Preparation

Commercial iron ore that used as starting raw material for iron making by reduction of iron ore method was coming from the south coast of the java island, precisely in lumajang which has chemical content in table 1.

Table 1. Chemical Content of Iron ore measured by XRF

Chemical content (Wt%)	Al	Si	P	K	Ca	Ti
	2.2	7.03	0.17	0.38	2.38	6.35
	V	Cr	Mn	Bi	Fe	
	0.6	0.01	0.58	10	69.07	

Reducing agent used in this research was charcoal which has fixed carbon content approximately 89.81% and the small amount of sulfur. Charcoal was mashed into microscopics scale up to 100 mesh in size using Ball Milling (Retsch PM-400 MA type).

B. Reduction Process

Before reduction process, iron ore must be washed several times using aquades to decrease the imprurities content. Then it was crushed up to microscopic scale using Ball Milling (Retsch PM-400 MA type) and sieved in 100 mesh in size using Vibrating Sieving (Fritsch Analysette 3 PROvibratory Sieve-shaker). The iron ore powder was separated with other impurities using permanent magnet repeated three times. The High purity of iron ore was mixed with charcoal and calcium oxides (CaO) using composition ratio 1:4:8. The homogeneous mixture weighing 60 grams was put in crucible and treated by microwave heating with various of power supply 2000, 3000 watt and irradiation time 40, 50, 60 min. Heating rate doesn't increase constantly. So it must be observed every 10 minutes by infrared Thermometer (SANFIX).

C. Characterization

The structural characterization of the sample include chemical compound and phase transformation was carried out with an XRD (PANalytical X-Ray Diffraction) with Cu-Kα radiation ($\lambda = 1.54178$ A^o) in wide range 2θ approximately 10^o – 90^o. The morphologies of the samples were obtained using Field-Emmision SEM (INSPECT S50) with a field emission gun operating at 15 – 20kV. The spectroscopy to determine amount of element and Fe content level were carried out with XRF (SPECTRO iQ II).

III. RESULT AND DISCUSSION

D. Micrwave Heating Characteristics

Heating process in microwave heating was caused by atomic vibration in most of atom in materials when microwave was conducted. The increasing temperature behavior in the reduction of iron ore under microwave irradiation was constructed in Fig.1

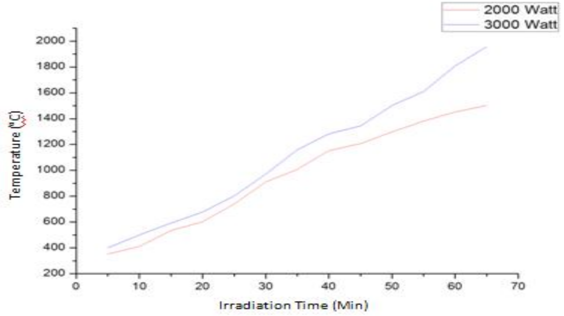

Fig .1 Effect of Irradiation time and power supply to heating process

Microwave energy is a nonionizing electromagnetic radiation with frequencies in the range of 300 MHz to 300 GHz. Microwave frequencies include three bands : the ultra high frequency (UHF: 300 MHz to 3 GHz), the superhigh frequency (SHF: 3 GHz to 30 GHz) and the extremely high frequency (EHF: 30 GHz to 300 GHz). Currently, 2450 MHz is the most commonly utilized frequency for the home microwave oven [2]

E. Themodynamics Study

Thermodynamics give information about more reaction that was possible to occur based on two parameters such as temperature and pressure. Iron oxides include hematite (Fe_2O_3), Magnetite (Fe_3O_4) dan Wustite (FeO) which all of them got aquilibrium state with Fe, C, CO dan CO_2. It was determined by Boudroud equation refer to (1)

$$C + CO_2 \rightarrow 2CO \quad \Delta G_T = 40800 - 41{,}7\ T \text{ kal/mol} \quad \ldots(1)$$

So that, reduction was possible to occur in widely temperature range 600 – 1100 ^{o}C and atmospheric condition.

Generally, reduction of Iron ore by CO_2 from charcoal has many steps. At 570 oC or highest Fe_2O_3, Fe_3O_4, FeO in the iron ore would be reduced chronologically refer to (2 – 4)

$$3\ Fe_2O_3 + CO \rightarrow 2Fe_3O_4 + CO_2 \quad \Delta H = -12636 \text{ cal} \quad \ldots(2)$$

$$Fe_3O_4 + CO \rightarrow 3FeO + CO_2 \quad \Delta H = 8664 \text{ cal} \quad \ldots(3)$$

$$FeO + CO \rightarrow Fe + CO_2 \quad \Delta H = -\ 4236 \text{ cal} \quad \ldots(4)$$

F. Effect of Microwave irradiation

Iron ore has so many contents include Fe_2O_3, SiO_2 and Fe_3O_4. It was proofed by XRD analysis in Fig. 2 that conducted to the sample before microwave treatment.

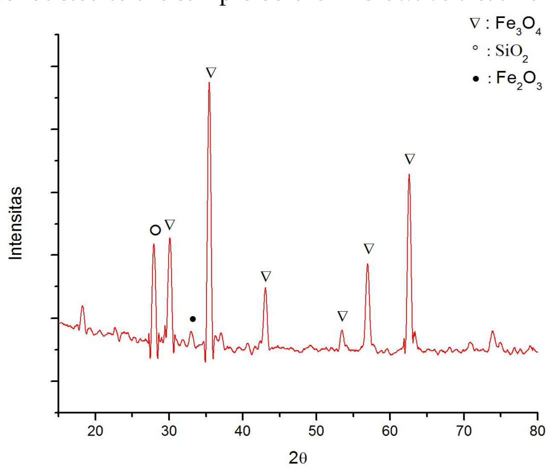

Fig.2 XRD Data of the Iron ore before Microwave Irradiation

Microwave heating process was conducted in various of power supply and irradiation time. Table 2 show the XRF data of the sample after microwave heating 2000 watt in various of irradiation time. The XRF data is main point of this research.

Table 2. XRF data of the sample (2000 Watt)

Irradiation time (min)	Amount of element (Wt%)					
	Mn	P	S	Si	Fe	C
40	0.55	0.75	0.01	12.96	72.62	4.17
50	0.31	0.65	0.02	14.71	77.29	3.53
60	0.35	1.50	0.01	9.13	76.58	3.99

Based on table 2, the longer irradiation time, the greater Fe content in the sample. The highest Fe content was reached by microwaves heating for 50 minutes in 2000 Watt power supply, approximately 77.29%. The number of Fe content after reduction was effected by phase transformation that occurred observed in Fig.3.

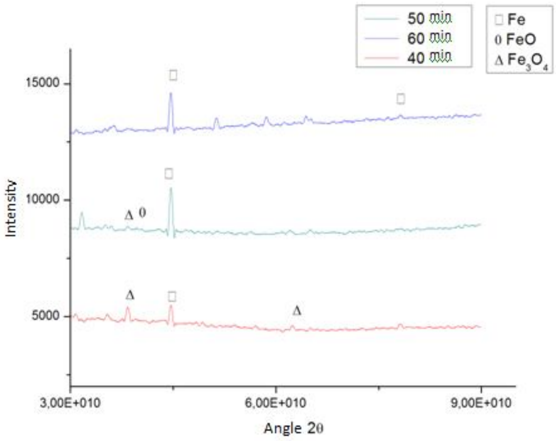

Fig.3 XRD Data of the Iron ore after Microwave Irradiation 2000 watt with Various of Irradiation time

The XRD data in Fig.3 indicated that iron ore tansform to Fe (2θ = 44.66) , Wustite (FeO) and magnetite (Fe_3O_4) in various amount influence with irradiation time. Fe content has the highest intensity in all of the irradiation time, but the traedline of amount of Fe content was not linearly with the irradiation time. The XRD data determine that intensity of Fe content increased from 40 min to 50 min irradiation time and decreased in 60 min irradiation time. This XRD data has correlation with XRF data. It is caused by at the high temperature 1700 ^{o}C, not all of Fe is reduced by carbon, but some of them react with magnesium and silica in castable, it is observed by EDS. Fig 4. Show morphology of the sample treated by 2000 watt microwave heating in various of irradiation time. Iron ore after reduction for 60 minutes melted partially (Fig.4b). whereas in 50 minutes microwave heating is not melt, just paticle bonding (Fig.4a). From the EDS observation, Fe distribution is not prevalent. In Fig.4a, EDS was carried out in two spot, it is regoion I which has Fe Content 74.4% and region II which has Fe content approximately 73.39%. Whereas in Fig.4b has different Fe content from approximately 79% and 71%.

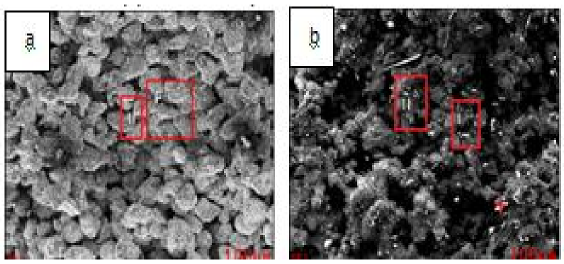

Fig 4. Morphology of the sample after microwave irradiation 2000 Watt for (a) 50 minutes, (b) 60 minutes

Table 3 indicate the XRF data of the sample after microwave heating 3000 watt in various of irradiation time.

Table 3. XRF data of the sample (3000 Watt)

Irradiation time (min)	Amount of element (Wt%)					
	Mn	P	S	Si	Fe	C
40	0.44	0.71	0.01	14.7	77.12	4.58
50	0.31	0.62	0.01	12.94	80.68	3.98
60	0.9	0.65	0.06	6.18	81.67	3.59

Based on XRF data in table 3, the Fe content increase linearly with various of irradiation time. The highest level of Fe content in 3000 watt power supply is 81.67% reached by 60 minutes irradiation time. The phase tansformation that occur after microwave heating is observed by XRD analysis that show in Fig 5.

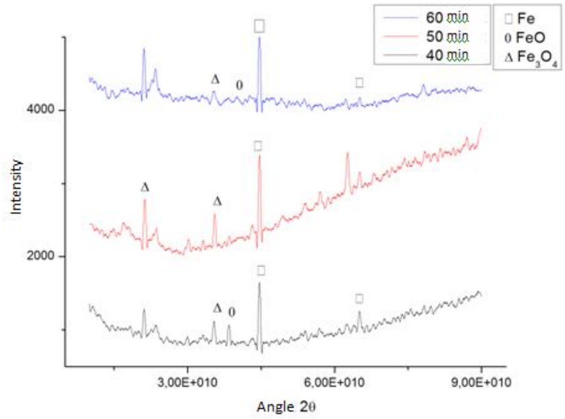

Fig.5 XRD data of the Iron ore after Microwave irradiation 3000 watt with various of irradiation time

The XRD data in Fig 5. Indicated that iron ore transform to magnetite (Fe_3O_4), wurstite (FeO) and Fe (2θ = 44.66).

Fig 6. Show morphology of the sample treated by 3000 watt microwave heating in various of time. Iron ore after reduction for 50 minutes melted in most region (Fig.6a). and in 60 minutes microwave heating is melt perfectly (Fig.6b). Fe distribution is observed by EDS. In Fig.6a, EDS was carried out in two spot, it is regoion I which has Fe Content 83.1% and region II which has Fe content approximately 75.84 %. Whereas in Fig.6b has different Fe content approximately 75.84% and 84.75%.

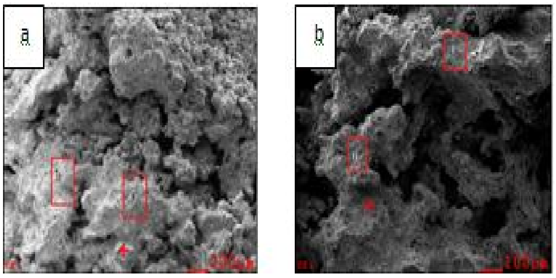

Fig.6 Morphology of the sample after microwave irradiation 3000 Watt for (a) 50 minutes, (b) 60 minutes

According to all data of the research and its analysis, the effect of irradiation time and power supply to reduction process by charcoal under microwave heating is described in Fig.7

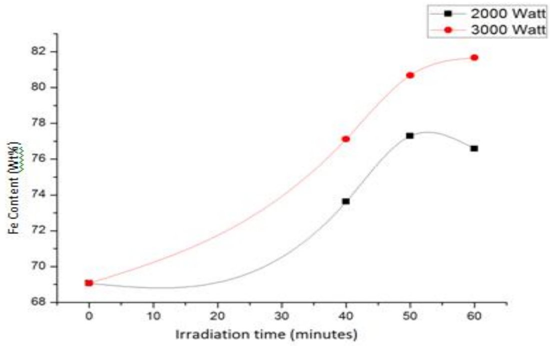

Fig.7 Effect of irradiation time and Power Supply to Fe Content

The curve was plotted in irradiation time at most 60 min. Treadline of curve in Fig.7 is not linear but 2^{nd} order polynomial behavior. It is mean the process has optimum state, which is influenced by various of irradiation time and power supply.

CONCLUSION

Iron ore has various chemical content include Fe as main content and others as slag. In extractive metallurgy iron ore was reduced by various model, generally using Cupola furnace. But nowadays, reduction process can be carried out by microwave heating and Charcoal as reducting agent. When iron ore was reduced by charcoal under microwave irradiation, all of phase in iron ore will transform which increase Fe content more than ordinary state.

Power supply and irradiation time is main parameters in the process. The optimum reduction process is consisted by 3000 watt power supply and 60 min irradiation time. This state can incease Fe content up to 81.67%.

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