CHAPTER III
EXPERIMENTAL

3.1 Raw Materials

3.1.1 Iron Ore

Iron ore was obtained from Xieng Khouang Lao PDR and used in the iron production. In the present work, a low grade Iron ore was selected and used; it has %Fe between 40-60 %. The iron ore (XK-01) was characterized for the wt % of elements by EDX (Energy Dispersive X-Ray Fluorescence Spectrometer), the structures by XRD (X-Ray Diffraction Spectrometer), and the compositions by the Wet Chemical Analysis. The average wt % element and SD of the XK-01 iron ore are shown in Table 3-1. All characteristic results of iron ore are shown in Appendix A.

Table 3-1 Wt % Element of XK-01

Elements	Average (wt %)	SD
C	5.95	4.52
O	32.70	0.98
Al	1.69	0.10
Si	2.28	0.26
K	0.26	0.06
Fe	55.99	5.60
Zr	0.87	0.05
Mn	0.00	0.00
Ca	0.00	0.00
Ti	0.03	0.03
Au	0.00	0.00
Cu	0.05	0.05
P	0.18	0.06
S	0.00	0.00
Mg	0.00	0.00
Na	0.00	0.00
Cl	0.00	0.00

3.1.2 Reductant

Reductant is a substance used to reduce oxygen in an iron ore. In the present study, Dai coal, obtained from Xieng Khouang Lao PDR, was analyzed for the Proximate Analysis and the structures by the XRD diffraction analysis (X-Ray diffraction spectrometer). The Dai coal Proximate Analysis result is shown in Table 3-2. All of the characteristic results of Dai coal are shown in Appendix A.

Table 3-2 Proximate Analysis of the Dai coal

	Parameter	Unit	Result
As received basis	Moisture	% by weight	21.29
	Ash	% by weight	5.97
	Volatile matter	% by weight	35.47
	Fixed carbon	% by weight	37.26
	Sulphur	% by weight	0.89
	Gross calorific value	MJ/kg Kcal/kg	20.97 5012
	Net calorific value	MJ/kg Kcal/kg	19.94 4766

3.1.3 Flux

Limestone is a flux used to separate iron from slag or other components during the reduction process. Limestone was obtained from CP® (Thailand) and used in our experiment; the specification of the Limestone is shown in Appendix A.

3.1.4 Binder

Bentonite is a binder used in making a pellet. The sodium bentonite was obtained from Volclay Siam Ltd. and used in our experiments. The specification of a Bentonite from Volclay Siam Ltd. is shown in Appendix A.

3.2 Equipments

1. Crushing machine: Ball mill (The chamber has a diameter 70 cm and a length 100 cm by using spherical grinding media; 1Kg×20 balls, The critical speed was 60 rpm, Rajamangala University of Technology Krungtehep.)
2. Hydraulic pressure pelletizer (Carver, model 3851–0, Petroleum and Petrochemical College Chulalongkorn university)
3. Oven (Memmert, model 600, Petroleum and Petrochemical College Chulalongkorn university)
4. High temperature furnace (Nabertherm,LHT 02/17 at the department of materials engineering, Kasetsart University and Nabertherm,HTCT 08/15 at the department of metallurgical engineering, Chulalongkorn University)
5. Energy Dispersive X-Ray Fluorescence (EDXRF) (Horiba, model 51-ADD0014)
6. X-Ray Diffraction (XRD) (Rigaku D/max; model 2000)

3.3 Characterization

All of the characterization methods are shown in Appendix A-2.

3.3.1 Energy Dispersive X-Ray Fluorescence (EDXRF)

Samples were characterized for wt % of elements by EDX (Horiba, model 51-ADD0014), an Energy Dispersive X-Ray Fluorescence Spectrometer (Hitachi, model S-4800), connected to a scanning electron microscope.

3.3.2 X-Ray Diffraction (XRD)

The sample was characterized for its structures by XRD or an X-Ray diffraction spectrometer (Rigaku D/max; model 2000).

3.3.3 Scanning Electron Microscope (SEM)

Sample particle sizes were measured by SEM (Hitachi, model S-4800), a scanning electron microscope. The SEM accelerating voltage, current and magnification are specified in the figures below.

3.3.4 Wet Chemical Analysis

XK-01 iron ore was also analyzed for its compositions by the Wet Chemical Analysis at the Rock and Mineral Analysis Department of Mineral Resources, Ministry of Natural Resources and Environment Thailand.

3.3.5 Proximate Analysis

Dai coal was analyzed by the Proximate Analysis by the Electricity Generating Authority of Thailand, Mah Moh, Lampaeng.

3.3.6 Microstructure Analysis

The Iron nugget from suitable condition was prepared the surface by using P1200 glass paper and Aluminum powder. After the surface was clear, the 2 % Nitral was used to etching. The microstructure of Iron nugget was measured by SEM (Hitachi, model S-4800).

3.3.7 Density of Iron Nugget

All of the iron nuggets were measured the density by pycnometer.

3.4 Methodology

3.4.1 Grinding Raw Materials

The XK-01 iron ore and the Dai coal were grinded by a dry cylindrical ball mill in which the chamber has a diameter 70 cm and a length 100 cm. Grinding media is of the, 1Kg spherical type, with 20 balls, and the critical speed was 60 rpm. Raw materials of 20 kg were added in each batch. The product was screened by a mesh 17 (1.494 mm) and the oversize was grinded again. The grinding steps are

shown in Figure 3-1. The particle size after grinding was measured by SEM (Hitachi, model S-4800).

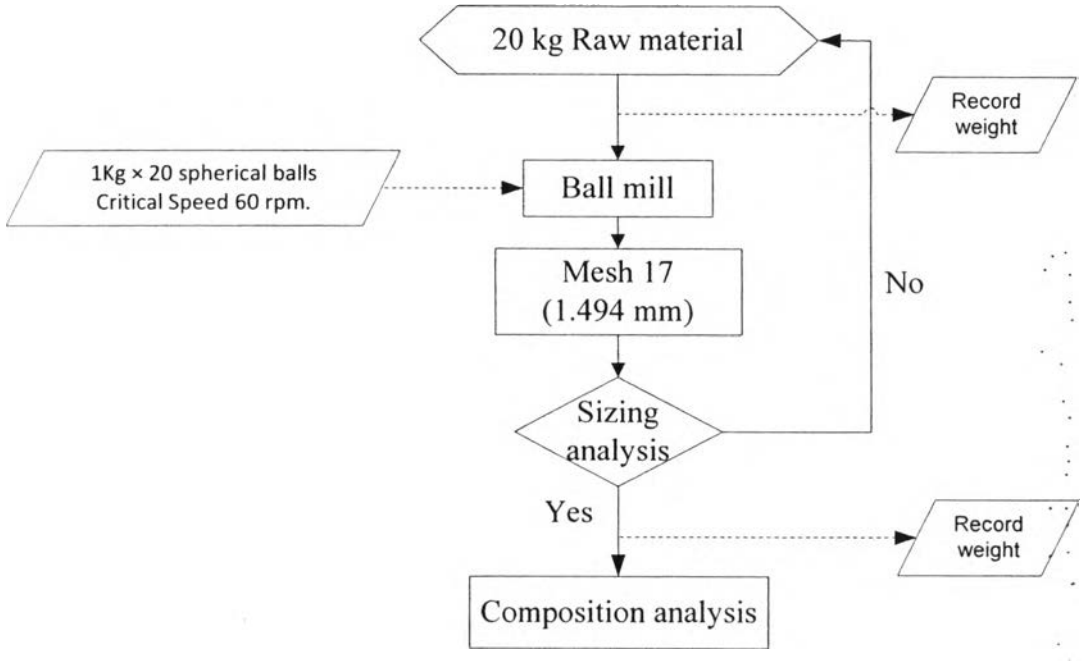


Figure 3-1 Grinding steps.

3.4.2 Mixing and the Pellet Preparation

3.4.2.1 Amounts of the raw materials in the mixtures of the experiments 1–7

XK-01, Dai coal, Limestone, Bentonite were mixed in which the amounts of the raw materials in the mixture are shown in Table 3-5. Water of 10 % by weight of the mixture was added to the mixture of the raw materials. The mixture was well mixed until a homogenous mixture was obtained.

Table 3-3 Amounts of the raw materials in the mixtures of the experiments 1–7

Experiment	No.	Mol ratio				Weight				Mold
		Fe	C/Fe	Limestone/Al_2O_3+SiO_2	Bentonite	XK-01 (g)	Dai coal (g)	Limestone (g)	Bentonite (g)	
1		1	0.49	0.89	0.02	1000.00	147.00	99.31	13.99	A
2		1	1.72	0.89	0.02	300.06	155.53	29.78	4.22	B
3		1	1.72	0.89	0.02	300.06	155.53	29.78	4.22	A
4		1	1.72	0.89	0.02	300.05	155.5	29.79	4.20	A
5	1	1	1.24	0.89	0.02	200.00	74.87	19.86	2.80	A
	2		1.34				80.63			
	3		1.44				86.39			
	4		1.53				92.15			
	5		1.63				97.91			
	6		1.72				103.67			
	7		1.82				109.43			
	8		1.91				115.19			
6	1	1	1.53	0.50	0.02	200.00	92.15	11.20	2.80	A
	2			0.60				13.39		
	3			0.70				15.62		
	4			0.75				16.80		
	5			0.89				19.86		
	6			1.00				22.31		
	7			1.26				28.00		
	8			1.76				39.21		
	9			2.26				50.41		
7	1	1	1.53	0.45	0.02	200.00	92.15	16.80	2.80	A
	2			0.55						
	3			0.65						

3.4.2.2 Pellet Preparation

The mixture was fed into a cylindrical mold (Mold A has a diameter of 4 cm and 7 cm high, Mold B has a diameter of 2.5 cm and 4 cm high) for making pellets that is shown in Figure 3-3. The pellet was compressed in the mold at 6,000 psi, 60 sec and then dried at 80°C and for 20 hr. Steps of the pellet preparation are shown in Figure 3-2.

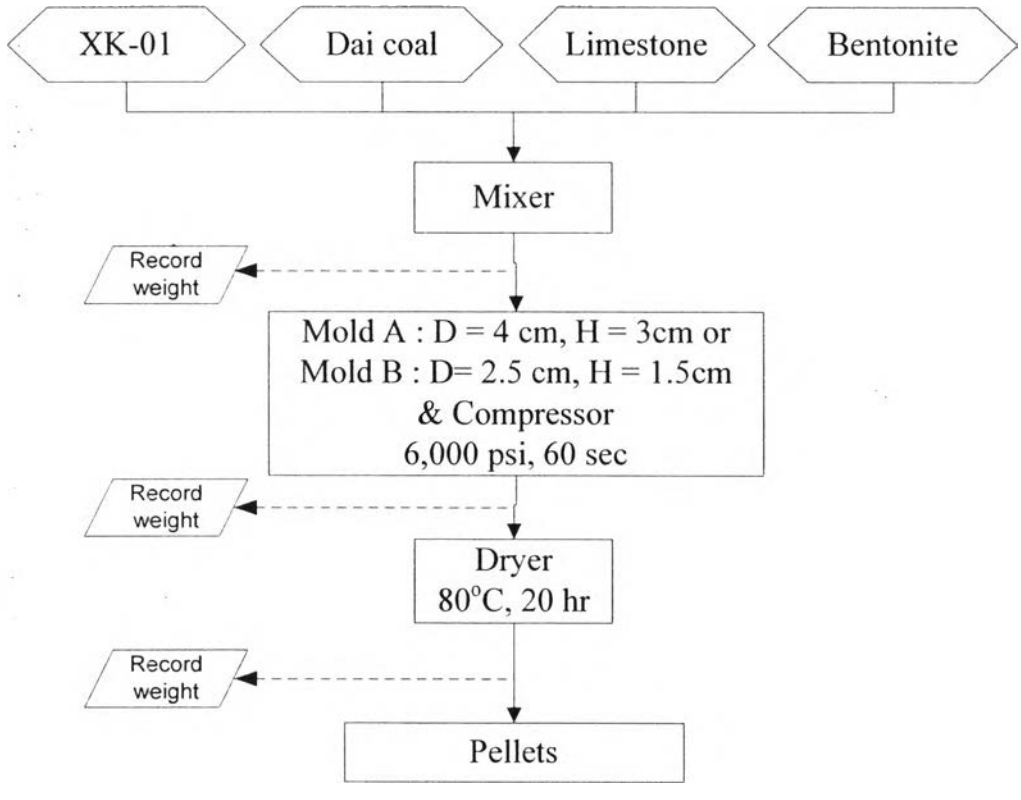
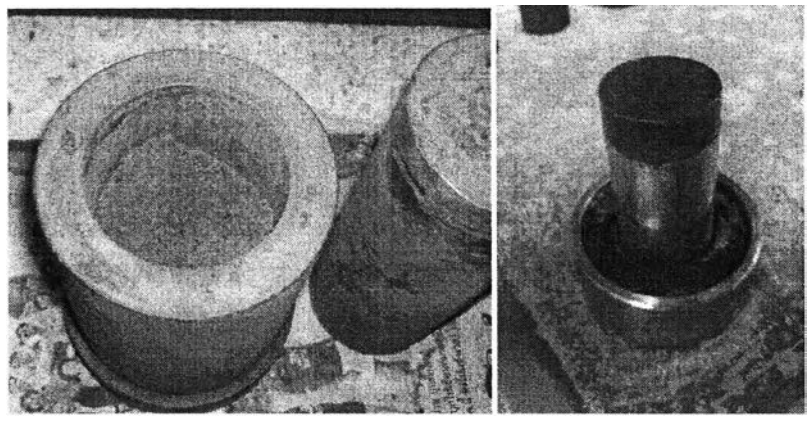


Figure 3-2 Step of pellet preparation.



Mold A Mold B

Figure 3-3 Cylindrical molds A and B.

3.4.3 Reduction

3.4.3.1 Experiments 1 and 2

All pellets of experiments 1–2 were reduced by a furnace (Nabertherm,LHT 02/17). The pellet was heated from room temperature (30°C) to the reduction temperature at a heating rate of 10 °C/min and then held on during the reduction time (Error of furnace +/- 5 °C). The temperatures profile of furnace is shown in Figure 3-4. The reduction conditions are shown in Tables 3-4 and 3-5. The sample was taken out from the furnace at a temperature below 200 °C.

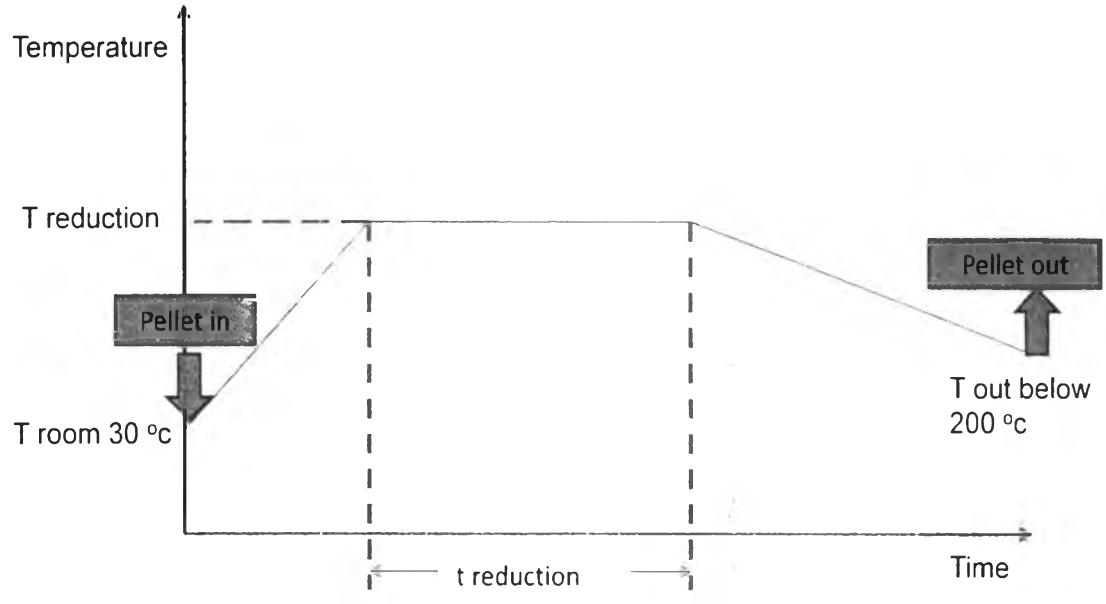


Figure 3-4 Temperature profile of the furnace for the experiments 1 and 2.

3.4.3.2 Experiments 3–7

The pellet was reduced in a furnace (Nabertherm,HTCT 08/15); it was fed at the reduction temperature which was held on during the reduction period. After the reduction period, the sample was taken out immediately. The temperature profile of the furnace is shown in Figure 3-5. The reduction conditions are shown in Tables 3-6 – 3-10.

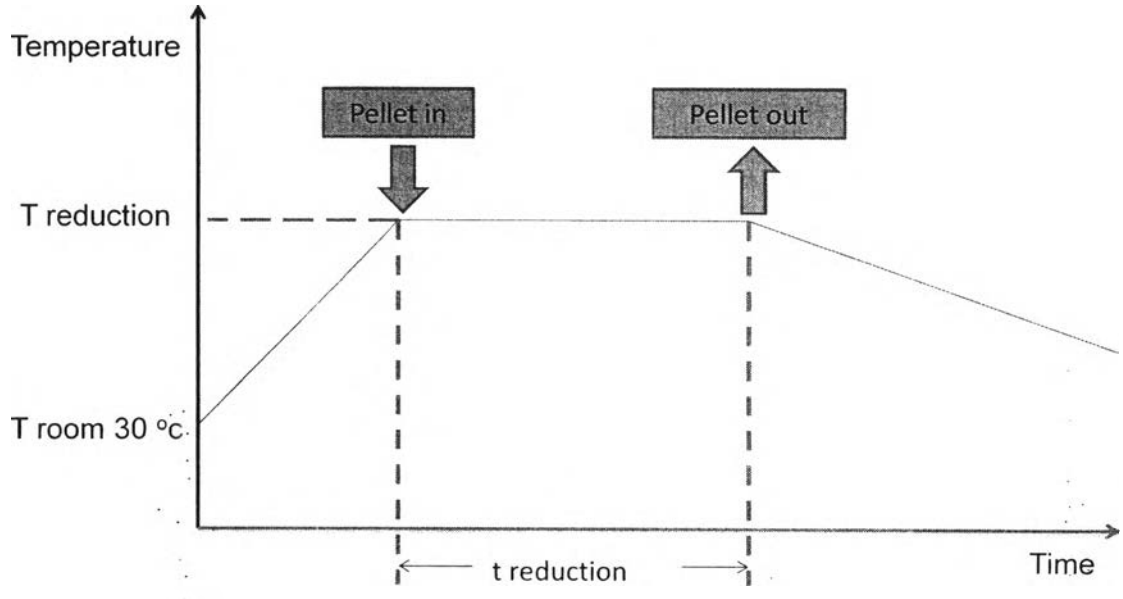


Figure 3-5 Temperature profile of the furnace for the experiments 3–7.

Table 3-4 The reduction conditions of the experiment 1

No.	Reduction Temperature (°C)	Time (min)		
		Heating from room temperature (30°C)	Hold on reduction temperature	Cool down (hr)
1	1500	147	50	20
2	1400	137	50	
3	1300	127	50	
4	1200	117	50	
5	1100	107	50	
6	1000	99	50	

Table 3-5 The reduction conditions of the experiment 2

No.	Reduction Temperature (°C)	Time (min)		
		Heating from room temperature (30°C)	Hold on reduction temperature	Cool down (hr)
1	1500	147	50	20
2	1450	137	50	
3	1400	127	50	
4	1300	117	50	
5	1200	107	50	

Table 3-6 The reduction conditions of the experiment 3

No.	Reduction Temperature (°C)	Hold on reduction temperature (min)
1	1500	50
2	1450	50
3	1400	50
4	1375	50
5	1350	50
6	1300	50
7	1200	50

Table 3-7 The reduction conditions of the experiment 4

No.	Reduction Temperature (°C)	Hold on reduction temperature (min)
1	1450	40
2	1450	30
3	1450	20
4	1450	10
5	1425	30
6	1425	20
7	1425	15
8	1425	10
9	1400	40
10	1400	35
11	1400	30
12	1375	40

Table 3-8 Experiment 5, varying the mol ratio C/Fe using suitable conditions from the experiment 4

No.	Reduction Temperature (°C)	Hold on reduction temperature (min)	Mol ratio C/Fe
1	1425	20	1.24
2			1.34
3			1.44
4			1.53
5			1.63
6			1.72
7			1.82
8			1.91

Table 3-9 Experiment 6, varying the mol ratio of Limestone/Al_2O_3+SiO_2 using suitable conditions from the experiments 4–5

No.	Reduction Temperature (°C)	Hold on reduction temperature (min)	Mol ratio C/Fe	Mol ratio Limestone/Al_2O_3+SiO_2
1	1425	20	1.53	0.50
2				0.60
3				0.70
4				0.75
5				0.89
6				1.00
7				1.26
8				1.76
9				2.26

Table 3-10 The reduction conditions of experiment 7 using suitable conditions from the experiments 4–6

No.	Reduction Temperature (°C)	Hold on reduction temperature (min)	Mol ratio C/Fe	Mol ratio Limestone/Al_2O_3+SiO_2
1	1425	20	1.53	0.45
2				0.55
3				0.65