CHAPTER III
EXPERIMENTAL

3.1 Raw Materials

3.1.1 Iron Ore

Iron ore, obtained from Phu Khoud, is the iron oxide used in the iron production. In the present work, a low grade Iron ore was used which has %Fe between 40-50 %. The iron ore (XK-03) was characterized for the wt % of elements by XRF (X-ray fluorescence spectrophotometer) and the particle diameter by PSA (Particle Size Analyzer). The average wt % element of the XK-03 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-03

Element	% wt
Al	4.824
Ca	0.07098
Cs	0.5234
Fe	41.62
K	0.8889
Mg	0.3062
Mn	2.911
Na	0.1928
O	36.91
P	0.3598
S	0.026
Si	11.2
Ti	0.1676

3.1.2 Reductant

Reductant is a substance used to reduce oxygen in an iron ore. In the present study, FIRST coal, obtained from FIRST Co., Ltd., was analyzed for the fixed carbon by the proximate analysis and the particle diameter by PSA (Particle Size Analyzer). The FIRST coal Proximate Analysis result is shown in Table 3.2.

Table 3.2 Proximate analysis of FIRST coal

Moisture	% Total Moisture	1.23
Properties : Air dried basis	% Inherent Moisture	0.97
	% Ash	23.84
	Volatile Matter	9.89
	% Fixed Carbon	65.30
	% S	0.49
	Gross Calorific Value (Kcal/kg)	6199.00

3.1.3 <u>Flux</u>

Limestone is a flux that used to separate the iron from the slag or the other components during the reduction process. Limestone was obtained from Petch Thai Chemical Co., Ltd. (Thailand) and used in our experiment; the specification of the Limestone is shown in Appendix A.

3.1.4 <u>Binder</u>

Bentonite is a binder that used in making a pellet. Sodium bentonite was obtained from Dhebkaset Industry Co., 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 diameter 70 cm and a length 100 cm by using spherical media; 1 kg of 15 balls, The critical speed is 60 rpm, Rajamangala University of Technology Krungtehep)

2. Hydraulic pressure pelletizer (Caver, model 3851-0, The Petroleum and Petrochemical College Chulalongkorn University)

3. Oven (Memmert, model 600, The Petroleum and Petrochemical College Chulalongkorn University)

4. High temperature furnace (Protherm Furnace, model PLF130/6)

3.3 Characterization

All of the characterization methods are shown in Appendix A2.

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 Fluorescence Spectrometer (XRF)

Samples were characterized for wt % of elements by XRF (PANalytical, model AXIOS PW4400).

3.3.3 Particle Size Analyzer (PSA)

Samples were characterized for size of diameter sizes by PSA (Malvern, Mastersizer X).

3.3.4   Gas Pycnometer (GP)

Samples were characterized for density by GP (Quantachrome, Ultrapycnometer 1000). The samples were weight and placed in the chamber. The Helium was used as a medium gas to determine the volume of the sample. The density of samples can be calculated from weight and volume.

3.3.5   Polarized Optical Microscope (POM)

Samples were analyzed the surface appearances by POM (Leica, CH-9453). The samples were place on the holder. The POM provide light source to the samples and reflect to the objective lens, thought to the detector.

3.3.6   Scanning Electron Microscope (SEM)

Sample were analyzed the morphology by SEM (Hitachi, model S-4800). The SEM accelerating voltage, current, and magnification are specified in the SEM figures.

3.3.7   X-Ray Diffraction (XRD)

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

3.3.8   Wet Chemical Analysis

XK-03 iron ore was also analyzed for its compositions by the Wet Chemical Analysis at the Thai Pride Cement Co., Ltd.

3.3.9   Proximate Analysis

The FIRST coal was analyzed by the Proximate Analysis at the Thai-Pride Cement Co., Ltd.

3.4 Methodology

3.4.1 Analysis and Preparation of Raw Materials

The XK-03 and FIRST coal were grinded by a cylindrical ball mill. The diameter and length of the chamber are 70 cm and 100 cm, respectively. The media is 1 kg of spherical metallic ball, with 15 balls. The critical speed is 60 rpm. The product was screened by a mesh 20 (300 µm) and the oversize was grinded again. The grinding step is shown in Figure 3-1. The particle size after grinding was measured by PSA (Malvern Mastersizer X).
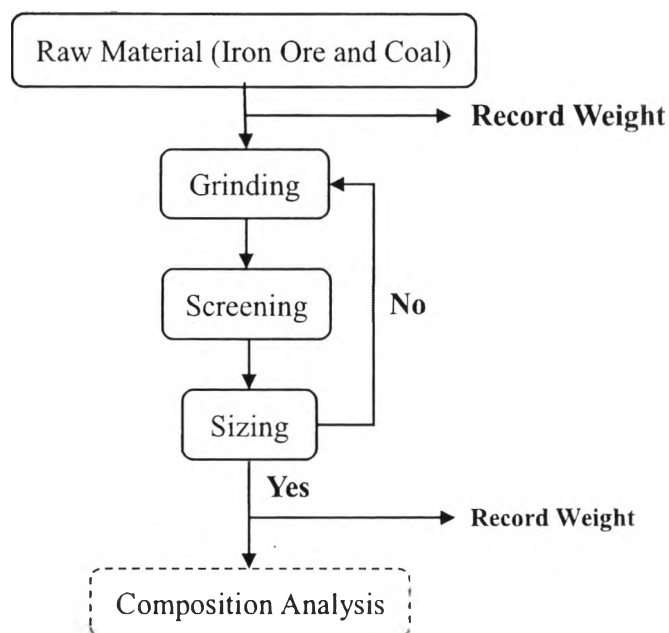
Figure 3.1 Grinding step of raw material.

3.4.2 Mixing of Raw Materials

XK-03, FIRST Coal, Limestone, and Bentonite were mixed by using molar ratios that tabulated in Table 3.3. Water of 10% by weight of the mixture was added. The mixture was mixed until it becomes homogenous phase.

Table 3.3 Amounts of the raw materials in the mixtures of the experiment 1-7

Experiment	No.	Mol ratio				Weight (g)			
		Fe	C/Fe	Limestone/Al_2O_3+SiO_2	Bentonite/Fe	XK-03	FIRST Coal	Limestone	Bentonite
1	1-2	1	1.6	0.49	0.016	200	44.91	47.52	2.03
	3-6				0.075				3.39
	7-10				0.085				4.74
2	1-3	1	1.6	0.49	0.038	200	44.91	47.52	4.74
	4-6				0.082				10.17
3	1-4	1	1.6	0.49	0.038	200	44.91	47.52	4.74
4	1	1	1.4	0.75	0.035	200	38.38	73.26	4.34
	2		1.6				43.86		
	3		1.8				49.34		
	4		2.0				54.83		
	5		2.2				60.31		
5	1	1	1.6	0.45	0.035	200	43.86	43.95	4.34
	2			0.55				53.72	
	3			0.65				63.49	
	4			0.75				73.26	
	5			0.85				83.03	
	6			0.95				92.79	
	7			1.05				102.56	
6	1-3	1	1.6	0.65	0.035	200	43.86	63.49	4.34
7	1-3	1	1.6	0.65	0.025	200	43.86	63.49	3.10
	4-6				0.035				4.34
	7-9				0.045				5.58

3.4.3 Pellet Preparation

The mixture was compress by cylindrical mold (4 cm of diameter) for making pellets. The pellet was compressed at 6000 psi, 2 minutes and then dried at 80°C for 24 hr. The pelletizer step is shown in Figure 3.2.
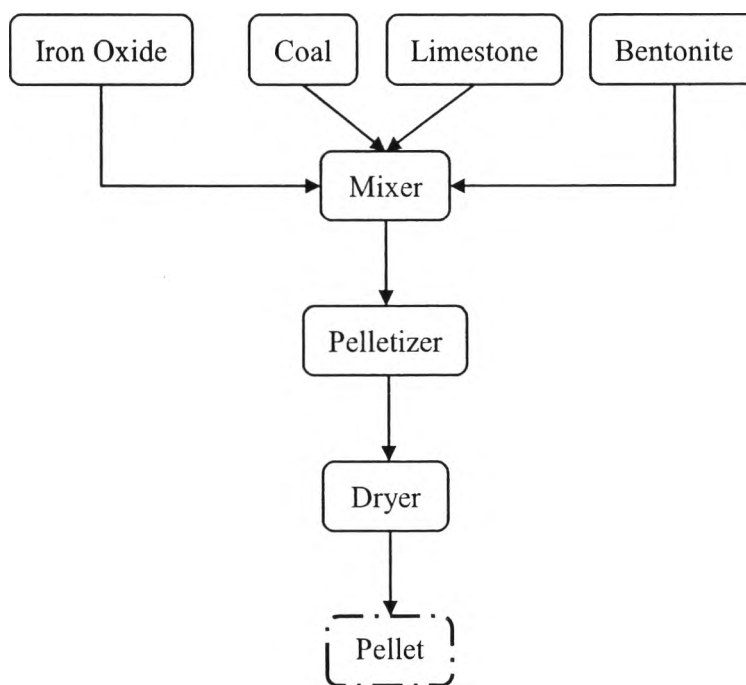
Figure 3.2 Preparation of the pellet.

3.4.4 Drop Test of the Pellet

The pellet of experiment 1, 2, and 7 were dropped to the ground (polished stone floor) from one meter high at room temperature until it breaks. The molar ratios of bentonite/Fe were varied to determine the strongest condition of the pellet.

3.4.5 Reduction

3.4.5.1 Experiment 3 and 6

All pellets of experiments 3 and 6 were reduced by furnace. The pellets were heated from room temperature (30°C) to the reduction temperature and then held on during the reduction time. The reduction conditions of experiment 3 and 6 are shown in Table 3.4 and 3.5, respectively. The samples were cooled down for 24 hours.

Table 3.4 The reduction conditions of the experiment 3

No.	Reduction Temperature (°C)	Soaking Time at Desire Temperature (mins)
1	1200	30
2		60
3	1300	30
4		60

Table 3.5 The reduction conditions of the experiment 6

No.	Reduction Temperature (°C)	Soaking Time at Desire Temperature (mins)
1		45
2	1300	75
3		90

3.4.5.2 Experiment 4 and 5

All pellets were reduced by furnace. The pellet were heated from room temperature (30°C) to 1300°C and held on during the reduction time of 60 minutes. The mole ratios of C/Fe and Limestone/$Al_2O_3+SiO_2$ were varied, which showed in Table 3.3. The samples were cooled down for 24 hours.