CHAPTER III

EXPERIMENTAL PROCEDURE

3.1 Experimental conditions and experimental procedure flow chart

Batch compositions of red clay, sand, grog and alkali materials are shown in Table 3.1.

Table 3.1. Batch compositions of red clay, sand, grog and alkali materials.

formula	clay (wt.%)	sand (wt%)			grog (wt%)			fluxes (wt%)
		raw*	#50	#100	raw*	#50	#100	
mixed clay 1	87.0	5.0	-	-	8.0	-	-	-
mixed clay 2	87.0	-	5.0	-	-	8.0	-	-
10% sand	82.0	10.0	-	-	8.0	-	-	-
15% sand	77.0	15.0	-	-	8.0	-	-	-
#50 sand	87.0	-	5.0	-	8.0	-	-	-
#100 sand	87.0	-	-	5.0	8.0	-	-	-
10% grog	85.0	5.0	-	-	10.0	-	-	-
12% grog	83.0	5.0	-	-	12.0	-	-	-
#50 grog	87.0	5.0	-	-	-	8.0	-	-
#100 grog	87.0	5.0	-	-	-	-	8.0	-
3% NaO	92.2	4.9	-	-	-	-	-	3.0
1% CaO	94.1	5.0	-	-	-	-	-	1.0
2% glass	93.1	4.9	-	-	-	-	-	2.0
4% glass	91.2	4.8	-	-	-	-	-	4.0
6% glass	89.3	4.7	-	-	-	-	-	6.0
8% glass	87.4	4.6	-	-	-	-	-	8.0
S50G5	90.0	-	5.0	-	5.0	-	-	-
SGG	85.7	-	4.8	-	4.8	-	-	4.8

* detail of particle size is shown in appendix A

The process flow chart is shown in Fig. 3.1. Plastic red clay, sand and grog were supplied from factory (Siamese Merchandise Co., Ltd.) and alkali materials were purchased from market. The particle size distribution, chemical composition (XRF) and crystal phase (XRD) of these materials were characterized. These raw materials were mixed according to the proportion shown in Table 3.1. Hand mixed clay body was extruded by a piston extruder machine in to a cylindrical shape of 10 mm in diameter and 10 cm in length and drying shrinkage was measured. These specimens were sintered at 900, 950, 1000, 1050 and 1100°C with a heating and cooling rates of 3 and 5 °C/min, respectively, and with a soaking time of 2 h in an electric furnace. Fired color, crystal phase, water absorption, bulk density, bending strength, capillary pores volume and microstructure were observed and characterized.

The optimum formula with low water absorption, high bulk density and high strength were selected to produce specimens using production equipment in a factory. Mixed clay body was formed with roller machine in to pottery shape and fired at production temperature (950°C approximately) in the gas furnace. Fired color, water absorption, bulk density and microstructure of the product were observed and freeze-thaw test (DIN 52252 standard method) was performed.

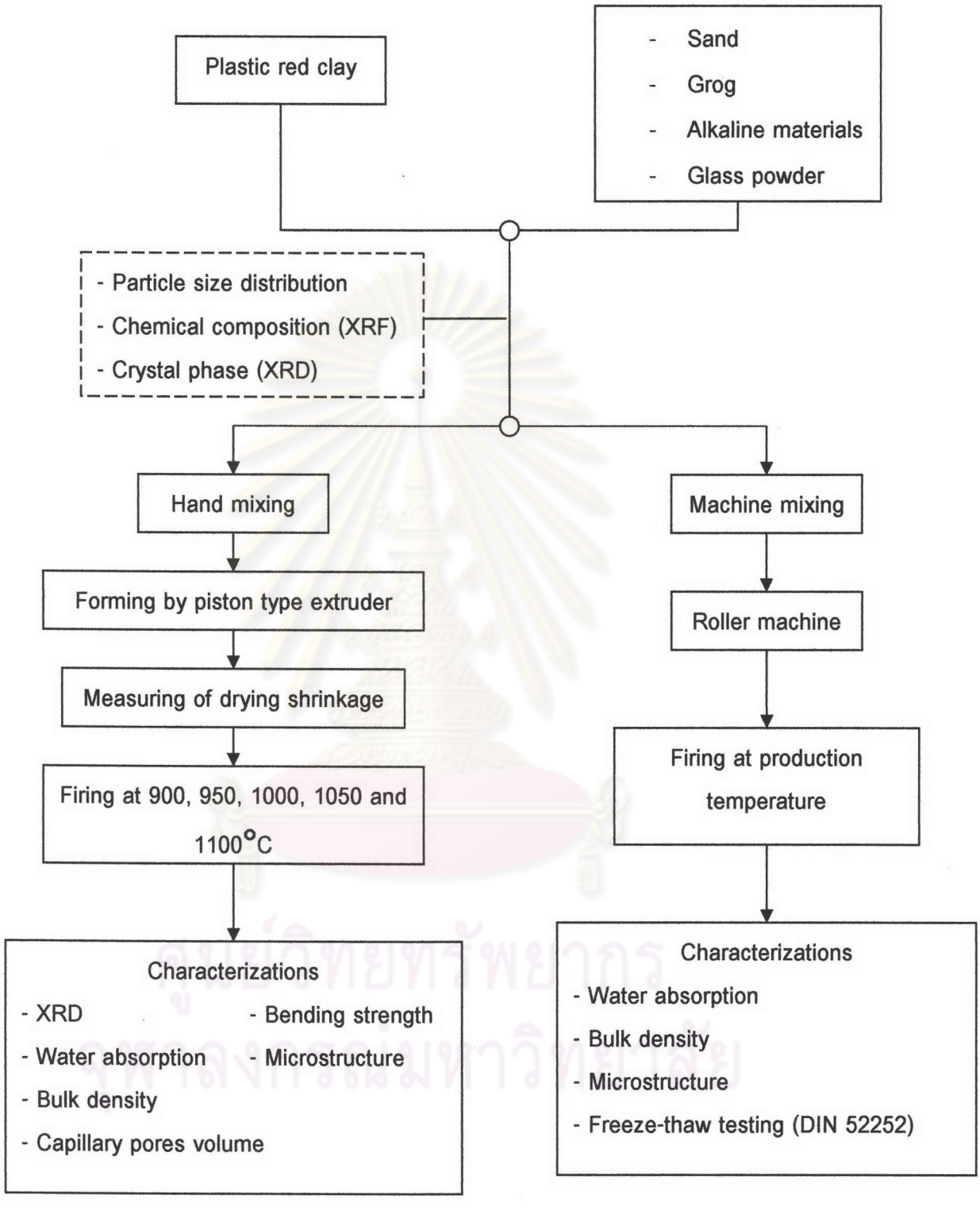

Fig. 3.1. Flow chart of specimen preparation and characterization.

3.2 Property measurements and characterizations

3.2.1 Particle size distribution of starting materials

Sedimentation method (Horiba LA-920) was used for investigation of particle size distribution of red clay powder. Raw clay was dissolved in water with dispersant, hexa-meta-phosphoric acid 0.2 wt. % solution. The solution is agitated for 15 min in a mixer and let to settle into test tubes.

Sand and grog were colleted from factory (Siamese Merchandise Co., Ltd.). The particle size distribution was obtained by wet sieving using 20, 35, 50, 100 140, 200, 230 and 325 mesh screens.

3.2.2 Water content of starting materials

Red clay, sand and grog were dried at 110°C for 24 h and then water content was calculated as in equation (1)

$$water\ content\ (\%) = \frac{before\ drying\ wt. - dried\ wt.}{dried\ wt.} x\ 100 \quad \text{---------- (1)}$$

3.2.3 Chemical analysis (XRF)

The red clay was dried at 110°C for 24 h while commercial products and soda-lime glass were manually crushed and then sieved through a 100 mesh screen (150 μm). Chemical compositions of these powder were characterized by X-ray fluorescence spectrometer, Philips model PW 2400.

3.2.4 Crystal phase analysis (XRD)

The commercial products and experimental specimens were crushed and sieved through a 100 mesh (150 μm) screen. Crystal phases were analyzed by X-ray diffractometer (D8 – Advance, Bruker), operated at 40 kV, 30 mA. The value of 2θ was from 10-60°, and the scanning speed was 4°/min.

3.2.5 Physical properties

The experimental specimens were extruded by piston extruder (Hydraulic type) into cylindrical rod, 10 mm in diameter. The extrusion operation was performed without vacuum and the specimens were cut to 10 cm in length.

3.2.5.1 Shrinkage

The lengths of specimens were measured before and after drying at ambient temperature in non-controlled atmosphere for 48 h and successively drying in an electric oven at 110°C for 24 h, for the measurement of the drying shrinkage. The lengths of specimens were measured before and after firing at 900, 950, 1000, 1050 and 1100°C for the determination of firing shrinkage.

3.2.5.2 Water absorption and bulk density

Water absorption and bulk density were measured by Archimedes' method.

3.2.5.3 Bending strength

Modulus of rupture of fired specimens was measured by three points bending mode according to ASTM C 674-88. The span of measurement was 80 mm.

3.2.6 Microstructure (optical microscope)

The cross section of rod specimen was observed by optical microscope (Olympus, BX60M). Specimens selected for observation are show in Table 3.2.

Table 3.2. Specimens selected for optical microscope.

commercial products	experimental products	experimental specimens						
			temperature °C					
		formula	850	900	950	1000	1050	1100
SM* Portugal German	S50G5 SGG	Mixed clay 1		•	•	•	•	•
		Mixed clay 2		•	•	•	•	•
		1% CaO	•	•	•	•	•	•
		4% glass	•	•	•	•	•	•

*SM=Siamese Merchandise Co.,Ltd.

3.2.7 Capillary pore volume

Capillary pore volume measured by pore size analyzer (SA 3100, COULTER). The method used is based on the BJH (Barrett, Joyner and Halenda) gas sorption method where gas molecules of known size are condensed by the process of capillary condensation. The quantity of gas condensed and the resultant sample pressure are recorded and used for subsequent calculation.

3.2.8 Freeze-thaw testing (DIN 52252 standard method)

Freeze-thaw test was performed as follows; (1) check initial condition of the commercial products (cracks and damage marked), (2) dry for 24 h at 105°C and then cooled to room temperature, (3) Immersed in water at ambient temperature for 48 h, (4) freezed in the refrigerator immediately at -15°C for 60 minutes and (5) thawed for 60 minutes in water at 20 ± 2°C. The freeze-thaw cycle was repeated 25 times.