CHAPTER 3

METHODOLOGY

This chapter describes the experimental procedures employed in fabricating and characterizing brick specimens. The experiments are divided into three parts: 1. the preparation and characterization of raw materials, 2. the production of specimens, and 3. tests of physical and mechanical properties and microstructure of specimens after firing at various temperatures including tests of their thermal behavior.

3.1 Preparation of Raw Materials

In this experiment, Hang Dong clay and charcoal used as raw materials are obtained from Hang Dong district in Chiang Mai province. First of all, all raw materials were crushed and sieved into different particle sizes. For Hang Dong clay, the particle size distribution was obtained by dry sieving through No. 60 mesh while the particle sizes of charcoal additive were sieved step by step through No. 35, 40 and 45 meshes to obtain various sizes in the range of 2-3 mm, 1-2 mm and less than 0.5 mm. Then, all raw materials were examined for various properties namely, using XRF for characterizing chemical compositions, XRD for mineral compositions, particle size analyzer for particle size distribution and SEM for microstructure (Figure. 3.1).

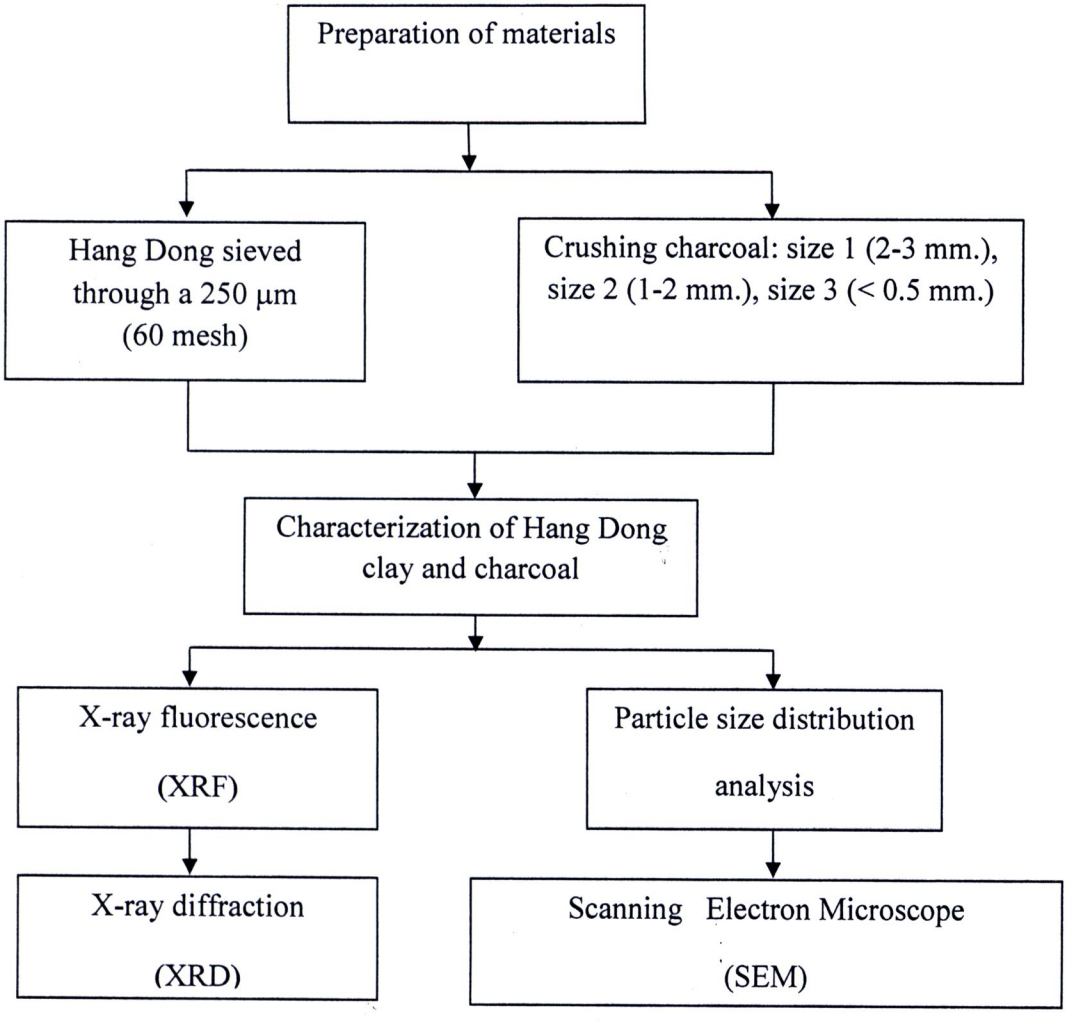

Figure. 3.1 A schematic preparation of Hang Dong clay and charcoal.

3.2 Specimen preparation

In order to determine the extent of the pore-forming effects of the charcoal particle sizes ranging 2-3 mm. (size 1), 1-2 mm. (size 2) and less than 0.5 mm. (size 3), they were added into raw brick clay and divided into five different specimens mixed with increasing charcoal (0%, 2.5%, 5.0%, 7.5% and 10%) content (Table 3.1). Each specimen was mixed in a porcelain ball mill in order to ensure homogenous mixing. Then, each was formed (mixed with about 20-30% water to plastic condition to obtain the desired shape), into soft-mud rectangle-shaped specimens with an internal dimension of 5.0 cm x 9.5 cm x 3.0 cm using brick hand-molding. Specimens were air dried at room temperature for 24 hrs, and then over dried at 110 ± 5 °C for another 24 hrs to remove water content. Then, each group of green specimen was fired at four different temperatures: 900, 950, 1000 and 1100 °C with two hours soaking time in a gas kiln furnace. The specimens were naturally cooled down to room temperature in the furnace. Then, physical and mechanical properties and microstructure of specimens were tested. Finally, certain specimens fired at certain temperatures were selected to be tested on their thermal expansion coefficients, thermal conductivity and specific microstructural characteristics respectively. (Figure. 3.2)

Table 3.1 Mixture proportions of the specimens (%by weight).

Specimens	Charcoal size	Charcoal (%)	Hang Dong clay (%)
1	original Hang Dong clay	0	100.0
2*	mixed with charcoal size1 mixed with charcoal size2 mixed with charcoal size3	2.5	97.5
3*	mixed with charcoal size1 mixed with charcoal size2 mixed with charcoal size3	5.0	95.0
4*	mixed with charcoal size1 mixed with charcoal size2 mixed with charcoal size3	7.5	92.5
5*	mixed with charcoal size1 mixed with charcoal size2 mixed with charcoal size3	10.0	90.0

*Mixed with charcoal particles: size 1 (2-3 mm.), size 2 (1-2 mm.) and size 3 (less than 0.5 mm.).

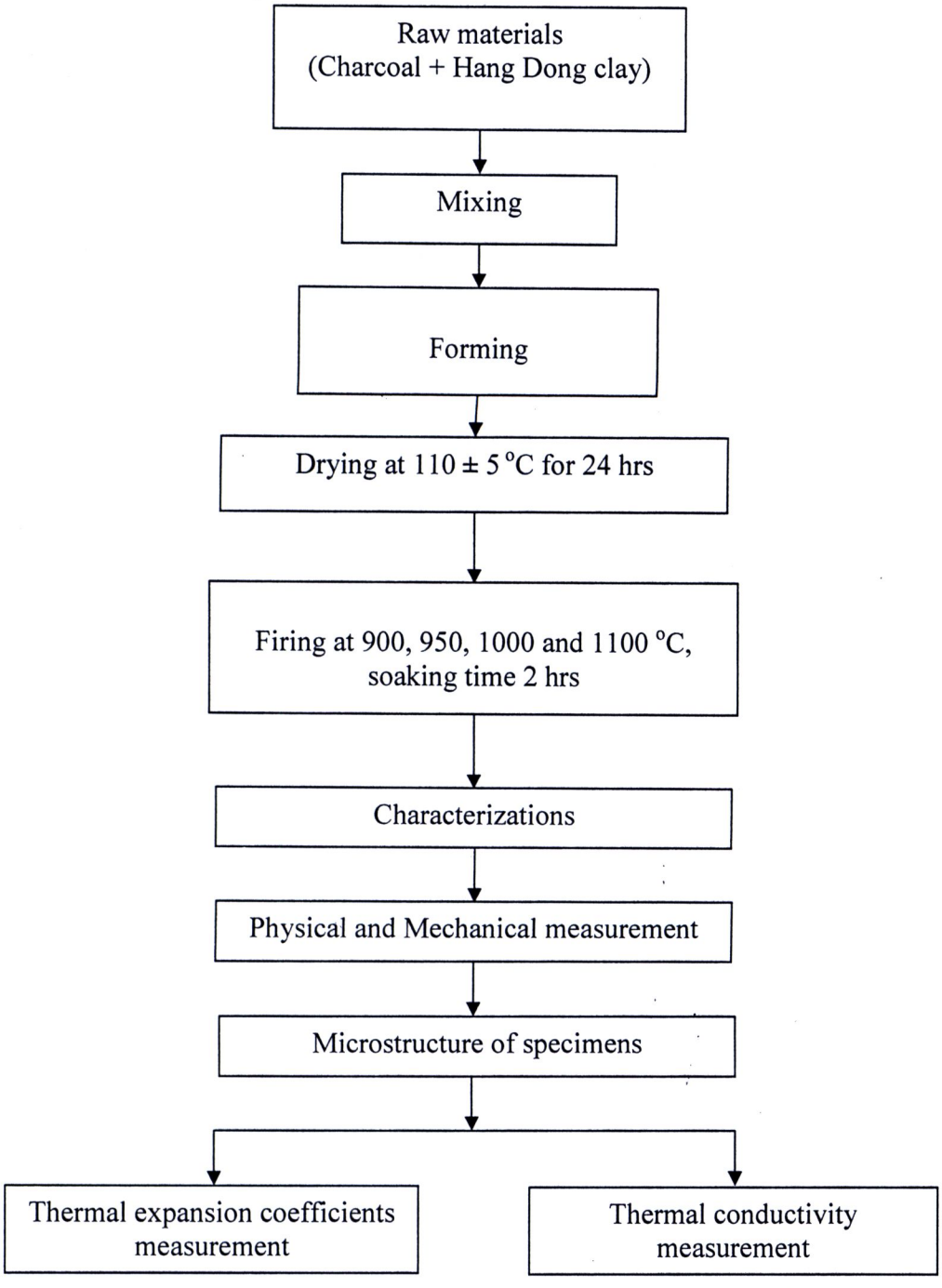

Figure. 3.2 Experimental procedures of specimen preparation and characterization.

3.3 Specimen characterization

3.3.1 Determination of chemical composition

The chemical composition of materials (Hang Dong clay and charcoal) in a form of powders was studied using an X-ray fluorescence (XRF: Mesa-500W, Horiba, Japan).

Specimen preparation

The starting materials (Hang Dong clay and charcoal) were sieved through a 250 μm mesh or 60 mesh, then, analyzed for their chemical composition.

3.3.2 Determination of mineral composition

The crystalline structure of Hang Dong clay and charcoal was determined using an X-ray diffraction technique (XRD: X' Pert Pro MPD, Philips, Netherlands).

Specimen preparation

All specimens were ground into powders, placed in the holder of diffractometer and scanned with Cu Kα radiation. X-ray was performed on powder specimens using a Bruker D8 Advance diffraction. Patterns were recorded from 2 to 80° 2θ with a step interval of 0.04° 2θ and counting time of 1 s per step.

3.3.3 Particle size distribution analysis

The particle size distribution was measured using a particle size analyser (Mastersizer 2000+Hydro 2000 MU, Malvern Instruments Limited, UK).

Specimen preparation

An average particle size of Hang Dong clay sieved through a 250 μm mesh or 60 mesh was analyzed by a laser diffraction. Charcoal additive particles in a range of sizes: 2-3 mm, 1-2 mm and less than 0.5 mm were obtained from dry sieving step by step through No. 35, 40 and 45 meshes.

3.3.4 Determination of microstructure

The microstructure of Hang Dong clay, charcoal and fired clay specimens were examined and analyzed using a scanning electron microscope (SEM) (LEO 1460 VP,Zeiss).

Specimen preparation

For microstructural analysis, Hang Dong clay, charcoal powder and cross section fired brick specimens were mounted on stubs, gold-coated in vacuum and viewed under scanning electron microscope.

3.3.5 Thermal analysis (TGA/DTA)

For the thermal analysis of specimens, Hang Dong clay mixed with increasing amount of charcoal 0%, 2.5%, 5.0%, 7.5% and 10%, were studied using thermogravimetric analysis (TGA) and differential thermal analysis (DTA). These techniques were used to detect the weight loss and endothermic or exothermic changes of the specimens.

Specimen preparation

The specimens were carried out using thermogravimetric analyzer (851^e STAR^e Thermobalance, Mettler Toledo, Switzerland) at a heating rate of 3 °C/min, heated from 25 °C to 950 °C atmosphere air flow rate 50 ml/min. The curve of resulting weight changes versus temperatures gave the information on the thermal stability and composition of the original Hang Dong clay specimen, the intermediate compounds, and the residues.

3.3.6 Thermal expansion coefficients (COE)

To study thermal expansion coefficients, clay specimens mixed with increasing amount of charcoal 0%, 2.5%, 5.0%, 7.5% and 10% were pressed into rod shape specimens (5 mm diam, 25 mm long), which were then fired in an electrical muffle furnace at 950 °C for 3 hrs. All specimens were polished to produce a smooth and parallel shape. Then they were measured to determine a function of temperature using a dilatometer (DIL 420 C, Netzsch, Germany). COE was calculated between 25-575 °C (ASTM C372-94) [57] using equation (3.1).

$$COE = l \times dT/dl \tag{3.1}$$

Where dl is the dimension change of fired briquette specimens, l is the original dimension of fired briquette specimens and dT is the temperature change.

3.3.7 Thermal conductivity analysis (k)

The thermal conductivity measurement test was conducted according to an adapted experimental procedure of international standards ASTM C 177-97 [58]. The thermal conductivity was calculated by using the following equation:

$$\frac{dq}{dA} = k\frac{dT}{dx} \tag{3.2}$$

Where q is the rate heat flow in direction normal to surface (W), k is the thermal conductivity (W/m K), A is the surface area (m^2), dT is the temperature difference, the thickness (K) and dx are the distance measured normal to surface (m). Fired sheet specimens mixed with increasing amount of charcoal 0%, 2.5%, 5.0%, 7.5% and 10%. All specimens were fired at 950 °C and the size of 30 cm wide x 30 cm long and 25 mm. thick for the measurement of thermal conductivity respectively as shown in Figure. 3.3.

Figure. 3.3 Thermal conductivity testing.

3.3.8 Densification analysis

3.3.8.1 Measurement of physical properties

The fired shrinkage of all specimens was determined by direct measurement of a length of a specimen before and after firing at 900-1100 °C. The linear drying shrinkage and total linear shrinkage specimens compared to the length before shrinkage were measured using the standard of ASTM C362-82 (2002) [59]. The specimens were calculated as follows.

$$\% \text{ Linear drying shrinkage} = S_d = (L_p - L_d)/L_p * 100 \quad (3.3)$$

$$\% \text{ Total linear shrinkage} = S_t = (L_p - L_f)/L_p * 100 \quad (3.4)$$

where: S_d = Linear drying shrinkage (%), S_t = Total linear shrinkage after drying and firing (%), L_d = Dry length of test specimen, L_p = Plastic length of test specimen and L_f = Fired length of test specimen.

The water absorption, bulk density, apparent density and apparent porosity were measured as recommended by the ASTM C373-88 (2002) [60]. The specimen was calculated according to the equation as follows:

$$\% \text{ Water absorption} = (W_3 - W_1)/W_1 * 100 \quad (3.5)$$

$$\% \text{ Apparent porosity} = (W_3 - W_1)/(W_3 - W_2) * 100 \quad (3.6)$$

$$\text{Apparent density} = W_1/(W_1 - W_2) \ (g/cm^3) \quad (3.7)$$

Bulk density = $W_1/(W_3-W_2)$ (g/cm^3) (3.8)

where: W_1 = Weighed at dry state (then boiled in water for 5 h, cooled), W_2 = Weighed a second time in water and W_3 = Weighed again at the saturated wet state in air.

3.3.9 Measurement of mechanical properties

3.3.9.1 Compressive strength test of fired brick specimens

The compressive strength of specimens was measured using the standard of ASTM C773-88 (2002) [61].

Compressive strength = C = W/A (kg/cm^2) (3.9)

Compressive strength is important for determining the load bearing capability of a brick where: C = Compressive strength of the specimen (kg/cm^2), W = Maximum load (kg), indicated by testing machine, A = Average of the gross areas of the upper and lower bearing surfaces of the specimen (cm^2).

Need a conclusion for chapter 3

The main goal of experimental procedures of this study is to determine XRF, XRD, Particle size distribution, Microstructure (SEM), TGA/SDTA, COE, Thermal conductivity, Physical and Mechanical properties by using ASTM C372-94, ASTM C177-97, ASTM C373-88, ASTM C773-88, respectively. The properties of the specimens are found.