CHAPTER III

EXPERIMENTAL PROCEDURE

3.1 Process Flow Chart and Experimental Conditions

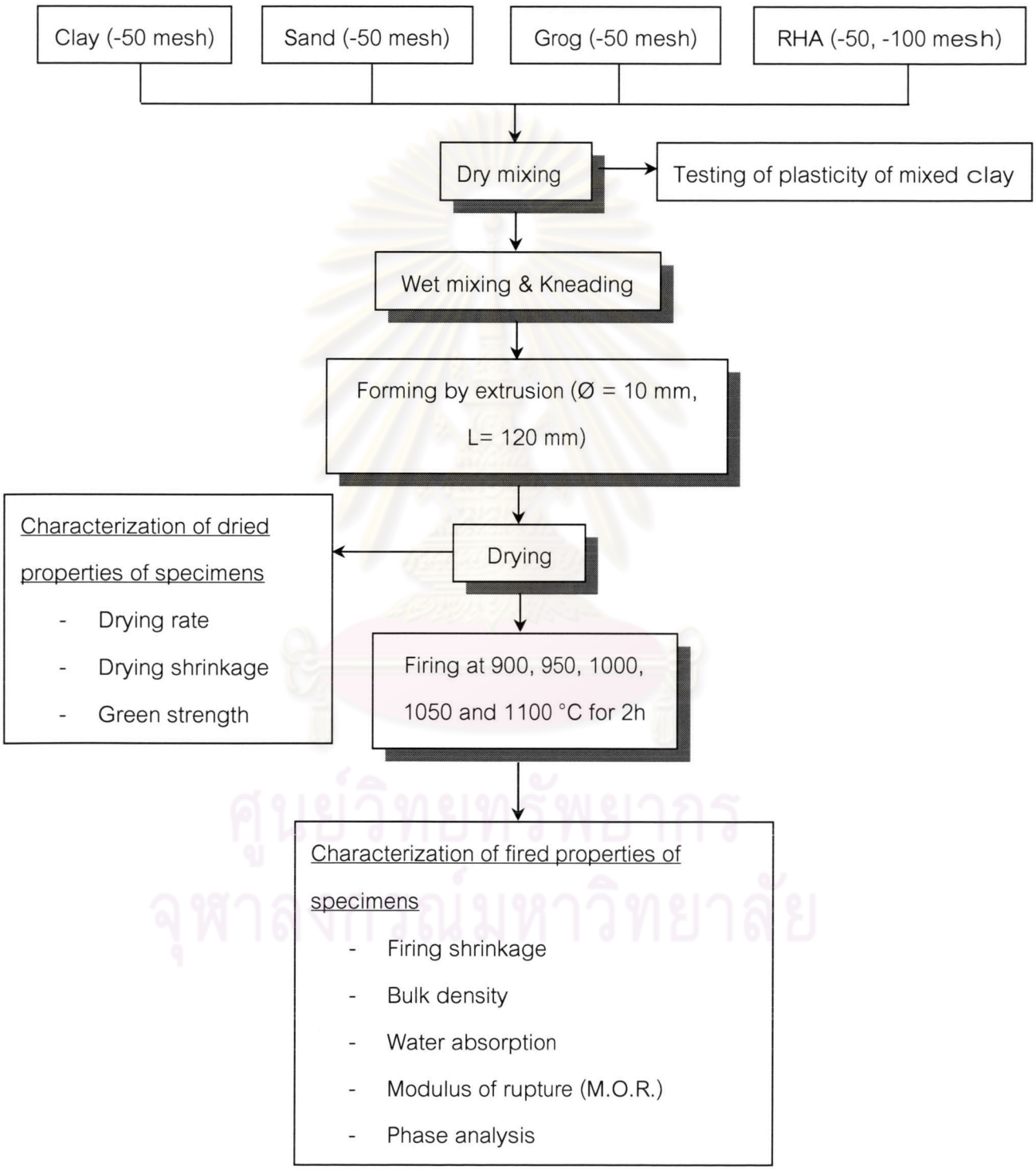


Fig. 3.1 Flow chart of specimen preparation and characterization

The process flow chart of the experiment is shown in Fig. 3.1. Firstly all of the starting materials, clay (-50 mesh), grog(-50 mesh), sand (-50 mesh) and rice husk ash (RHA) (-50, -100 mesh) were mixed in dry condition to get the homogeneous composition. All compositions of mixed clay bodies are shown in Table 3.1. Dry mixed bodies were sampled for the plasticity measurement. Then, the wet mixing was performed by adding about 20 wt% of water. After that the wet mixture were kneaded by hand to get a dough for forming. The hardness of the dough was measured by hardness tester (see Fig. 3.2) in the range of 7 of scale number. If the hardness of the dough was over 7, a small amount of water was added and the dough was kneaded by hand again. On the contrary, if the hardness was less than 7, the dough was dried in open air and mixed again until the hardness become 7 of scale number.

The specimens were extruded into round rods approximately 10 mm in diameter and 120 mm in length. Then, specimens were dried at room temperature for 2 days. After that, the specimens were dried in an oven at 110 °C for 24 h. In this drying process, the drying shrinkage and weight loss of specimens were measured. Dried specimens were fired at 900, 950, 1000, 1050 and 1100 °C for 2 h, at a heating rate of 3 °C/min. Then, the firing shrinkages were measured.

Bulk density, water absorption and apparent porosity of fired specimens were measured by Archimedes' method. Phase composition was analyzed by X-ray diffractometor (XRD). Modulus of rupture (M.O.R.) of green and fired specimens were measured by 3-point bending strength.

Fig. 3.2 Hardness tester for plastic clay body

Table 3.1 Compositions of mixed clay bodies of this experiment

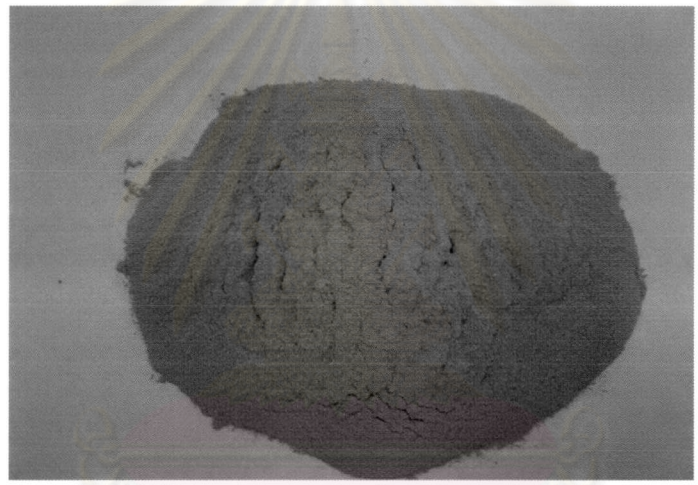
Formulas	Compositions (wt%)				
	Clay(-50 mesh)	Sand(-50 mesh)	Grog(-50 mesh)	RHA (-50 mesh)	RHA(-100 mesh)
NC	100	-	-	-	-
NF	90	5	5	-	-
NS50	90	-	5	5	-
NS100	90	-	5	-	5
3RHA50	87	5	5	3	-
3RHA100	87	5	5	-	3
6RHA50	84	5	5	6	-
6RHA100	84	5	5	-	6
9RHA50	81	5	5	9	-
9RHA100	81	5	5	-	9

3.2 Starting Materials

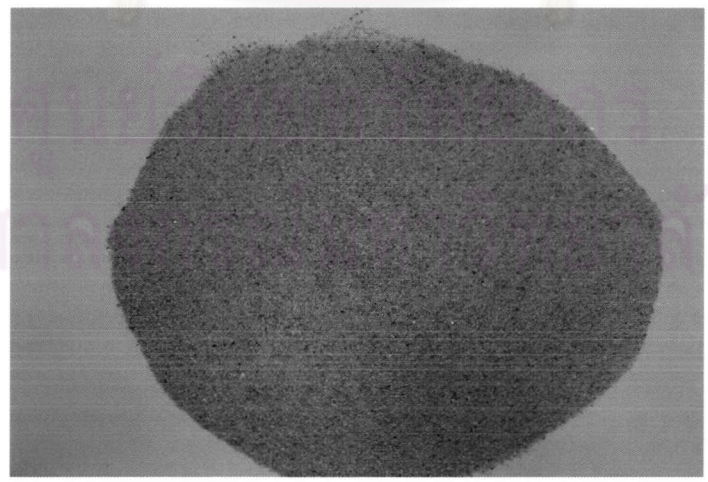
The starting materials used in this experiment are shown in Table 3.2. The appearance of materials are shown in Fig. 3.3.

Table 3.2 Starting materials used in this experiment

Materials	Properties	Sources
Clay	Particle size < 50 mesh	Ratchaburi, Thailand
Sand	Particle size < 50 mesh	Maeklong river, Ratchaburi
Grog	Particle size < 50 mesh	Siamese merchandise Co.,Ltd., Thailand
Rice husk ash (RHA)	Particle size < 50 and 100 mesh	Pathum rice mill and granary Co., Ltd., Thailand

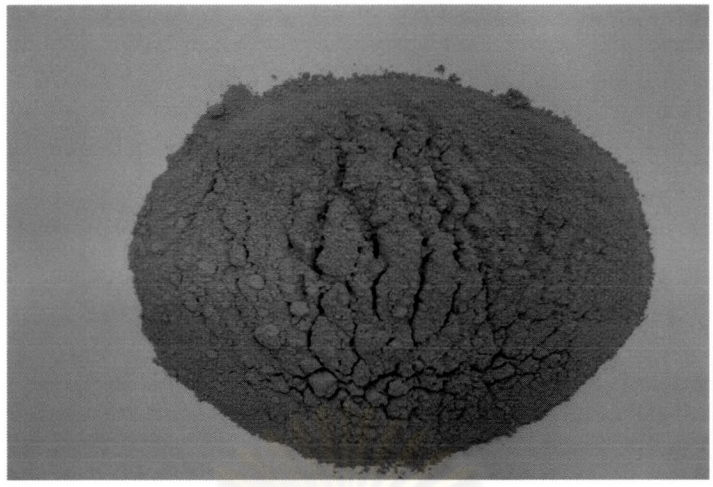


(a) Clay



(b) Sand

Fig. 3.3 Appearance of starting materials



(c) Grog

(d) Rice husk ash

Fig. 3.3 Appearance of starting materials (cont.)

All of starting materials were characterized as follows:

3.2.1 Chemical composition

The chemical composition of starting materials was analyzed by X-ray fluorescence, XRF (ARL 9400, Switzerland) at Siam Research and Development Co.,Ltd.

3.2.2 Crystal Phase

Phase composition of starting materials was analyzed by X-ray diffractometor, XRD (X'pert, Philips) at Siam Research and Development Co.,Ltd., Thailand. The starting materials were crushed and ground to powder under 100 mesh. The qualitative analyses

of the starting materials were determined at the following conditions; target: Cu, voltage 8.5 kVA, 2θ : around 5-70°, and the time step 0.12°/sec.

3.2.3 Microstructure observation

Microstructure and particle size of clay and rice husk ash were observed by scanning electron microscope, SEM (JSM 5410L, JEOL Co.,Ltd.) at Scientific and Technological Research Equipment Center, Chulalongkorn University. The particle size distribution of clay was measured by particle size analyzer in Japan.

3.2.4 Thermal analysis

Thermal analysis of clay and rice husk ash was performed at the heating rate of 10°c/min by using thermal analyzer, which is a combined TG-DTG-DTA unit (Simultaneous thermal analysis, STA 409C/3/E) at Scientific and Technological Research Equipment Center, Chulalongkorn University.

3.3 Property Measurement and Characterizations of Mixed Clay and Fired Specimens

3.3.1 Plasticity of clay and mixed clays

Liquid limit (L.L.), plastic limit (P.L.) and plasticity index (Ip) of clay and mixed clay were measured by Atterberg's limit tester in conformity with ASTM D4318-00 *(18)* at Soil Mechanics Laboratory, Department of Civil Engineering, Faculty of Engineering, Chulalongkorn University. The picture of equipment is shown in Fig. 3.4. The plasticity index of clay was calculated as follows.

$$Ip = L.L. - P.L.$$

Where:

Ip = plasticity index, %

L.L. = liquid limit, %

P.L. = plastic limit, %

Fig. 3.4 Atterberg's limit test equipment

3.3.2 Drying rate and drying shrinkage

Some formulas of mixed clay bodies were selected for drying rate measurement. Drying rates of green body at room temperature were measured by measuring the weight of green specimens every day up to 12 days. Then, specimens were dried at 110 °C for 24 h to measure the total water content in specimens at the starting stage. The water content of specimens was plotted as a function of time. This graph was interpreted as drying rate curve.

Linear drying shrinkage (S_d) of specimens was measured by measuring the length of specimens before drying (L_p) compared with the length of specimens after dried at 110 °C (L_d) according to ASTM C326-82.*(19)* The percentage of linear drying shrinkage was calculated as follows.

$$S_d = (L_p - L_d)/ L_p \times 100$$

where :

S_d = linear drying shrinkage, %

L_p = length of specimen before drying, and

L_d = length of dried specimen

3.3.3 Firing shrinkage

The percentage of linear firing shrinkage was calculated as follows.

$$S_f = (L_d - L_f)/L_d \times 100$$

where :

S_f = linear drying shrinkage, %

L_d = length of dried specimen, and

L_f = length of fired specimen

3.3.4 Bulk density and water absorption

Bulk density and water absorption of fired specimens were measured by Archimedes' method according to ASTM C373-88.*(20)* The detail was omitted.

3.3.5 Modulus of rupture of green body and fired specimens

Moduli of rupture of green body and fired specimens were measured by 3-point bending test in conformity with ASTM C 674-88.*(21)* All tests were performed by using LLOYD 500, Intro enterprise Co., Ltd. The crosshead speed was constant at 0.5 mm/min. The modulus of rupture of each specimen was calculated as follows.

$$M = 8PL/\pi d^3$$

Where:

M = modulus of rupture, MPa

P = load at rupture, N

L = distance between supports, in mm

d = diameter of specimen, in mm

3.3.6 Crystal phase of fired specimens

The qualitative analyses of fired specimens were determined by the same X-ray diffractometor and at the same conditions designated in 3.2.2.