CHAPTER 3
METHODOLOGY

This chapter describes the scientific experimental procedure to contemplate characterization and properties of the ancient Nan pottery. The experiment was divided into three parts: the first part was to studied characterization and physical properties of ancient Ban Bo Suak potteries. The second part was to investigate different clay sources of Ja-Manas kiln (JQA), Doi Fuang Moh kiln (FQB), Dong Poo Ho kiln (PQC) and Nong Tom kiln (NQD) and to select the clay that had similar properties to ancient potteries. The last part was to measure deterioration of SiO_2 content in selected time by autoclave and bury test.

3.1 Characterization and physical properties of ancient Ban Bo Suak potteries

The ancient pottery from Ban Bo Suak was used as the reference sample in this study. Different sources of the ancient kiln sites, such as Ja-Manas kiln (JQA.SH), Doi Fuang Moh kiln (FQB.SH), Dong Poo Ho kiln (PQC.SH) and Nong Tom kiln (NQD.SH) were selected as reference data for investigation. The ancient pottery sherds were cross-section into small pieces, size approximately 5 x 5 x 5 mm. (W x L x H). The chemical compositions of the ancient samples were then measured by XRF technique. Phase structure of the ancient samples was determined by XRD technique. In addition, microstructure was studied by using Scanning Electron Microscope (SEM) and Light Microscope (LM) (Figure 3.1).

3.1.1 Microstructure of ancient pottery

Ancient pottery were cut to size 5 x 5 x 5 mm (W x L x H). They were then dried in an oven pre-set at 110 °C for 24 hours to remove moisture. The samples were mounted on stubs and cross-section surface was coated by gold in vacuum chamber. Finally, microstructure was studied by using Scanning Electron Microscope (SEM, EDX : LEO 1460 VP, Zeiss) and Light Microscope (LM).

3.1.2 Characterization of ancient pottery

The samples were cut to obtain dimensions of 5 x 5 x 5 mm. (W x L x H). They were then dried in an oven pre-set at 110 °C for 24 hours to remove moisture. Chemical composition of the samples was specified by using X-ray Fluorescence (XRF). In the part phase structure, dried samples were placed in the holder and scanned with Cu Kα radiation (XRD: X' Pert Pro MPD, Philips, Netherlands). X-ray was performed on samples using Bruker D8 Advance diffraction. Patterns were recorded from 2-80° with a step interval of 0.04° and counting time of 1 s per step.

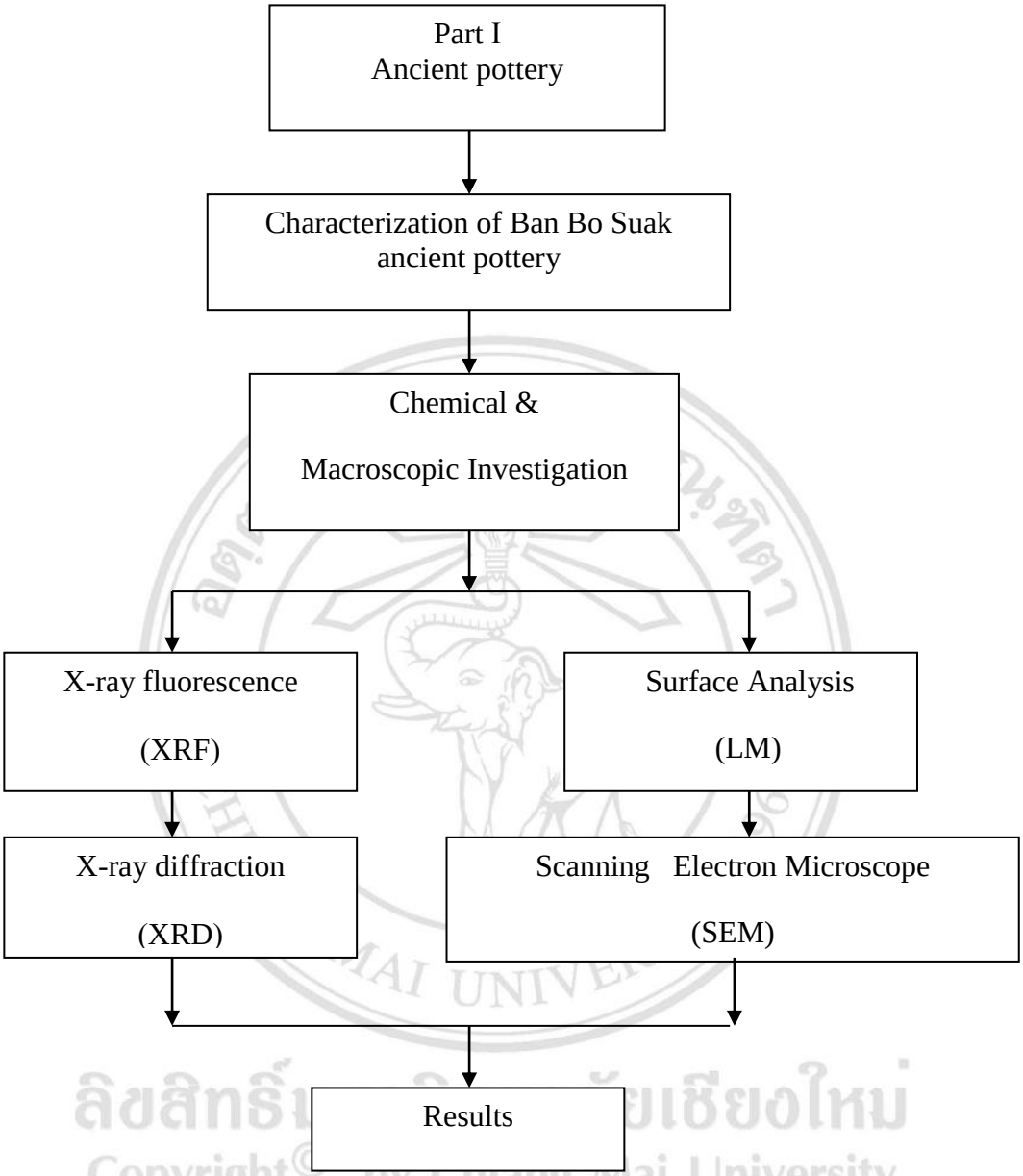

Figure 3.1 Experimental procedures preparation of Ban Bo Suak ancient pottery

3.2 Characterization and properties of different clay sources

Different clay samples were collected near kiln sites from Ja-Manas kiln, Doi Fuang Moh kiln, Dong Poo Ho kiln and Nong Tom kiln at Suak sub-district, Nan Province, Thailand. The samples were crushed and sieved through a 60 mesh stainless steel sieve to get rid of large particle size (>250 μm). All clay sources were then dried in an oven pre-set at 110 °C for 24 hours to eliminate moisture (Figure 3.2).

3.2.1 Particle size distribution analysis

Clay samples were crushed and sieved through 60-325 mesh stainless steel to eliminate large particle size (>250 μm) and residue. Thus, the remaining residue was obtained in this part. After clays were sieved through 60 meshes (250 μm), they were then analyzed the average particle size and the particle size distribution by laser diffraction technique (Mastersizer 2000 Hydro 2000 MU, Malvern Instruments Limited, UK).

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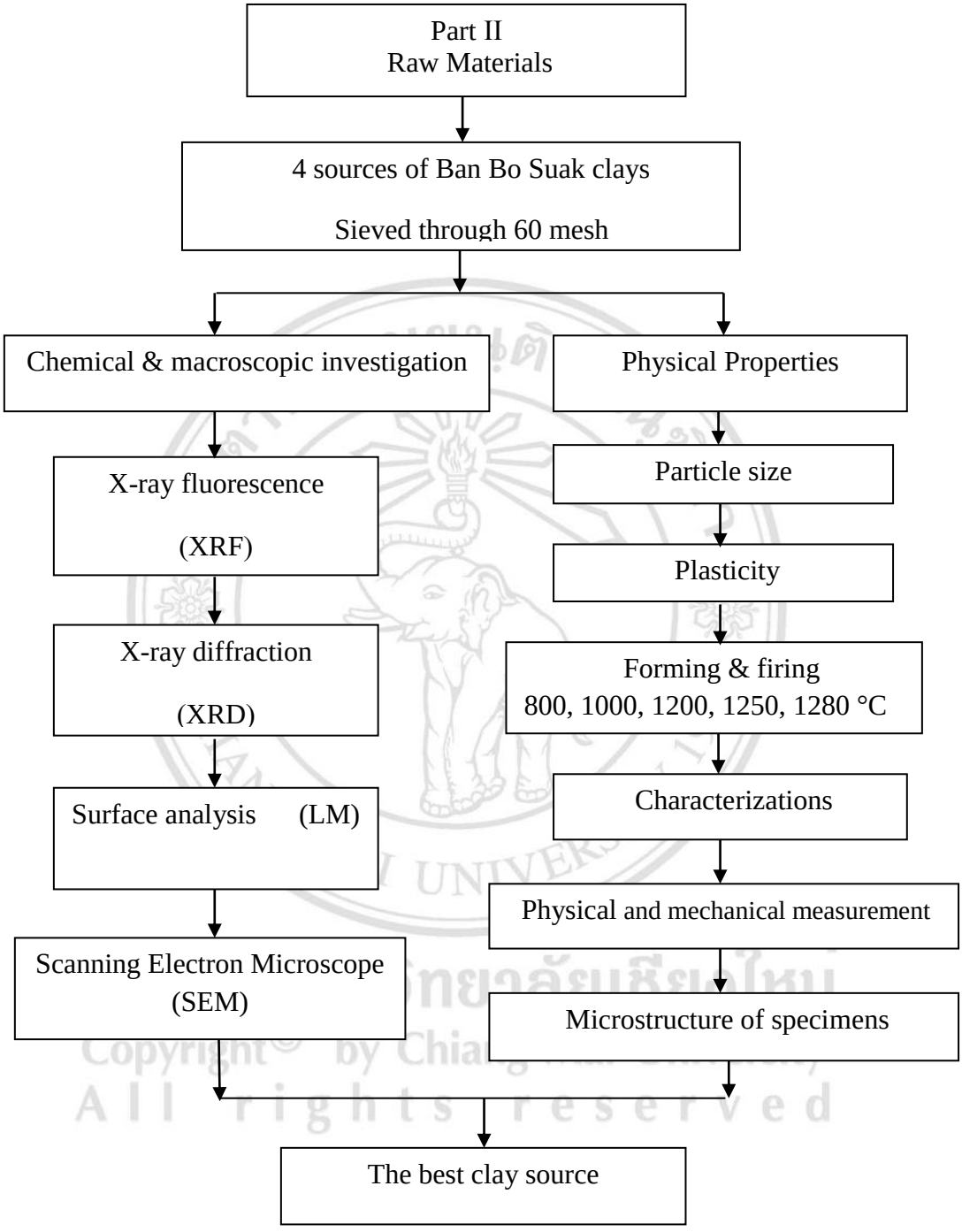

Figure 3.2 Experimental procedures of specimen preparation and characterization.

3.2.2 Physical properties of different clay sources

Different clay powders were studied and modified from sieved method. The clay powders were mixed in water with different water contents to achieve the optimum condition for forming process. These test methods covered the determination of liquid limit, plastic limit, and plasticity index. The liquid limit of a soil containing substantial amounts of organic matter decreased dramatically when the soil was oven-dried before testing. Comparison of the liquid limit of a sample before and after oven-drying could therefore be used as a qualitative measure of organic matter content of a soil. Clays were thoroughly mixed with water and shaped into dimensions of 10 cm x 1 cm and then coiled to check surface cracks (Figure 3.3).

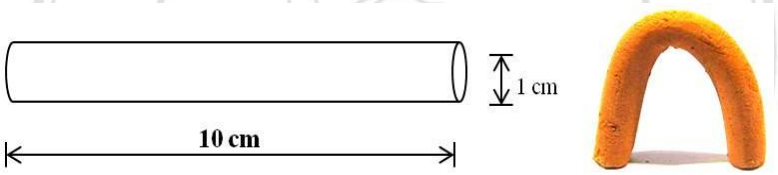

Figure 3.3 Example of plasticity testing.

3.2.3 Specimen preparation

Clays were thoroughly mixed with water and pressed in a plaster mold to obtain rectangular bars with dimensions of 12 cm x 2 cm x 1 cm (Figure 3.4). The bars were left dried at the room temperature for 24 hour to avoid rapid shrinkage. The samples were then dried in an oven pre-set at 110 °C for 24 hours, prior to firing at 800 °C, 1000 °C, 1200 °C, 1250 °C and 1280 °C to investigate basic physical properties.

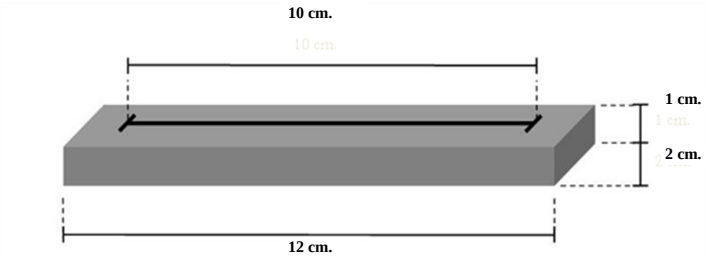

Figure 3.4 Example of plaster mould for forming process.

3.2.4 Water absorption

To understand the densification, water absorption and apparent porosity, samples were tested and results calculated by using the Archimedes method, which was recommended by the ASTM C373-88 (2002) [43]. In this process, the samples were weighed under different conditions. Firstly, the samples were weighed in air atmosphere, dry weigh (W_1). The samples were then immersed in the water and boiled for 5 hours until the water transferred on to the surface pores. Secondly, the samples were weighed in the water (W_2). Lastly, the saturated samples were weighted in air atmosphere (W_3). These values were used to calculate water absorption, apparent porosity, apparent density and bulk density of the samples according to the equation as follow:

$$\% \text{ Water absorption} = (W_3-W_1)/W_1 *100 \quad (3.1)$$

When: W_1 = Weighed at dry state

W_2 = Weighed the second time in water

W_3 = Weighed again at the saturated wet state in air.

3.2.5 Linear shrinkage

The shrinkage values of all samples were obtained by calculating the different length of samples before and after firing at 800 °C , 1000 °C , 1200 °C and 1250 °C respectively, according to the standard of ASTM C326-82 (2002) [42]. The length of samples before and after firing at different temperature was calculated as follow:

$$\% \text{ Linear drying shrinkage} = S_d = (L_p - L_d)/L_p * 100 \quad (3.2)$$

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

When: 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

L_f = Fired length of test specimen.

3.2.6 Softening point

Softening point of the samples was studied from the sagging distance of samples after being fired at 1280 ºC. The clay is mixed with water to normal plasticity and 10 test bars measuring 1x2x12 cm are formed. The test bars should be supported so that the free span equals the distance between the cross-lines of the test bar. If possible cones should be placed next to the test bars to show the temperature. After firing the amount of bending is compared with the cones and results from former tests. When testing new clay the test bar should be placed so that it can be viewed through a spyhole and the approximate temperature at which bending starts is noted [77]. The test bars are placed in the kiln as shown in (Figure 3.5).

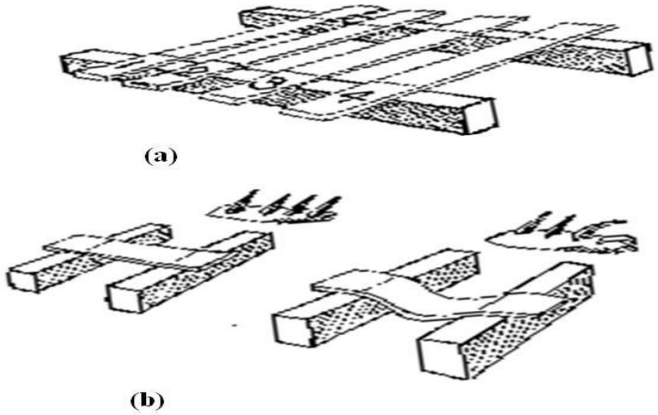

Figure 3.5 Setting of the test bars for firing (a) the span equals the distance between the cross-lines of the test bar (b) bending of the bars is compared with the bending of bars [77].

3.2.7 Characterization of fired samples

Experimental part aimed to examine the characterization of the samples after firing at different temperatures: 800 °C, 1000 °C, 1200 °C and 1250 °C. The chemical composition was studied using X-ray fluorescence. Phase structure of samples was proved by using X-ray diffraction technique and microstructure was studied by the Scanning Electron Microscope.

3.3 Study deterioration using autoclave and bury tests

This experimental section aimed to study deterioration of the ancient Nan pottery by using autoclave and bury methods. The specimens were of dimensions 5x5x1 cm (W x L x H). They were dried in an oven preset at 110 °C for 24 hours. Experimental procedures test deterioration using autoclave and bury tests as shown in (Figure 3.6).

3.3.1 Autoclave tests

The samples were autoclaved in the water with the constant temperature of 126 °C and pressure at 127 Psi. The times were varied as 0, 6, 12 and 18 hours (Figure 3.7). The observed of reactive in the presence of water which percolates through the pores. After that, mass lost of the samples after being autoclaved were weighted to compare with the fired samples before test.

3.3.2 Bury tests

The locations of Ban Bo Suak in the experiment of bury shown in Figure 3.8. They were buried in the cavity with the depth of about 1 m. (W x L x H) for 0, 90, 180 and 360 days (Figure 3.9). After that, they were analyzed for chemical composition and phase transformation by using XRF and XRD, respectively. Microstructures of the samples were analyzed by using SEM. Moreover, mass lost of the samples after being buried were weighted to compare with the fired samples before test.

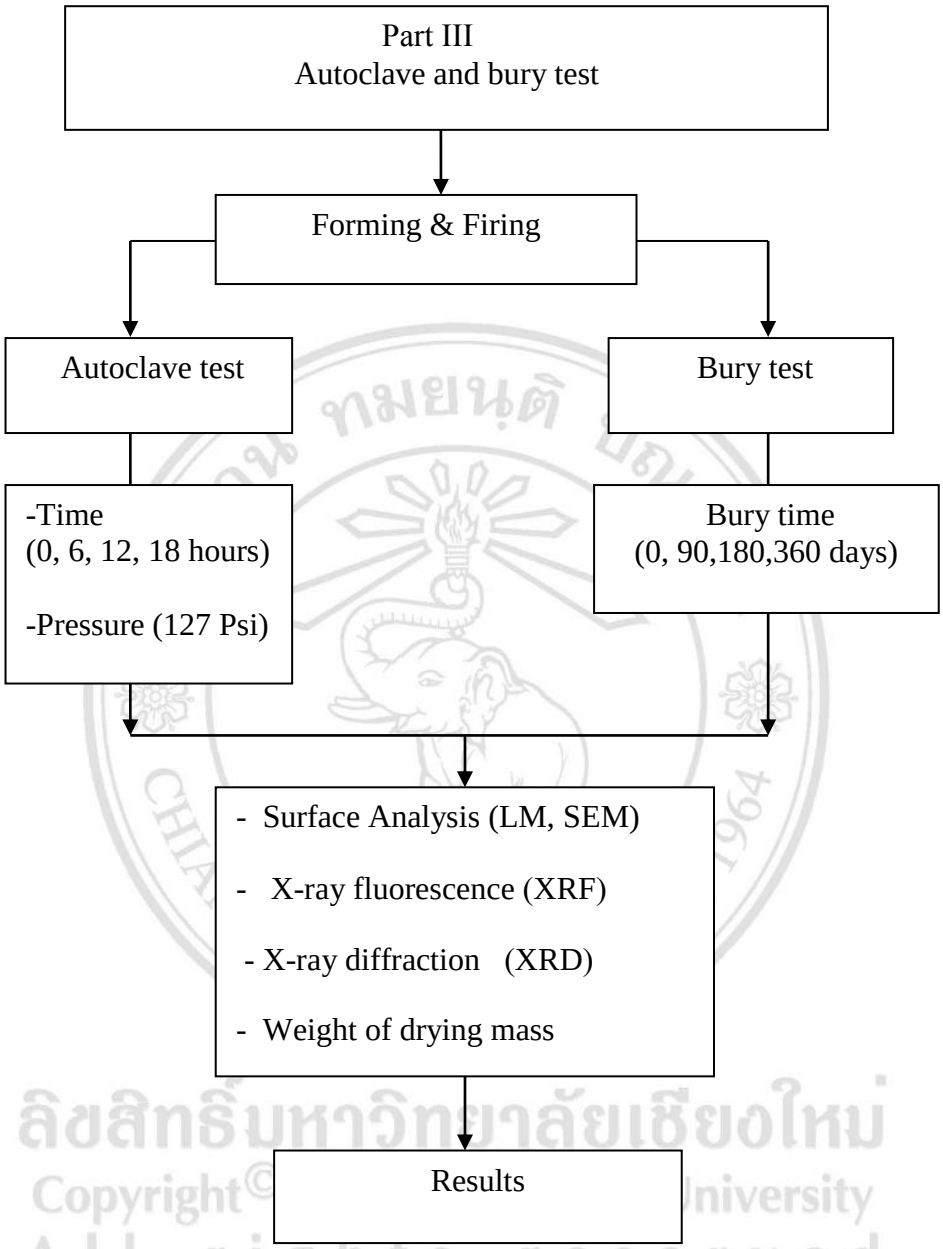

Figure 3.6 Experimental procedures to test deterioration using autoclave and bury tests.

Figure 3.7 Autoclave test

Figure 3.8 Locations of Ban Bo Suak kiln site in bury experiment.

Figure 3.9 Experimental procedures of bury test.