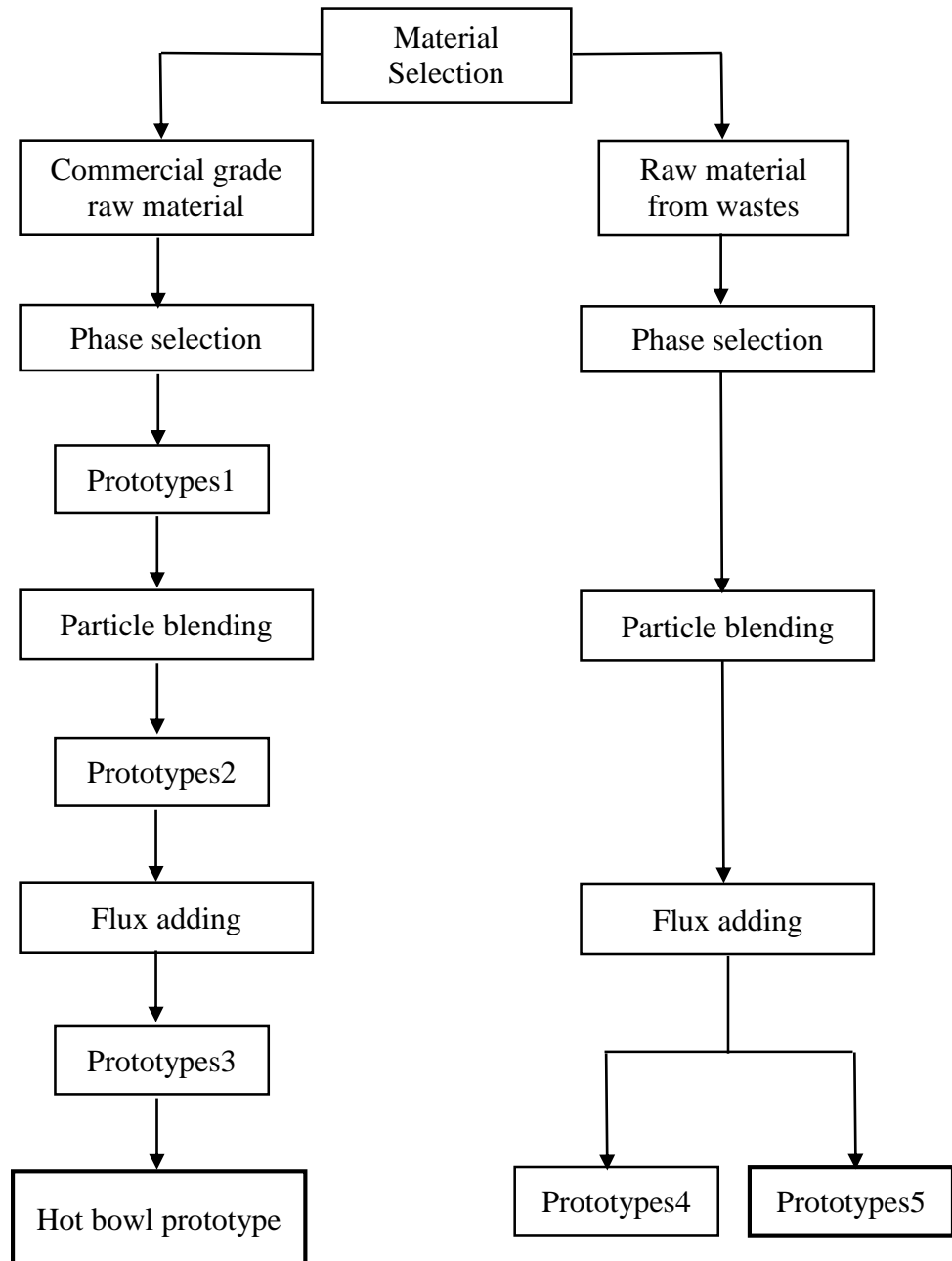


## CHAPTER 4 EXPERIMENTAL PROCEDURE AND RESULT

Clay body prototypes were developed as shown below.



**Figure 4.1** It shows prototype development steps

## 4.1 Raw Materials

Two different types of raw materials used are

1. The commercial grade raw materials.
2. The industry-waste raw materials.

### 4.1.1 The commercial grade raw material

All were supplied by C.K. Ceramics Co. Ltd., Thailand;

1. Alumina; Sumitomo Chemical Co. Ltd., Japan (A-21).
2. Kaolin; Hadsompan, Ranong province, Thailand.
3. Ball clay; Bannasan, Surajthanee province, Thailand.
4. Talc; Kaoka, Lampang province, Thailand.
5. Spodumene; Shenzhen Quanjin Trade Co., Ltd

### 4.1.2 The industry-waste raw material

1. Rice husk ash; Somboon Karnkha rice mill factory, Sampharn, Nakhonpathom province,
2. Sludge cake from aluminum scrap and dross recycling industry (Aw); Bang-bon, Bangkok khun thian, Bangkok

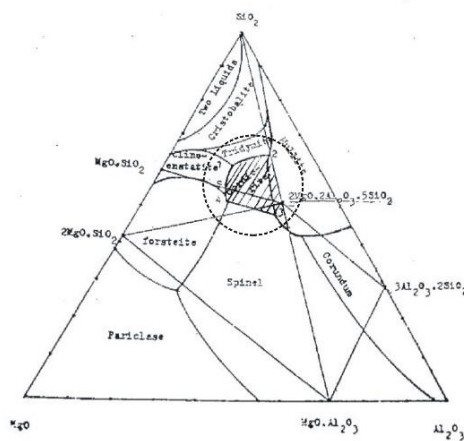
All the industry-wastes raw materials were calcined at 800 °C before blending in the clay body prototype.

## 4.2 Characterization/Testing; instrument

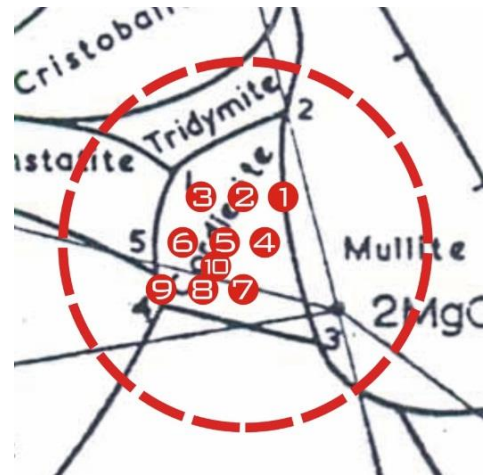
1. Chemical compositions of raw materials from wastes; Wavelength Dispersive type X-ray Fluorescence Spectroscopy (WD-XRF using Rh radiation in a Bruker® S4 Pioneer).
2. Surface area comparison of clays; Methylene Blue Index in accordance with ASTM C837-09
3. Particle sizes and surface areas of raw materials; BETTERSIZ 9300
4. Crystal structures and their relative amount; X-ray diffraction using  $\text{CuK}\alpha$  radiation with a Bruker® D8 Advance Diffractometer.
5. Coefficient of thermal expansion(COE); Anter Unitherm dilatometer 1161 ranging between 25-1000 °C at a heating rate of 3°C/min. Porosity of all samples were observed after HF 0.1 % surface etching.
6. Point analysis; FEI NanoSEM450 Field Emission Scanning Electron Microscope,Energy Dispersive Spectrometry
7. Thermal shock resistance; JIS S 2400
8. Linear shrinkage; ASTM C326-03 (Reapproved 1999)
9. water absorption; ASTM C373-88 (Reapproved 1999)

## 4.3 Experimental procedures

The Phase diagram (Figure 4.2a) was used to identify the compositions that could form the cordierite phase (the circled area in Figure 4.2a was enlarged as shown in Figure 4.2b). The ranges of each component are 10-20% of MgO, 25-35% of Al<sub>2</sub>O<sub>3</sub>, and 50-60% SiO<sub>2</sub>. 10 points in the Figure 4.2b were the chosen compositions for this study.



(a)



(b)

**Figure 4.2** (a) Phase Diagram of MgO - Al<sub>2</sub>O<sub>3</sub> - SiO<sub>2</sub> [25] and (b) Ten points represented the chosen compositions.

### 4.3.1 Prototype1

Compositions, which give cordierite phase, were selected using MgO - Al<sub>2</sub>O<sub>3</sub> - SiO<sub>2</sub> phase diagram. The chemical compositions were shown in Table 4.1 and the batch compositions of the samples were shown in Table 4.2

**Table 4.1** The chemical compositions of Prototype1 clay body in weight %

Sample no.	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	MgO
1	44.00	38.00	18.00
2	48.00	34.00	18.00
3	52.00	30.00	18.00
4	48.00	38.00	14.00
5	52.00	34.00	14.00
6	56.00	30.00	14.00
7	52.00	38.00	10.00
8	56.00	34.00	10.00
9	60.00	30.00	10.00
10	51.40	34.90	13.70

**Table 4.2** The batch compositions of Prototype1 clay body 1-10 in weight %

Sample no.	Talc	Kaolin	Ball clay	Alumina
1	54.01	8.17	8.17	29.64
2	53.41	12.12	12.12	22.36
3	52.81	15.98	15.98	15.23
4	40.86	19.87	19.87	19.41
5	40.41	23.58	23.58	12.43
6	39.97	27.21	27.21	5.60
7	28.41	30.94	30.94	9.71
8	28.10	34.44	34.44	3.02
9	28.16	33.82	33.82	4.20
10.	41.24	20.86	20.86	17.03

### 4.3.2 Prototypes2

Prototypes2 were the selected Prototype1 and ground within the period of 1 to 7 hours as shown in Table 4.3.

**Table 4.3** The Prototype2 clay body of different grinding times

Sample no.	s7-1	s7-2	s7-3	s7-4	s7-5	s7-6	s7-7	s7-8
Grinding Time	0 Hr.	1 Hr.	2 Hr	3 Hr.	4 Hr.	5 Hr.	6 Hr.	7 Hr.

### 4.3.3 Prototype3

Prototypes3 were the selected Prototype2 in which Flux (Spodumene : sodium feldspar ratio of 5:1) of 10, 15, 20, 25, 30 and 40 parts per hundred weight were added as shown in Table 4.4.

**Table 4.4** The Prototype3 clay body of different flux compositions in parts per hundred weight

Sample no.	s7-6	s7-6-10	s7-6-15	s7-6-20	s7-6-25	s7-6-30	s7-6-40
Flux	0 %	10%	15%	20%	25%	30%	40%

### 4.3.4 Prototype4

Cordierite composition, #10 in Figure 4.2 (b), was used as Prototype4. Three different clay bodies were prepared using SiO<sub>2</sub> from different sources as shown in Table 4.5. Each contained SiO<sub>2</sub> as follows:

- Quartz, SiO<sub>2</sub> 99.5% by weight
- Rice husk, SiO<sub>2</sub> 66% by weight
- Kaolin, SiO<sub>2</sub> 44.85% by weight
- Ball clay, SiO<sub>2</sub> 51.5% by weight

**Table 4.5** The batch composition of clay bodies with cordierite composition in weight%

Sample no.	Quartz	Rice husk ash	Kaolin	Ball clay	Talc	Alumina
Q	24.00	-	-	-	41.10	34.90
R	-	24.00	-	-	41.10	34.90
C	-	-	20.86	20.86	41.24	17.03

\*\* Q; batch composition with quartz, R; batch composition with rice husk ash, C; batch composition with commercial clay.

### 4.3.5 Prototype5

Cordierite composition, #10 in Figure 4.2 (b), was used as Prototype5. Two different clay bodies were prepared using Al<sub>2</sub>O<sub>3</sub> from different sources as shown in Table 4.6. Each was as follows:

- Sludge cake from aluminum scrap and dross recycling industry, Al<sub>2</sub>O<sub>3</sub> 78.8% by weight
- Commercial grade Al<sub>2</sub>O<sub>3</sub>

**Table 4.6** The batch composition of clay bodies with cordierite composition in weight%

Sample no.	Aw	Alumina	Kaolin	Ball clay	Talcum
W	21.31	-	20.74	20.74	37.22
C	-	17.03	20.86	20.86	41.24

\*\*W; batch composition with sludge cake from aluminum scrap and dross recycling industry (Aw), C; batch composition with commercial raw material.

## 4.4 The sample Preparation

### 4.4.1 Prototype1

1. Each composition in Table 4.2 was mixed with water. The ratio of the weight of each batch composition to that of water was 50:50. The mixture was ground in ball mill for 1 hour. All specimens were slip casted and fired at the heating rate of 80°C/Hr. to the temperatures of 1250°C and soaked at the highest temperature for 1 hour. Specimens with the dimension of 5 cm x 7.5 cm x 0.7 cm were used in thermal shock resistance and water absorption tests. Specimens with the dimension 2.5 cm x 12 cm x 1 cm were used in linear shrinkage tests
2. The clay body which gave the best results or Prototype1 was selected. Different blending times of 0-7 hours were applied to the Prototype1. Forming and testing of the specimens were the same as those mentioned in #1.

### 4.4.2 Prototype2

1. The Prototype1 which gave the best results was selected as Prototype2. Flux of 10-40 parts per hundred by weight were added to the mixture. Forming and testing of the specimens were the same as those mentioned in #1.

### 4.4.3 Prototype3

1. The Prototype2 which gave the best results was selected as Prototype3. The clay body was slip casted into hot bowl of 8-inch diameter.

### 4.4.4 Prototype4

1. The batch compositions made use of rice husk ash shown in Table 4.5 was ground in ball mill for 1 hour. The clay body was pressed using hydraulic compression and fired at the heating rate of 80°C/Hr. to the temperatures of 1250-1300°C and soaked at the highest temperature for 1 hour. Specimens with the dimension of 5 cm x 7.5 cm x 0.7 cm were used in thermal shock resistance and water absorption tests. Specimens with the dimension 2.5 cm x 12 cm x 1 cm were used in linear shrinkage test.

### 4.4.5 Prototype5

1. The batch compositions made use of sludge cake from aluminum scrap and dross recycling industry shown in Table 4.6 was ground for 5 hours. The clay body was press using hydraulic compression and fired at the heating rate of 80°C/Hr. to the temperatures of 1200-1325°C and soaked at the highest temperature for 1 hour. Forming and testing were the same as those mentioned in #5.