CHAPTER 3 PRODUCT DESIGN AND PROTOTYPE CONSTRUCTION

Implementation of research and development of ovenware ceramics clay body for Thai SMEs involved the followings:

3.1 Conceptual Design
3.1.1 Identification Customer Needs
3.1.2 Crystal Structure
3.1.3 Product Specification
3.2 Materials Selection

3.1 Conceptual Design

In order to provide clay body that could meet the requirement of Thai SMEs for ovenware ceramics manufacturing, technical information of ovenware ceramics in the market is helpful in identifying product specification which leads to materials selection for the clay body.

3.1.1 Identification Customer Needs

It is clearly stated in chapter 2 that Thai SMEs need clay body for ovenware ceramics which could be manufactured employing the existing technology. To them, being able to produce comparable ovenware ceramics would be the first step to go for other alternatives in the future. By so doing, seven different directed fire tableware ceramics were selected from market for reverse engineering expecting to understand the structure ceramics from China, Japan, France, and Thailand. Product characteristics and properties are shown in Tables 3.1-3.6 and Figures 3.1-3.3.

Physical appearances of the products were measured and observed and presented in Table 3.1.

Table 3.1 Ovenware Samples from the market (Source :2006, Year in A.D.)

Sample	**Physical appearance (Shape)**	**Diameter/Volume (cm.)/(cc.)**	**Thickness (mm.)**	**Clay body (color)**	**Glaze (color)**
Japanese.1 (Direct fire)		26.5/850	5	white	White Matt
China.1 (Direct fire)		18/800	5	Light brown	Light brown

Table 3.1 Ovenware Samples from the market (Source :2006, Year in A.D.) (Ext.)

Sample	**Physical appearance (Shape)**	**Diameter/Volume (cm.)/(cc.)**	**Thickness (mm.)**	**Clay body (color)**	**Glaze (color)**
France.1 (Direct fire)		18/1200	5	cream	Red/orange
Thailand.1 (Direct fire)		13.5/850	5	cream	Light brown transparency
Thailand.2 (Direct fire)		11/525	11	cream	brown
Thailand.3 (Direct fire)		12/200	11	cream	brown
Thailand.4 (Direct fire)		15/525	15	cream	brown

Table 3.2 The summary of physical properties of all Products were selected from the market

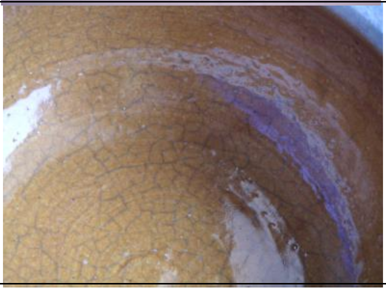
Sample	**Shock Resistance (°C)**	**Water Absorption (%)**	**After test**	**Crystalline phase**	**COE ($x10^{-6}$ $^{\circ}C^{-1}$)**
Japanese1	290 ± 5.5	11.79		Mullite	1.524
China1	210 ± 5.5	13.91		Mullite, Cordierite	4.917
France1	150 ± 5.5	0.54		N/A	7.754
Thailand1	190 ± 5.5	0.35		Mullite	6.004
Thailand2	170 ± 5.5	8.32		N/A	4.771

Table 3.2 The summary of physical properties of all Products were selected from the market (Ext.)

Sample	Shock Resistance (°C)	Water Absorption (%)	After test	Crystalline phase	COE (x10^{-6} °C^{-1})
Thailand3	170 ± 5.5	5.96		N/A	4.868
Thailand4	270 ± 5.5	7.37		N/A	4.898

Thermal shock resistance of the products from Table 3.2 depicted that only the Japanese ceramics can withstand shock resistance temperature as high as 290°C. According to JIS S 2400 (Table 3.3) the Japanese ceramics thus was categorized as direct fire high heat- resistance class. Thermal shock resistance temperatures of the rest were below 290 °C but greater than150 °C and were categorized as direct fire class.

Table 3.3 Thermal- shock resistance according to standard JIS S 2400 [10]

Class	Test temperature difference °C(t_2-t_1)
Direct fire (high heat Resistance)	280 to 290
Direct fire	150 to 160
Oven (max. 300 °C)	150 to 160
Oven (max. 200 °C)	120 to 128

Three samples with the highest thermal shock resistance temperature, the Japanese1 (J1), China1(C1), and Thailand1(T1), were selected for further analysis. Even though Thailand1 seemed to have lower thermal shock resistance than that of Thailand4, it was selected because it was the thinnest product of the group of Thailand which could stand the high temperature test.

3.1.2 Crystal Structure

Figure 3.1 shows x-ray diffraction patterns of J1, C1, and T1. The phases of the samples were cordierite (C), Corundum (Co), Spinel (Sp), and Cristobalite (Cr).

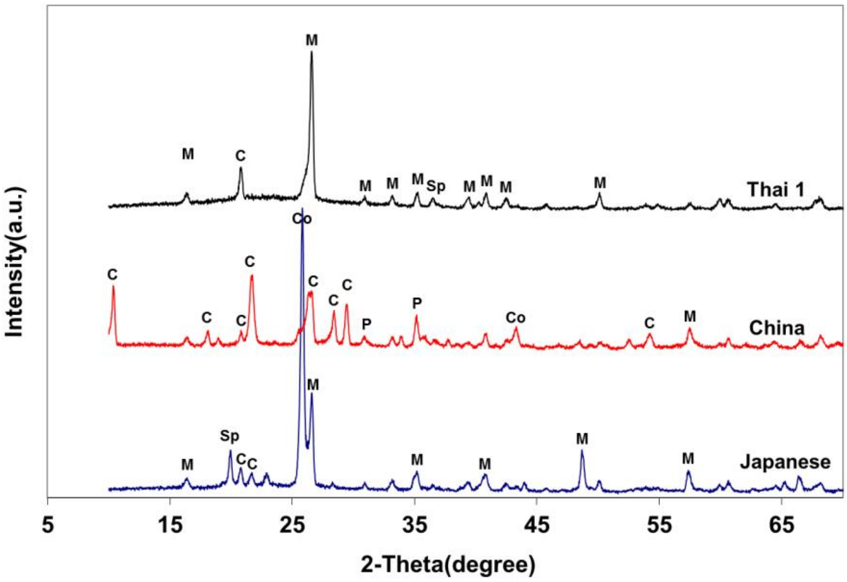


Figure 3.1 It showed XRD patterns of selected samples from the market

J1 and T1 had Mullite as major phase even though the patterns were different, whereas, C1 had cordierite composites as major phase. The findings were confirmed by the result of chemical compositions shown in Table 3.4. The chemical compositions of the three selected samples were evaluated by Wavelength Dispersive type X-ray Fluorescence Spectroscopy (WD-XRF using Rh radiation in a Bruker® S4 Pioneer). High compositions of Al_2O_3 and SiO_2 of mullite were observed in J1 and T1 whereas the composition of Al_2O_3, SiO_2 and MgO of cordierite was found for sample C1.

Table 3.4 The mineral composition at different positions

	SiO_2	Al_2O_3	K_2O	Fe_2O_3	Na_2O	TiO_2	CaO	MgO	P_2O_3	ZnO	SO_3	CuO
J1	57.5	30.5	1.06	0.92	0.64	0.53	0.19	0.11	0.06	0.06	0.04	0.03
C1	49.6	29.2	1.54	1.79	0.49	1.26	1.34	7.71	0.21	0.09	0.07	0.04
T1	61.9	21.3	4.17	1.13	1.35	0.19	0.53	0.45	0.07	0.08	0.15	0.05

Compositions at different positions of samples were observed using SEM-EDS. Figure 3.2 showed the randomly selected positions of sample J1. The compositions of all positions were shown in Table 3.5 Positions P.1, P.2, P.4, P.5 and P.6 had strong signals of Al_2O_3 and SiO_2 which implied the composition of mullite. The amount of Al_2O_3 and SiO_2 were about the same for all positions. Only position P.3 had the high percentage of Al_2O_3 which is the composition of alumina or corundum.

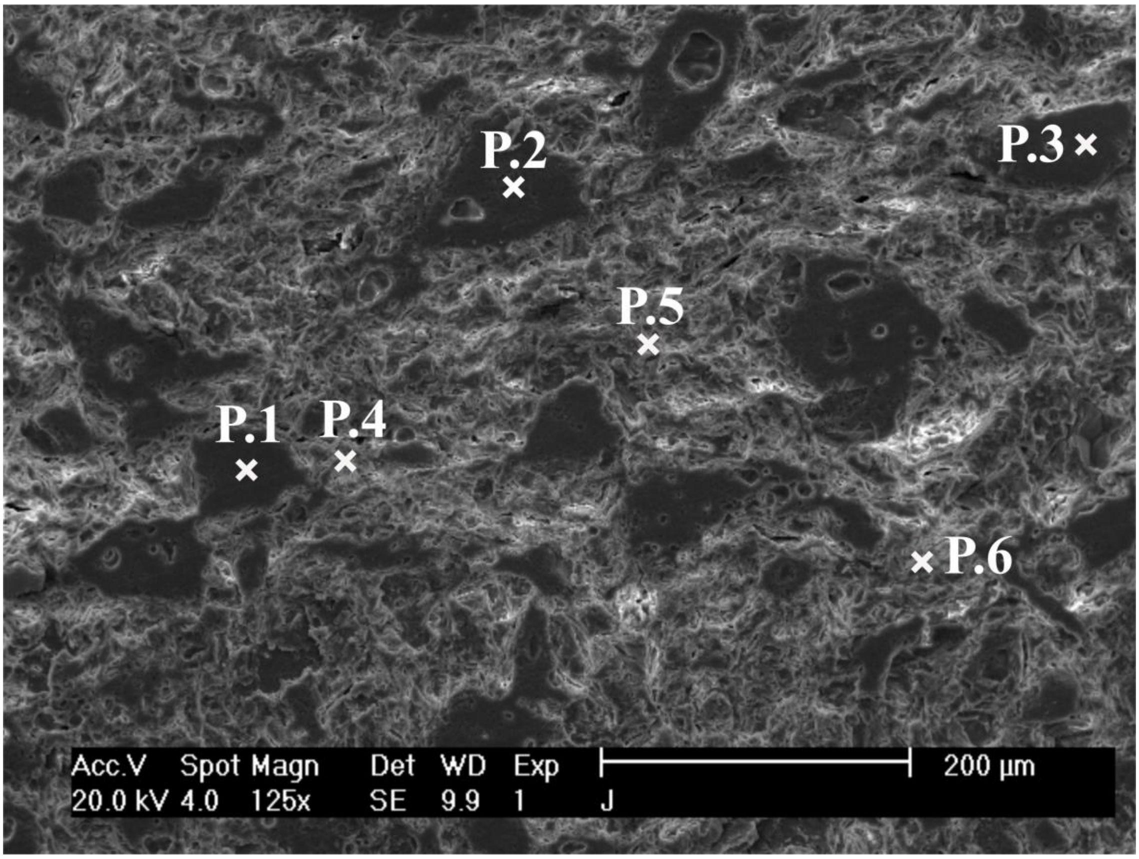


Figure 3.2 SEM micrographs of samples J1. (The etching surfaces)

Table 3.5 The mineral composition at different positions

Element	P.1 (Wt %)	P.2 (Wt %)	P.3 (Wt %)	P.4 (Wt %)	P.5 (Wt %)	P.6 (Wt %)
Na_2O	5.35	4.93	11.09	4.35	5.06	-
Al_2O_3	19.25	18.93	88.91	34.98	22.83	39.71
SiO_2	71.14	68.51	-	56.06	70.25	51.94
K_2O	4.25	5.65	-	0.93	1.10	3.66
CaO	-	0.86	-	-	-	-
Fe_2O_3	-	1.11	-	1.34	-	3.11
TiO_2	-	-	-	2.34	0.76	1.57
Total	100.00	100.00	100.00	100.00	100.00	100.00

Figure 3.3 showed the randomly selected positions of sample C1. The compositions of all positions were shown in Table 3.6. Positions P.6 and P.7 had strong signals of Al_2O_3, SiO_2 and MgO which implied the composition of cordierite. Positions P.1 and P.5 had stong signals of Al_2O_3 and SiO_2 which implied the composition of Mullite. Only point P.2 showed the high percentage of Al_2O_3 which is the composition of alumina or corundum.

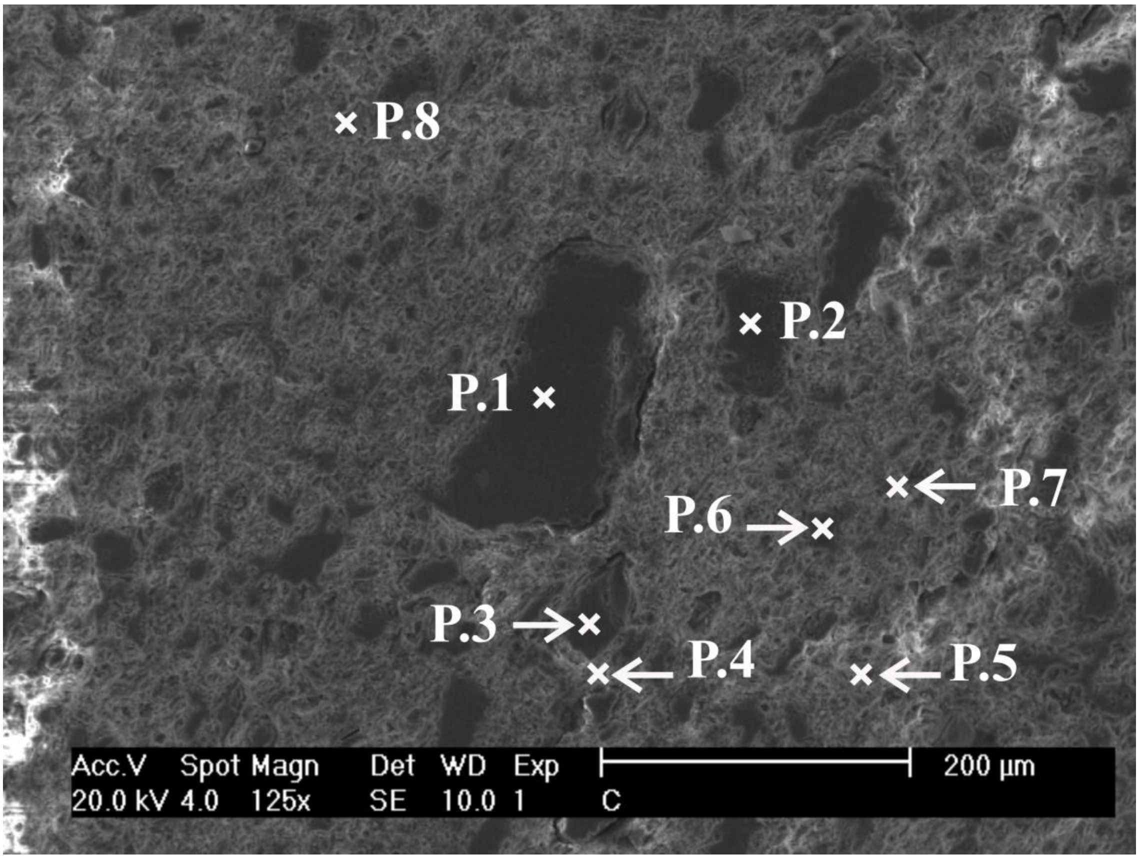


Figure 3.3 SEM micrographs of samples C1. (The etching surfaces)

Table 3.6 The mineral composition at different positions

Element	**P.1 (Wt %)**	**P.2 (Wt %)**	**P.3 (Wt %)**	**P.4 (Wt %)**	**P.5 (Wt %)**	**P.6 (Wt %)**	**P.7 (Wt %)**	**P.8 (Wt %)**
Na_2O	-	-	-	-	2.02	1.66	-	-
MgO	-	2.81	2.32	39.08	2.95	10.13	16.28	34.86
Al_2O_3	55.09	93.09	43.77	2.36	42.64	27.54	36.02	17.44
SiO_2	42.30	2.34	48.89	58.56	47.03	57.57	44.942	24.71
K_2O	1.52	-	1.97	-	2.64	0.82	0.98	-
Fe_2O_3	-	1.10	1.28	0	1.96	1.22	1.18	18.33
TiO_2	1.09	0.66	1.76	0	0.76	1.05	0.61	4.65
Total	100	100	100	100	100	100	100	100

The results observed from all techniques applied can be deduced that the major phase of J1 was mullite and the major phase of C1 was cordierite.

3.1.3 Product Specification

From the front end study and the data obtained above, clay body is needed to meet the following requirements of ovenware ceramics manufacturing:

1. White color
2. Easy to clean.
3. Water absorption is below 1%[26]
4. COE is below 4-5 x 10^{-6} $°C^{-1}$

5. Withstanding sharp change of temperature from 150°C in the oven into cold water immediately JIS S 2400 [10]
6. Passing TIS 601-2546 Ceramic Containers for Food: Earthen ware [26]
7. Sintering at the lowest possible temperature (environmental concern)
8. Forming and firing can be done by middle and small-sized entrepreneurs

3.2 Materials Selection

The reverse engineering informed that mullite is the phase that gives high heat resistance ceramics. However, stated so in Chapter 2 that mullite phase is found at sintering temperature of about 1400°C and is fully achieved at 1600°C [25,30]. Another phase that shows potential to withstand high heat is cordierite. Generally, cordierite composites were synthesized at sintering temperatures between 1250-1350 °C [25,30]. In order to obtain cordierite phase at the lower side of synthesis temperature, more work has to be done in the processing stage, i.e., attaining optimum particle sizes of raw materials [31,32].

Cordierite (2MgO.2Al2O3.5SiO2) comprises magnesium oxide, alumina, and silica.[4] Raw materials can be obtained from many different sources. Some of the starting raw materials reported can be classified as follow:

1.) raw material-laboratory grade
2.) raw material-commercial grade
3.) raw material from waste

Raw material from waste is intriguing. The use of waste causes clay body be environmentally friendly in addition to the sintering at the lowest temperature possible. Moreover, value added waste is also gained.

Alumina and silica are the major proportions in cordierite. Moreover, alumina is the only raw material in clay body which has to be imported. Alternative sources from waste for alumina and silica are thus beneficial. In this study, rice husk ash was used for silica source and sludge cake from aluminum scrap and dross industry for alumina source.

Fluxing agent is also play role in ceramics processing-property relationships. In terms of COE, spodumene has been known to do the adjustment well. Hence, spodumene was selected as fluxing agent in this study [7,19,33,34].