

### Chapter 3

#### Results and Discussion

The relationships between particle size and various specific properties of the raw and fired material were established.

#### 3.1 Sample selection

For this research representative white clay and pottery stone from Kao Pangka in Lampang were selected from the main production centers of white clay and pottery stone. A representative sample was taken from the deposit place and was thoroughly evaluated. This sample was graded in size range  $< 63$  microns,  $< 40$  microns and  $< 10$  microns, so that any differences due to particle size, including ceramic-technological behavior, could be evaluated.

#### 3.2 Sample preparation

A representative sample of Lampang white clay was separated into three samples by particle size. Wet sieve screens and an air cyclone were used to make samples. The samples with particle size  $< 63$  microns,  $< 40$  microns were made using wet sieve screens. The sample with particles  $< 10$  microns was made using an air cyclone (the type Turbo classifier TC-15M). After separation the properties of the dry clay were measured. The results are shown in Table 3.1 :

Table 3.1: Fractions of Lampang white clay with various particle sizes

Sieved with 63 micron		Sieved with 40 micron		Air separate with 10 micron	
Retained	Passed	Retained	Passed	Retained	Passed
+ 63	- 63	+ 40	- 40	+ 10	- 10
15 %	85 %	30 %	70 %	63 %	37 %
100 %		100 %		100 %	

It is of interest that about 70 % of the clay has particles < 40 microns. The fraction < 40 microns is of special interest because of desirable industrial properties.

### 3.3 Characterization

#### 3.3.1 Grain size<sup>5,6</sup>

The first investigation of the raw material was to measure grain size distribution. The results of grain size distribution for the different fractions of Lampang clay after sieving is shown in Figure 3.1.

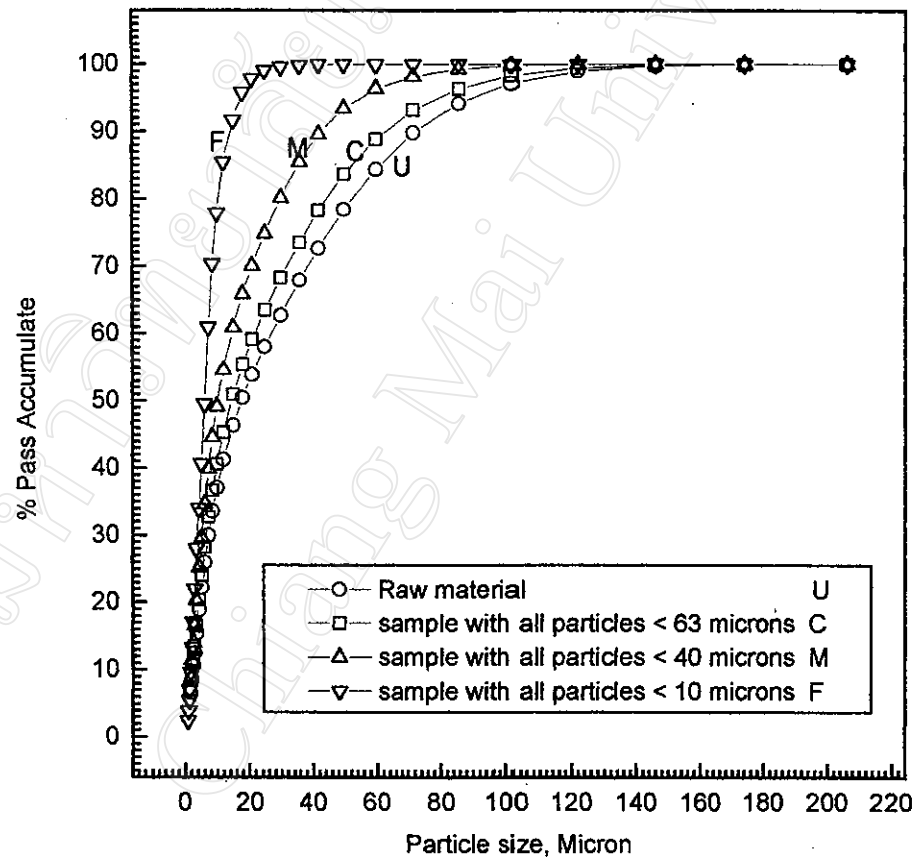


Figure 3.1: Particle size distribution of Lampang white clay

Grain size was investigated with standard sieves. It was found that most of the representative raw clay is composed of coarse and medium grain size that can be recovered with a sieve screen of 40 microns. The grain size larger than 40 microns contain mostly quartz sand and feldspar<sup>30</sup>. The material with grain size smaller than 40

microns was composed of fine quartz sand, illite and kaolinite, etc. Grain size was also investigated by means of a laser instrument (Laser Particle Size Analyzer, type Sympatec HELOS H0392) shown by Figure 3.1. The particle size distribution of each of the three samples was determined.

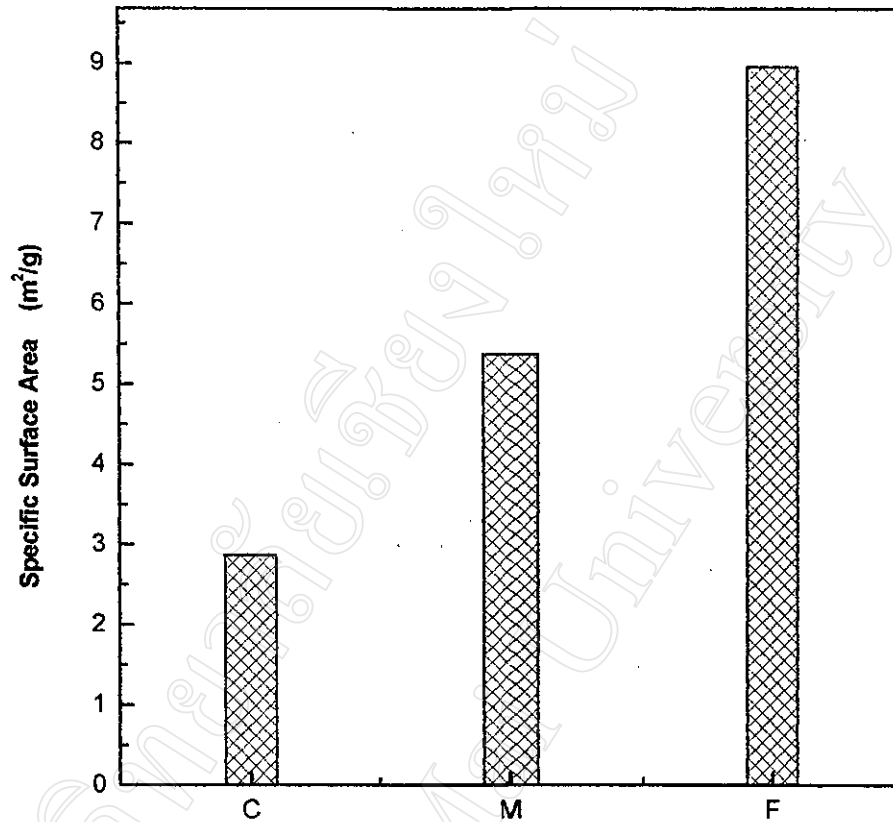
From the amount and particle size distribution of each sample it is seen that the M sample (particles < 40 Microns) is the sample of greatest interest for commercial production.

### 3.3.2 Specific surface area<sup>5,11-12</sup>

The results of specific surface area for each of the three samples is shown in Table 3.2 and Figure 3.2.

Table 3.2: Specific surface area of Lampang white clay after sieving

	Courses sample, C < 63 microns	Medium sample, M <40 microns	Finest sample, F <10 microns
Specific surface area, m <sup>2</sup> /g	2.87	5.38	8.96



**Figure 3.2: Specific surface area for each of three samples of Lampang white clay**

The differences of specific surface area shown in Table 3.2 and Figure 3.2 respectively are correlated with the differences in particle size distribution in the clay mineral, especially for the finest sample (particles finer than 10 microns). Larger amounts of illite found in this sample.

### 3.3.3 Chemical composition<sup>5</sup>

Chemical composition is important because of its effect on ceramic behavior. The chemical composition for the three samples of Lampang clay are shown in Table 3.3 and Figure 3.3.

Table 3.3 : Chemical composition of Lampang white clay in percentage

	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	CaO	MgO	K <sub>2</sub> O	Na <sub>2</sub> O	L.O.I.	Σ
Raw material	76.62	15.30	0.60	0.06	0.06	0.14	4.20	0.67	2.75	100.0
< 63 micron	75.96	15.46	0.55	0.08	0.04	0.11	4.36	0.54	2.90	100.0
< 40 micron	69.52	19.69	0.69	0.11	0.07	0.16	5.34	0.79	3.63	100.0
< 10 micron	52.01	31.34	1.09	0.11	0.06	0.27	8.23	1.02	5.87	100.0

L.O.I. = Loss on Ignition

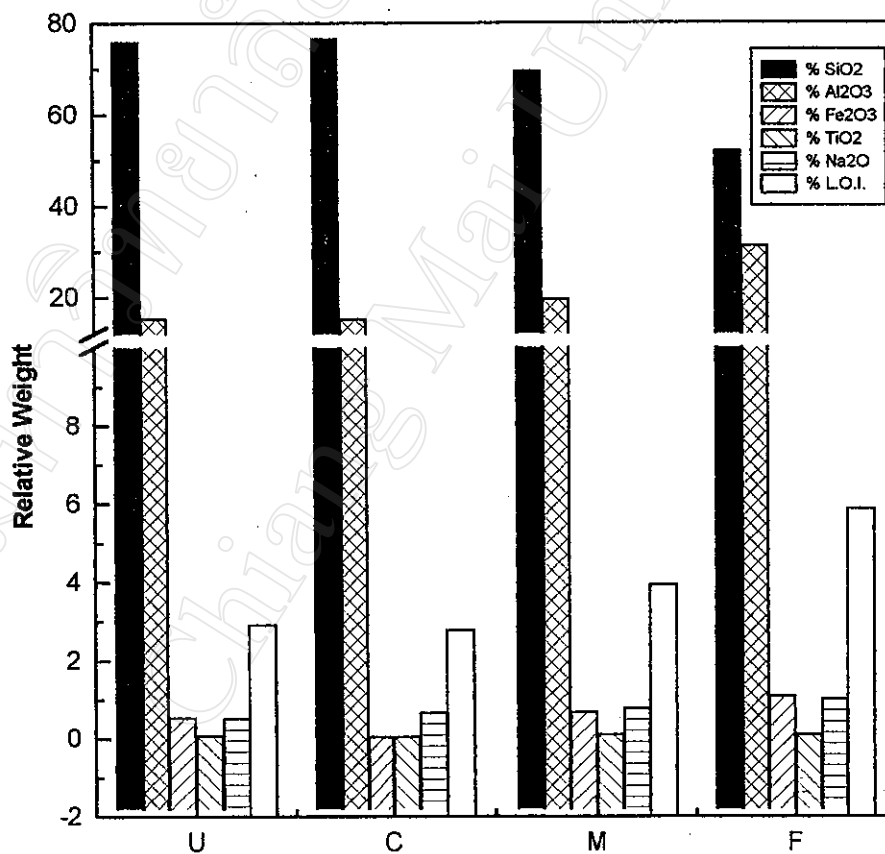


Figure 3.3: Chemical composition of Lampang Clay

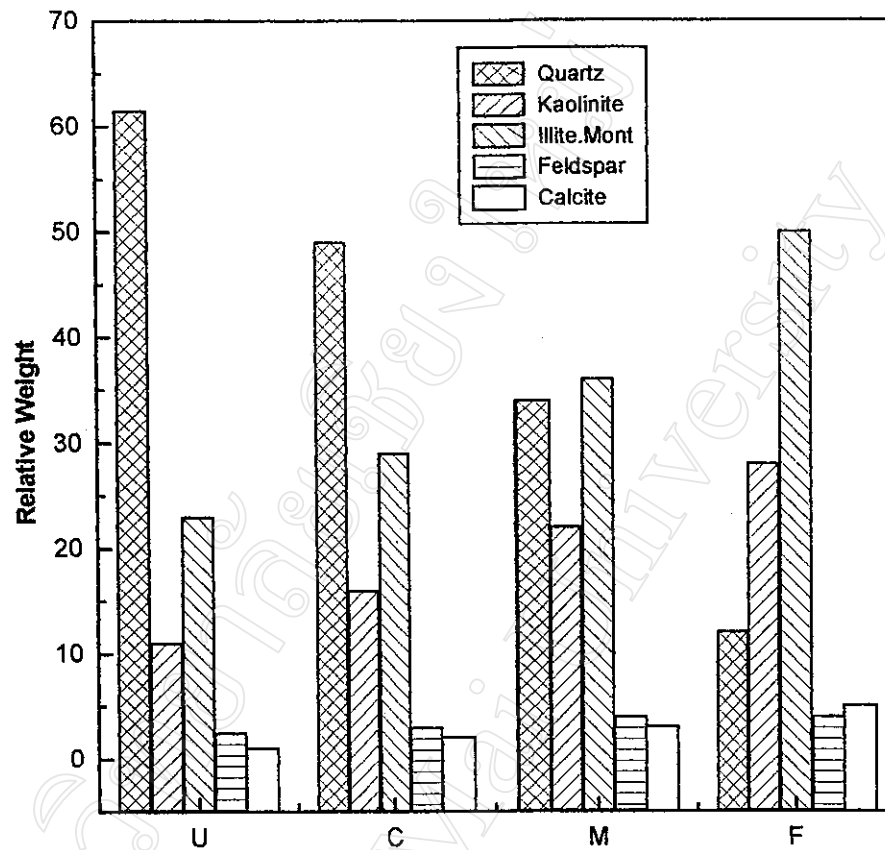
The chemical composition of each sample is shown in Table 3.3 and Figure 3.3. This makes it clear that the sample < 63 microns is comparable to that of the raw material. The sample < 40 microns shows less  $\text{SiO}_2$  than the original material, more  $\text{Al}_2\text{O}_3$ , and a slightly more of the other oxides. The sample < 10 microns contain less  $\text{SiO}_2$  and more  $\text{Al}_2\text{O}_3$  than the other samples. The amounts of  $\text{Fe}_2\text{O}_3$ ,  $\text{K}_2\text{O}$  and  $\text{Na}_2\text{O}$  in this sample all are larger than in the other samples. Possibility the most significant observation is that  $\text{Fe}_2\text{O}_3$  (an undesirable constituent) is concentrated in the fine sample, making it possible to improve the raw clay by merely eliminating the finest particles. Chemical composition and particle size are related to  $\text{SiO}_2$  decreases with increased grain size (part of the  $\text{SiO}_2$  has a grain size larger than 40 microns).  $\text{Al}_2\text{O}_3$  is higher in the fine sample. It appears that  $\text{Al}_2\text{O}_3$  was formed as fine particles like some of the other oxides, namely,  $\text{K}_2\text{O}$  and  $\text{Na}_2\text{O}$ .

#### 3.3.4 Mineralogical composition<sup>5</sup>

The third feature studied was mineral composition. Mineral composition for each sample is shown in Table 3.4 and Figure 3.4.

Table 3.4 : Mineralogical composition of Lampang white clay in percentage

	Quartz	Feldspar	Calcite	Illite	Kaolinite	Anatas	$\Sigma$
Raw material	61.5	2.5	1.0	23.0	11.0	1.0	100.0
Sample passing < 63 microns screen	49.0	3.0	2.0	29.0	16.0	1.0	100.0
Sample passing < 40 microns screen	34.0	4.0	3.0	36.0	22.0	1.0	100.0
Sample passing < 10 microns screen	12.0	4.0	5.0	50.0	28.0	1.0	100.0



**Figure 3.4: Mineral composition of Lampang Clay Samples**

The mineralogical composition of each sample of Lampang clay is shown in Table 3.4 and Figure 3.4. Quartz is more prominent in the fraction with grain size > 10 microns. In the sample which passed through 63 microns sieve, two and three layer clay-minerals are prominent. In contrast to kaolinite, illite-montmorillonite increases with decreasing grain size. These differences in the mineral constituents reflect the differences in chemical composition, shown in Table 3.3. The small amounts in the alkali salts correlates with the small amount of a feldspar as shown in Figure 3.4. It appears that there has been no essential incorporation of alkali-ions in the clay-mineral lattice. The larger fraction of Fe-ions occur in the finer grain sizes. This is conspicuous and probably important.

### 3.3.5 Exchange Capacity

The fourth parameters to be investigated is the cation exchange capacity (CEC). This parameter is an important measure ceramic behavior of the raw material. Cation exchange capacity for the three samples of Lampang clay are shown in Table 3.5 and Figure 3.5.

Table 3.5 : Adsorption of cations and exchange capacity of Lampang white clay

	Na <sup>+</sup>	K <sup>+</sup>	Ca <sup>+</sup>	Mg <sup>+</sup>	Total	NH <sub>4</sub> <sup>+</sup>	C.E.C.(meq./100g)
Raw Material	0.032	0.020	0.131	0.013	0.196	0.10	29.8
< 63 microns	0.030	0.015	0.130	0.019	0.195	0.16	35.5
< 10 microns	0.064	0.044	0.255	0.027	0.391	0.28	67.1

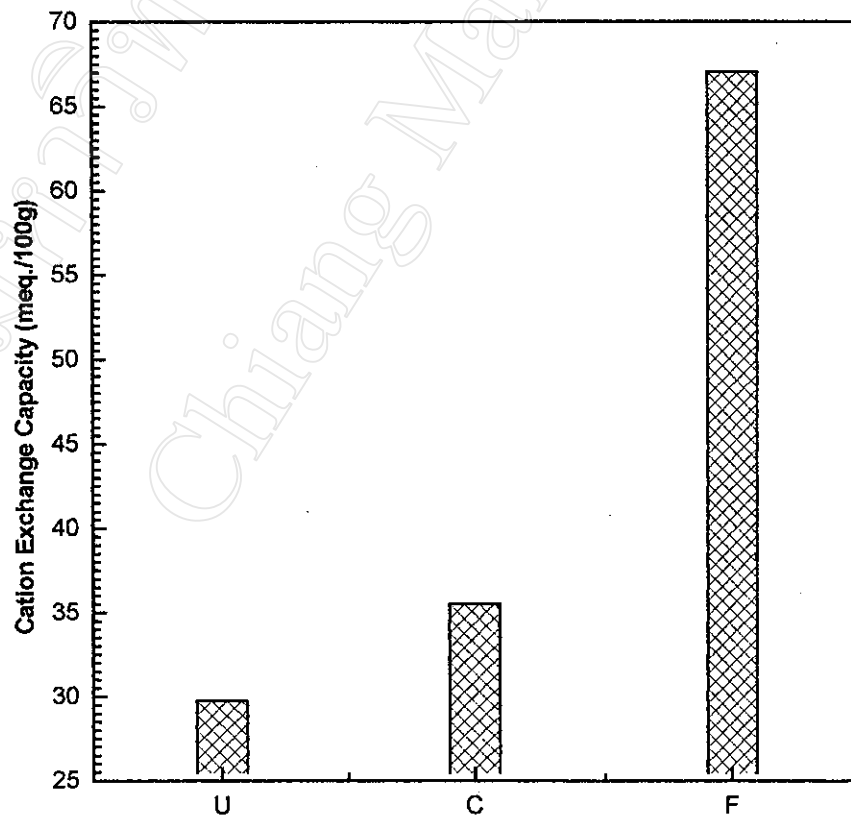


Figure 3.5 : Cation exchange capacity of Lampang Clay



The differences in adsorption of cations and in exchange capacity are shown in Table 3.5 and Figure 3.5, respectively. These are correlated with particle size. Particles finer than 10 micron have a large illite content and the highest cation exchange capacity.

### Summary of Bulk properties of Raw clay<sup>5,30-55</sup>

Raw Lampang white clay is a quartz-rich clay consisting primarily of kaolinite clay-mineral (in addition to illite and montmorillonite). The quartz grain size in this clay is usually larger than 10 microns. As the particle size decreases the illite-montmorillonite clay-mineral increases. Quartz is preferentially found in samples with the largest particles. A small increase of the amount of feldspar present correlates with the small increase of the  $K_2O$  content. It is arguable that no incorporation of alkali ions has taken place in the clay-mineral lattice.

### 3.3.6 Thermal Properties

#### Differential thermo analysis (DTA)

One of the important ways in which clay minerals are characterized is in terms of differential thermal behavior. For the present work three samples and the original raw material were studied. The results are showed in Figure 3.6:

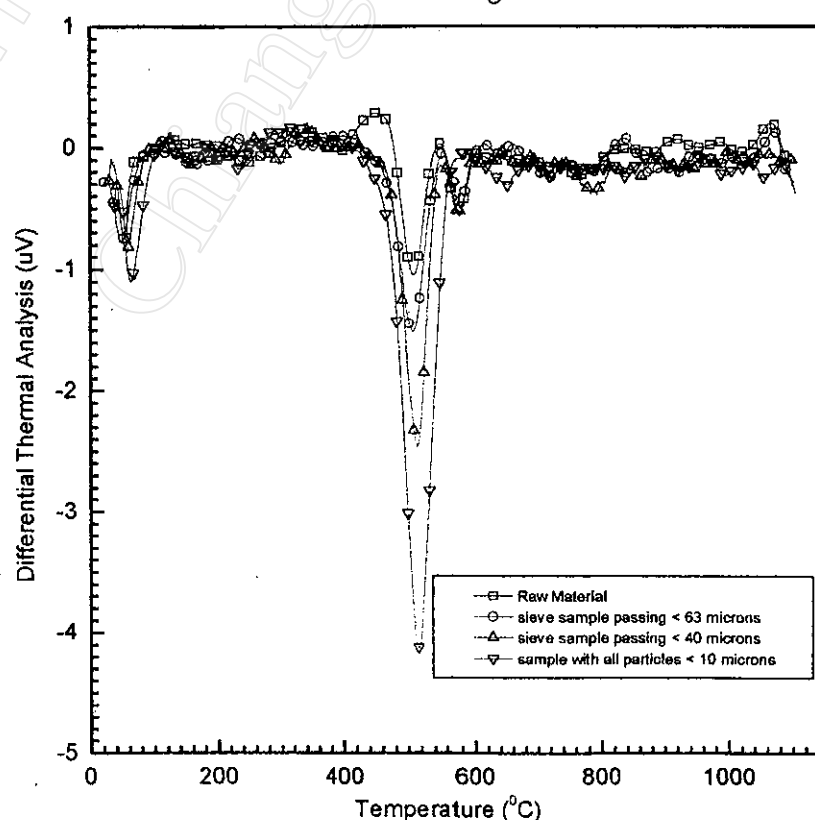


Figure 3.6 : Differential thermo analytical behavior of Lampang clay

The differences in DTA behavior between the original and sieved samples are related and well correlated with the differences in mineralogical and chemical properties. The first peak at about  $100^{\circ}\text{C}$  shows the endothermic evaporation of physically adsorbed water from the surface of the clay particles. The fraction with the finest particles ( $< 10$  microns) has a high content of two layer and three layer clay-minerals and evaporation of the water from these particles includes water which is chemically bonded between the layers of the three layered clay-minerals which are present in the samples. The result is a broad peak and small shift in the characteristic temperature. The second peak at about  $500^{\circ}\text{C}$  is due to a strong endothermic evaporation of the chemically combined water (or structure water) of crystalline kaolin as kaolin ( $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ ) changes to meta-kaolin ( $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ ). This peak is related to the amount of kaolin and is most pronounced in the fine fraction. The third peak, at about  $570^{\circ}\text{C}$ , is due to a small endothermic event, namely, the phase change of alpha-quartz to beta-quartz. This peak correlates with the amount of quartz in the clay material and is smallest in the fine fraction. The fourth peak at about  $1100^{\circ}\text{C}$  is caused by a small exothermic event, probably the change of meta-kaolin to be Al-Si spinell ( $\text{Al}_2\text{O}_3 \cdot \text{SiO}_2$ ).

Another way to characterize clay material is differential thermal gravimetric analysis (TGA). Results of the present study are shown in Figure 3.7:

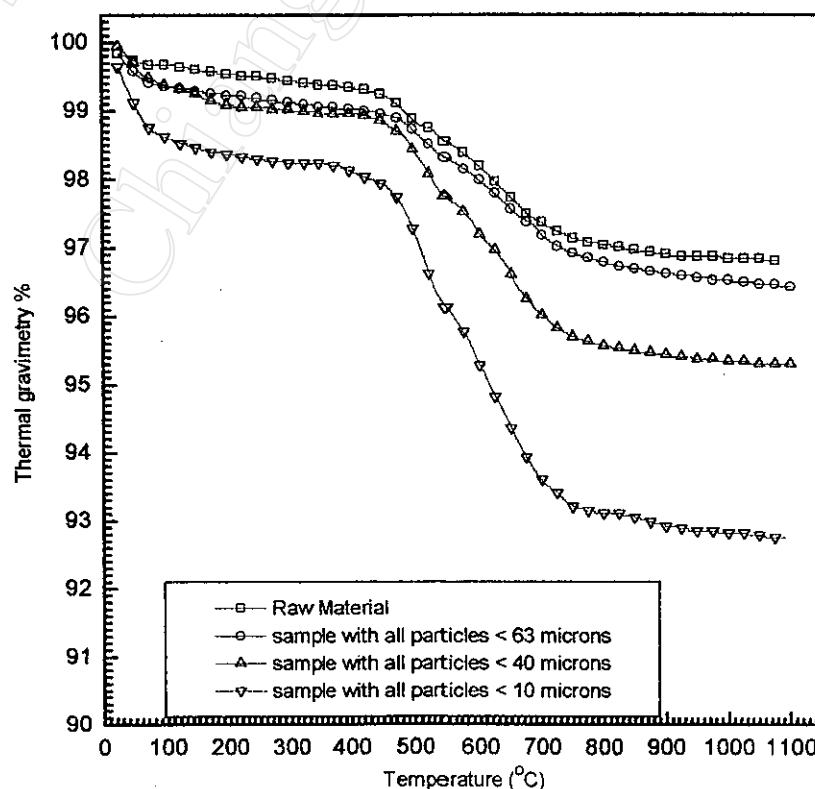


Figure 3.7 : Differential thermal gravimetry of Lampang white clay

Figure 3.7 shows that particles of different sizes behave differently. The first change (between room temperature until about 80°C) is evaporation of physically adsorbed water on the particle surface. The Lampang clay sample < 10 microns shows the larger change from about 100°C to about 450°C we still see small decreases in sample weight. This can be explained as the evaporation of physically absorbed water, especially water between the layers of the clay-minerals and thermal decomposition of organic matter contained in clay-mineral. From about 450°C to 750°C we see strongly decreasing weight of samples which can be explained as the evaporation of chemically combined water or structure water of the clay-minerals, especially as kaolin ( $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ ) changes to meta-kaolin ( $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ ). The size of this peak is related to the amount of kaolin present. There is more kaolin in the fine fraction than the courser fractions.

Another way to characterize the clay material is by expansion-shrinkage behavior. Results are showed in Figure 3.8:

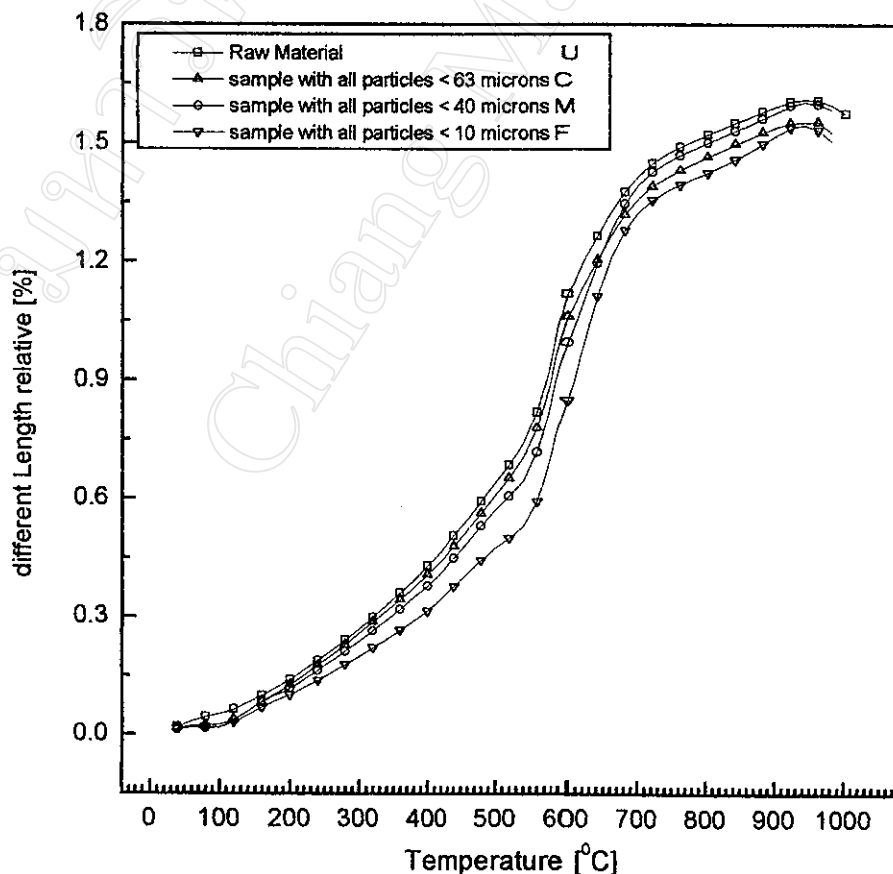


Figure 3.8 : Thermal Expansion of Lampang white clay samples

Figure 3.8 shows the change of linear expansion-shrinkage with temperature. All samples show the same pattern of change. From room temperature until approximately 550°C all show a slow expansion. From 550°C to about 700°C the expansion is more rapid, explained by the conversion of quartz from alpha-quartz (cubic) to beta-quartz (tetragonal). This conversion is accompanied by a volumetric change of 0.8% to 1.3%. The third change, from about 700°C to 950°C, is the slow expansion of samples with increasing temperature. At about 950°C the alkali-clay-mineral, especially illite and feldspar, start to melt and the volume decreases. After 1100°C, all samples melt and lose porosity which shows up as shrinkage.

### 3.3.7 Scanning electron microscope (SEM)

The scanning electron microscope was used to study various samples of Lampang clay. Typical results are shown in Figure 3.9 – 3.12:



Figure 3.9 : Picture of raw Lampang clay at room temperature, 3000x magnification



Figure 3.10 : Picture of Lampang clay sample with all particles < 63 microns room temperature at 3000x magnification.

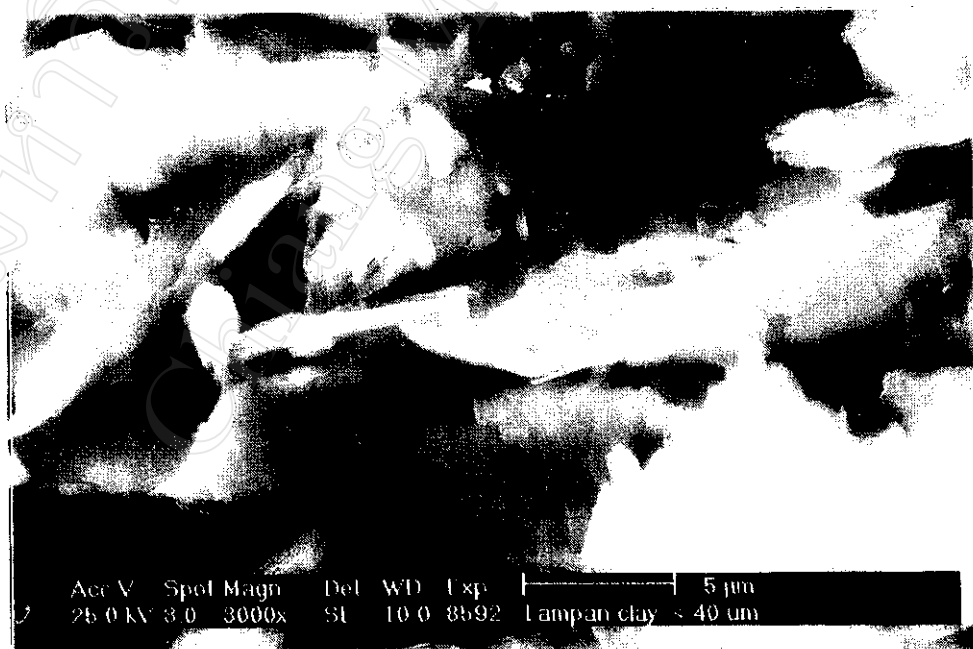


Figure 3.11 : Picture of the Lampang clay sample consists of particles < 40 microns at room temperature, 3000x magnification.

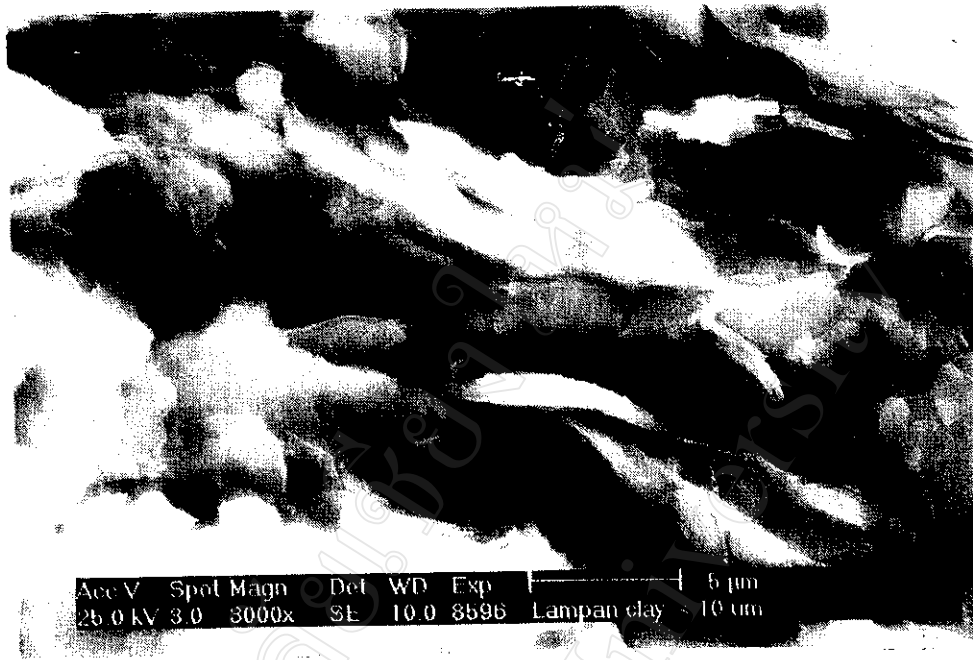


Figure 3.12 : Picture of the Lampang clay sample with all particles < 10 microns at room temperature, 3000x magnification.

The scanning electron microscope pictures of raw Lampang clay show the course grain of quartz and plate particles of two and three layers. These are clay-minerals that are consistent with the results of the mineral composition analysis that showed about 64% pure mineral and about 36 % clay-mineral (see Figure 3.9). Figure 3.10 shows more plate particles and less of the course grain mineral and corresponds to approximately 52 % mineral and 48 % clay-mineral. Figure 3.11 shows still more plate particles and still less course grain particles. The sample has approximately 28 % mineral and 62 % clay-mineral. Figure 3.12 shows that most particles are fine plate particles that have about 16 % mineral and about 84 % clay-mineral. Note, the mineral composition was measured by means of X-ray analysis. SEM results are included for completeness.

### 3.3.8 Behavior when heated to various temperatures.

One important characterization of all fractions of Lampang clay are the changes observed with a change in temperature. Results are shown in Table 3.6.

Table 3.6: Weight percent of various phases as a function of temperature in raw material and various fraction (< 63 microns, < 40 microns, < 10 microns)

	Quartz	Feldspar	Illite	Kaolinite	Calcite	Anatas	Mullite	Amorphous	$\Sigma$
Raw material	61.5	2.5	23.0	11.0	1.0	1.0	0.0	0.0	100.0
400°C	61.0	2.0	19.0	11.0	0.0	0.0	0.0	7.0	100.0
700°C	56.0	2.0	0.0	0.0	0.0	0.0	0.0	42.0	100.0
1100°C	54.0	1.0	0.0	0.0	0.0	0.0	0.0	45.0	100.0
1200°C	42.0	0.0	0.0	0.0	0.0	1.0	8.0	50.0	100.0
< 63 microns	49.0	3.0	29.0	16.0	2.0	1.0	0.0	0.0	100.0
400°C	48.0	2.0	27.0	16.0	2.0	0.0	0.0	5.0	100.0
700°C	45.0	2.0	0.0	0.0	0.0	0.0	0.0	53.0	100.0
1100°C	41.0	1.5	0.0	0.0	0.0	0.0	0.0	57.0	100.0
1200°C	40.0	0.0	0.0	0.0	0.0	0.0	9.0	51.0	100.0
< 40 microns	34.0	4.0	36.0	22.0	3.0	1.0	0.0	0.0	100.0
400°C	34.0	4.0	27.0	22.0	2.0	0.0	0.0	11.0	100.0
700°C	32.0	3.0	0.0	0.0	0.0	0.0	0.0	97.0	100.0
1100°C	30.0	1.5	0.0	0.0	0.0	0.0	5.0	98.5	100.0
1200°C	27.0	0.0	0.0	0.0	0.0	0.0	10.0	63.0	100.0
< 10 micron	12.0	4.0	50.0	28.0	5.0	1.0	0.0	12.0	100.0
400°C	14.0	4.0	33.0	28.0	2.0	0.0	0.0	19.0	100.0
700°C	10.0	2.0	0.0	0.0	0.0	0.0	0.0	88.0	100.0
1100°C	9.0	1.5	0.0	0.0	0.0	0.0	0.0	89.5	100.0
1200°C	6.0	0.0	0.0	0.0	0.0	0.0	16.0	78.0	100.0

From Table 3.6 it can be observed that the amount of quartz and feldspar mineral are decreased when the clay is sieved with a fine screen. On the other hand, the amount of illite and kaolinite clay-mineral increases. When heated, all fractions show no change in mineral composition between room temperature and 400°C. Up to 400°C, the reaction in the sample is only drying (the loss of physically adsorbed water evaporating from the surface of the particles). By 700°C, feldspar has not changed, quartz changes phase from alpha-quartz to beta-quartz. At this temperature, clay-minerals (illite, kaolinite, calcite and anatas) change phase and become amorphous. For example, kaolinite will change to be meta-Kaolin. And the X-ray peaks associated with kaolin disappear. By 1000°C, the reaction of alkali clay-mineral will start and all phases become glassy. At this temperature, mullite begins to form from the glassy phase. If the total amount of  $\text{Al}_2\text{O}_3$  is insufficient, mullite will not form at all. By 1200°C, feldspar is completely melted and the amount of quartz is reduced and the amount of mullite is increased in all fractions. The fine sample (all particles < 10 microns) shows the amount of mullite is more than in the other fractions because this fraction has more kaolinite. Note: the fraction of the mullite structure is important when ceramic materials mature when fired.

### Conclusion

The medium and fine fractions of white clay from Lampang have properties which should make them useful to the ceramic industry.

#### 3.3.9 Properties of slip made from Lampang clay.

One standard method for ceramic production is hollow castings made in plaster molds (slip is allowed to stay in the plaster mold until solid clay material has collected on the plaster surfaces. The mold is inverted and the slip, which has not adhered to the plaster mold, is allowed to flow out).

Rheological behavior of clay slip (clay mixed with water) is related to quality control in ceramic processing and body preparation. When clay-mineral particles are dispersed in water, the resulting clay slurry can be investigated in standard ways. Deflocculant demand, viscosity and flow ability can be measured.



### 3.3.9.1 Deflocculant demand

All fractions of Lampang white clay studied had suitable demand for deflocculant and should make good slurries (see Figure 3.13).

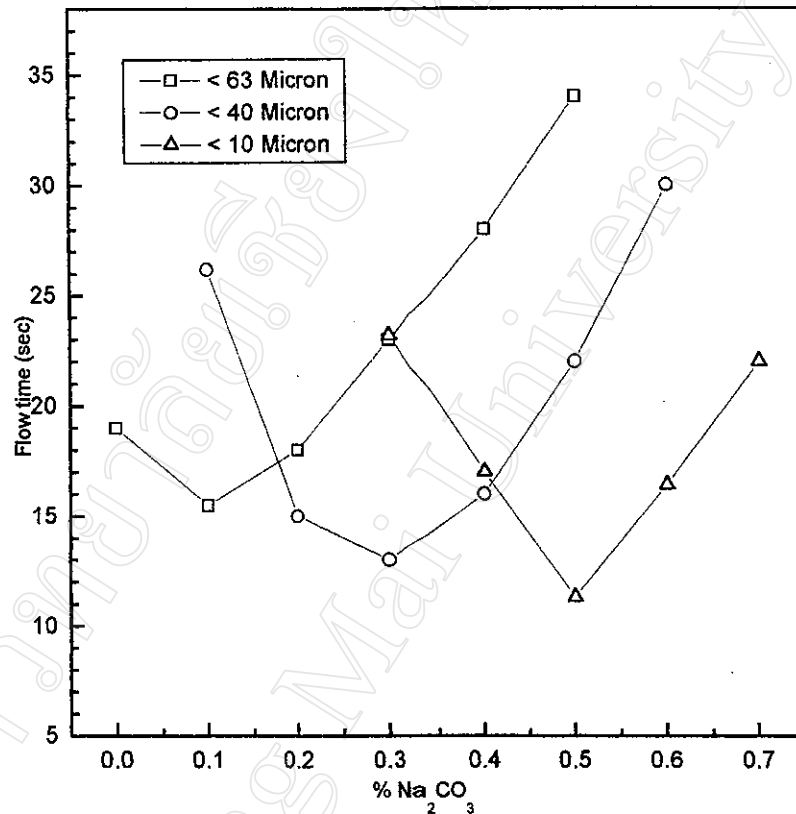


Figure 3.13 : Deflocculant demand of Lampang white clay after size grading.

From Figure 3.13 shows that fine fraction of Lampang clay needs more deflocculant than the coarse fraction. It is likely that this is because of a greater number of particles, a higher specific surface area, and higher cat-ions exchange capacity. To be dispersed, the fine Lampang clay sample needs more deflocculant. The sample < 63 microns has a very rapid sedimentation rate and flow rate. The sample < 40 microns has a board curve that shows that it can be readily dispersed in water. It can be seen that the fraction < 40 microns is of greatest interest for the ceramic industry because preparation and control of such material would be easiest.

### 3.3.9.2 Viscosity

Another important rheological characteristic is viscosity. If viscosity is plotted against shear rate the observed value depends on the history of the sample, that is, hysteresis is demonstrated. The appropriate viscosimeter has a rotary cylinder and viscosity is indicated by the extent to which the liquid material retards rotation. This retardation is found to depend on the rate at which the measuring cylinder turns and whether the cylinder is turning faster or moving more slowly. Hysteresis loops are measured and have the largest area for samples which are the most thixotropic. Note: thixotropy is not a desirable property if one is trying to produce ceramic greenware by casting clay slip.

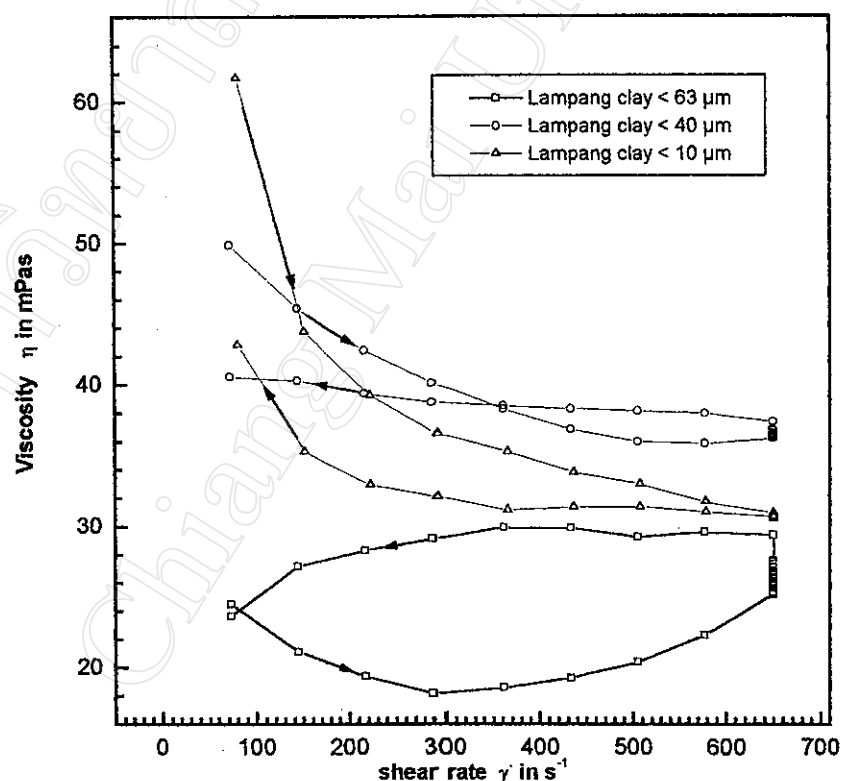


Figure 3.14 : Viscosity curve of Lampang clay.

All three samples of Lampang white clay have some degree of thixotropic behavior. For the sample with particles  $< 63$  microns (see Figure 3.14) if the applied shear rate is increased (the viscometer turns initially at about 300 revolutions per minute)

the viscosity decreases. The explanation is that particles form new arrangements and flow as larger units. But, when the shear rate is high enough, the viscosity increases again because the coarse grains of the quartz retard the rotating cylinder of the viscometer. If the revolutions per minute are in excess of 650 and then the slurry is allowed to rest for one minute, the viscosity decreases and it is necessary to apply only reduced shear to reach an equilibrium point. With sedimentation of the quartz, the viscosity increases suddenly from approximately 25 mPa.s to 29 mPa.s and we say that the slurry has high thixotropy. After 650 revolutions per minute the viscosity decreases with a decrease in applied shear rate but we find a large difference between the stirred material and the material which has been allowed to rest. It can be said that the sample with particles < 63 microns has viscosity behavior that would make it difficult to control in an industrial setting. For the fraction < 40 microns behavior is similar but increasing the applied shear rate to about 360 revolutions per minute produces a smaller change in viscosity that is, it is less thixotropic. That can be explained by the fact that the sample with particles < 40 microns has smaller quartz particles and more clay-mineral with plate structure than the sample with particles < 63 microns. Regarding quality control in the ceramic process, the sample with particles < 40 microns has a large range of shear rates that must be controlled. For the sample with particles < 10 microns, a different behavior is encountered. In this sample, if the applied shear rate for the slip is increased, then the viscosity first decreases rapidly and then increases as the shear rate is increased to 650 revolutions per minute. It can be said that the sample with particles < 40 microns has a desirable viscosity and acceptable thixotropy for use in ceramic production.

### 3.3.9.3 Thixotropy

Thixotropy is another important rheological property of slip (see Figure 3.15, 3.16 and 3.17). Thixotropy is often measured at high concentrations (about 60 % solid). The data for Figure 3.14 should be compared with the data for Figure 3.15, 3.16 and 3.17.

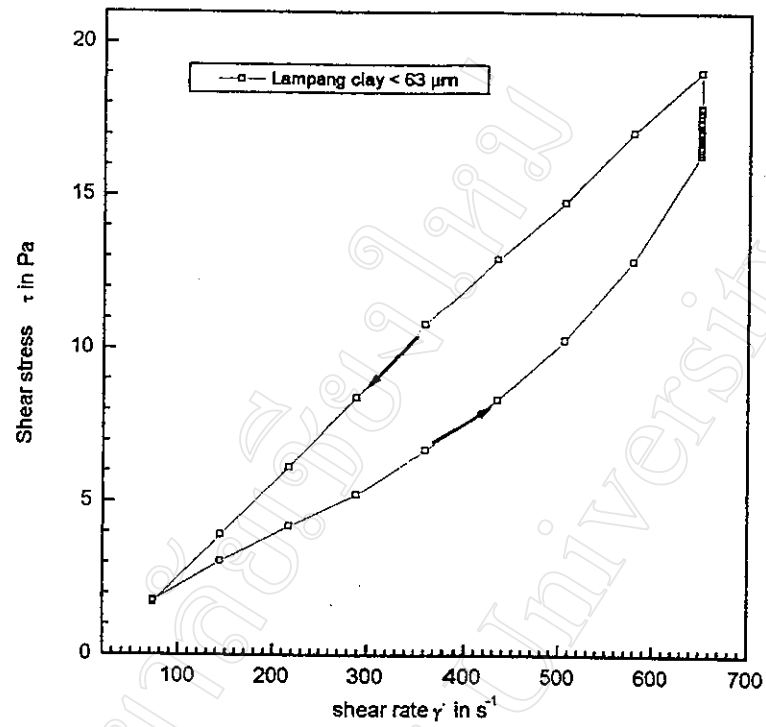


Figure 3.15 : Flow curve of Lampang clay < 63 microns.

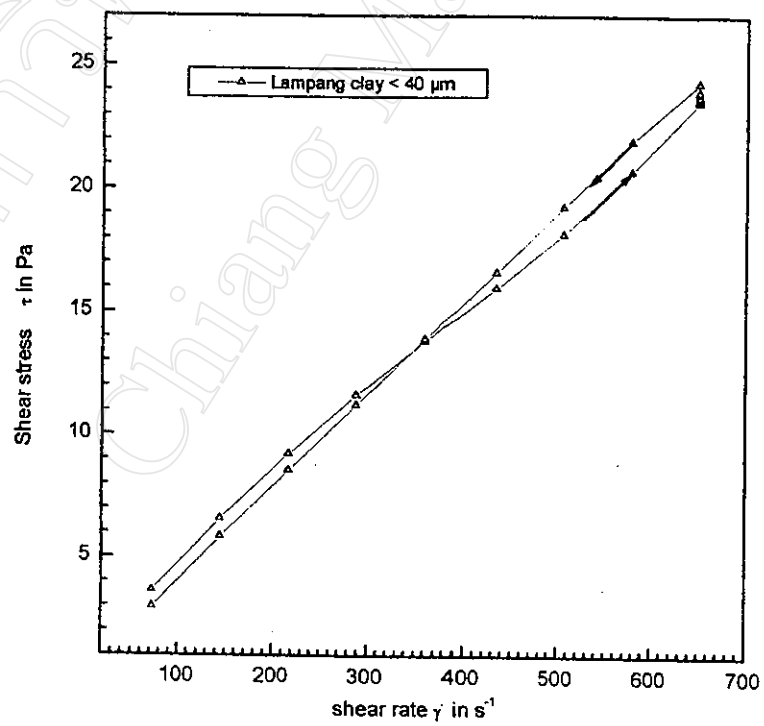


Figure 3.16 : Flow curve of Lampang clay < 40 microns.

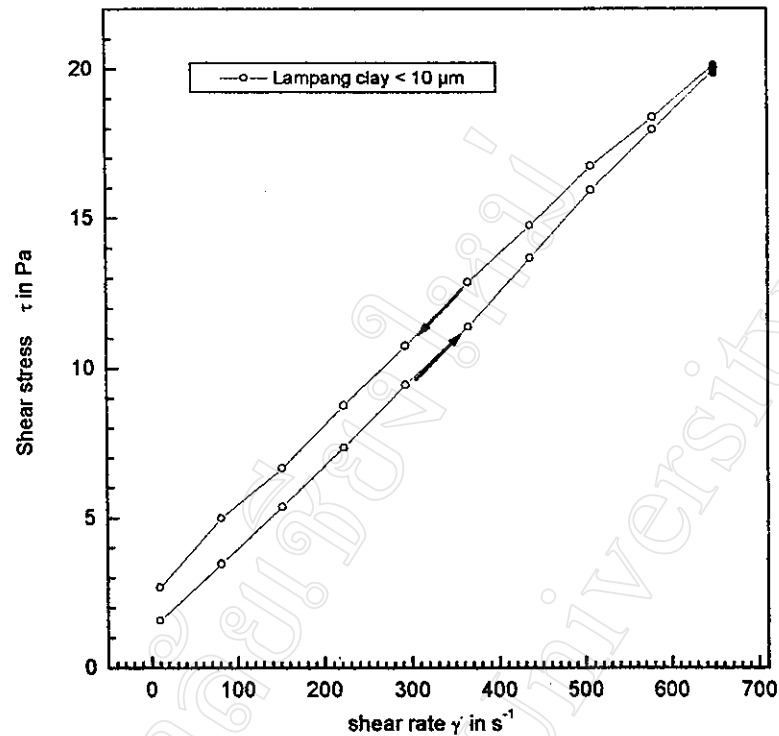


Figure 3.17 : Flow curve of Lampang clay < 10 microns.

Figure 3.15, 3.16 and 3.17 show the flow behavior curve or flow ability of the slip with 50 % water for each sample. Data for C, the sample < 63 microns, in Figure 13, we note that if the applied shear rate applied by the viscometer at the beginning is about 300 revolutions per minute, the shear stress of slip is increased. This can be explained by noting that particles undergo abrasion against the moving cylinder of the measuring instrument. After 650 revolutions per minute letting the slip rest for one minute at a time and applying a shear rate from 650 revolutions per minute back to 70 revolutions per minute. It is found the slip undergoes sedimentation, primarily of quartz, and the shear stress increases suddenly from approximately 16 Pascal to 19 Pascal. We say that the slip has high thixotropy. After 650 revolutions per minute the shear stress decreases with a decrease applied shear but shows a big difference with any increase in applied shear rate. It can be said that the fraction of < 63 microns is difficult to control in ceramic processing. The fraction < 40 microns in Figure 14 and < 63 microns fraction shows similar behavior but the pathway to reduce applied shear rate crosses the pathway to

increase the applied shear rate at about 360 revolutions per minute. For the fraction  $< 10$  microns in Figure 3.17 behavior is different. Here if the applied shear rate for the slip is 650 revolutions per minute and the slip is allowed to rest for one minute intervals the shear stress slowly decreases. This can be understood if we note that the fraction  $< 10$  microns has a high content of fine plate like clay-mineral particles and only a small amount of fine quartz. Plate-like structure particles have a slippery behavior and exhibit reduced abrasion between particles. Thixotropy decreases and the flow becomes more viscous. For quality control in ceramic processing the fraction  $< 40$  microns is seen to have Newtonian flow behavior, little thixotropy, which makes it the best choice for ceramic production.

#### 3.3.10 Casting behavior

Casting behavior of clay slip is important for quality control in the ceramic forming process. When a clay-mineral slip is poured into a gypsum mold, the gypsum mold will adsorb water and the clay particles are deposited on the mold. The casting rate can be measured. All samples from slip with 50 % water were measured and are shown in Figure 3.18.

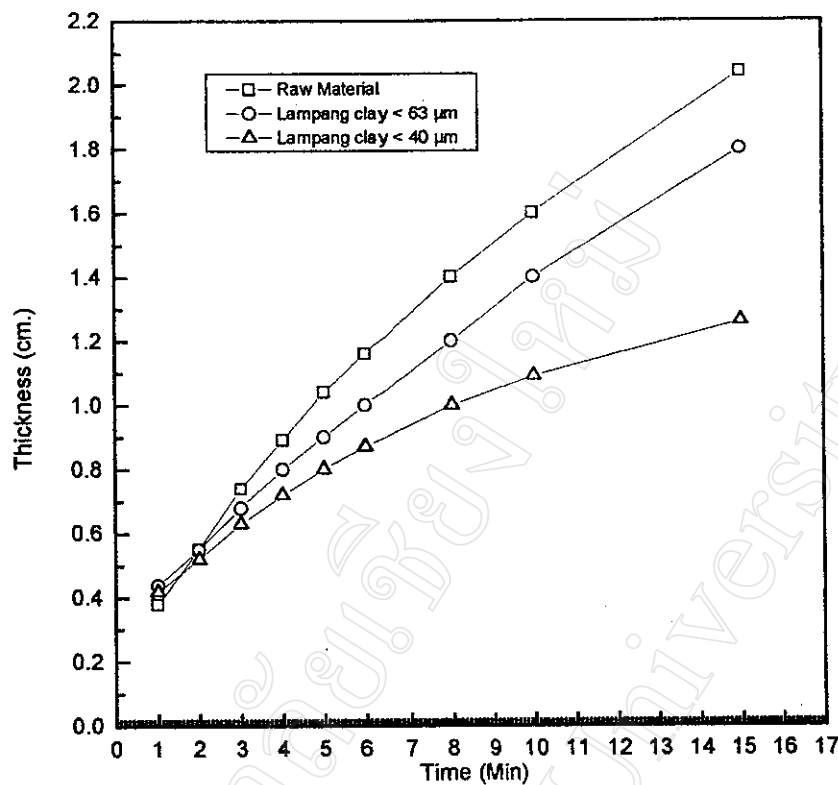


Figure 3.18 : Casting rate of Lampang clay.

Figure 3.18 shows casting rates for slip (50 % water) made with Lampang clay. The coarsest sample had the highest casting rate. Water flows easily through the deposited clay layer. The sample < 63 microns had a casting rate slower than the raw material because the original clay had some very coarse material which was discarded. The deposit of clay is thicker and the arrangement of clay particles is better. In overview, it can be said that the raw material and the sample < 63 microns had high sedimentation rates. The next sample, < 40 microns, had the slowest casting rate because it contains more of the fine plate structure particles with some of the fine quartz. The fine particles are expected to have good particle arrangement and produce a dense clay layer that reduces the flow rate of water to the gypsum mold. For ceramic forming by slip casting we need a suitable casting rate. From the casting behavior of all samples from Lampang white clay, the best casting rate is seen in the sample < 40 microns. Measurements were

not make on the finest fraction because the total amount of material available for this measurement was insufficient.

### 3.3.11 Plastic behavior of moist clay samples

Plastic behavior is a very important technological property of clay. Plastic behavior of all the clay samples in this study is indicated in Figure 3.19.

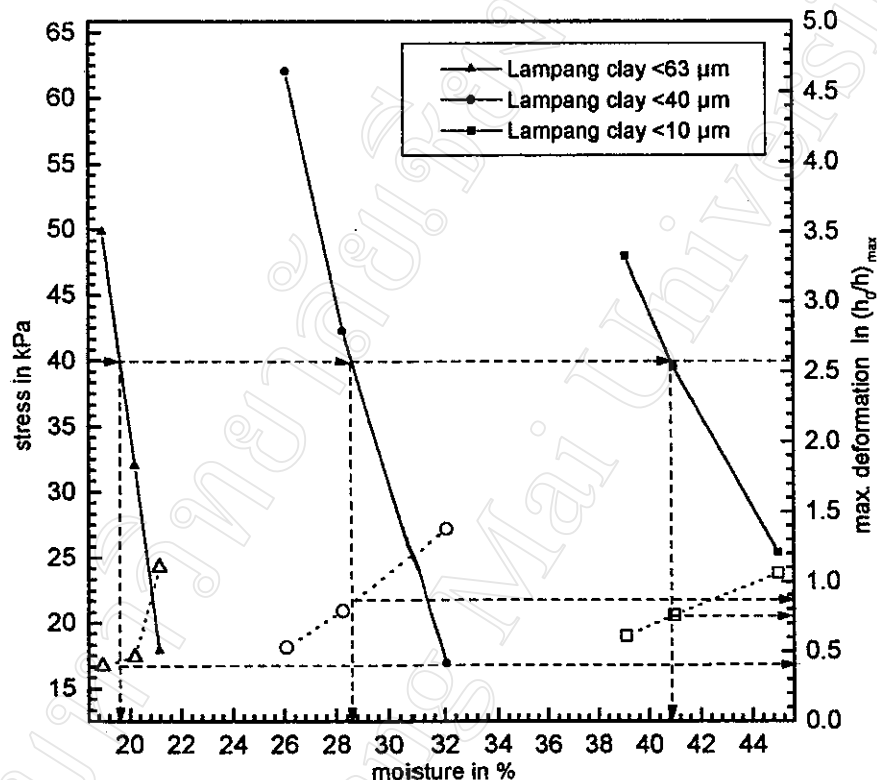


Figure 3.19 : Stress and maximum deformation of most Lampang clay as a function of water content.

Figure 3.19 shows plasticity of Lampang clay. It can be seen that samples with finer grain sizes hold more water. For the fraction < 63 microns the moisture content is least and we can see from slope that sample stress changes very fast with only a small change of moisture content. The fraction < 40 microns has an intermediate moisture content but reaches the largest stress before breaking. The fraction < 10 microns shows a smaller change in physical property with changing moisture content. That means the



fraction < 40 microns would be easiest to use in usual preparation process. The fine fraction needs a higher moisture content when compared to the other fractions. The maximum deformation of all fractions occurs at stress 40 kilo Pascal. The fraction < 63 microns shows 0.42 maximum deformation at 19.5 % moisture. The fraction < 40 microns shows 0.85 maximum deformation with 28.7 % moisture and the fraction < 10 microns has 0.75 maximum deformation with 41.0 % moisture. This means that the fraction < 40 microns has highest deformation capability and is the easiest of the samples studied to control in the usual forming processes in the ceramic factory.

### 3.3.12 Drying behavior

Another important technological property is behavior on drying. Drying behavior is important for the processing and quality control. There are two properties which are usually used to characterize drying behavior : dry shrinkage and dry strength.

#### 3.3.12.1 Dry shrinkage

Dry shrinkage is occurs as mechanically held water evaporates from the green product. Dry shrinkage of all of the samples of Lampang clay which have studied are shown in Figure 3.20.

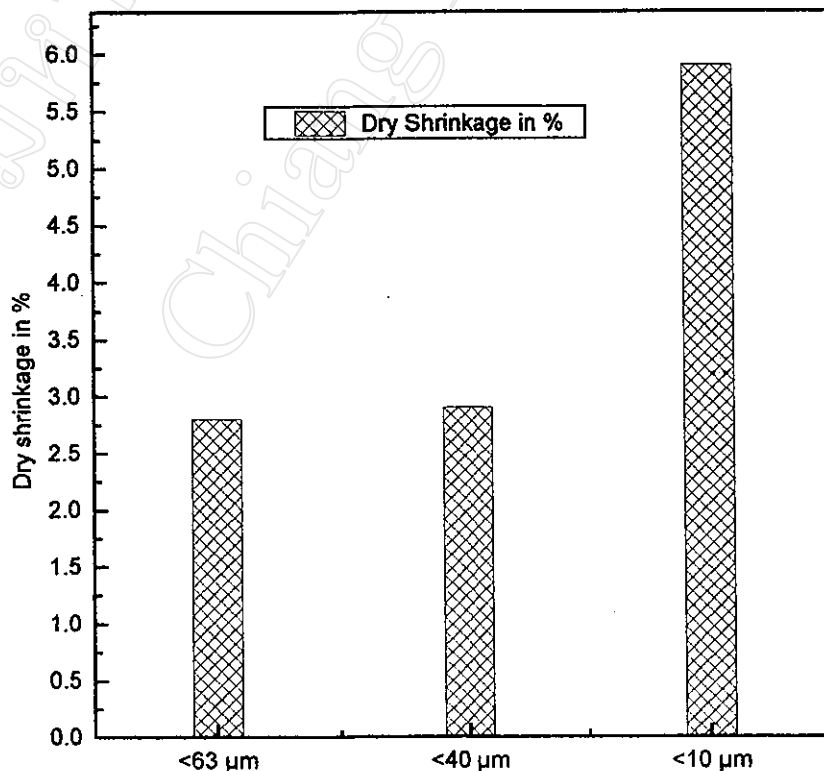


Figure 3.20 : Dry shrinkage of Lampang clay for each of three samples.

From Figure 3.20 it is seen that the sample < 63 microns and the sample < 40 microns have similar dry shrinkage, about 2.8 %. However, the sample < 10 microns shrinks much more, about 5.9 %. This can be explained by the high amount of fine plate like clay-minerals contained by this sample. According to dry shrinkage the most suitable fraction for use in ceramic body production is the sample < 40 microns. It does not have much dry shrinkage. Note that shrinking is associated with warping of green ware. Clay with particles < 10 microns should not be used for ceramic production if possible.

### 3.3.12.2 Dry strength

Dry strength is a property of clay materials that is measured after water mechanically included, evaporates from the green product. Dry strength is important to industry because the green product must be transported to the next process after forming. Dry strength of all samples of Lampang clay studied are shown in Figure 3.21.

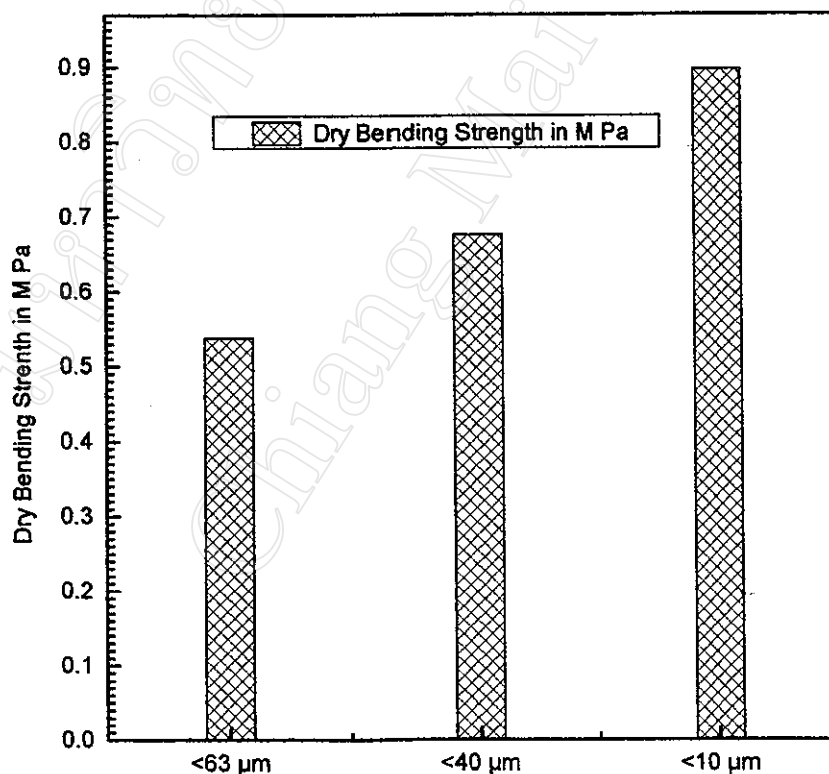


Figure 3.21 : Dry bending strength of Lampang clay for each of three samples.

Figure 3.21 shows that dry strength increases somewhat as particle size decreases. That can be explained by the evaporation of mechanical water and the consequent greater contact and closer packing of particles.<sup>5,24-26,30-55</sup> The way that particles are associated and the physical-chemical attachment of the particle surface are all factors in dry strength. The sample with all particles < 10 microns has a high content of fine plate like particles that arrange in a dense pattern. These fine particles are strongly bonded together. The fraction of < 40 microns has fewer fine plate particles are some fine quartz that mixes with the fine particles that results in improved strength.

From both of these properties it can be recognized that sample M (particles < 40 microns) is of most interest for use in making ceramic bodies. This fraction has acceptable dry shrinkage and sufficient dry strength.

### **3.3.13 Property of the Fired Clay**<sup>5,21-55</sup>

The most important characteristic for ceramic raw materials is the behavior of these materials when fired. Firing behavior is measured in terms of shrinkage due to firing, bending strength, density, porosity and whiteness after firing.

#### **3.3.13.1 Fired shrinkage**

Fired shrinkage of ceramic materials is used to establish the sintering temperature of the clay material. Shrinkage that results from firing must be known before the clay can be used to advantage. The size and shape of the product after firing will be strongly dependent on shrinkage in the kiln. Fired shrinkage, that is, the shrinkage that occurs in the kiln, is shown for all the samples of Lampang clay in Figure 3.22.

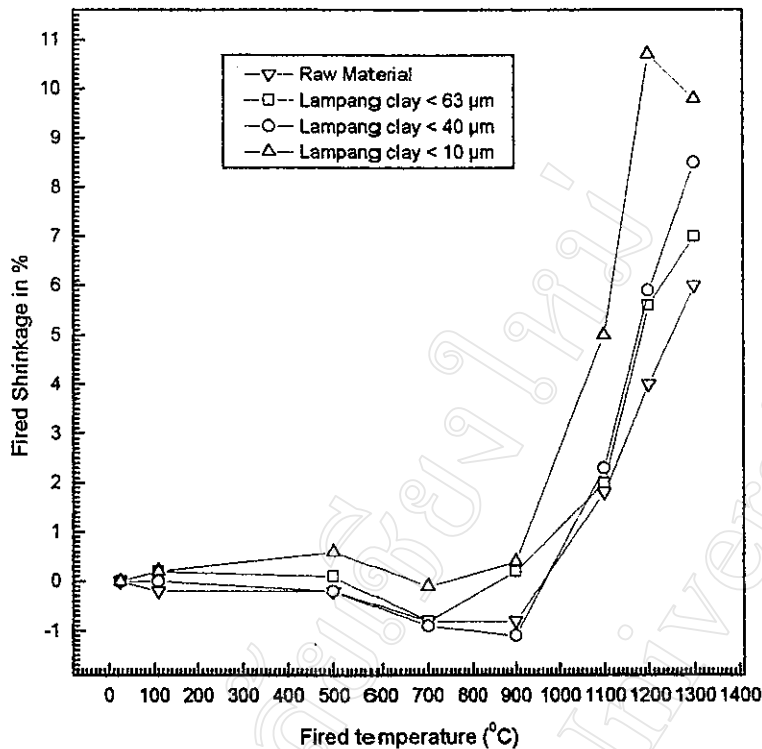


Figure 3.22 : Fired shrinkage of Lampang clay after sieving and firing.

Figure 3.22 shows that all samples have a similar pattern of shrinkage. In the firing process (room temperature up to 1200°C) all fractions shrink. The shrinkage from room temperature to 500°C is small and results from burning out organic matter and evaporation of water. From 500°C to 700°C, fired shrinkage decreases, that is, the samples expand. (In all samples, quartz changes phase and expands). From 700°C to 900°C small increases in shrinkage are seen. From 900°C up to 1200°C we have strongly increasing effects from sintering and a conversion to a glassy phase. From 1200°C to 1300°C, sample U (raw material) and sample C (particles < 63 microns), sample M (particles < 40 microns) are still shrinking. That means there will be more shrinkage as the temperature is increased higher than 1300°C. The sintering process is not yet finished at this temperature. For the F sample (particles < 10 microns) at 1200°C shrinkage reach a maximum and after that decrease. This can be explained by the fact

that the sintering condition is reached at about  $1200^{\circ}\text{C}$  and after  $1200^{\circ}\text{C}$  the glassy phase is expands as the temperature is further increased.

### 3.3.13.2 Strength of fired clay

Other characteristics of interest and importance concern bending strength. For industrial purposes, the fired strength must be known. Otherwise, the firing process cannot be controlled and the quality of the product will be variable. The fired strength of all the samples of Lampang clay is shown in Figure 3.23.

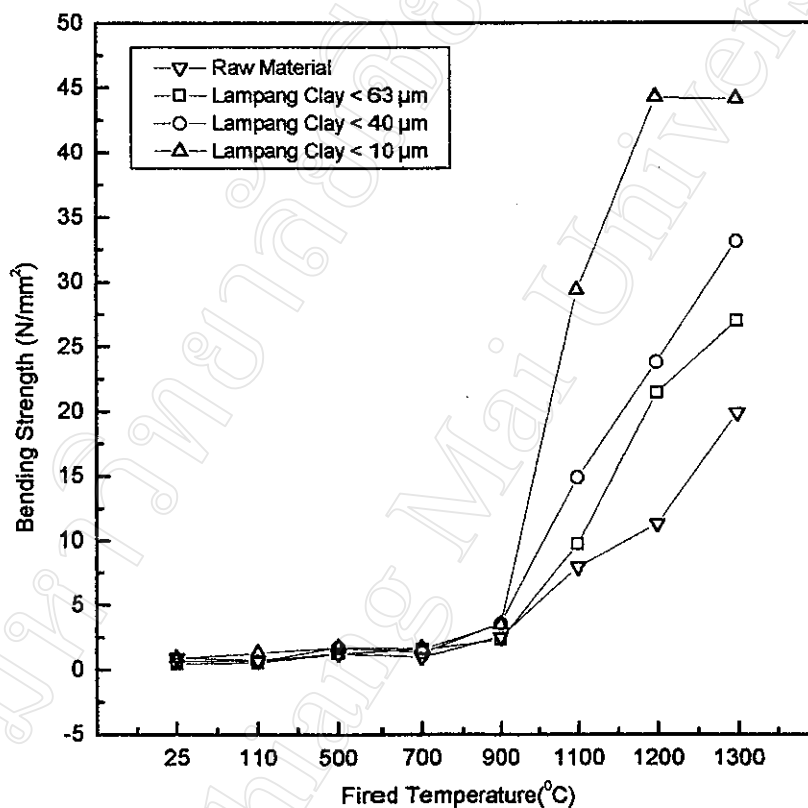


Figure 3.23 : Bending strength of Lampang clay after sieving and firing.

From Figure 3.23 we see that the pattern of fired bending strength correlates with fired shrinkage. From room temperature to  $700^{\circ}\text{C}$ , fired bending strength of all fractions seems to be constant. From  $700^{\circ}\text{C}$  to  $900^{\circ}\text{C}$  there is a small increase. From  $900^{\circ}\text{C}$  until  $1300^{\circ}\text{C}$ , sample U, sample C (particles  $< 63$  microns) and sample M (particles  $< 40$  microns) increase in strength to about  $33\ \text{N/mm}^2$  and can be increased further by

increasing the temperature. But fraction < 10 microns reaches its highest fired strength (of about  $44 \text{ N/mm}^2$ ) at  $1200^\circ\text{C}$  and decrease if the temperature is raised any more. This behavior correlates with fired shrinkage behavior.

### 3.3.13.3 Density after firing

Density after firing is another important characteristic. For processing purposes, density after firing must be known so that the firing process can be stopped as soon as possible to save fuel costs and prevent the product from melting. Density after firing for all samples are shown in Figure 3.24. Two values for density are given in each case. The lower density figure is obtained for the whole test piece and includes effects due to porosity. The higher value is obtained after the test piece is finely ground and the effect of porosity is eliminated.

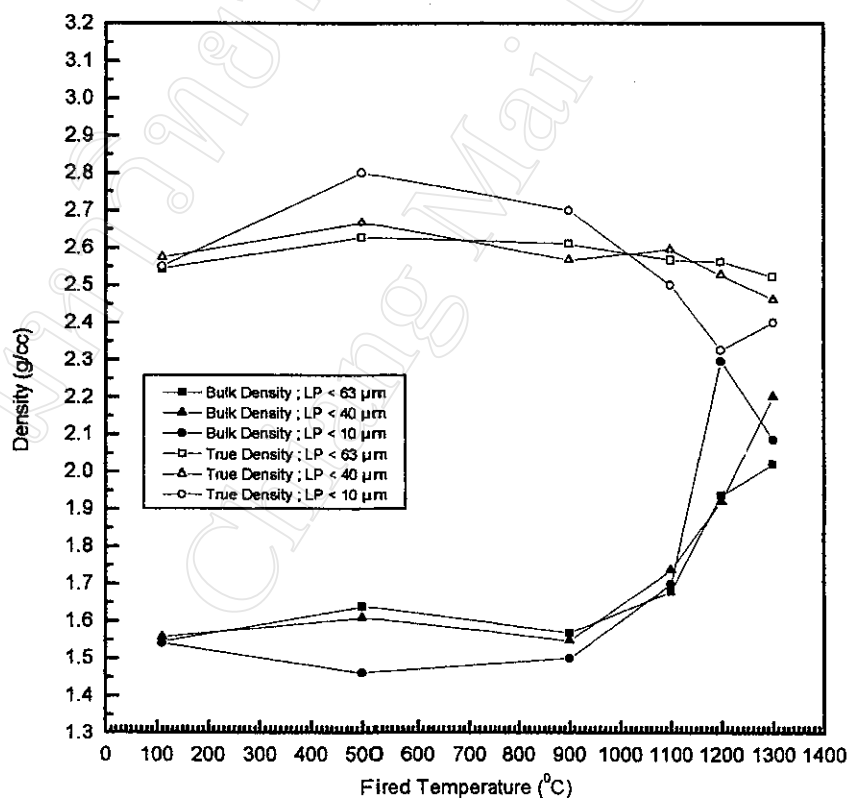


Figure 3.24 : Bulk density and true density of Lampang clay.

Figure 3.24 shows density before and after grinding. All samples are fired Lampang clay. Differences in density are on indication of porosity. For the sample C (all particles < 63 microns), from room temperature until 500°C both densities increases. From 500°C to 900°C density after grinding decreases slightly but density of whole test pieces is constant. This can be explained by the burning of organic matter and evaporation of water. From 900°C to 1300°C, the density of the ground samples decreases slightly but density of the whole test pieces increases significantly. It can be seen that material becomes denser when temperature is increased. At 1300°C there is still a gap between both densities; that means that the whole test pieces still have pores and the material has not yet reached the sintering condition. For sample M (particles < 40 microns), the pattern of both densities are similar to sample C but with a smaller gap at high temperature (> 900°C). At 1300°C there is still a difference between the two densities but smaller than that for sample C. That means we are still below the sintering condition and the clay can be fired to a still higher temperature. For the sample F (particles < 10 microns), from room temperature until 1200°C a similar pattern can be seen. The interesting point is 1200°C. At that temperature the gap between the two densities is small and that means that by 1200°C the material reaches the sintering condition and has a dense body. After 1200°C the gap increases again which means that gas bubbles are beginning to form in the glassy phase material.

#### 3.3.13.4 Porosity after firing

Porosity after firing is implied by the density measurements but can be measured independently. The importance of porosity measures is that they give information about sintering that occurs when the clay is fired. For processing purposes, porosity after firing must be investigated. Results from this study are shown in Figure 3.25.

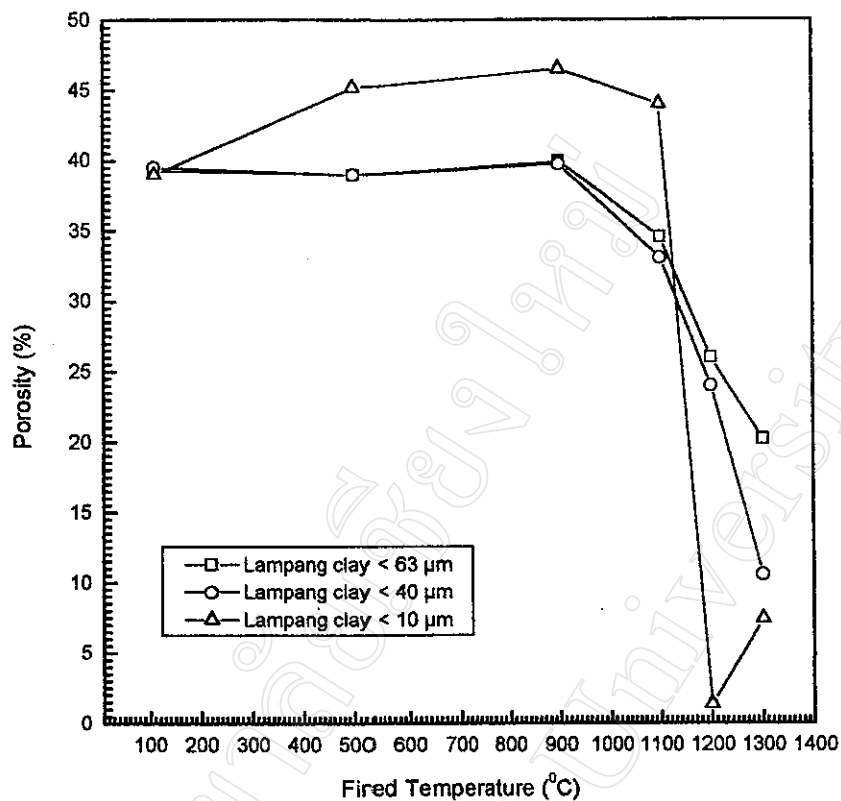


Figure 3.25 : Porosity of Lampang clay after firing.

Figure 3.25 shows porosity after firing for all samples. For sample C (particles < 63 microns), from room temperature to 900°C, porosity is seen to be constant at about 39 %. From 900°C to 1300°C porosity of all samples decreases but residual porosity in the sample C seems to be a little higher. This means that sample C can be fired to higher temperatures than the other samples. It is of interest that the sample F (particles < 10 microns) from room temperature until 900°C, shows a porosity increase of about 5 %. From 900°C to 1100°C there is a small decreased in porosity, but from 1100°C to 1200°C the porosity decreases sharply from about 44 % to be about 1.5 %. After this minimum the porosity increases again to about 7 %. That can be explained by noting that between 1100-1200°C there is a very rapid sintering of alkali-material which reduces the porosity and after 1200°C, the clay is completely sintered and becomes glassy and melts. The production of gas bubbles appears to increase the porosity again. If sample F (particles



< 10 microns) is used or mixed with other materials to make a ceramic body, it must be fired very carefully especially after  $1100^{\circ}\text{C}$ . From its firing behavior alone, the fine fraction can be seen to be of no interest for ceramic production.

### 3.3.13.5 Whiteness

The most important characteristic of ceramic raw material especially Lampang clay which fires to a white color, is the degree of whiteness after firing. That depends on the temperature reached and the atmosphere during firing as well as iron content. For raw materials that are used for white products, for example porcelain, degree of whiteness after firing is of critical importance. Even trace amounts of iron degrades whiteness. The whiteness of all samples of Lampang clay is shown in Figure 3.26.

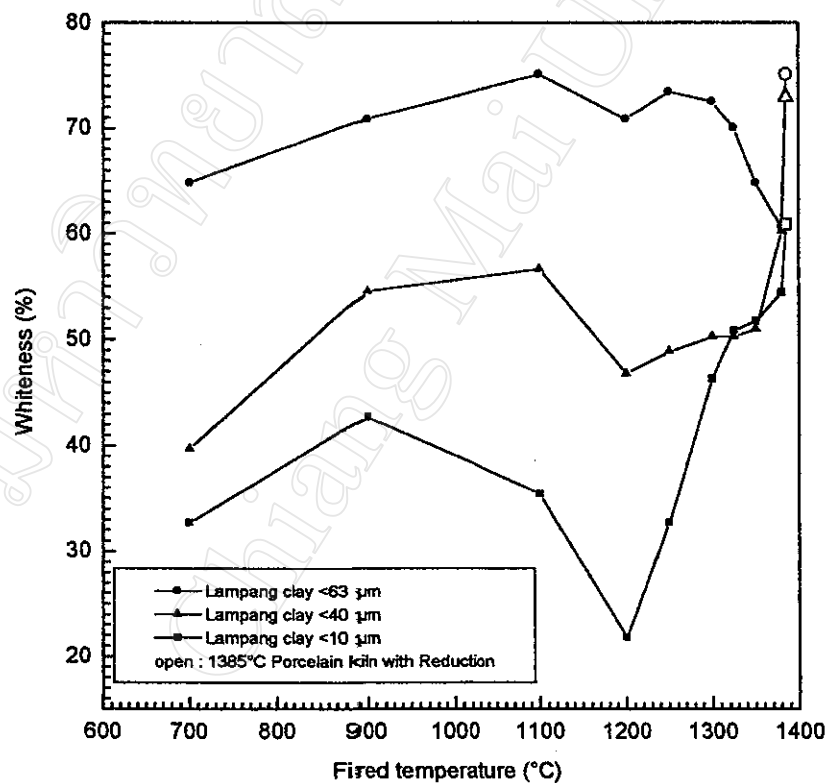


Figure 3.26 : Whiteness of Lampang clay after firing.

Figure 3.26 shows the whiteness of Lampang clay fractions after firing to different temperatures in an oxidation atmosphere with an electric kiln. Whiteness achieved by firing to  $1385^{\circ}\text{C}$  in a reduction atmosphere in a gas kiln is also shown. The whiteness of the test pieces in the oxidation and the reduction firing correlates well with  $\text{Fe}_2\text{O}_3$  content. The temperature of approximately  $1000^{\circ}\text{C}$  causes a brownish color in all test pieces through formation of  $\text{Fe}_3\text{O}_4$ . Through reduction to  $\text{FeO}$  above  $1200^{\circ}\text{C}$  the whiteness increases again. Using reduction firing above  $1100^{\circ}\text{C}$ , completes  $\text{FeO}$  reduction and results in 65 - 70 % improvement in whiteness.

### 3.3.13.6 Scanning electron microscope (SEM)

The SEM can be used as one more line of investigation to determine the behavior of ceramic materials. SEM micrographs of each sample after firing have been taken. Figure 3.27-3.29 show the Lampang clay sample C (particles < 63 microns), sample M (particles < 40 microns) and sample F (particles < 10 microns). All samples were fired to  $1350^{\circ}\text{C}$  in an electric kiln with an oxidation atmosphere.

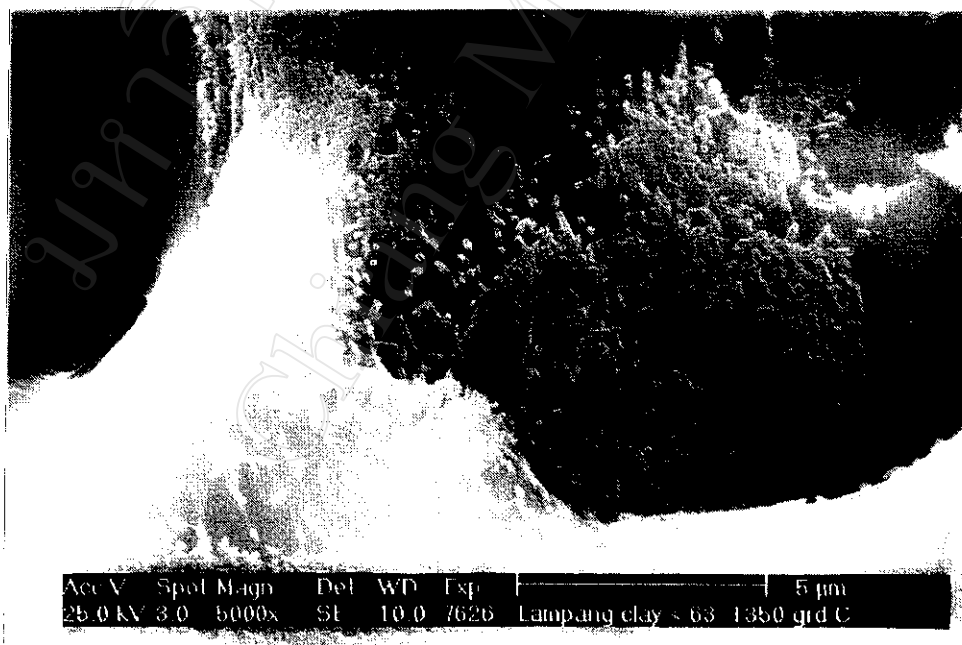


Figure 3.27: Scanning electron microscope of Lampang clay sample C (particles < 63 microns) fired to  $1350^{\circ}\text{C}$  at 3000x magnification.

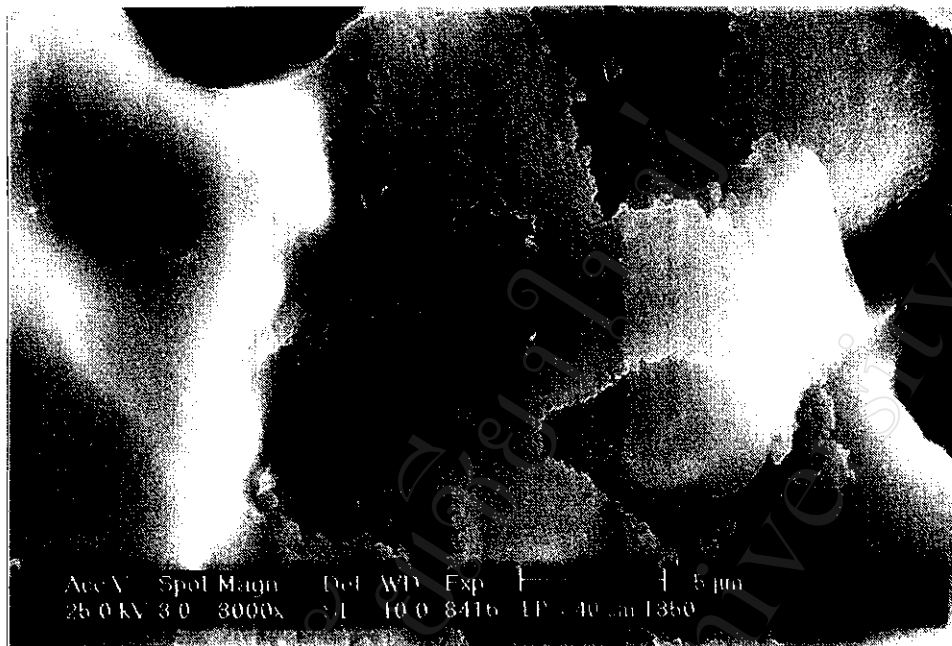


Figure 3.28: Scanning electron microscope of Lampang clay sample M (particles < 40 microns) after firing to 1350°C at 3000x magnification.

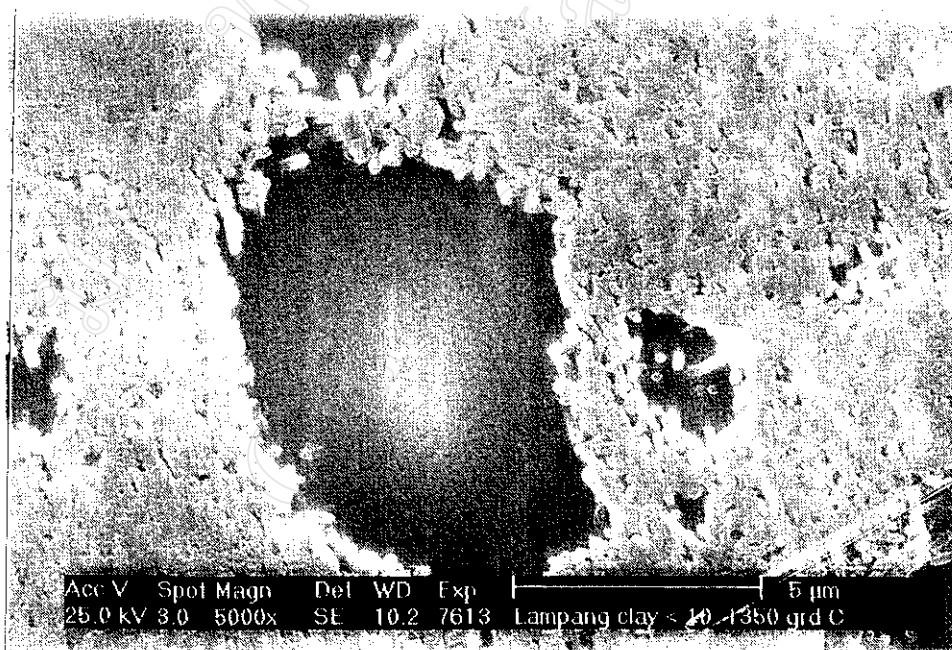


Figure 3.29: Scanning electron microscope of Lampang clay sample F (particles < 10 microns) after firing to 1350°C at 3000x magnification.

Scanning electron microscope pictures show that (see Figure 3.27) that secondary mullite is dispersed in a glassy phase and still has porosity. Figure 3.28 shows more secondary mullite than Figure 3.27 and that there is still porosity and quartz particles in material. Figure 3.29 shows secondary mullite in a glassy phase.

### **Conclusion**

The sample M (particles < 40 microns) has the best firing behavior and best properties after firing. It can be used to make white ceramic products, for example, white stoneware or porcelain. A suitable firing temperature for this material is about 1380°C.

### **General conclusion**

From all the investigations of Lampang white clay, the fraction with particles < 40 microns has the best characteristics for use in making high grade ceramic bodies.

## **3.4 Options for the improvement of the quality of the white clay from Lampang.**

From the characteristics of Lampang clay it can be seen that the elimination of the finest fraction will greatly reduce the amount of iron impurity but how this will affect other properties is not yet known. More study is needed. The possible use of chemical and magnetic treatments were also investigated.

### **3.4.1 Physical refinement; separation by particle size<sup>30-54</sup>**

#### **3.4.1.1 Wet vibration sieve screen**

A first and easy method for classification and separation of the clay particles and quartz sand smaller than 40 microns is the wet vibration sieve screen shown in Figure 3.30.

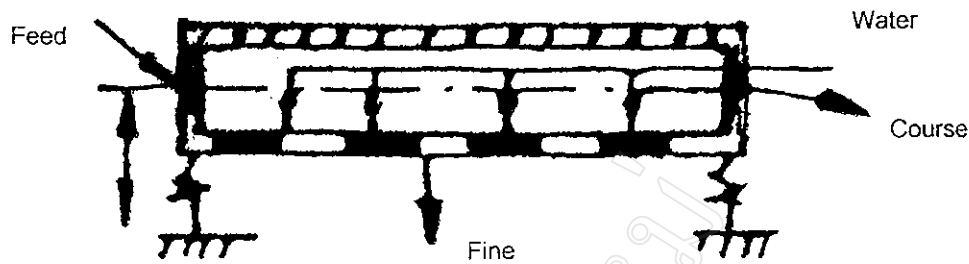


Figure 3.30 : Scheme of wet vibration sieve screen<sup>30</sup>

The idea is that raw clay will be suspended in water in a mixing tank, coarse quartz sand will separate out by sedimentation and a screw conveyor will remove the coarsest material. The slurry can be delivered to a vibration sieve tower. The smallest sieve size to be used is 40 microns which will pass all particles smaller than 40 microns including the finest particles. This method is easy and does not require the use of any technology which might be difficult to manage. This is the cheapest method of separation and is easy to control, but the sieve screens are easily destroyed or damaged with the result that the quality of the product may not be consistent and probably would not be suitable for large-scale productions. If the sieve screen has a defect, it will pass coarse particles, with a corresponding loss of quality.

#### 3.4.1.2 Wet tunnel sieve screen

A second easy and better method to separate clay particles and quartz sand is the wet tunnel sieve screen of the kind shown in Figure 3.31.

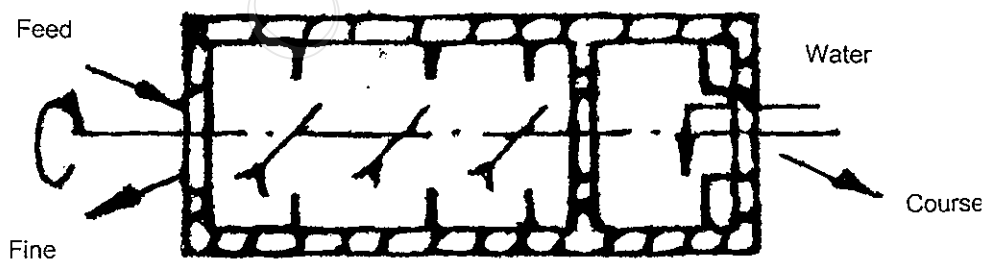


Figure 3.31 : Scheme of wet tunnel sieve screen<sup>30</sup>

The process with the tunnel screen is basically similar to that of the tower screen. Raw clay is suspended with water in a mixing tank. Coarse quartz sand is withdrawn by a screw conveyor. The raw clay without the coarsest particles is fed to a multiple layer tunnel sieve. Raw Lampang clay can be separated into fractions by particle sizes by means of sieve of different sizes. The smallest sieve size used should be 40 microns and the will yield is all particles smaller than 40 microns. This method, is still easy and does not depend on high technology. Production is continuous. But here we have the same problem, namely, these fine sieve screens are easy to destroy. The quality exported is not good enough for large industrial production.

### 3.4.1.3 Hydrocyclone

A third process that is better than sieving is the hydrocyclone, as shown in Figure 3.32 and Figure 3.33.

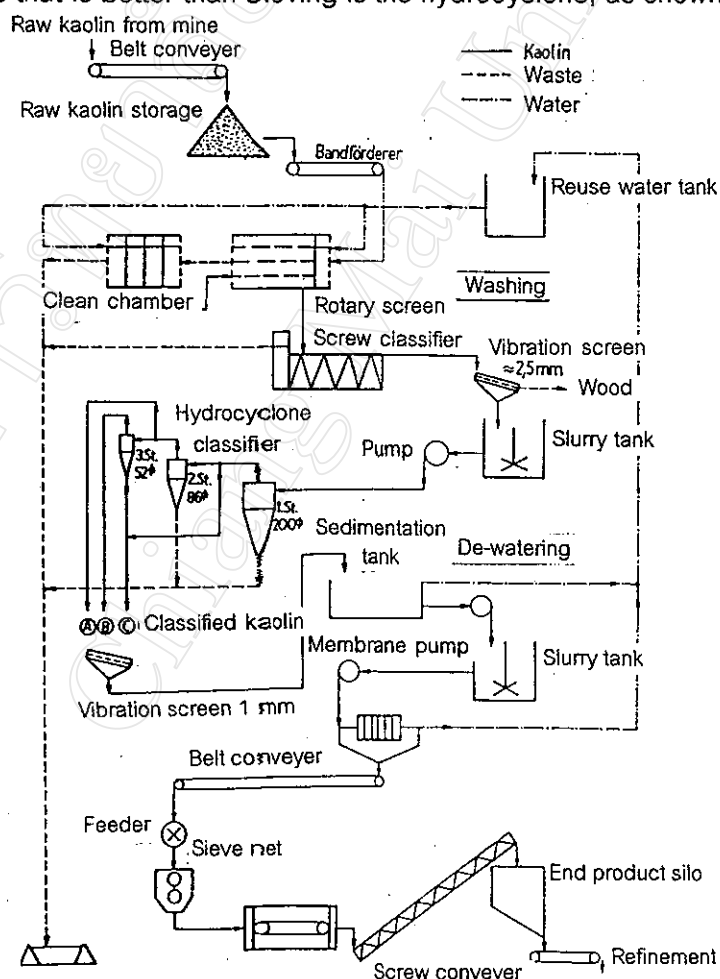


Figure 3.32 : Schematic diagram of the technological process for Kaolin mine in

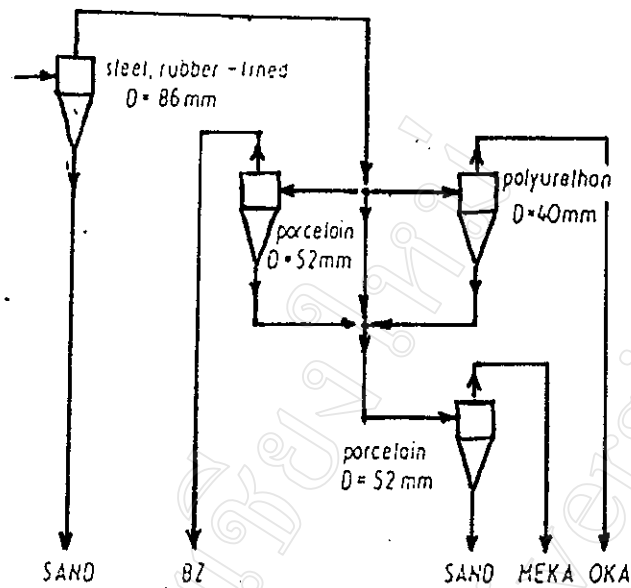


Figure 3.33 : Schematic diagram of kaolin classification by means of hydrocyclones

Figure 3.32 shows the technological process for kaolin production in Germany. Raw kaolin from the mine is transported in various loads. These are blended to insure consistency. The blended raw kaolin is transported to a tunnel sieve. Water is added to separate out any big stones or wood. The output from the tunnel sieve is passed through a sedimentation tank which separates the coarse sand. The remaining slurry flows to a coarse vibration sieve screen (2.5 mm size) to separate any remaining wood from clay slurry. The clay slurry then flows to a mixing tank and is pumped to the first hydrocyclone, which is about 200 mm diameter. This separates sand and most minerals from the clay. The clay slurry overflow may be called C-grade product. Then it is pumped to a secondary hydrocyclone one with 86 mm diameter. Fine quartz sand is removed here. The improved slurry, call it B-grade, is pumped to a third hydrocyclone with 52 mm diameter. This third hydrocyclone separates illite and mica. The overflow is called A-grade and is the best quality kaolin that is produced at that factory. A multi cyclone process could be used to refine Lampang clay.

In Figure 3.33 multiple-stage of kaolin classification by hydrocyclone as used in Germany is shown. After washing in a screw washer, kaolin is subjected to multiple-stage hydrocyclone classification as illustrated in Figure 3.33 resulting in three grades of kaolin.

### 3.4.2 Chemical Treatment<sup>54-56</sup>

In the research reported here the iron oxide in various fractions of Lampang clay was measured. Table 3.7 gives the results of this study.

Table 3.7 : Percentage of iron oxide form in samples of Lampang clay

	Raw clay (%Fe <sub>2</sub> O <sub>3</sub> )	%Fe <sup>2+</sup> (with HF/H <sub>2</sub> SO <sub>4</sub> )	%Fe <sup>3+</sup> (Fe(total)-Fe <sup>2+</sup> )	%Fe-Ion on particle Surface (Dithionit Method)
< 63 micron	0.60	1.25%(0.0075)	98.75%(0.5925)	58.0% (0.3480)
< 40 micron	0.69	1.70%(0.0117)	98.30%(0.6783)	65.0% (0.4485)
< 10 micron	1.09	1.45%(0.0158)	98.55%(1.0742)	73.0% (0.7957)

Raw clay (%Fe<sub>2</sub>O<sub>3</sub>) = % of the total amount Fe<sub>2</sub>O<sub>3</sub> found in the raw clay

%Fe<sup>2+</sup> (with HF/H<sub>2</sub>SO<sub>4</sub>)<sup>56</sup> = % of total Fe that is Fe<sup>2+</sup>

%Fe<sup>3+</sup> (Fe(total)-Fe<sup>2+</sup>) = % of total Fe that is Fe<sup>3+</sup>

Note that iron in any oxidation state is an unwanted impurity that degrades the color of white clay when it is fired. From Table 3.7 we see that iron oxide contamination of the fine fraction of Lampang clay is greatest. With the HF/ H<sub>2</sub>SO<sub>4</sub> method<sup>56</sup> the %Fe<sup>2+</sup> is found. Trace amount of Fe<sup>2+</sup> are found in all samples of Lampang clay. We can assume that Fe(total) - Fe<sup>2+</sup> = Fe<sup>3+</sup>. Most of the iron oxide contamination is in form Haematite (Fe<sub>2</sub>O<sub>3</sub>)<sup>54</sup> but that amount is too small to detect and identify by means of X-ray diffraction. The dithionit method<sup>56</sup> is used to analyze %Fe-Ion on the particle surface. It is found that more than 50 % of the iron oxide is attached to the surface of the clay particles and is



greatest in the fine particle fraction. This result shows that most of iron oxide in natural Lampang clay is in the 3+ oxidation state, (probably Haematite ( $\text{Fe}_2\text{O}_3$ )) and is attached to the surface of the clay particles. <sup>54</sup>Haematite also has magnetic properties and possibly can be separated from Lampang clay by means of a high field magnetic separator. If this is possible, then Lampang clay can be greatly improved at very little cost. This interesting possibility is theoretical but worth investigating.

<sup>54</sup>However, purification of clay by chemical means is not possible with any simple and low cost scheme. Chemical refinement is difficult to control, it is expensive, and without elaborate precautions results in the introduction of new impurities. Chemical refinement of clay is not recommended. If the clay is so impure, then it could be better to start with raw material from some other source.

### 3.4.3 Magnetic Treatment<sup>54</sup>

The removal of the iron impurity by the use of a high field magnetic separator, especially the fraction of < 40 microns which has other desirable properties, has been explored. This fraction was processed with a magnetic separator of 5 T Magnetic field. Slurry was processed at a rate of 1 cm/s and a processing time of 60 seconds. Results are shown in Tables 3.8 to 3.10.

Table 3.8 : Chemical composition of treated Lampang clay sample M.

	$\text{SiO}_2$	$\text{Al}_2\text{O}_3$	$\text{Fe}_2\text{O}_3$	$\text{TiO}_2$	$\text{CaO}$	$\text{MgO}$	$\text{K}_2\text{O}$	$\text{Na}_2\text{O}$	L.O.I.
Original material	69.52	19.69	0.69	0.11	0.07	0.16	5.34	0.79	3.63
Passed through the separator	68.30	21.60	0.50	0.03	0.07	0.17	4.93	0.43	3.97
Retained by the separator	66.20	21.22	2.28	0.12	0.08	0.24	5.02	0.34	4.50

Table 3.9 : Mineral composition and whiteness of treated Lampang clay sample M.

	Quartz	Feldspar	Calcite	Illite	Kaolinite	Anatas	%Whiteness
Original material	49.0	3.0	2.0	29.0	16.0	1.0	48.11
Passed through the separator	47.0	3.0	2.0	30.0	17.0	1.0	61.11
Retained by the Separator	44.0	3.0	2.0	31.0	19.0	1.0	28.88

Table 3.10 : Whiteness after firing of M sample showing differences of material before and after magnetic processing. Different test pieces have been fired to different temperatures.

Temperature, °C	700	900	1100	1200	1250	1300	1325	1350	1380	1385/RF
Before treatment	68.5	63.8	53.7	54.7	56.0	54.2	55.6	56.1	59.9	74.5
After treatment	73.4	73.3	75.1	71.9	69.7	67.4	69.3	69.4	70.8	81.1

If the M sample of Lampang clay is passed through a magnetic separator, the amount of  $\text{Fe}_2\text{O}_3$  decreases, however, not all of the iron can be removed by this method (see Table 3.8). Table 3.9 shows only a small effect on mineral composition, the amount of quartz is decreased; illite and kaolin are increased. This can be explained by the iron oxide being preferentially attached to the surface of illite and kaolin plates. Iron oxide is still a contaminant. Some of the iron content is removed by this method. Table 3.9 also shows that whiteness changed with the partial removed of iron. Table 3.10 shows changes in whiteness after firing of raw < 40 microns fraction. This removal of some of the iron increases the whiteness significantly. In each instance, processing with a magnetic separator improves whiteness. The whitest sample is obtained with reduction firing.

#### 3.4.4 Preliminary conclusions

If we need higher quality material, we can use a magnetic separator in the process and hydrocycones. Chemical refinement is not suitable for Lampang clay because this method is expensive and chemicals use in the process contaminate the clay and change its properties. However, all results so far are laboratory scale. In the future, pilot plant scale might be studied.

### 3.5 Use of refined materials for the production of porcelain and other new types of ceramic products.

This study indicates that the middle size fraction is most suitable for ceramic production. Properties are improved by rejecting the fine particles of Lampang clay.

#### 3.5.1 Use of Lampang clay for the production of porcelain

The M sample of Lampang clay has been selected as being the most suitable for making porcelain body, but the mineralogical composition is deficient in clay-mineral content. Kaolin must be added. For this purpose we use a high grade of kaolin. Thus, the composition of a mixture of natural Lampang clay and ground Lampang stone can be changed. Some properties of kaolin from Germany and Lampang stone are given in Appendix II. Table 3.11 compares the composition (mineral and the chemical) of various commercial porcelain bodies. (See appendix III). Porcelain of various thickness produced by casting are shown in Table 3.14. Both of two experimental compositions increase in thickness at suitable rates. Figures 3.34 and 3.35 show the secondary mullite suspended in glassy phase and some grains of quartz. Both are porous and show cracks between quartz grains and the glassy phase due to quartz inversion during the cooling period process. The test pieces of both experimental porcelain compositions were fired to 1385°C in a reduction atmosphere. The fired ware, formed by casting technique, are shown in Figure 3.36, (left is P-1 and right is P-2).

### 3.5.1.1 Possible porcelain formulations

Two possible porcelain formulations using Lampang clay and ground Lampang stone are shown in Table 3.11.

Table 3.11 : Possible porcelain formulas

Sample	Lampang clay < 40 micron	Lampang stone < 100 micron	Kaolin
P-1	50 %	40 %	10 %
P-2	40 %	40 %	20 %
P-3	60 %	40 %	-
P-4	50 %	50 %	-

German Kaolin was used, Grade medium called MEKA Kaolin

All samples can be forming by casting technique, but samples P-3 and P-4 are deformed when fired. Only P-1 and P-2 are observed for other properties.

The mineral composition and chemical composition of both possible porcelain samples are shown in Table 3.12 and 3.13.

Table 3.12 : Mineral composition of both possible porcelain formulas.

Sample	%Quartz	%Feldspar	%Kaolinite	%Calcite	%Illite
P-1	40	10	17	3	30
P-2	38	12	22	3	25

Table 3.13 : Chemical composition of both possible porcelain formulas

Sample	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	CaO	MgO	K <sub>2</sub> O	Na <sub>2</sub> O	L.O.I.
P-1	70.6	19.3	0.6	0.0	0.2	0.2	4.0	1.3	3.8
P-2	70.1	19.9	0.5	0.1	0.2	0.2	3.3	1.2	4.5

### 3.5.1.2 Properties of porcelain types

From Table 3.11, Test pieces formed by pressing the plastic clay and by slip casting were made from both porcelain mixtures. The thickness of test pieces with casting technique are shown in Table 3.14.

Table 3.14 : Body forming from slurry by cast shaping

minute	1	2	3	4	5	6	8	10	15
P-1, cm	0.34	0.48	0.50	0.61	0.69	0.72	0.85	0.93	1.18
P-2, cm	0.26	0.36	0.41	0.46	0.52	0.55	0.67	0.76	0.99

Formed samples were fired in a laboratory kiln under an oxidation atmosphere with different firing temperatures. The heating rate was 5 °K/min and with soaking 30 minutes at the final temperature. In the same way, the same samples were fired in an industrial porcelain kiln under a reduction atmosphere and other porcelain firing conditions. The total firing time in the industry porcelain kiln is 4.5 hours with final temperature of 1370 °C. Sample properties, such as shrinkage on firing, bending strength and whiteness after firing are shown in Table 3.15 – 3.17.

Table 3.15 : Shrinkage of both possible porcelain formulas

Temp., °C	Dry	110	500	700	900	1100	1200	1250	1300	1350	1380
P-1, %	1.6	3.8	3.6	2.0	0.6	7.2	12.0	14.2	14.4	13.2	12.2
P-2, %	-	3.8	4.0	3.2	2.8	7.4	13.2	14.8	14.4	13.0	11.8

Table 3.16 : Bending strength of both possible porcelain formulas

	Oxidation atmosphere											
Temp., °C	Dry	110	500	700	900	1100	1200	1250	1300	1350	1380	1370,RF
P-1, N/mm <sup>2</sup>	0.8	1.0	1.9	1.6	2.9	23.7	29.6	75.2	53.2	20.8	20.2	56.9
P-2, N/mm <sup>2</sup>	0.7	0.9	1.3	1.2	2.3	22.6	31.4	74.1	57.7	18.3	17.8	61.5

Table 3.17 : Whiteness after firing of both possible porcelain formulas

Oxidation atmosphere										
Temp., °C	700	900	1100	1200	1250	1300	1325	1350	1380	1370,RF
P-1, %	75.8	53.5	62.5	53.7	55.1	63.6	68.0	68.8	70.9	79.5
P-2, %	75.8	60.6	68.4	60.3	60.2	63.4	67.4	70.6	73.5	81.4

Table 3.15 and 3.16 show that shrinkage depends on temperature. Under an oxidation atmosphere at 1250 °C the test pieces reach maximum density (the sintering condition) but the test pieces still have open porosity. Over 1250 °C the density again decreases because oxygen is produced and this free gas expands forming gas bubbles in the glassy body and porosity increases. With a reducing atmosphere, the oxidation of  $\text{Fe}^{2+}$  to  $\text{Fe}^{3+}$  is retarded. The result is better properties, especially, increased whiteness and these are shown in Table 3.16 and 3.17. Under a reducing atmosphere, maximum whiteness is observed. Other properties of the porcelain test pieces are shown in Table 3.18.

Table 3.18 : Density, Water absorption and phase composition after firing of two possibility porcelain formulas, reduction atmosphere, industrial kiln.

	Density g/cm <sup>3</sup>	Water absorption %	Phase composition	
			%Mullite	%Quartz
P-1	2.25	1.4	14	6
P-2	2.45	0.3	18	7

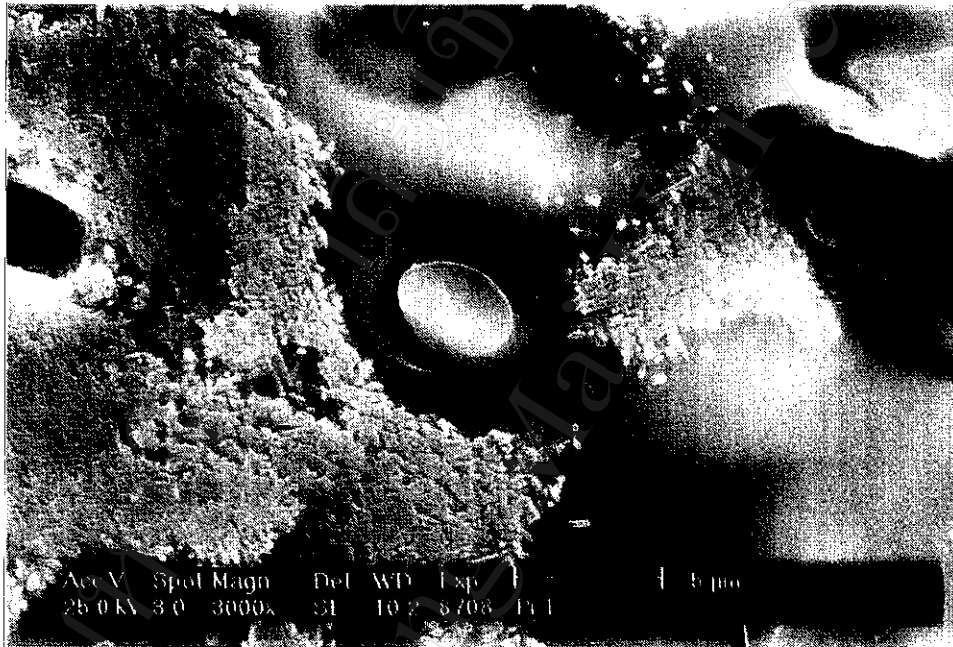


Figure 3.34 : Scanning electron microscope of sample P-1 after firing to 1370°C in a reducing atmosphere

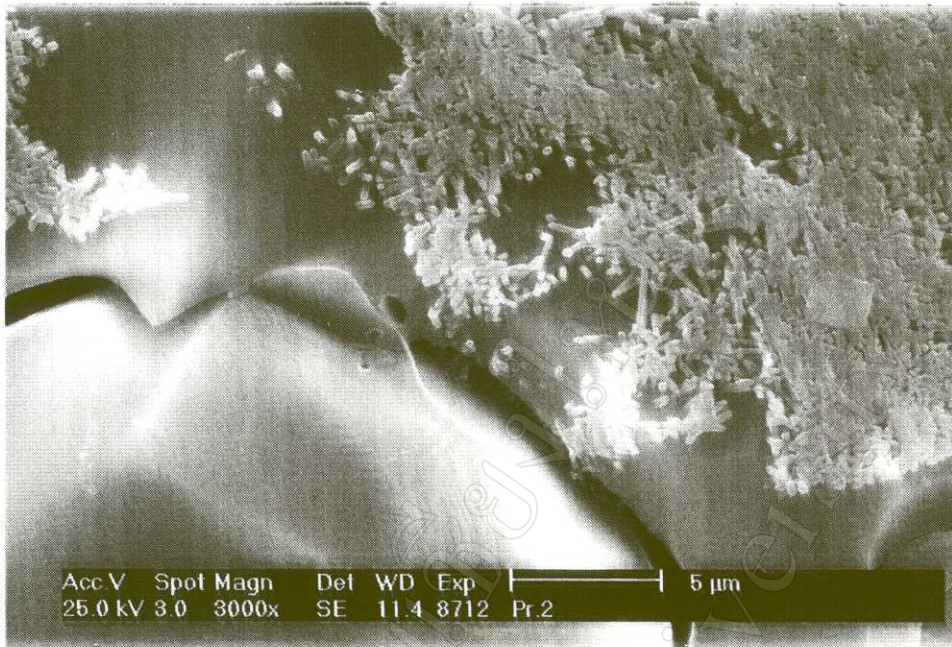


Figure 3.35 : Scanning electron microscope of sample P-2 after firing to 1370°C in a reducing atmosphere

From Table 3.18 it can be said that the P-2 mixture has more mullite and other better properties than mixture P-1. The properties and scanning electron microscopes shows that both mixtures are porcelain bodies.



Figure 3.36 : Sample crucibles of both porcelain compositions (fired to 1370°C in a reducing atmosphere) are shown. The crucible on the left is P-1 and the one on the right is P-2.



### 3.5.1.3 Conclusion

The chief result of this study of Lampang clay is the finding that the fraction < 40 microns with additional kaolin can be used for the production of a white porcelain body. The Lampang clay with particles < 40 microns is entirely satisfactory for porcelain production. But a whiter body is likely if the fines (< 10 microns) are rejected. All of this work had been done at the bench scale. Any industrial applications should come after pilot plant scale studies. In order to make the best use of these results, the technology must be kept simple and low cost.

### 3.5.2 Application for floor tile<sup>71</sup>

The fraction of Lampang clay with particles greater than 40 microns but not greater than 160 microns can be used to produce ceramic floor tile. The fraction 40 - 160 microns accounts for about 30 % of raw Lampang clay. Thus, finding a suitable use of this fraction is necessary.

#### 3.5.2.1 Possible floor tile formulations

Formulations for possible floor tile production from Lampang clay are shown in Table 3.19. In the ceramic floor tile body, Lampang clay of 40 - 160 microns, ground Lampang stone and Maetan clay have been used to insure that forming and sintering properties are satisfactory.

Table 3.19 : Some floor tile formulations

Sample	Lampang clay 40-160 micron	Lampang stone > 100 micron	Maetan clay
T-1	70 %	30 %	-
T-2	80 %	20 %	-
T-3	40 %	30 %	30 %
T-4	50 %	20 %	30 %
T-5	30 %	30 %	40 %

### 3.5.2.2 Properties of floor tile types

The floor tile clay was mixed with 3 % water and then pressed with pressure about 20 MPa to make plate shaped test pieces (1.5 cm width x 2.0 cm length x 0.5 cm thick). Test pieces were fired in a laboratory kiln to different final temperatures, a heating rate of 5 degrees K/minute and 30 minute soaking at the final temperature was used. Properties of all test pieces are shown in Table 3.20 to 3.23.

Table 3.20 : % Water absorption after firing

sample	T-1	T-2	T-3	T-4	T-5
at 1150°C	12.7	12.9	14.5	14.3	-
at 1200°C	17.8	16.2	13.9	12.4	6.3
at 1250°C	-	-	-	-	4.1

Table 3.21 : % Open porosity after firing

sample	T-1	T-2	T-3	T-4	T-5
at 1150°C	23.7	23.6	27.1	26.5	-
at 1200°C	32.4	29.5	26.5	24.0	14.0
at 1250°C	-	-	-	-	9.5

Table 3.22 : Bulk density after firing (g/cm<sup>3</sup>)

sample	T-1	T-2	T-3	T-4	T-5
at 1150°C	1.87	1.83	1.86	1.86	-
at 1200°C	1.83	1.82	1.90	1.93	2.21
at 1250°C	-	-	-	-	2.30

Table 3.23 : Bending strength after firing ( $\text{N/mm}^2$ )

sample	T-1	T-2	T-3	T-4	T-5
at $1150^\circ\text{C}$	5.61	4.79	5.00	4.21	-
at $1200^\circ\text{C}$	6.42	6.86	8.38	4.82	22.17
at $1250^\circ\text{C}$	-	-	-	-	30.94

The best composition for floor tile using Lampang clay with particles between 40-160 microns appears to be composition T-5. This composition has better properties than other samples and  $1250^\circ\text{C}$  seems to be the best final temperature compared with the standard temperatures for firing floor tile as seen in appendix IV. But all properties probably can be improved by grinding before mixing.

### 3.5.2.3 Conclusion

The fraction of 40-160 microns from Lampang clay can be used for floor tile.

### 3.5.3 Conclusion regarding the use of refined materials

Refined materials from Lampang clay can be classified in two parts,  $< 40$  microns and material between in the 40-160 micron range. Part of  $< 40$  microns can be used to make porcelain products. Part of 40-160 microns can be use for floor tile. More study is desirable in both instances.