#### CHAPTER IV

#### RESULTS AND DISCUSSIONS

### 4.1 General

In order to determine the factors affecting fineness of mill base, a total of 45 experiments were conducted. Process variables which were considered to be the main factors were mixing time, fluid viscosity and grinding flow rate. The experiments were separated into two main steps: mixing and grinding.

After all raw materials were weighted at predetermined composition depending on specified formula, they were transferred into stirred mixing tank. The fineness of mill base at the start of the mixing process is usually formed to be higher than 100 microns. The raw materials are mixed at three mixing times of 60, 90 and 120 minutes. Viscosity of mill base was adjusted in mixing tank by addition of Solvesso 150. Five viscosity values of 70, 72, 74, 76 and 85 KU were selected. Samples were taken at the end of mixing to analyze for viscosity and fineness.

After each batch mixing the mixture was transferred to grinding step where it was circulated between receiving tank and grinding machine until fineness of mill base was under 10 microns. The grinding machine is operate at three grinding flow rates of 16, 18 and 20 kg./min. After two hours of grinding, samples were taken every one hour to analyze fineness.

### 4.2 Instrumental and Experimental Error

### 4.2.1 Instrumental Error

Grind gauge meter used for measurement of fineness is checked for its accuracy by conducting the measurement of the same sample twice. The results are

shown in Table 4.1. It is observed that the measured value of fineness can present an error up to  $\pm 12.5\%$ .

Table 4.1 Precision of fineness measurement of mill base

Instrumental Error						
sample	fineness of mill base (microns)					
	test-1	test-2	average	% error		
1	15	17	16±1	±12.5		
2	14	15	14.5 <u>+</u> 0.5	<u>+</u> 6.9		
3	13	12	12.5±0.5	<u>±</u> 8		
4	10	10	10±0	0		
5	9	• 10	9.5 <u>±</u> 0.5	<u>+</u> 10.53		

## 4.2.2 Experimental Error

Several experiments were conducted twice to find possible deviation in experimental results. The results are summarized in Table 4.2. It is observed that error in conducting experiments can be as high as  $\pm 10.53\%$ .

Table 4.2 Repeatability of experiment

Experimental Error						
experiment	fineness of mill base (microns)					
no.	batch-1	batch-2	average	% error		
1	15	15	15 <u>±</u> 0	0		
2	14	13	13.5 <u>+</u> 0.5	<u>+</u> 7.4		
3	13	12	12.5±0.5	<u>±</u> 8		
4	10	10	0 <u>+</u> 0	0		
5	9	10	9.5±0.5	±10.53		

### 4.3 Discussion

Experimental results obtained from each experiment are summarized in Appendix C. The main variable in this study are mixing time, viscosity, flow rate of grinding machine and grinding time. Each variable will be discussed separately.

### 4.3.1 Effect of mixing time

In mixing step, mixing time varies from 60, 90 to 120 minutes. In this step, many raw materials are mixed together such as pigment, resin, solvent by using high speed dispersers. After mixed, sample of paint is determined fineness by grind gauge meter. The experimental data from Appendix C can be plotted in graph and shown from Figures 4.1 to 4.5.

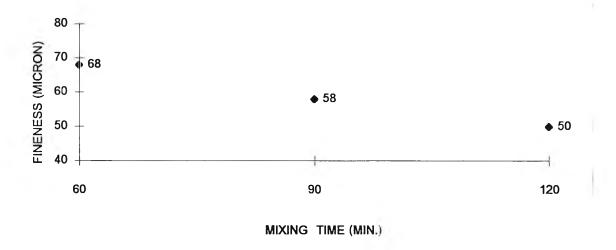


Figure 4.1 Effect of mixing time before grinding at viscosity 70 KU

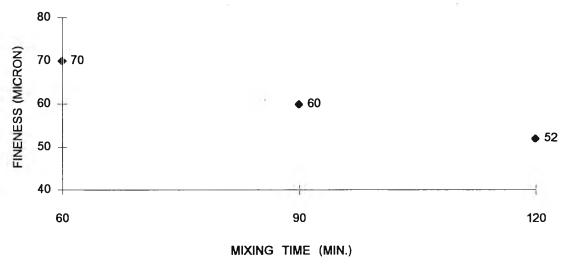


Figure 4.2 Effect of mixing time before grinding at viscosity 72 KU

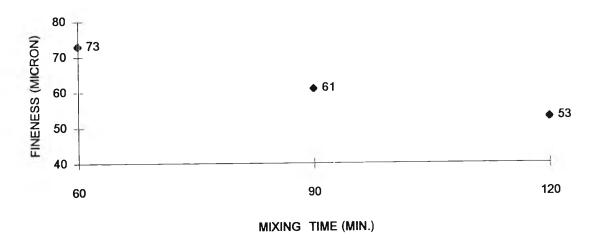


Figure 4.3 Effect of mixing time before grinding at viscosity 74 KU

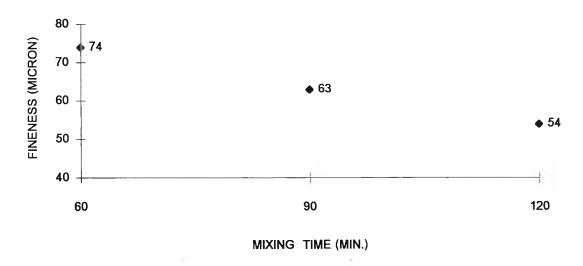


Figure 4.4 Effect of mixing time before grinding at viscosity 76 KU

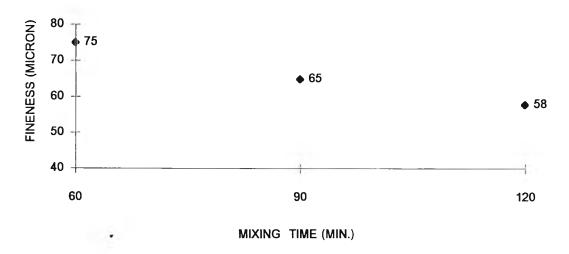


Figure 4.5 Effect of mixing time before grinding at viscosity 85 KU

Graph of mixing time (minutes) versus fineness (micron) are prepared on normal co-ordinates. All correlation dot as shown in Appendix C are obtained at Figures 4.1 to 4.5, which show that a longer mixing time generates better fineness of paint than a shorter mixing time. The data showed the same result as of W. Carr and A. Kelly experiment.

W. Carr and A. Kelly [8] studied factors which affect the efficiency of sand grinding (sand mill). Their study used pigment particle size measurements as the criterion of dispersion levels and grinding efficiency. The effect of many factors on the efficiency of sand grinding of decorative paint stainers is determined. W. Carr and A. Kelly used β-phthalocyanine blue and a long oil soya penta alkyd resin. The data which W. Carr and A. Kelly obtained from experiments showed that the pre-mixing stage was the fifth most significant parameter for grinding process. In this pre-mixing stage, pre-mixing time varied from 1 to 5 hours. The results shown that pre-mixing stage at high level was better. The highest color strength, k/s values, in the experiments was 1.020, is significant.

Figures 4.1 to 4.5 shown that, at the same viscosity, the fineness values of long mixing time was better than that of short mixing time. So, results of these experiments and the result of W. Carr and A. Kelly were consistency.

#### 4.3.2 Effect of viscosity

In mixing step, pigment, resin and solvent are charged to be stirred by propellers for dispersion in homogeneous paint. Solvent was raw material for adjusting viscosity according to the conditions before grinding step. The viscosity is varied from 70, 72, 74, 76 to 85 KU. Paint, which is adjusted viscosity, is passed to grinding process by grinding machine. After grinding process, sample of paint is determined fineness. The experimental data from Appendix C can be plotted in bar graphs and shown from Figures 4.6 to 4.14.

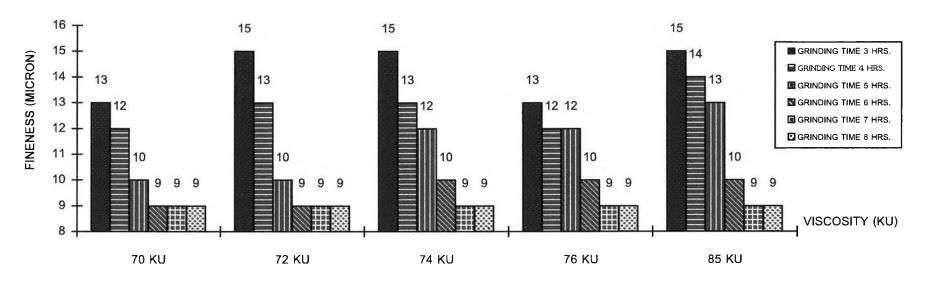


Figure 4.6 Effect of viscosity on fineness of mill base at mixing time 60 min., flow rate 16 kg./min.

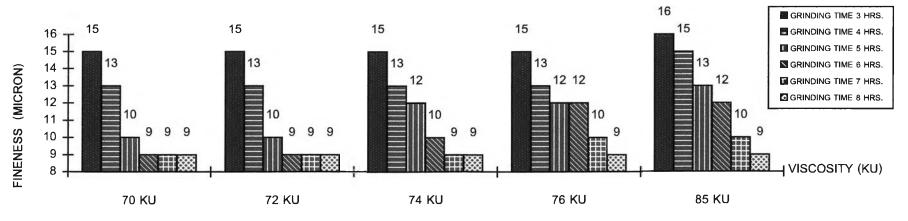


Figure 4.7 Effect of viscosity on fineness of mill base at mixing time 60 min., flow rate 18 kg./min.

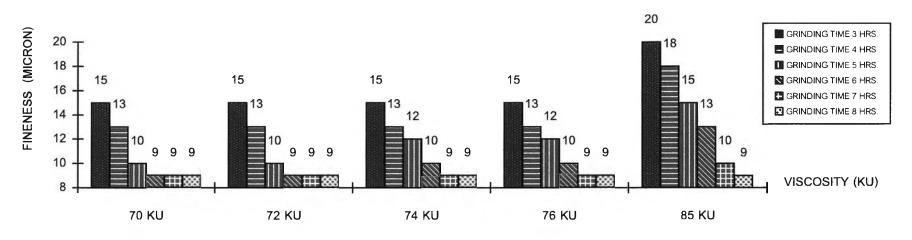


Figure 4.8 Effect of viscosity on fineness of mill base at mixing time 60 min., flow rate 20 kg./min.

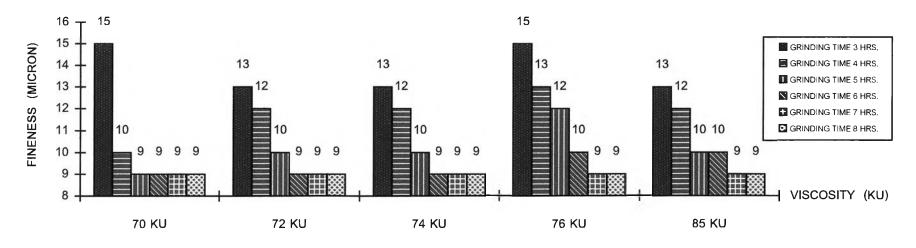


Figure 4.9 Effect of viscosity on fineness of mill base at mixing time 90 min., flow rate 16 kg./min.

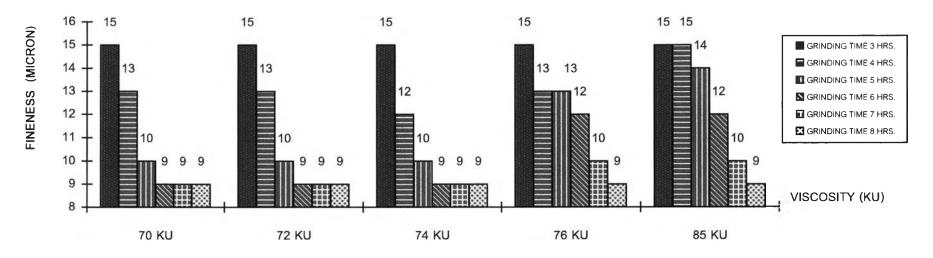


Figure 4.10 Effect of viscosity on fineness of mill base at mixing time 90 min., flow rate 18 kg./min.

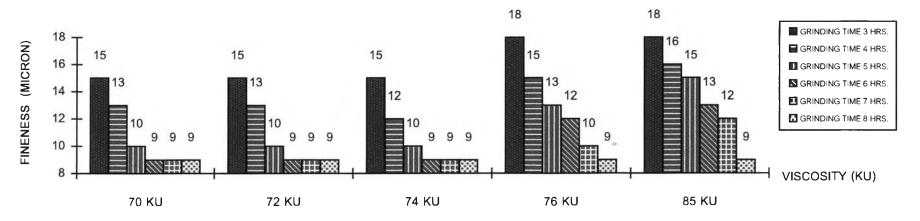


Figure 4.11 Effect of viscosity on fineness of mill base at mixing time 90 min., flow rate 20 kg./min.

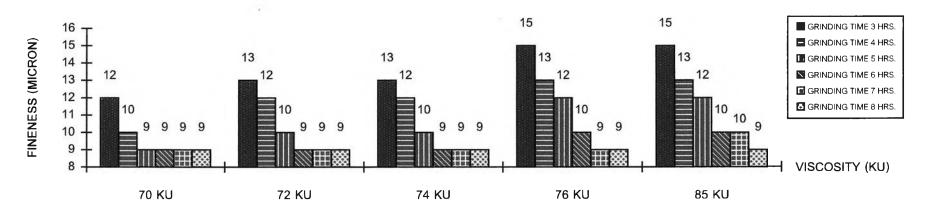


Figure 4.12 Effect of viscosity on fineness of mill base at mixing time 120 min., flow rate 16 kg./min.

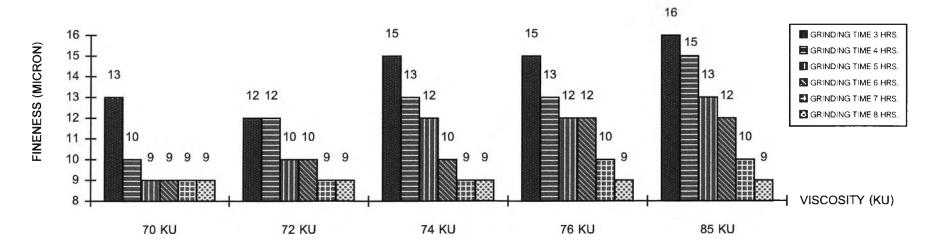


Figure 4.13 Effect of viscosity on fineness of mill base at mixing time 120 min., flow rate 18 kg./min.

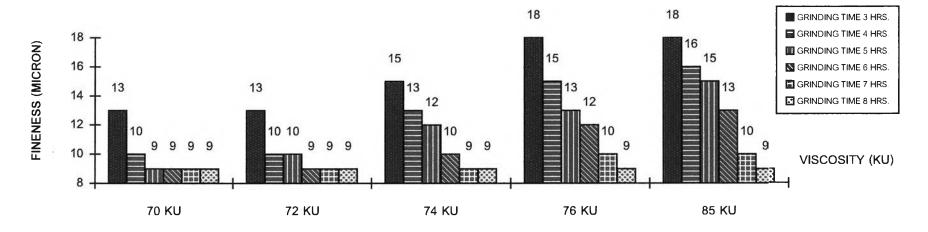


Figure 4.14 Effect of viscosity on fineness of mill base at mixing time 120 min., flow rate 20 kg./min.

Graphs plot viscosity (KU) versus fineness (micron). Figures 4.6 to 4.14 shown correlation that fineness of the low viscosity paint decreased better and faster in grinding process than the high viscosity one. (grinding time about 3-5 hours)

W. Carr [9] studied factors which affect the efficiency of ball milling. Mill base composition was an investigating factors which related to viscosity of paint before grinding. Viscosity was proportional to percentage of pigment when quantity of resin fixed. W. Carr obtained from his experiments that after grinding step, 15% pigment given 1.7 k/s value, and mean particle size was 0.280 microns. But 30% pigment given 1.21 k/s value, and mean particle size was 0.375 microns. Then the results of his study shown that the low viscosity gave the mean particle size.

Figures 4.6, 4.7, 4.9, 4.11, 4.14 shown that fineness of low viscosity paint decreased better and faster than high viscosity one after grinding process which related to the result of W. Carr. Nevertheless, in this experiment, the viscosity affected only on period of grinding time.

## 4.3.3 Effect of flow rate

After mixing process, paint is passed to grinding process by grinding machine. Flow rate, manually adjusted, of paint varied from 16,18 to 20 kg/min.. The experiment data from Appendix C plotted in graphs and shown from Figures 4.15 to 4.23.

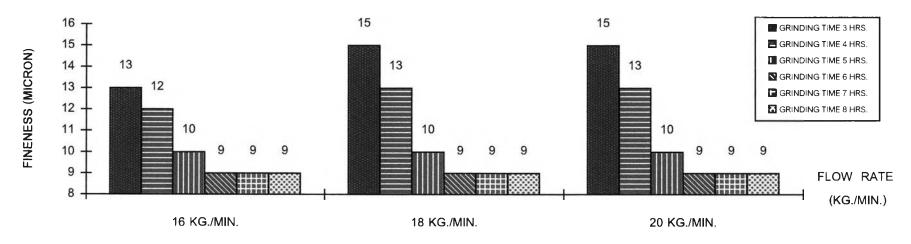


Figure 4.15 Effect of flow rate in grinding step on fineness of mill base at mixing time 60 min., viscosity 70 KU

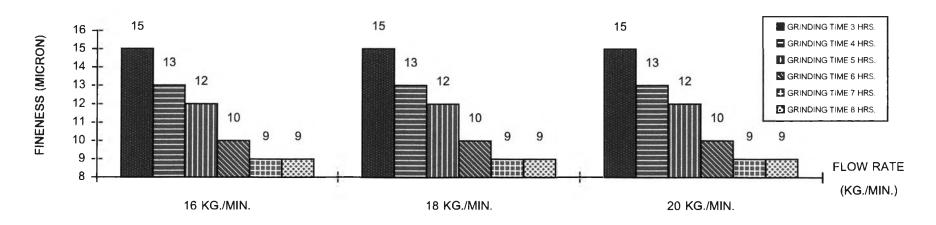


Figure 4.16 Effect of flow rate in grinding step on fineness of mill base at mixing time 60 min., viscosity 74 KU

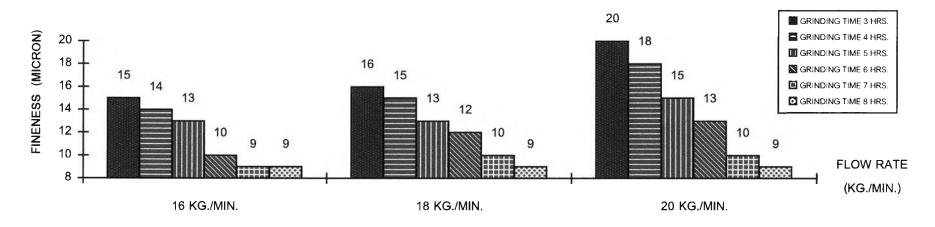


Figure 4.17 Effect of flow rate in grinding step on fineness of mill base at mixing time 60 min., viscosity 85 KU

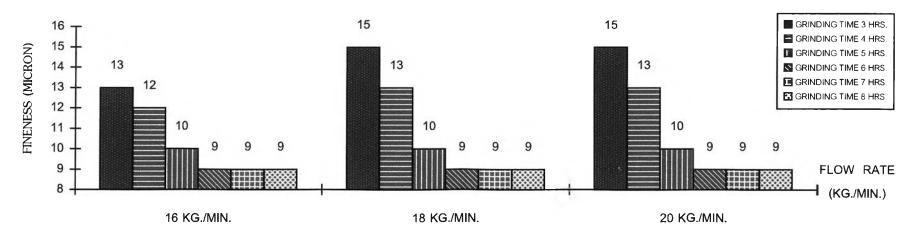


Figure 4.18 Effect of flow rate in grinding step on fineness of mill base at mixing time 90 min., viscosity 72 KU

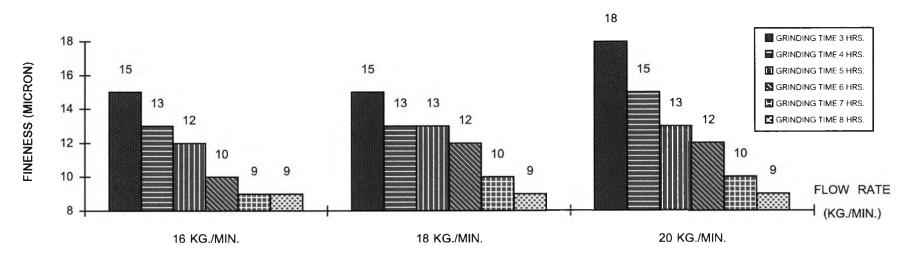


Figure 4.19 Effect of flow rate in grinding step on fineness of mill base at mixing time 90 min., viscosity 76 KU

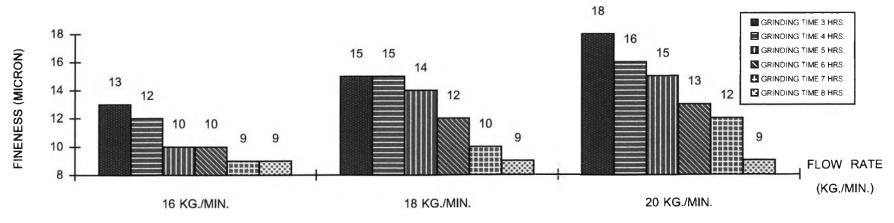


Figure 4.20 Effect of flow rate in grinding step on fineness of mill base at mixing time 90 min., viscosity 85 KU

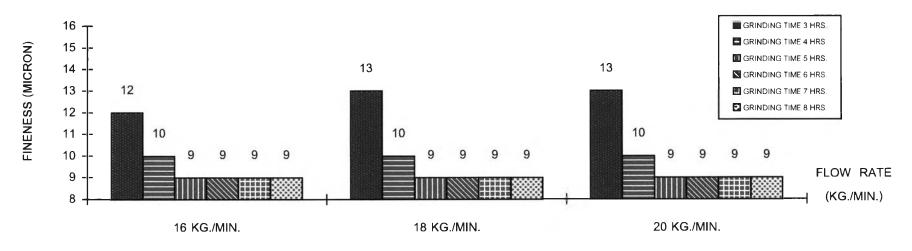


Figure 4.21 Effect of flow rate in grinding step on fineness of mill base at mixing time 120 min., viscosity 70 KU

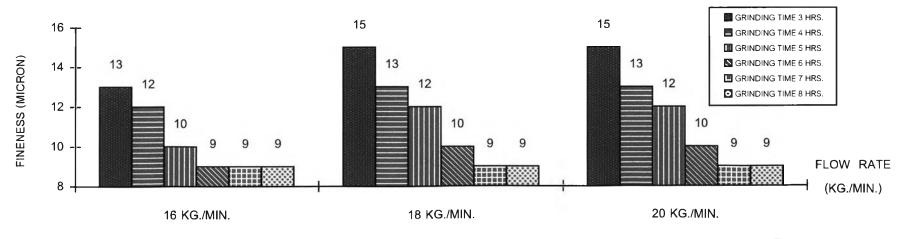


Figure 4.22 Effect of flow rate in grinding step on fineness of mill base at mixing time 120 min., viscosity 74 KU

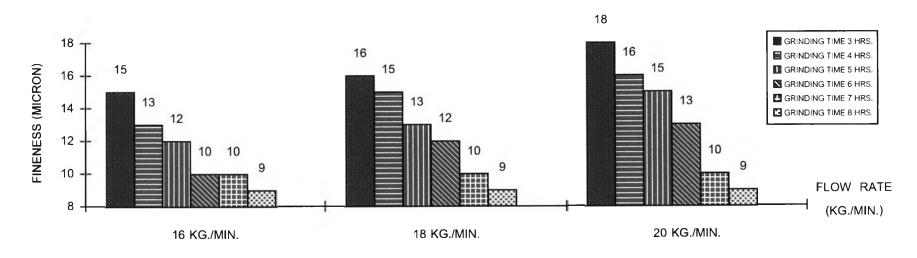


Figure 4.23 Effect of flow rate in grinding step on fineness of mill base at mixing time 120 min., viscosity 85 KU

From the previous tables, flow rate (kg/min.) versus fineness (micron) is plotted in bar graphs. Figures 4.15 to 4.23 show that flow rate hardly affects, or no effect, on fineness of paint in varied conditions of the experiments. In case of effecting as shown in Figures 4.17 and 4.20, fineness decreased at the low flow rate faster than at the high flow rate.

# 4.3.4 Effect of grinding time

For grinding process, paint is deaggregated by grinding machine. Grinding time is varied from 3, 4, 5, 6, 7 to 8 hours. After grinding process passed 2 hours, sampling is taken every 1 hour to determine fineness by using grind gauge meter. The experimental data in Appendix C shown from Figures 4.24 to 4.29.

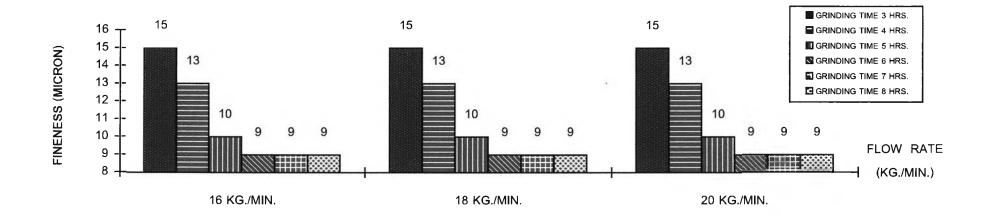


Figure 4.24 Effect of grinding time on fineness of mill base at mixing time 60 min., viscosity 72 KU

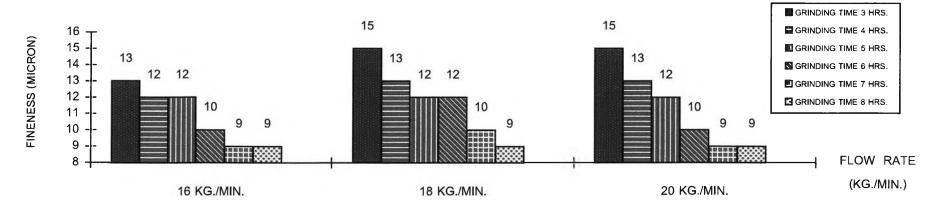


Figure 4.25 Effect of grinding time on fineness of mill base at mixing time 60 min., viscosity 76 KU

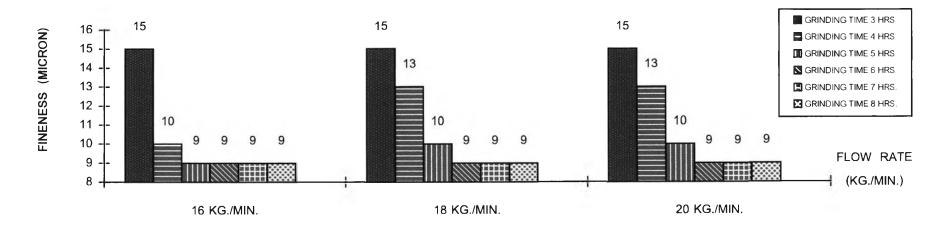


Figure 4.26 Effect of grinding time on fineness of mill base at mixing time 90 min., viscosity 70 KU

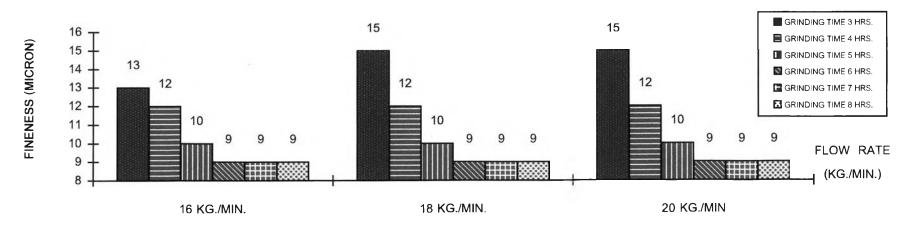


Figure 4.27 Effect of grinding time on fineness of mill base at mixing time 90 min., viscosity 74 KU

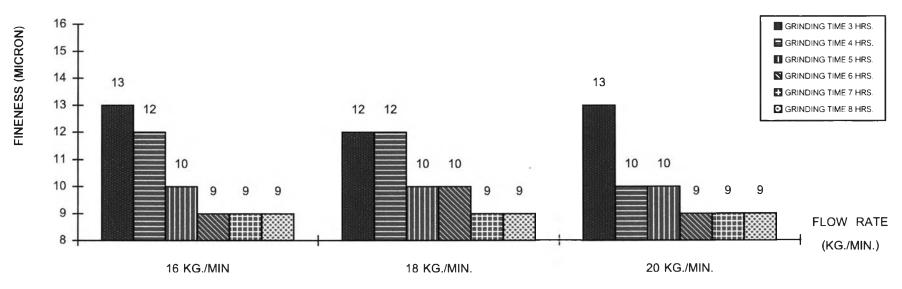


Figure 4.28 Effect of grinding time on fineness of mill base at mixing time 120 min., viscosity 72 KU

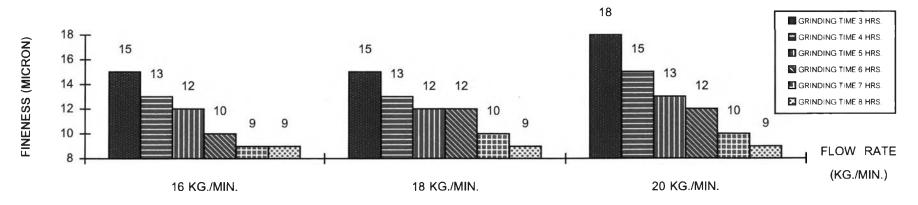


Figure 4.29 Effect of grinding time on fineness of mill base at mixing time 120 min., viscosity 76 KU

Graphs between flow rate (kg/min.) and fineness (micron) are plotted. Figures 4.24 to 4.29 shown that fineness was getting smaller subject to longer grinding time.

W. Carr [10] studied sand grinding versus ball milling in relation to pigment particle size. His study used three organic pigments to compare the relative efficiencies of ball milling and sand grinding. The Joyce-Loebl-ICI disk centrifuge is used to measure particle size distribution. The data which W. Carr has obtained from experiments has shown that from both sand mill and ball mill, if grinding time or milling time was longer, the fineness of pigment is low. However this effect achieved only a limited level of dispersion process.

Figures 4.24, 4.26, 4.27 shown consistency of effect from grinding time. When grinding time was long, the fineness of pigment was too low. This effect is called grinding time inverted to fineness. Nevertheless, the fineness decreased to a limit of dispersion, and this result was same as the result of W. Carr study.