

## CHAPTER 8

### EXPERIMENT FOR FINDING SUITABLE CONDITIONS

#### 8.1 Experiment and Data Collection

The two levels of melamine crystal pH, which are pH 8.0-8.7 and pH 8.7-9.5, and the three levels of F/M ratio, which are 1.6, 1.8, and 2.0, are fixed for the analysis of variance of the fixed effects model.

#### Curing time (in seconds)

		pH of Melamine Crystal			
		8.0-8.7		8.7-9.5	
F/M ratio	1.6				
	1.8				
	2.0				

Table 8.1 : The Table for Data Collection of the Experiment for Finding Suitable Conditions

And the eight observations, which are collected from testing in each condition as shown in Table 8.1, will be taken in random order of melamine crystal for the company's production in any three months, following the concept of completely randomized experimental design.

## 8.2 Collected Data

The collected data from the experiment are shown in Table 8.2.

Curing time (in seconds)

		pH of Melamine Crystal			
		8.0-8.7		8.7-9.5	
F/M ratio	1.6	246	253	232	219
		250	238	221	227
		236	242	230	216
		249	239	230	219
	1.8	195	210	183	188
		207	201	192	199
		207	208	185	196
		198	195	200	191
	2.0	198	186	168	176
		184	192	178	185
		183	195	183	180
		192	180	171	172

Table 8.2 : The Data of the Experiment for Finding Suitable Conditions.

- Remark : 1) NaOH 2.5 % concentration of 6.2 milliliters used for a batch of reactor in the condition of F/M ratio of 1.6 and melamine crystal pH 8.0-8.7.
- 2) NaOH 2.5 % concentration of 6.6 milliliters used for a batch of reactor in the condition of F/M ratio of 1.8 and melamine crystal pH 8.0-8.7.
- 3) NaOH 2.5 % concentration of 7.0 milliliters used for a batch of reactor in the condition of F/M ratio of 2.0 and melamine crystal pH 8.0-8.7.
- 4) NaOH 2.5 % concentration of 5.2 milliliters used for a batch of reactor in the condition of F/M ratio of 1.6 and melamine crystal pH 8.7-9.5.
- 5) NaOH 2.5 % concentration of 5.6 milliliters used for a batch of reactor in the condition of F/M ratio of 1.8 and melamine crystal pH 8.7-9.5.
- 6) NaOH 2.5 % concentration of 6.2 milliliters used for a batch of reactor in the condition of F/M ratio of 2.0 and melamine crystal pH 8.7-9.5.

### 8.3 Data Analysis of Experiment

#### 8.3.1 Analysis of Variance

The analysis of variance for this experiment is summarized in Table 8.3.

Source of Variation	Sum of Squares	Degrees of Freedom	Mean Square	$F_o$
F/M ratio (A)	22,568.00	2	11,284.00	293.07
pH of Melamine crystal (B)	2,451.02	1	2,451.02	63.66
Interaction	190.17	2	95.08	2.47
Error	1617.13	42	38.50	
Total	26,826.31	47		

Table 8.3 : The Analysis of Variance for Experiment for Finding Suitable Conditions.

In Table 8.3, since  $F_{0.05, 2, 42} = 3.22$  and  $F_{0.05, 1, 42} = 4.07$ , we conclude that there is no significant interaction between F/M ratio and melamine crystal pH; however, it is clear that F/M ratio and pH of melamine crystal significantly affect the curing time.

And, in Figure 8.1, 8.2, 8.3, and 8.4, a normal probability plot of the residuals and the three plots of residuals versus F/M ratio, melamine crystal pH, and fitted values. These plots do not reveal any model inadequacy or unusual problem with the assumptions of the analysis of variance for this experiment.

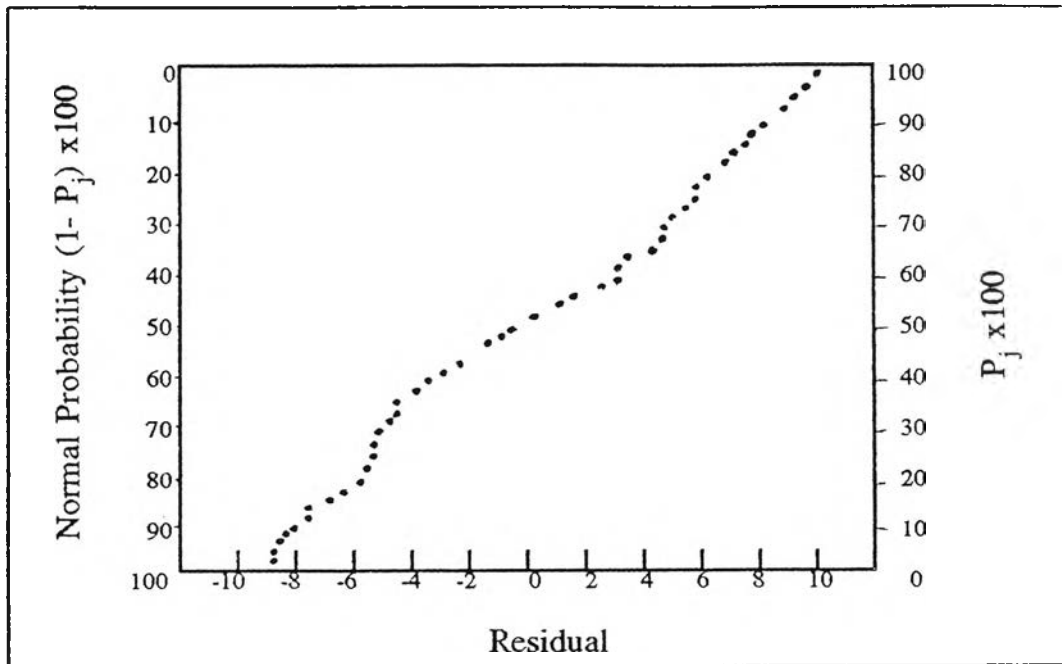


Figure 8.1 : The Normal Probability Plot of Residuals  
for Experiment for Finding Suitable Conditions.

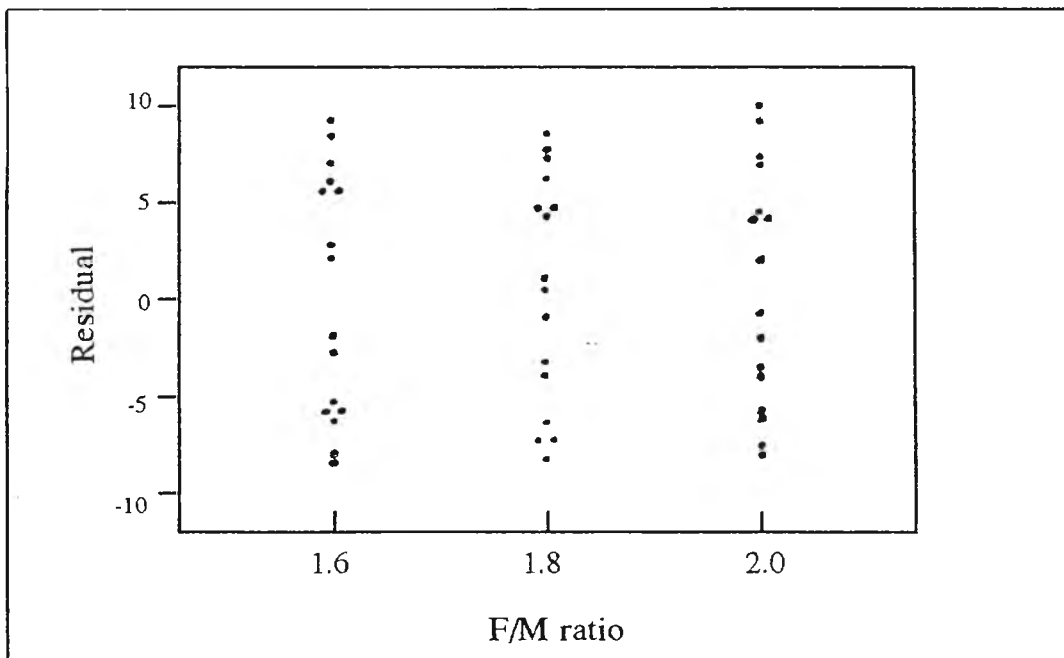


Figure 8.2 : The Plot of Residuals versus F/M ratio  
for Experiment for Finding Suitable Conditions.

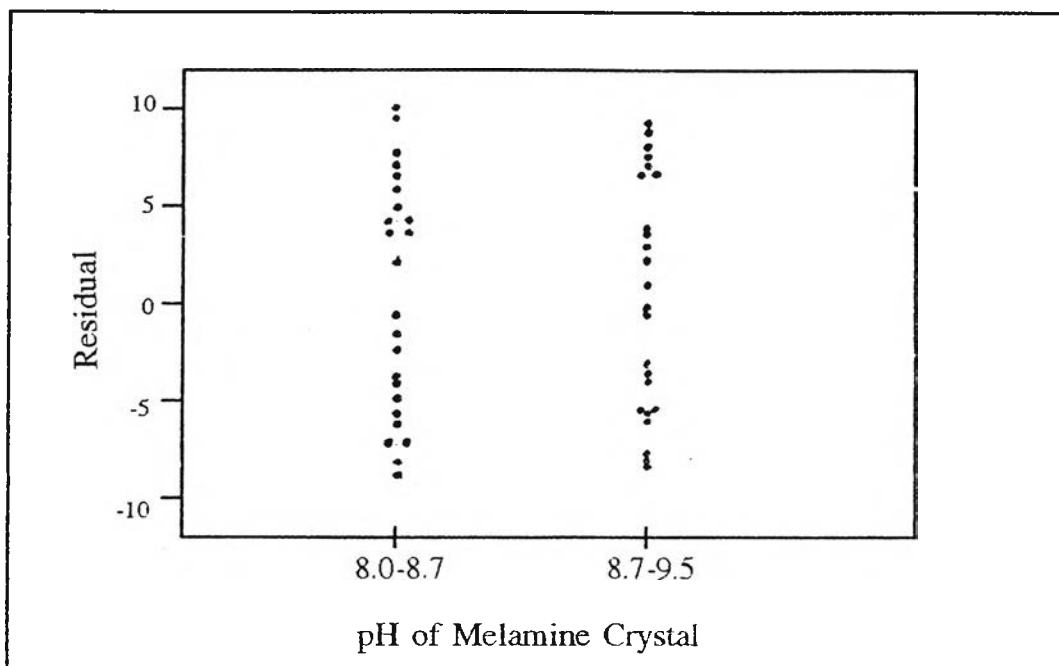


Figure 8.3 : The Plot of Residuals versus pH of Melamine Crystal for Experiment for Finding Suitable Conditions.

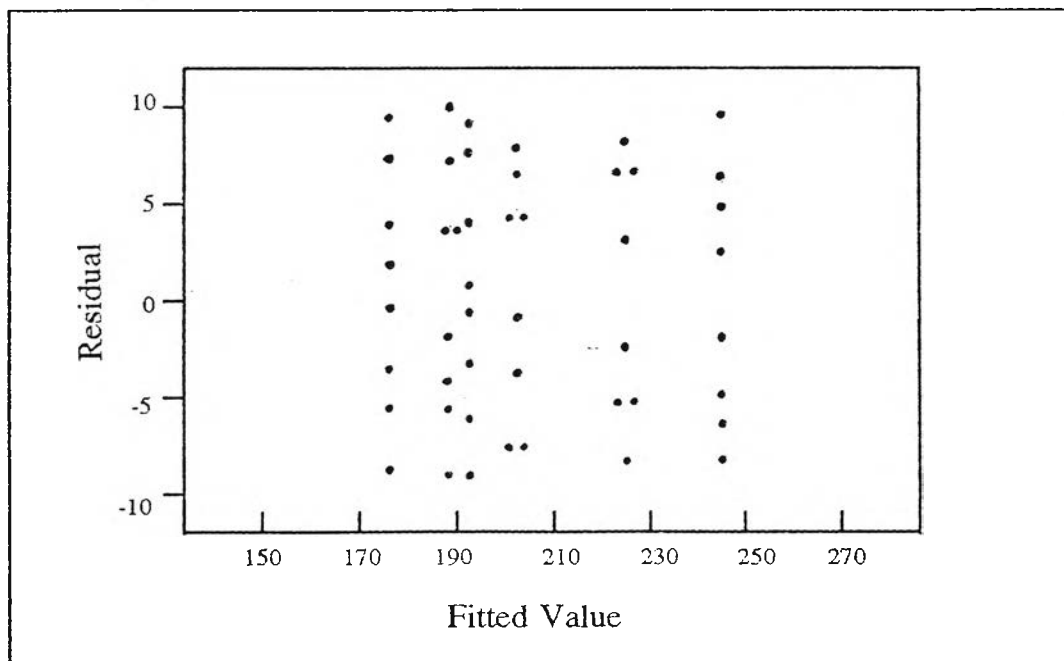


Figure 8.4 : The Plot of Residuals versus Fitted Values for Experiment for Finding Suitable Conditions.

And, the graph of the average curing times at each treatment combination is constructed to assist in interpreting the results of this experiment as shown in Figure 8.5.

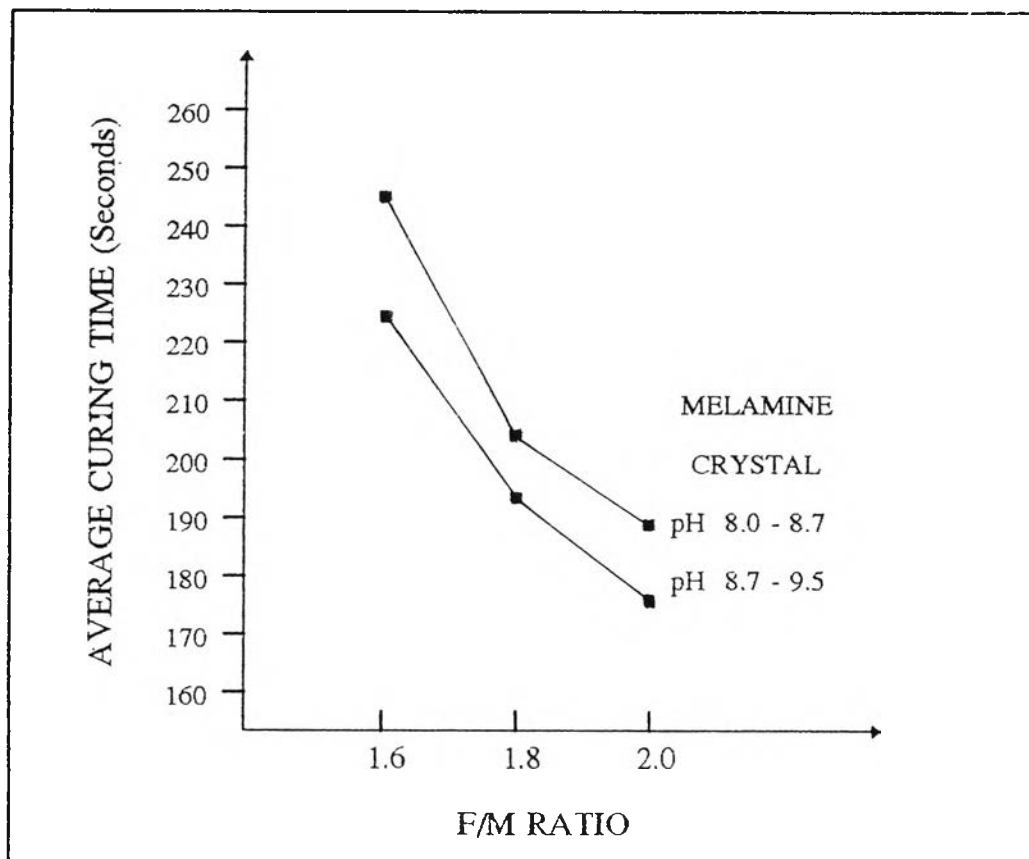


Figure 8.5 : The Graph of the Average Curing Times versus the Levels of F/M Ratio for Each Melamine Crystal pH Level.

In Figure 8.5, the absence of interaction is evident in the parallelism of the two lines. And the average curing time decreases from F/M ratio of 1.6 to F/M ratio of 2.0. Also the melamine crystal pH 8.0-8.7 gives higher average curing time of about 15 seconds than the melamine crystal pH 8.7-9.5 in the range of F/M ratio 1.6 to 2.0.

### 8.3.2 Cheking Number of Replicates

The sample size or the number of replicates is examined using the operating characteristic curve by computing

$$\begin{aligned}\Phi^2 &= \frac{naD^2}{2b\sigma^2} \\ &= \frac{n \times 3 \times 15^2}{2 \times 2 \times 7^2} \\ \therefore \Phi^2 &= 3.44n\end{aligned}$$

The minimum value of  $\Phi^2$ , the numerator degrees of freedom  $(b-1) = 1$ , denominator degrees of freedom  $(ab(n-1))= 6(n-1)$ , and  $\alpha = 0.05$  are used to find  $\beta$  in the operating characteristic curve that can be summarized in Table 8.4.

$n$	$\Phi^2$	$\Phi$	$\omega_1 =$ Numerator Degrees of Freedom	$\omega_2 =$ Error Degrees of Freedom	$\beta$
2	6.88	2.62	1	6	0.15
3	10.32	3.21	1	12	0.01

Table 8.4 : Table for Choice of Sample Size.

From Table 8.4, at least three replicates, which give a  $\beta$  risk of about 0.01, are enough to accept that the difference in the curing time mean of the two levels of melamine crystal pH as large as 15 seconds and the standard deviation is no larger than 7 seconds. Therefore, the eight replicates could be enough for this experiment.



### 8.3.3 Mutiple Comparisons

The comparisons between the individual row or column means in this experiment should be done to discover the specific differences of the curing time in each condition by using Duncan's multiple range test as follows.

The curing time means in each condition of this experiment are summarized in Table 8.5.

		Melamine Crystal pH	
		8.0-8.7	8.7-9.5
F/M ratio	1.6	244.13	224.25
	1.8	202.63	191.75
	2.0	188.75	176.63

Table 8.5 : The Curing Time Means in Each Condition.

The standard error of these treatment means is

$$S_{\bar{y}} = \sqrt{\frac{MS_E}{n}} = \sqrt{\frac{38.50}{8}} = 2.19$$

since each mean contains  $n= 8$  observations. And the value  $r_{0.05}(2, 42) = 2.85$  and  $r_{0.05}(3, 42) = 3.00$  can be obtained from the table of significant ranges for Duncan's multiple range test. The least significant ranges are

$$R_2 = t_{0.05}(2, 42)S_y = 6.24$$

$$R_3 = t_{0.05}(3, 42)S_y = 6.57$$

and the comparisons between the row means in the column of melamine crystal pH 8.0-8.7 are as follows.

$$\text{F/M ratio 1.6 vs. 2.0} = 244.13 - 188.75 = 55.38 > 6.57 \text{ (R3)}$$

$$\text{F/M ratio 1.8 vs. 2.0} = 202.63 - 188.75 = 13.88 > 6.24 \text{ (R2)}$$

$$\text{F/M ratio 1.6 vs. 1.8} = 244.13 - 202.63 = 41.50 > 6.24 \text{ (R2)}$$

This analysis indicates that there are significant differences between all pairs of the curing time means at the melamine crystal pH 8.0-8.7.

In the same way, the comparisons between the row means in the column of melamine crystal pH 8.7-9.5 are as follows.

$$\text{F/M ratio 1.6 vs. 2.0} = 224.25 - 176.63 = 47.62 > 6.57 \text{ (R3)}$$

$$\text{F/M ratio 1.8 vs. 2.0} = 191.75 - 176.63 = 15.12 > 6.24 \text{ (R2)}$$

$$\text{F/M ratio 1.6 vs. 1.8} = 224.25 - 191.75 = 32.50 > 6.24 \text{ (R2)}$$

This analysis indicates that there are significant differences between all pairs of the curing time means at the melamine crystal pH 8.7-9.5.

And the comparison between the column means in the row of F/M ratio of 1.6 is

$$\text{Melamine Crystal pH 8.0-8.7 vs. 8.7-9.5} = 244.13 - 224.25 = 19.88 > 6.24 \text{ (R2)}$$

, the comparison between the column means in the row of F/M ratio of 1.8 is  
Melamine Crystal pH 8.0-8.7 vs. 8.7-9.5 = 202.63-191.75 = 10.88 > 6.24 (R2)

, and the comparison between the column means in the row of F/M ratio of  
2.0 is

Melamine Crystal pH 8.0-8.7 vs. 8.7-9.5 = 188.75-176.63 = 12.12 > 6.24 (R2)

It shows that there are significant differences in the curing time means due to the two different ranges of melamine crystal pH in each F/M ratio.

#### 8.4 Curing time Standard Deviation and Means Estimation

Since the error mean square ( $MS_E$ ) is an estimator of  $\sigma^2$ , the standard deviation,  $\sigma$ , of the curing time in the process could be estimated by

$$\sigma = \sqrt{MS_E} = \sqrt{38.50} = 6.20 \text{ seconds.}$$

And the curing time means in each condition are estimated by

$$\bar{y} \pm t_{\frac{\alpha}{2}, ab(n-1)} \sqrt{\frac{MS_E}{n}}$$

where,  $\bar{y}$  is a curing time average in each condition of the experiment,

a 100(1- $\alpha$ ) percent confidence interval on the mean,

for this experiment,  $\alpha = 0.05$ , a=3, b=2, n =8, and  $MS_E = 38.50$

Therefore, the curing time means in each condition are computed by the following equation.

$$\mu = \bar{y} \pm t_{0.025, 42} \sqrt{\frac{38.50}{8}}$$

$$\mu = \bar{y} \pm 4.43 \text{ seconds.}$$

$$\mu \approx \bar{y} \pm 5 \text{ seconds.}$$

From the collected data of the experiment for finding suitable conditions in the condition of F/M ratio of 1.6 and melamine crystal pH 8.0-8.7, the curing time average( $\bar{y}$ ) is 244.13 seconds. Therefore, the 95 % confidence interval of curing time mean of this condition is (239.70, 248.56) seconds, or the curing time mean is in the range of 239.70 to 248.56 seconds.

And the condition of F/M ratio of 1.6 and melamine crystal pH 8.7-9.5, the curing time average( $\bar{y}$ ) is 224.25 seconds. Therefore, the 95 % confidence interval of curing time mean of this condition is (219.82, 228.68) seconds, or the curing time mean is in the range of 219.82 to 228.68 seconds.

And the condition of F/M ratio of 1.8 and melamine crystal pH 8.0-8.7, the curing time average( $\bar{y}$ ) is 202.63 seconds. Therefore, the 95 % confidence interval of curing time mean of this condition is (198.20, 207.06) seconds, or the curing time mean is in the range of 198.20 to 207.06 seconds.

And the condition of F/M ratio of 1.8 and melamine crystal pH 8.7-9.5, the curing time average( $\bar{y}$ ) is 191.75 seconds. Therefore, the 95 % confidence interval of curing time mean of this condition is (187.32, 196.18) seconds, or the curing time mean is in the range of 187.32 to 196.18 seconds.

And the condition of F/M ratio of 2.0 and melamine crystal pH 8.0-8.7, the curing time average( $\bar{y}$ ) is 188.75 seconds. Therefore, the 95 % confidence interval of curing time mean of this condition is (184.32, 193.18) seconds, or the curing time mean is in the range of 184.32 to 193.18 seconds.

And the condition of F/M ratio of 2.0 and melamine crystal pH 8.7-9.5, the curing time average( $\bar{y}$ ) is 176.63 seconds. Therefore, the 95 % confidence interval of curing time mean of this condition is (172.20, 181.06) seconds, or the curing time mean is in the range of 172.20 to 181.06 seconds.

Finally, the curing means in each condition can be presented in the graph as shown in Figure 8.6

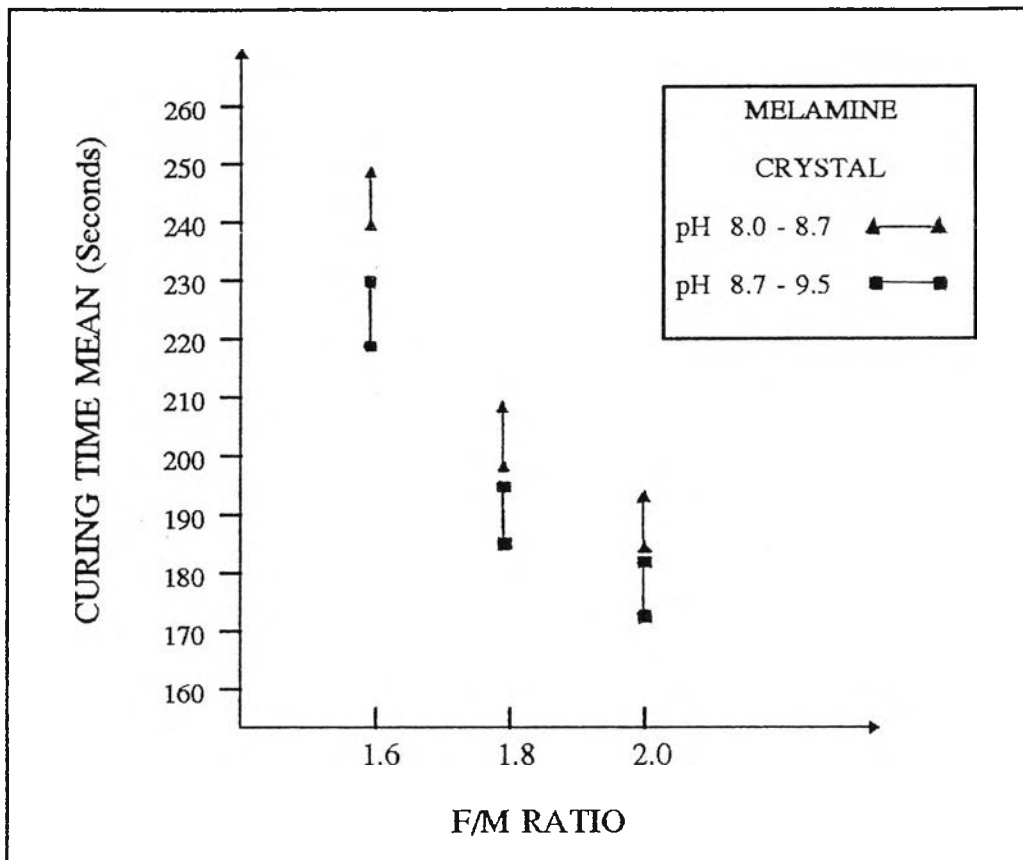


Figure 8.6 : The Graph of the Estimated Curing Time Means versus F/M Ratio and Melamine Crystal pH of Experiment for Finding Suitable Conditions.

### 8.5 Relationship of Curing Time Mean and Factors in Experiments

The relationship of the curing time mean based on 95 % confidence interval, F/M ratio, and melamine crystal pH in the factor screening experiments, the preliminary experiment, and the experiment for finding suitable conditions can be presented in Figure 8.7.

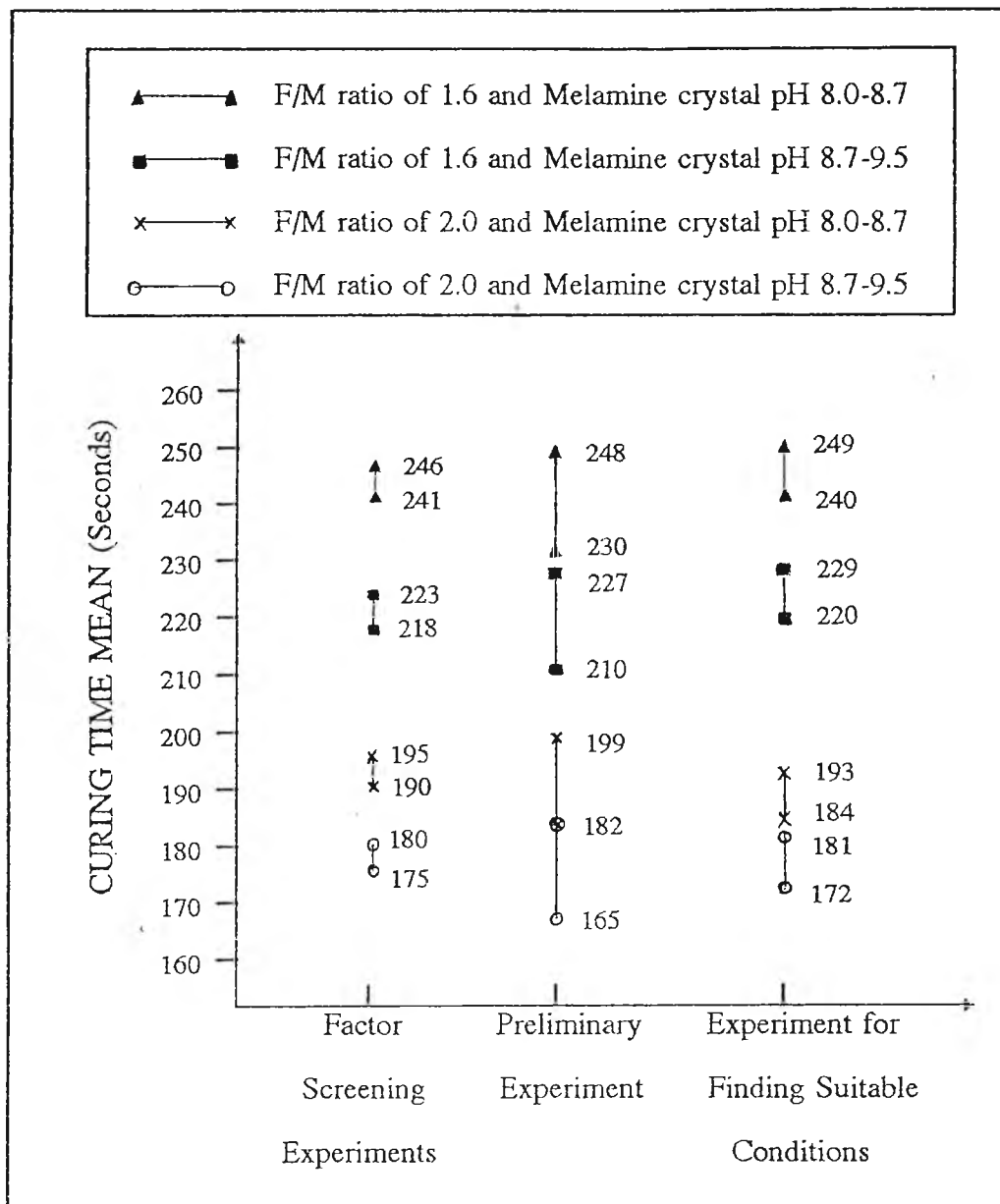


Figure 8.7 : The Relationship of Curing Time Mean and Conditions of F/M Ratio and Melamine Crystal pH in Experiments.

Moreover, the three curing time means in the same conditions of F/M ratio and melamine crystal pH of the three experiments are not different, based on level of significance of 0.05 by testing hypotheses.

## 8.6 Suitable Conditions

From the results of the experiment for finding suitable conditions, the suitable conditions for a reactor of the laboratory can be concluded as shown in Table 8.6.

	Condition					
	1	2	3	4	5	6
F/M Ratio	1.6	1.6	1.8	1.8	2.0	2.0
pH of Melamine Crystal	8.0-8.7	8.7-9.5	8.0-8.7	8.7-9.5	8.0-8.7	8.7-9.5
Sodium Hydroxide 2.5% Concentration (milliliters)	6.2	5.2	6.6	5.6	7.0	6.2
Curing Time Mean (seconds)	244	224	202	192	189	177

Remark : The accuracy of the curing time means is about  $\pm 5$  seconds based on 95% confidence interval.

Table 8.6 : The Suitable Conditions for Reactor of the Laboratory.