CHAPTER IV

RESULTS AND DISCUSSION

4.1 The Phase Behavior of Ternary Liquid System for IPA – Water – CHX.

4.1.1 Solubility Curve and Plait Point Composition at 26°C.

The volume of IPA was added in the mixture of CHX and water as feed composition according to Table 3.1 and Table 4.1.

According to the procedure in Chapter III (Table 3.2), the compositions of lower layer and upper layer were determined. Table 4.2 shows the composition of extract and raffinate phase. The GC. chromatograms are shown in Appendix A.

The experimental results from Table 4.1, and 4.2 are plotted on ternary phase diagram in weight percent. This curve is called solubility curve. Tie line and conjugate line were obtained from this curve. The plait point compositions were obtained by extending conjugate line to solubility curve. The plait point compositions obtained from Figure 4.1 are IPA = 27.08 %, CHX = 47.04 % and water = 25.88%.

Table 4.1 Feed composition at 26°C.

Sample No.	Feed (ml)			Feed (% wt)		
	Water	CHX	IPA	Water	CHX	IPA
1	3.0	27.0	5.0	10.73	75.24	14.02
2	6.0	24.0	9.0	18.89	58.88	22.22
3	9.0	21.0	12.0	25.88	47.04	27.08
4	12.0	18.0	13.3	32.91	38.45	28.63
5	15.0	15.0	14.0	39.82	31.01	29.17
6	18.0	12.0	13.0	47.92	24.90	27.16
7	21.0	9.0	10.0	58.56	19.55	21.89
8	24.0	6.0	7.0	70.24	13.67	16.09

Table 4.2 The compositions of raffinate phase (lower layer) and extract phase (upper layer) at 26°C.

Sample No.	Raffinate (Lower layer) (%)			Extract (Upper layer)(%)		
	Water	CHX	IPA	Water	CHX	IPA
1	86.22	0.011	13.76	0.027	99.42	0.55
2	75.68	0.099	24.22	0.33	96.29	3.38
3	57.67	2.93	39.40	1.31	90.89	7.80
4	35.73	16.35	47.92	3.02	82.85	14.13

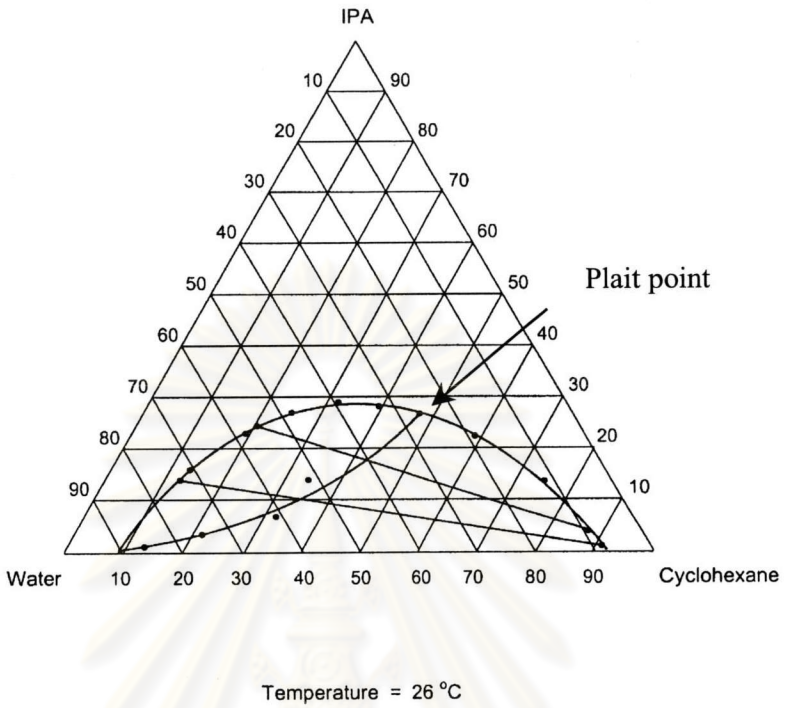

Figure 4.1 Ternary phase diagram of IPA – water – CHX , in weight percent, at 26°C which shown the plait point compositions.

4.1.2 Solubility Curve and Plait Point Composition at 30°C.

The amount volume of IPA was in the mixture of CHX and water as feed composition according to Table 3.1 and Table 4.3.

According to the procedure in Chapter III (Table 3.2), the composition of lower layer and upper layer were determined. Table 4.4 shows the composition of extract and raffinate phase.

In the same case of experiment 4.1.1, therefore, the results from Table 4.3 and 4.4 are plotted on ternary phase diagram as shown in Figure 4.2. The plait point compositions obtained at 30°C from Figure 4.2 are IPA = 27.58%, CHX = 46.72% and water = 25.70%.

Table 4.3 Feed composition at 30°C.

Sample No.	Feed (ml)			Feed (%wt)		
	Water	CHX	IPA	Water	CHX	IPA
1	3.0	27.0	6.1	10.41	72.97	16.62
2	6.0	24.0	10.0	18.43	57.45	24.12
3	9.0	21.0	12.3	25.70	46.72	27.58
4	12.0	18.0	13.6	32.70	38.20	29.10
5	15.0	15.0	15.3	38.77	30.19	31.04
6	18.0	12.0	14.3	46.67	24.24	29.09
7	21.0	9.0	11.7	56.47	18.85	24.68
8	24.0	6.0	9.0	67.17	13.07	19.76

Table 4.4 The compositions of raffinate phase (lower layer) and extract phase (upper layer) at 30°C.

Sample No.	Raffinate (Lower layer) (%)			Extract (Upper layer) (%)		
	Water	CHX	IPA	Water	CHX	IPA
1	98.56	0.003	1.43	0.036	99.16	0.80
2	87.23	0.14	12.63	0.39	94.97	4.64
3	70.70	2.18	27.11	1.62	88.31	10.07
4	29.44	25.95	44.56	6.03	74.15	19.82

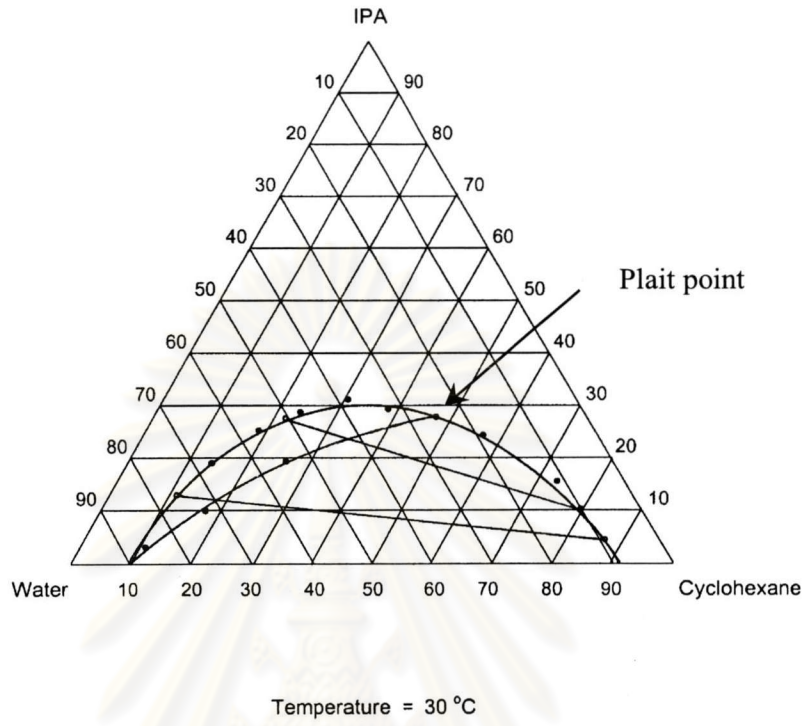

Figure 4.2 Ternary phase diagram of IPA – water – CHX , in weight percent, at 30°C which shown the plait point compositions.

4.2 Operating Conditions of Heterogeneous Azeotropic Distillation.

4.2.1 Effect of Feed Rate.

The effect of feed rate on the composition of bottom product, CHX phase and water phase at different feed rate is shown in Tables 4.5- 4.10 and Figures 4.3 - 4.8. The details of all data are shown in Appendix B and C.

When the feed rate was increased, percentage of IPA in product did not changed and percentage of CHX in product had tendency to decrease as shown in Figure 4.4.

Percentage of CHX in product and water phase is important because this indicated the CHX loss in product and in wastewater from stripping column. If product has high concentration of CHX, this lead to the loss of CHX and could not

be recycled to system. In the same way, when water phase has high concentration of CHX, CHX may be lost because the water phase was separated by stripping column. Only IPA and CHX in CHX phase were recycled to the top of azeotropic distillation column. Water from the bottom of stripping column normally was pure but sometimes contained with CHX because of operation.

To obtain the feed rate, which was suitable for operation, some effects should be considered. From Figure 4.6 at the feed rate of 9.5 and 12.0 L/min percentage of CHX in water phase was lower than other feed rates but percentage of CHX (0.004%) in product at feed rate 11.0 L/min was the lowest as shown in Figure 4.4. Although percentage of water in product was not the lowest (see Figure 4.5) it still suitable for used. The effect of feed rate on percentage of CHX in CHX phase as shown in Figure 4.7, it can be seen that percentage of CHX in CHX phase changed not much in each feed rate. The different operating conditions may cause very small variation of the percentage of CHX in CHX phase. Therefore in this experiment, the suitable feed rate was 11.0 L/min. And at this feed rate the percentage of IPA (99.335%) was high enough for the use in the production of nitrocellulose.

The relationship between feed rate and percent reflux rate is shown in Figure 4.8. It can be explained that increasing feed rate also need the increase of percent reflux rate, except at feed rate of 10.5 L/min, reflux rate was opened only 95% that was quite low, so it affected the percentage of IPA in product which was lower than other feed rates. It was necessary to increase percent reflux rate with increasing feed rate. It was found that amount of CHX, which used as the entrainer, should also be increased. Until the percent reflux rate increased to the optimum point, the higher purity of IPA in bottom product would be obtained and the purity of IPA should be more than 99%. After that, the increase of percent reflux rate would not affect distillation efficiency, the percentage of IPA in bottom product would be lower than at optimum point and CHX in bottom product was also found in product.

Table 4.5a The compositions of feed and product (IPA) at feed rate of 8.0 L/min.

Time (hr.)	Feed compositions (%)			Feed rate (L/min.)	Product compositions (%)			Product rate (L/min.)
	IPA	CHX	Water		IPA	CHX	Water	
2	81.28	0.64	18.08	8.0	99.987	0.005	0.008	9.0
4	81.34	0.74	17.91	8.1	99.989	0.003	0.011	7.6
6	80.89	0.90	18.21	8.0	99.641	0.346	0.013	9.4
8	81.41	0.77	17.82	8.0	99.985	0.005	0.010	9.5
Avg.	81.23	0.76	18.00	8.0	99.901	0.090	0.011	8.9

Table 4.5b The compositions of CHX phase and water phase at feed rate of 8.0 L/min.

Time (hr.)	CHX phase compositions (%)			Reflux rate (%)	Water phase compositions (%)		
	IPA	CHX	Water		IPA	CHX	Water
2	13.91	83.61	2.48	95	33.62	0.78	65.60
4	15.02	82.31	2.68	95	32.43	0.93	66.64
6	14.88	82.48	2.65	95	33.89	1.16	64.95
8	15.21	82.13	2.66	95	31.86	1.02	67.12
Avg.	14.76	82.63	2.62	95	32.95	0.97	66.08

Table 4.6a The compositions of feed and product (IPA) at feed rate of 9.0 L/min.

Time (hr.)	Feed compositions (%)			Feed rate (L/min.)	Product compositions (%)			Product rate (L/min.)
	IPA	CHX	Water		IPA	CHX	Water	
2	80.50	1.21	18.29	9.0	99.886	0.071	0.043	10.1
4	79.71	0.73	19.56	9.1	99.960	0.005	0.035	11.1
6	79.59	1.88	18.54	9.0	99.900	0.063	0.037	12.0
8	80.11	0.58	19.30	9.0	99.918	0.030	0.052	8.9
Avg.	79.98	1.10	18.92	9.0	99.916	0.042	0.042	10.5

Table 4.6b The compositions of CHX phase and water phase at feed rate of 9.0 L/min.

Time (hr.)	CHX phase compositions (%)			Reflux rate (%)	Water phase compositions (%)		
	IPA	CHX	Water		IPA	CHX	Water
2	13.65	84.30	2.23	97	18.85	0.26	80.89
4	14.51	82.94	2.55	97	27.52	0.48	72.00
6	13.80	83.89	2.31	97	27.77	0.66	71.57
8	13.43	84.59	1.98	97	27.39	0.56	72.05
Avg.	13.80	83.93	2.27	97	25.38	0.49	74.13

Table 4.7a The compositions of feed and product (IPA) at feed rate of 10.5 L/min.

Time (hr.)	Feed compositions (%)			Feed rate (L/min.)	Product compositions (%)			Product rate (L/min.)
	IPA	CHX	Water		IPA	CHX	Water	
2	83.35	1.00	15.65	10.4	99.416	0.000	0.584	10.9
4	82.29	2.01	15.71	10.5	98.775	0.000	1.225	9.8
6	82.90	1.43	15.68	10.6	98.205	0.002	1.793	8.8
8	83.19	1.13	15.69	10.5	98.197	0.027	1.776	12.2
Avg.	82.93	1.39	15.68	10.5	98.648	0.007	1.345	10.4

Table 4.7b The compositions of CHX phase and water phase at feed rate of 10.5 L/min.

Time (hr.)	CHX phase compositions (%)			Reflux rate (%)	Water phase compositions (%)		
	IPA	CHX	Water		IPA	CHX	Water
2	13.39	84.20	2.410	95	37.84	1.47	60.66
4	12.98	84.62	2.402	95	32.94	1.22	65.84
6	12.91	84.85	2.244	95	30.72	1.29	67.99
8	12.84	84.78	2.384	95	33.52	1.18	65.30
Avg.	13.03	84.61	2.360	95	33.75	1.29	64.95

Table 4.8a The compositions of feed and product (IPA) at feed rate of 11.0 L/min.

Time (hr.)	Feed compositions (%)			Feed rate (L/min.)	Product compositions (%)			Product rate (L/min.)
	IPA	CHX	Water		IPA	CHX	Water	
2	84.68	0.63	14.68	10.9	99.300	0.006	0.694	9.8
4	84.08	0.82	15.09	11.1	98.342	0.000	1.658	12.1
6	84.31	0.87	14.82	10.9	99.834	0.000	0.166	14.1
8	84.77	0.65	14.56	11.0	99.863	0.009	0.128	10.7
Avg.	84.46	0.74	14.79	11.0	99.335	0.004	0.662	11.7

Table 4.8b The compositions of CHX phase and water phase at feed rate of 11.0 L/min.

Time (hr.)	CHX phase compositions (%)			Reflux rate (%)	Water phase compositions (%)		
	IPA	CHX	Water		IPA	CHX	Water
2	12.66	85.07	2.27	99.5	32.78	0.41	66.81
4	13.33	84.21	2.47	99.5	37.22	1.35	61.43
6	15.41	81.95	2.64	99.5	34.15	0.89	64.96
8	17.74	79.69	2.58	99.5	32.97	0.97	66.06
Avg.	14.78	82.73	2.49	99.5	34.28	0.91	64.82

Table 4.9a The compositions of feed and product (IPA) at feed rate of 12.0 L/min.

Time (hr.)	Feed compositions (%)			Feed rate (L/min.)	Product compositions (%)			Product rate (L/min.)
	IPA	CHX	Water		IPA	CHX	Water	
2	81.77	0.66	17.58	12.1	99.904	0.015	0.081	11.6
4	80.10	1.15	18.75	12.1	99.692	0.001	0.307	12.6
6	80.61	0.70	18.69	12.0	99.971	0.004	0.025	12.8
8	79.59	0.67	19.74	11.9	99.949	0.020	0.031	11.9
Avg.	80.52	0.79	18.69	12.0	99.879	0.010	0.111	12.2

Table 4.9b The compositions of CHX phase and water phase at feed rate of 12.0 L/min.

Time (hr.)	CHX phase compositions (%)			Reflux rate (%)	Water phase compositions (%)		
	IPA	CHX	Water		IPA	CHX	Water
2	12.02	86.33	1.65	100	28.59	0.68	70.73
4	14.78	82.75	2.47	100	34.43	0.90	64.67
6	13.58	84.10	2.33	100	25.77	0.70	73.54
8	12.79	85.12	2.09	100	22.65	0.60	76.74
Avg.	13.29	84.57	2.13	100	27.86	0.72	71.42

Table 4.10a The average compositions of feed and product (IPA) at feed rate of 8.0, 9.0,10.5, 11.0 and 12.0 L/min.

Feed (L/min.)	Feed compositions (%)			Product compositions (%)			Product rate (L/min.)
	IPA	CHX	Water	IPA	CHX	Water	
8.0	81.23	0.76	18.00	99.901	0.090	0.011	8.9
9.0	79.98	1.10	18.92	99.916	0.042	0.042	10.5
10.5	82.93	1.39	15.68	98.648	0.007	1.345	10.4
11.0	84.46	0.74	14.79	99.335	0.004	0.662	11.7
12.0	80.52	0.79	18.69	99.879	0.010	0.111	12.2

Table 4.10b The average compositions of CHX phase and water phase at feed rate of 8.0, 9.0,10.5, 11.0 and 12.0 L/min.

Feed (L/min.)	CHX phase compositions (%)			Water phase compositions (%)			Reflux rate (%)
	IPA	CHX	Water	IPA	CHX	Water	
8.0	14.76	82.63	2.62	32.95	0.97	66.08	95
9.0	13.65	84.30	2.23	25.38	0.49	74.13	97
10.5	13.03	84.61	2.36	33.75	1.29	64.95	95
11.0	14.78	82.73	2.49	34.28	0.91	64.82	99.5
12.0	13.29	84.57	2.13	27.86	0.72	71.42	100

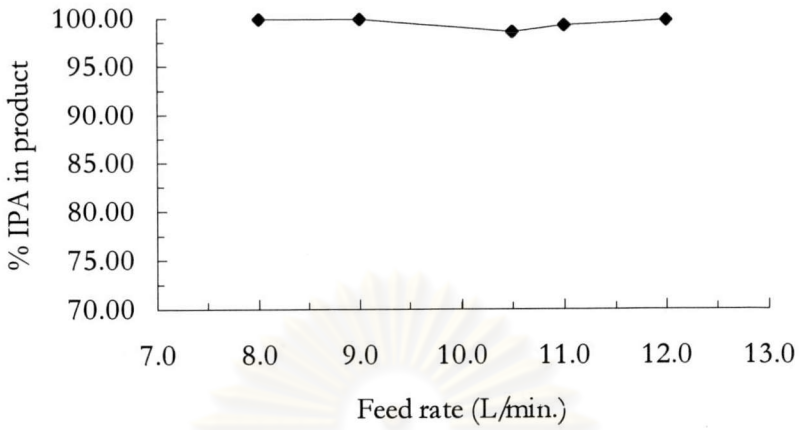

Figure 4.3 Effect of feed rate on percentage of IPA in product at 8.0, 9.0, 10.5, 11.0, and 12.0 L/min.

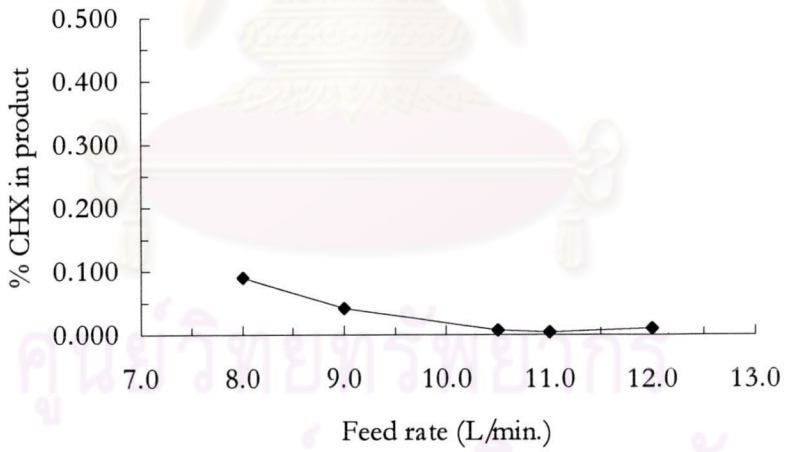

Figure 4.4 Effect of feed rate on percentage of CHX in product at 8.0, 9.0, 10.5, 11.0, and 12.0 L/min.

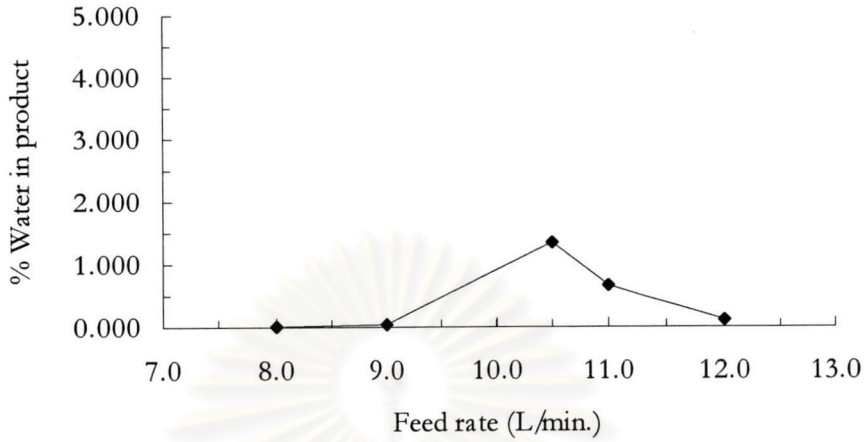

Figure 4.5 Effect of feed rate on percentage of water in product at 8.0, 9.0, 10.5, 11.0, and 12.0 L/min.

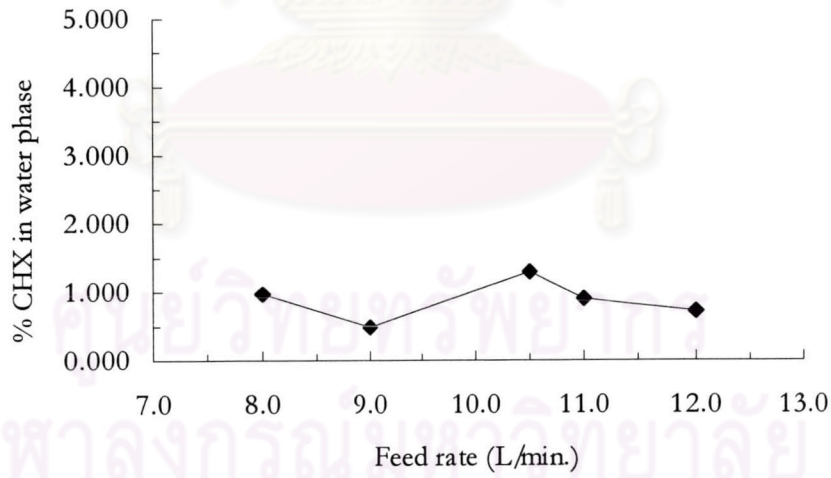

Figure 4.6 Effect of feed rate on percentage of CHX in water phase at 8.0, 9.0, 10.5, 11.0, and 12.0 L/min.

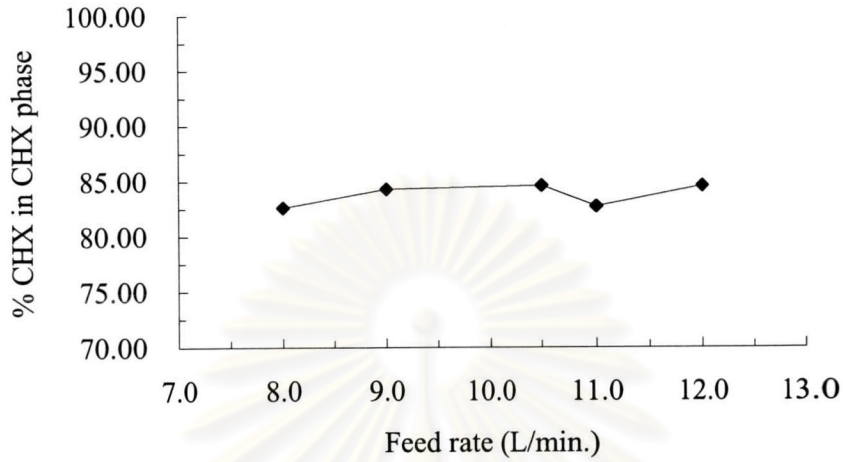

Figure 4.7 Effect of feed rate on percentage of CHX in CHX phase at 8.0, 9.0, 10.5, 11.0, and 12.0 L/min.

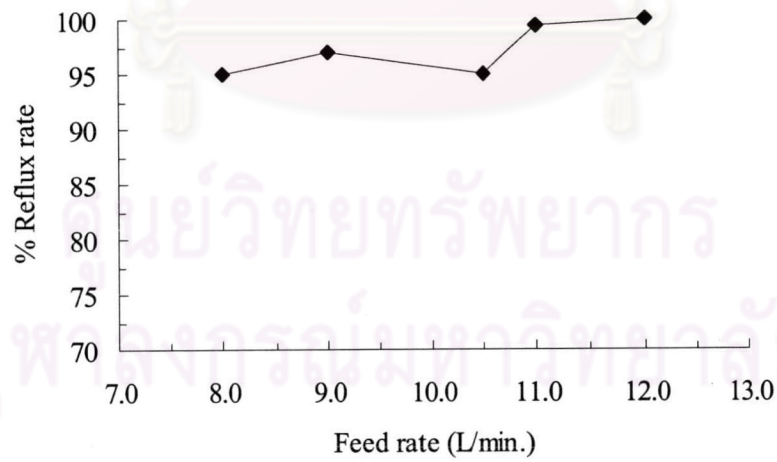

Figure 4.8 Relationship between feed rate and percent reflux rate.

4.2.2 Effect of Reflux Rate.

In this part, the feed rate was fixed then the reflux rate was varied in range of 94 – 99 %. The results are shown in Tables 4.11 - 4.13 and Figures 4.9 – 4.12. The details of all data are presented in Appendix D.

The effect of percent reflux rate on percentage of IPA in product is shown in Figure 4.9. The percent IPA in product was increased with increasing percent reflux rate and when 98% reflux rate was reached the percent IPA in product was almost no change.

However, for industrial plant, the efficiency of operation is important. For the optimum operating condition. The relationship between feed rate and reflux rate that lead to high percentage of IPA and low percentage of CHX in product must be obtained. Figure 4.10, it can be seen that percentage of CHX in product decreased with increasing percent reflux rate, especially at feed rate of 10.5 L/min. At feed rate of 9.5 L/min, percentage of CHX in product was almost no changed with increasing percent reflux rate.

From Figure 4.11, percentage of CHX in water phase was fluctuated with reflux rate. The feed rate, which give lowest percentage of CHX in water phase and in product was 11.0 L/min, because the first loss of CHX was in product and the second loss of CHX was in water phase. The results show that at feed rate of 11.0 L/min, the lowest percentage of CHX in product caused the feed compositions having the lowest percentage of CHX, as shown in Figure 4.12. Therefore, the results from this part could support the results in the part of varying feed rate.

At 98% reflux rate for every feed rate, it was found that percentage of IPA in product was high (more than 99%) and percentage of CHX in product was lower (less than 0.002%). Therefore, the optimum percent reflux rate should be 98%.

Table 4.11a The compositions of feed and product (IPA) at feed rate of 9.5 L/min.

Reflux rate (%)	Feed compositions (%)			Feed rate (L/min.)	Product compositions (%)			Product rate (L/min.)
	IPA	CHX	Water		IPA	CHX	Water	
94	83.26	0.67	16.06	9.5	99.603	0.007	0.390	7.6
95	82.87	0.66	16.47	9.5	99.447	0.009	0.544	7.5
96	83.12	0.56	16.32	9.5	99.676	0.008	0.316	12.0
97	82.41	0.52	17.07	9.5	99.739	0.002	0.259	7.2
98	83.77	0.42	15.80	9.5	99.760	0.008	0.232	10.6
99	82.15	1.12	16.73	9.5	99.886	0.008	0.106	6.5

Table 4.11b The compositions of CHX phase and water phase at feed rate of 9.5 L/min.

Reflux rate (%)	CHX phase compositions (%)			Water phase compositions (%)		
	IPA	CHX	Water	IPA	CHX	Water
94	12.29	85.74	1.97	34.56	1.01	64.43
95	14.87	83.09	2.04	37.36	1.08	61.56
96	12.07	85.97	1.96	34.81	0.94	64.25
97	12.48	85.46	2.06	36.49	1.02	62.48
98	12.75	85.09	2.16	37.84	1.01	61.15
99	13.44	84.51	2.05	35.27	0.93	63.80

Table 4.12a The compositions of feed and product (IPA) at feed rate of 10.5 L/min.

Reflux rate (%)	Feed compositions (%)			Feed rate (L/min.)	Product compositions (%)			Product rate (L/min.)
	IPA	CHX	Water		IPA	CHX	Water	
94	81.38	0.76	17.85	10.5	99.893	0.045	0.062	8.8
95	81.95	0.71	17.34	10.5	99.869	0.021	0.110	14.4
96	81.93	0.61	17.46	10.5	99.918	0.016	0.066	8.5
97	80.95	1.00	18.05	10.5	99.905	0.013	0.082	6.8
98	81.35	0.92	17.73	10.5	99.839	0.004	0.157	7.2
99	81.79	0.59	17.62	10.5	99.798	0.004	0.198	11.4

Table 4.12b The compositions of CHX phase and water phase at feed rate of 10.5 L/min.

Reflux rate (%)	CHX phase compositions (%)			Water phase compositions (%)		
	IPA	CHX	Water	IPA	CHX	Water
94	12.47	85.36	2.17	37.06	1.23	61.72
95	12.36	85.50	2.15	35.21	1.24	63.56
96	12.84	85.10	2.06	36.68	1.13	62.19
97	13.47	84.37	2.16	34.26	1.01	64.73
98	12.66	85.27	2.06	36.23	1.15	62.62
99	12.572	85.263	2.165	35.001	1.300	63.699

Table 4.13a The compositions of feed and product (IPA) at feed rate of 11.0 L/min.

Reflux rate (%)	Feed compositions (%)			Feed rate (L/min.)	Product compositions (%)			Product rate (L/min.)
	IPA	CHX	Water		IPA	CHX	Water	
94	80.74	0.46	18.81	11.0	99.970	0.001	0.029	7.0
95	82.22	0.48	17.30	11.0	99.981	0.001	0.018	8.2
96	81.32	0.46	18.22	11.0	99.965	0.002	0.033	17.1
97	81.262	0.47	18.27	11.0	99.977	0.001	0.022	13.0
98	81.89	0.46	17.66	11.0	99.976	0.000	0.024	18.6
99	80.95	0.50	18.55	11.0	99.974	0.001	0.025	11.3

Table 4.13b The compositions of CHX phase and water phase at feed rate of 11.0 L/min.

Reflux rate (%)	CHX phase compositions (%)			Water phase compositions (%)		
	IPA	CHX	Water	IPA	CHX	Water
94	12.93	84.71	2.36	27.33	0.80	71.87
95	11.93	85.69	2.38	28.07	0.83	71.11
96	12.32	85.40	2.28	25.09	0.80	74.11
97	11.62	86.13	2.26	26.92	0.88	72.20
98	12.76	84.93	2.31	25.96	0.78	73.26
99	12.08	85.75	2.17	29.90	0.89	69.21

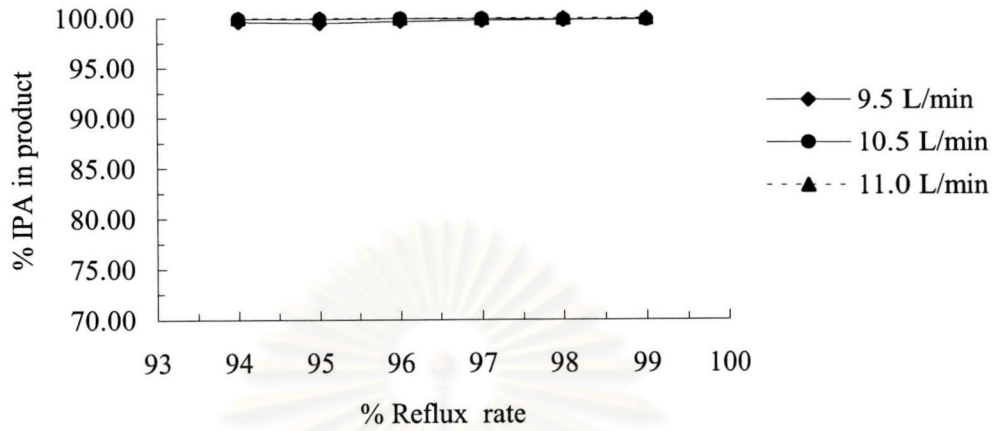

Figure 4.9 Effect of percent reflux rate on percent IPA in product at feed rate of 9.5, 10.5 and 11.0 L/min.

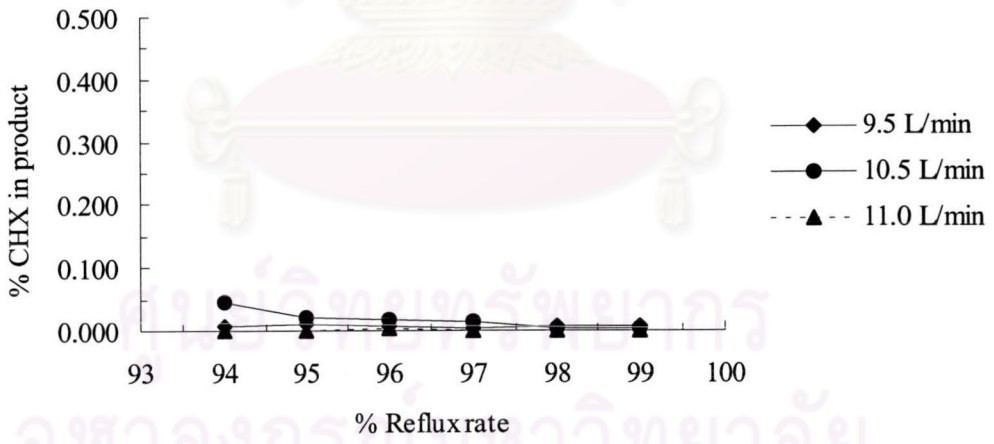

Figure 4.10 Effect of percent reflux rate on percent CHX in product at feed rate of 9.5, 10.5 and 11.0 L/min.

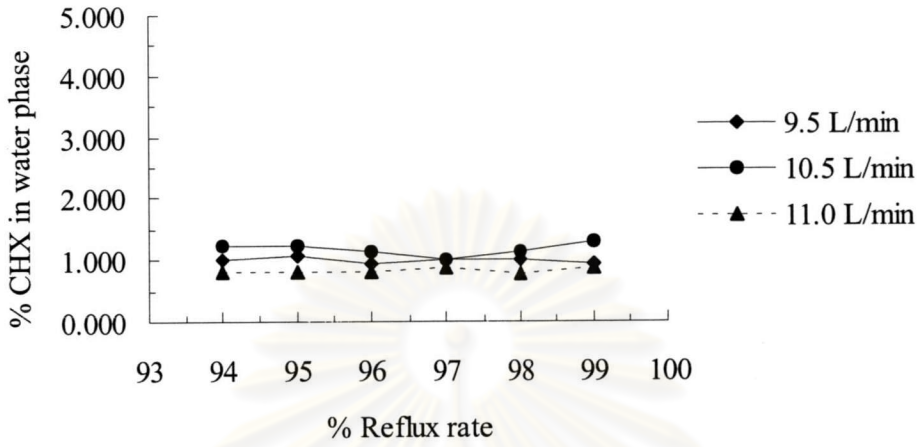

Figure 4.11 Effect of percent reflux rate on percent CHX in water phase at feed rate of 9.5, 10.5 and 11.0 L/min.

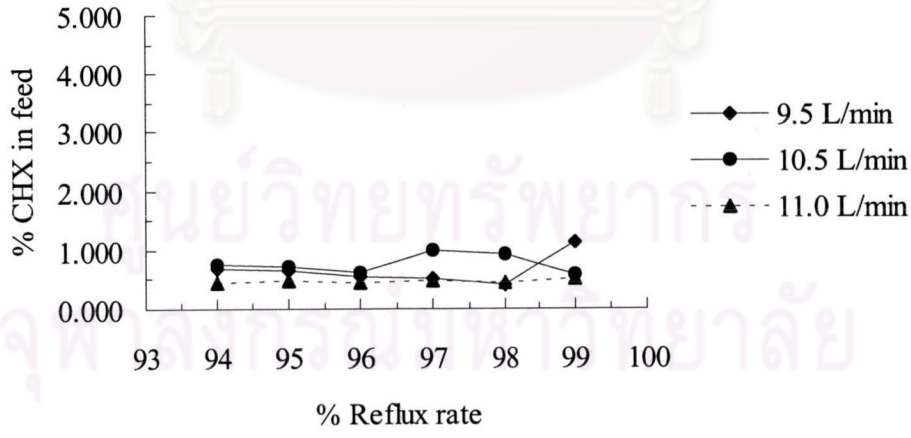

Figure 4.12 Effect of percent reflux rate on percent CHX in feed at feed rate of 9.5, 10.5 and 11.0 L/min.

4.3 The Optimum Feed Rate and Reflux Rate.

From the results of the effect of feed rate and effect of reflux rate (Section 4.2.1 and 4.2.2), the appropriate feed rate was 11.0 L/min. Therefore in this part, the optimum feed rate and reflux rate were at 11.0 L/min. and 98%, respectively. For comparative results, another feed rate was chosen to study at the same reflux rate of 98%, these feed rates were 9.5 and 12.5 L/min. The results obtained are shown in Tables 4.14 – 4.16 and Figures 4.13 – 4.16. The details of all data are shown in Appendix E.

From Figure 4.13, feed rate of 11.0 L/min gives the higher percentage of IPA in product than at feed rate of 9.5 and 12.5 L/min. In the same case, from Figure 4.14, at feed rate of 11.0 L/min, IPA product also has the lowest percentage of CHX. These results were similar to the results from Section 4.2.1 and 4.2.2. Thus the optimum feed rate should be at 11.0 L/min.

From Figure 4.15, the percentage of CHX in water phase at feed rate 11.0 L/min., was not the lowest value but it could be accepted because the main effect of CHX loss from the system was due to the percentage of CHX in product. It can be explained that amount of CHX in product cannot be recycled to the system but the percentage of IPA in water phase can be recycled to the system.

The relationship between feed rate and percentage of CHX in feed is shown in Figure 4.16. It can be seen that at the feed rate of 11.0 L/min, the percentage of CHX in feed is lowest value. It can be explained that in this time, water phase had low percentage of CHX, so it made the feed also low percentage of CHX.

Table 4.14a The compositions of feed and product (IPA) at feed rate of 9.5 L/min.

Time (hr.)	Feed compositions (%)			Feed rate (L/min.)	Product compositions (%)			Product rate (L/min.)
	IPA	CHX	Water		IPA	CHX	Water	
2	79.54	0.82	19.65	9.5	99.745	0.136	0.119	10.5
4	82.04	1.30	16.66	9.5	99.335	0.047	0.618	11.6
6	81.57	1.36	17.06	9.5	99.054	0.055	0.891	13.1
8	81.73	1.36	16.90	9.5	99.166	0.072	0.762	8.3
Avg.	81.22	1.21	17.57	9.5	99.325	0.078	0.597	10.9

Table 4.14b The compositions of CHX phase and water phase at feed rate of 9.5 L/min.

Time (hr.)	CHX phase compositions (%)			Reflux rate (%)	Water phase (%)		
	IPA	CHX	Water		IPA	CHX	Water
2	13.04	85.00	1.96	98	34.08	0.95	64.97
4	12.50	85.50	2.01	98	37.99	0.849	61.17
6	13.13	84.70	2.17	98	34.19	0.78	65.04
8	9.73	88.79	1.48	98	38.66	0.81	60.53
Avg.	12.10	86.00	1.90	98	36.23	0.85	62.93

Table 4.15a The compositions of feed and product (IPA) at feed rate of 11.0 L/min.

Time (hr.)	Feed compositions (%)			Feed rate (L/min.)	Product compositions (%)			Product rate (L/min.)
	IPA	CHX	Water		IPA	CHX	Water	
2	83.48	0.90	15.62	11.0	99.956	0.004	0.040	9.1
4	81.03	0.80	18.17	11.0	99.951	0.005	0.044	11.7
6	80.83	0.68	18.49	11.0	99.967	0.001	0.032	10.7
8	82.08	0.86	17.06	11.0	99.945	0.002	0.053	11.9
Avg.	81.86	0.81	17.33	11.0	99.955	0.003	0.042	10.8

Table 4.15b The compositions of CHX phase and water phase at feed rate of 11.0 L/min.

Time (hr.)	CHX phase compositions (%)			Reflux rate (%)	Water phase (%)		
	IPA	CHX	Water		IPA	CHX	Water
2	14.96	82.85	2.18	98	35.21	1.08	63.72
4	12.99	84.94	2.07	98	33.04	0.96	66.00
6	12.86	84.97	2.17	98	33.66	0.86	65.49
8	12.07	85.87	2.06	98	34.96	0.90	64.15
Avg.	13.22	84.66	2.12	98	34.22	0.95	64.84

Table 4.16a The compositions of feed and product (IPA) at feed rate of 12.5 L/min.

Time (hr.)	Feed compositions (%)			Feed rate (L/min.)	Product compositions (%)			Product rate (L/min.)
	IPA	CHX	Water		IPA	CHX	Water	
2	81.15	1.04	17.81	12.5	99.817	0.018	0.165	11.4
4	82.33	0.62	17.05	12.5	99.787	0.008	0.205	12.0
6	77.96	2.40	19.65	12.5	99.860	0.006	0.134	12.8
8	81.96	0.68	17.36	12.5	99.792	0.006	0.202	11.0
Avg.	80.85	1.18	17.96	12.5	99.814	0.009	0.176	11.8

Table 4.16b The compositions of CHX phase and water phase at feed rate of 12.5 L/min.

Time (hr.)	CHX phase compositions (%)			Reflux rate (%)	Water phase (%)		
	IPA	CHX	Water		IPA	CHX	Water
2	12.40	85.69	1.91	98	32.75	1.23	66.02
4	13.28	84.42	2.30	98	32.04	1.42	66.55
6	13.68	83.93	2.39	98	33.65	0.95	65.40
8	12.25	85.52	2.23	98	34.33	1.06	64.61
Avg.	12.90	84.89	2.21	98	33.19	1.16	65.65

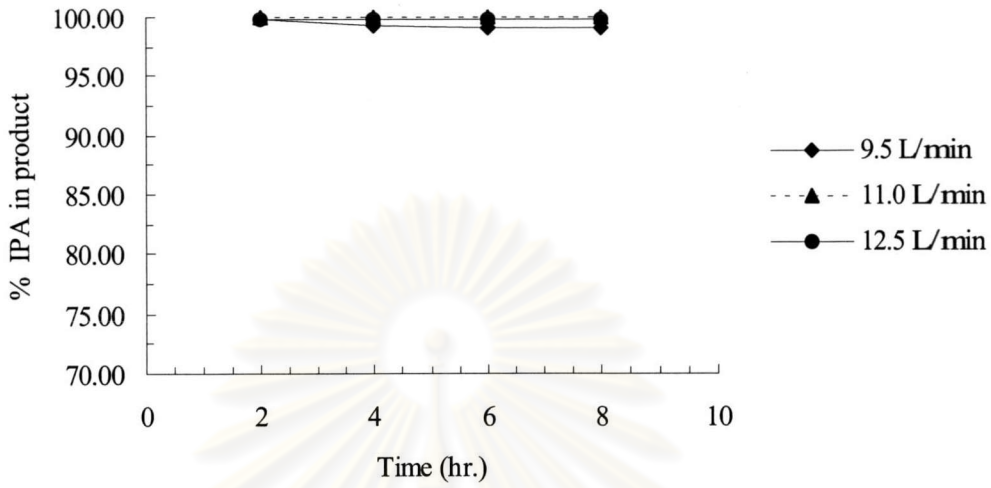

Figure 4.13 Percentage of IPA in product at feed rate of 9.5, 11.0 and 12.5 L/min.

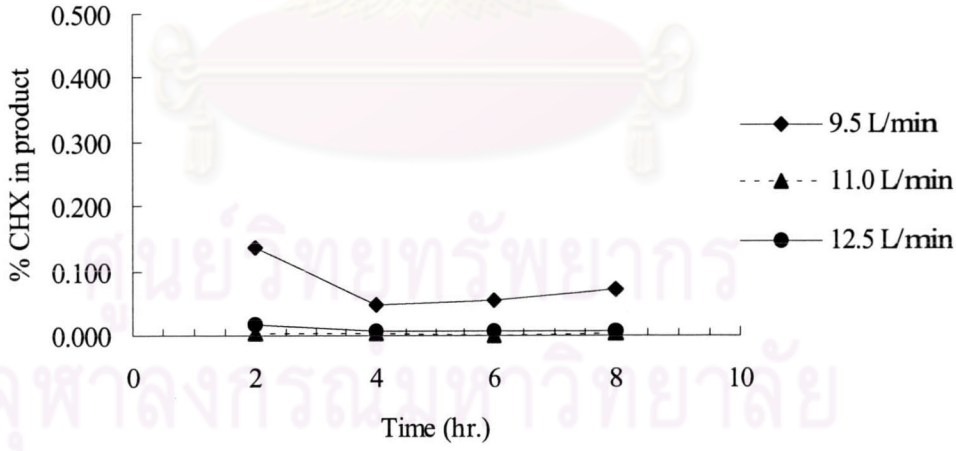

Figure 4.14 Percentage of CHX in product at feed rate of 9.5, 11.0 and 12.5 L/min.

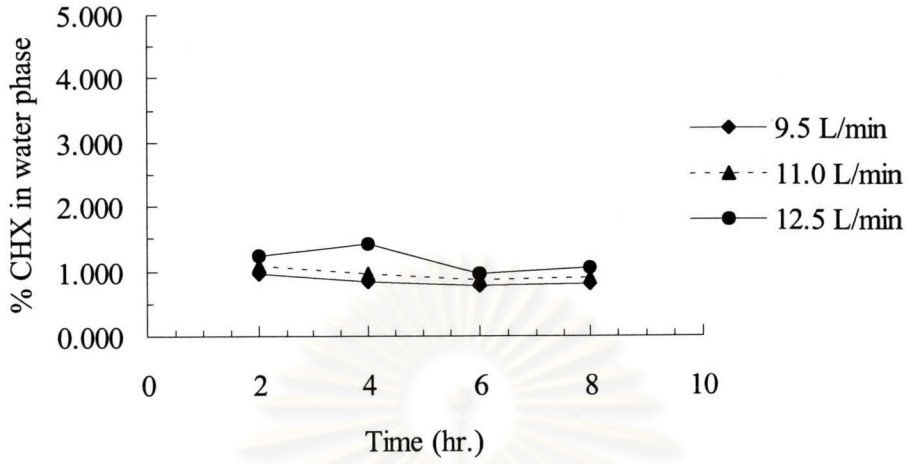

Figure 4.15 Percentage of CHX in water phase at feed rate of 9.5, 11.0 and 12.5 L/min.

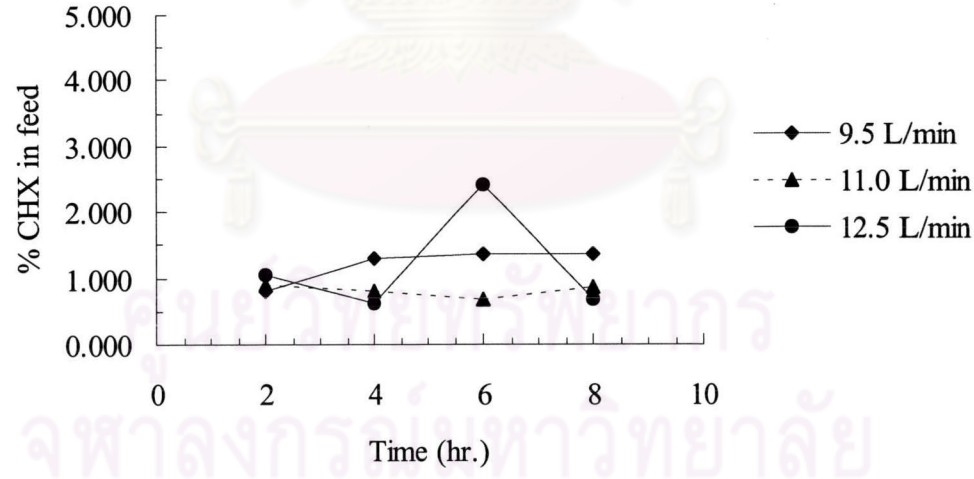

Figure 4.16 Percentage of CHX in feed at feed rate of 9.5, 11.0 and 12.5 L/min.

4.4 Material Balance.

One objective of this research work is to study phase behavior of the ternary liquid system. In this case, the vapor compositions at the top of distillation column was the interesting point but not determined by GC analysis (sample could not be collected). Therefore, calculation method by using material balance could solve this problem. From Section 4.3, at the optimum feed rate and optimum reflux rate, the vapor compositions of distillation top product calculated by material balance is presented in Table 4.17. The results show that the vapor compositions are close to the CHX phase compositions. The detail of calculation is shown in Appendix F.

Table 4.17 The vapor compositions at the top of distillation column.

Feed rate (L/min.)	Product rate (L/min.)	Reflux rate (%)		Compositions (% wt)			Total (% wt)
				IPA	CHX	Water	
9.5	10.9	98	Feed	81.22	1.21	17.57	100.0
			Product	99.325	0.078	0.597	100.0
			CHX phase	12.10	86.00	1.90	100.0
			Vapor*	10.11	86.61	3.28	100.0
11.0	10.8	98	Feed	81.86	0.81	17.33	100.0
			Product	99.955	0.003	0.042	100.0
			CHX phase	13.22	84.66	2.12	100.0
			Vapor*	12.13	84.16	3.71	100.0
12.5	11.8	98	Feed	80.85	1.19	17.96	100.0
			Product	99.814	0.009	0.176	100.0
			CHX phase	12.90	84.89	2.21	100.0
			Vapor*	11.98	83.98	4.04	100.0

* Calculated by material balance.

4.5 The Actual Cyclohexane Consumptions.

From the distillation facility operating record of the Nitro Chemical Industry, CHX actual consumptions are summarized in Table 4.18.

Table 4.18 Actual CHX consumptions under normal operations.

Volume of CHX (L)	Date/Month/Year	Period Time (days)	Avg. volume of CHX used per day (L)
200	11/01/2000 – 26/02/2000	20	10
200	27/02/2000 – 21/03/2000	23	8.7
200	22/03/2000 – 26/04/2000	35	5.7
200	03/05/2000 – 12/06/2000	40	5
200	13/06/2000 – 22/07/2000	49	4.1
200	05/01/2001 – 27/01/2001	22	9.1
200	28/01/2001 – 28/03/2001	60	3.3
200	29/03/2001 – 06/06/2001	66	3
200	07/06/2001 – 21/07/2001	45	4.4

At normal operation, the CHX in the system should not be lost. From Table 4.18, the data show that CHX loss in some period of operation was high (10 L/day) and some period of operation was low (3 L/day). It can be explained that the higher loss of CHX may be due the unefficient operation. Therefore, the best operation for industrial plant should give the lowest loss of CHX.