CHAPTER IV

RESULTS AND DISCUSSION

Each batch of mother liquor from process of amoxicillin trihydrate was kept in the tank without stirring. One batch was used for one experiment for comparison of results.

4.1 Fractional Distillation of Mother Liquor

Acetone and methylene chloride would be separated from 1,500 ml fresh mother liquor by fractional distillation and vigreux column was used as a fractionating column. Vigreux column was made with glass spikes sticking inwards for vaporization and condensation of the liquid mixture is shown in Figure G-1. The details of all data and calculations are shown in Appendix A.

Table 4.1 shows the temperature at the top of the column which remains constant at 42°C-59°C during the distillation. The percentage recovery of solvent was approximately 72.66% (total content of solvents in mother liquor was approximately 15 to 20%). Solvent had only a small amount of the methylene chloride. Recovered solvent composed of acetone (93.71%), methylene chloride (3.01%), water (2.89%) and impurity (0.40%). Recover acetone was used in process of amoxicillin trihydrate and sodium cloxacillin monohydrate.

Generally, for best fractional distillation, the difference of boiling points of liquid in the mixture should be greater than 10°C, the separation would be completed.

Table 4.1 Distillation of fresh mother liquor by control temperature-top column for methylene chloride and acetone separation.

Experiment	Temp.- Top. Column (°C)	Time (min)	Solvents in Mother Liquor (%)	Solvent Recovery (%)	Solvent DMK (%)	Solvent MDC (%)	Solvent H_2O (%)	Solvent Impurity (%)
1	42-55	0-25	18	7.41	37.16	60.96	1.69	0.19
	55-59	26-215	18	65.18	94.36	2.44	2.92	0.28
Total	42-59	215	18	72.59	-	-	-	-
2	43-55	0-15	17	7.84	41.34	56.36	2.10	0.20
	55-59	15-200	17	65.10	94.84	2.24	2.67	0.25
Total	42-59	200	17	72.94	-	-	-	-
3	43-55	0-20	17	7.84	39.39	58.39	1.96	0.26
	55-59	20-130	17	65.00	93.88	2.46	3.18	0.48
Total	42-59	130	17	72.94	-	-	-	-
4	42-55	0-20	16	6.67	44.63	52.50	2.55	0.32
	55-59	20-120	16	65.00	91.26	5.56	2.75	0.43
Total	42-59	120	16	71.67	-	-	-	-
5	43-55	0-20	17	6.67	50.11	53.13	2.38	0.21
	55-59	20-190	17	66.66	94.20	2.34	2.91	0.55
Total	42-59	190	17	73.33	-	-	-	-
Average	55-59	-	17	65.39	93.71	3.01	2.89	0.40

4.2 Effect of Separation of Triethylamine

The triethylamine salt in mother liquor of amoxicillin trihydrate process could be separated to the purified form of amine. The parameters of separation were temperature, reaction time and pH. The reaction of triethylamine is shown as follows:

$$(CH_3CH_2)_3\,N^{+}HCl^{-} + NaOH \longrightarrow CH_3CH_2)_3\,N: + NaCl + H_2O \ldots\ldots\ldots (4.1)$$

4.2.1 Effect of Mother Liquor Concentration on Triethylamine Separation

The effect of mother liquor concentration at percentage volume decreased 10-70% of original mother liquor volume on percentage recovery and triethylamine product composition is shown in Table 4.2 and Figures 4.1-4.4. The details of all data and recovery percentage calculations are shown in Appendix B. The parameters of separation are as follows:

- Residual of mother liquor : 75 ml
  (Volume decrease 10 - 70% of original of mother liquor)
- Reaction temperature : 75 °C
- Reaction time : 20 mins
- pH : 13
- NaOH concentration : 3.9 %w/v

For Figures 4.1-4.2, the higher concentration of mother liquor was resulted in the higher percentage recovery and purity of triethylamine product. Figures 4.3-4.4 show the decreased percentage of water and impurity of recovered triethylamine, because the triethylamine concentration increased with increasing the mother liquor concentration. At the volume decrease of 70%, the highest concentratrion of mother liquor, the higher percentage recovery and triethylamine concentration were 83.10% and 94.73%, respectively.

Table 4.2 Effect of mother liquor quantity on triethylamine separation from residual mother liquor.

Volume decrease	Mother Liquor		(%)															
	Original	Residual	Experiment 1.				Experiment 2.				Experiment 3.				Average			
(%)	(ml)	(ml)	Recovery	TEA	H_2O	Impurity	Recovery	TEA	H_2O	Impurity	Recovery	TEA	H_2O	Impurity	Recovery	TEA	H_2O	Impurity
10	2,000	1,800	64.58	88.19	3.49	8.32	68.75	90.79	2.33	6.78	72.92	89.19	3.20	7.61	68.75	89.39	3.01	7.57
20	1,725	1,533	64.81	92.26	2.63	5.11	70.37	91.45	2.41	6.41	70.37	91.74	2.40	5.86	68.52	91.82	2.48	5.79
30	1,458	1,276	69.35	92.84	2.56	5.60	69.35	92.58	2.50	4.29	77.42	92.53	2.56	5.12	72.04	92.65	2.54	5.00
40	1,201	1,025	77.78	92.69	2.23	5.08	79.17	94.16	2.04	3.80	77.78	93.13	2.33	4.54	78.24	93.33	2.20	4.47
50	954	795	78.16	93.94	2.08	3.98	81.61	93.83	1.92	4.25	83.91	93.82	2.08	4.10	81.23	93.86	2.03	4.11
60	720	576	78.79	94.33	2.22	3.45	81.48	94.47	2.01	3.52	81.48	94.35	2.10	3.65	80.55	94.38	2.11	3.54
70	501	376	82.64	94.85	2.15	3.00	82.64	94.83	2.05	3.12	84.02	94.51	1.99	3.50	83.10	94.73	2.06	3.21

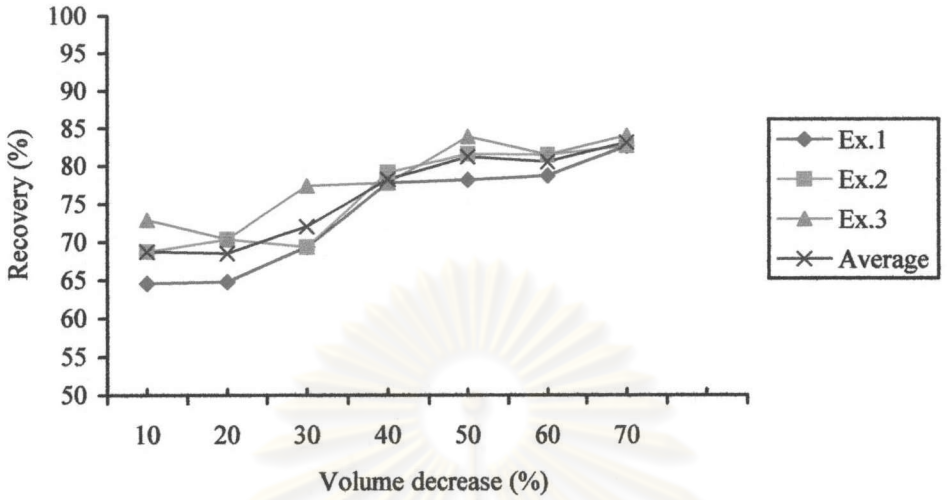


Figure 4.1 Effect of mother liquor quantity on the percentage recovery of triethylamine separation.

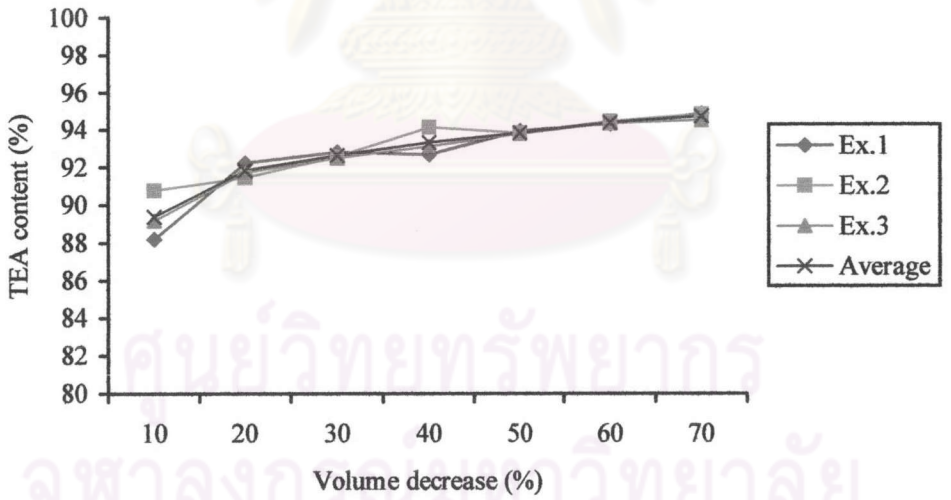


Figure 4.2 Effect of mother liquor quantity on the percentage TEA content of triethylamine separation.

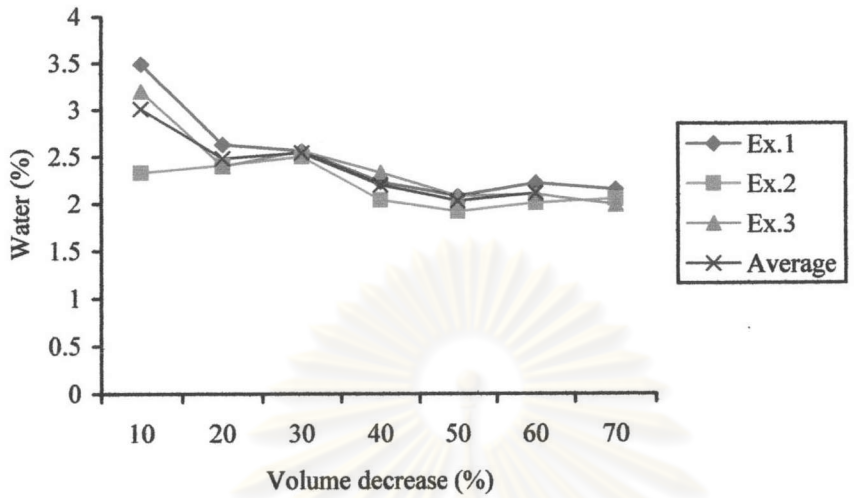


Figure 4.3 Effect of mother liquor quantity on the percentage water of triethylamine separation.

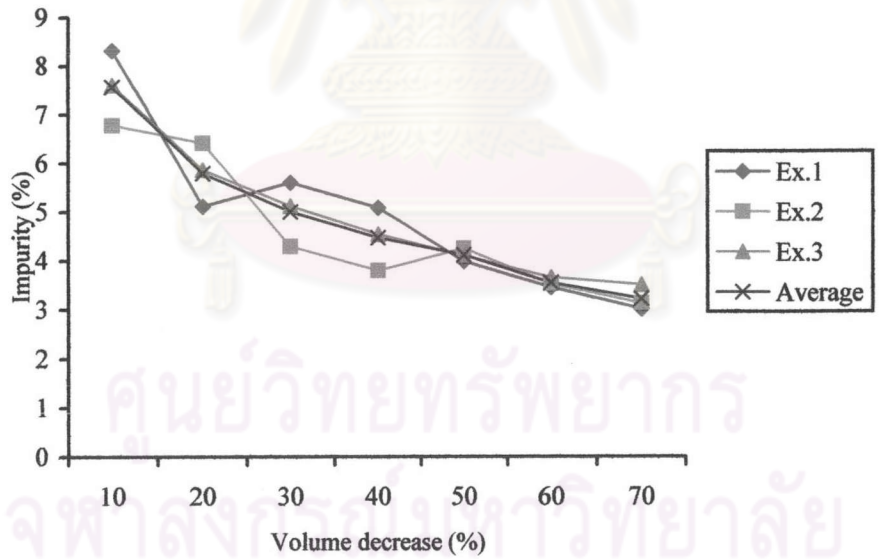


Figure 4.4 Effect of mother liquor quantity on the percentage impurity of triethylamine separation.

4.2.2 Effect of pH on Triethylamine Separation

The effect of pH ranged from 3 to13.5 on percentage recovery is shown in Table 4.3 and Figures 4.5-4.6. The details of all data and recovery percentage calculations are shown in Appendix C. The parameters of separation are as follows:

- Residual of mother liquor : 200 ml
  (Volume decrease 55 % of residual mother liquor after solvents separation)
- Reaction temperature : 75 ^{O}C
- Reaction time : 20 mins
- pH : 3 to 13.5
- NaOH concentration : 0 to 4.1 %w/v

Figure 4.5 shows that the percentage triethylamine recovery increases with increasing pH and the separation would be completed at approximately pH 13.

But the triethylamine recovery separated could not be 100%, because triethylamine was partially miscible in water and some triethylamine loss in process. If solvent extraction is used, it will have more impurity in recovered triethylamine and the impurity separation is difficult because the impurity mixed very well with triethylamine.

Figure 4.6 shows the effect of pH and concentration of NaOH (%w/v) on percentage recovery. It was found that, approximately pH 13 and 3.9% w/v of NaOH were appropriate condition of separation, the percentage triethylamine recovery was 82.96%.

Table 4.3 Effect of pH on triethylamine separation from residual of mother liquor.

NaOH	Experiment 1.			Experiment 2.			Experiment 3.			Average
(w/v)	pH	Recovery		pH	Recovery		pH	Recovery		Recovery
(%)		(ml)	(%)		(ml)	(%)		(ml)	(%)	(%)
0.00	3.23	-	-	3.17	-	-	3.25	-	-	-
0.25	8.05	-	-	8.16	-	-	7.65	-	-	-
0.50	9.33	-	-	9.26	-	-	9.23	-	-	-
0.64	9.63	-	-	9.50	-	-	9.80	-	-	-
0.98	9.69	-	-	9.59	2.40	8.24	10.04	-	-	2.81
1.22	9.77	1.90	6.67	9.77	4.30	15.09	10.12	1.90	6.67	9.48
1.46	9.83	3.80	13.33	9.83	6.20	21.75	10.14	3.80	13.33	16.14
1.69	9.93	4.70	16.49	10.00	7.60	26.67	10.31	4.30	15.09	19.42
1.92	10.02	8.10	28.42	10.38	11.50	40.35	10.43	6.20	21.75	30.17
2.15	10.15	9.10	31.93	10.96	14.80	51.93	10.54	7.20	25.26	36.37
2.38	10.31	11.90	41.75	11.89	17.70	62.11	10.75	7.60	26.67	43.51
2.61	10.54	16.70	58.60	12.50	21.00	73.68	11.13	12.40	43.51	58.60
2.83	10.97	20.50	71.93	12.73	22.00	77.19	11.96	13.40	47.02	65.38
3.05	12.05	23.40	82.11	12.93	23.00	80.70	12.64	16.70	58.60	73.80
3.27	12.53	23.80	83.51	13.06	23.50	82.46	12.91	17.70	62.11	76.13
3.49	12.76	23.80	83.51	13.12	23.60	82.81	13.08	20.50	71.93	79.42
3.70	12.92	24.40	85.61	13.22	23.80	83.51	13.24	22.00	77.19	82.10
3.92	13.01	24.40	85.61	13.27	23.80	83.51	13.32	22.50	78.93	82.69
4.13	13.06	24.40	85.61	13.33	23.80	83.51	13.38	22.50	78.93	82.69

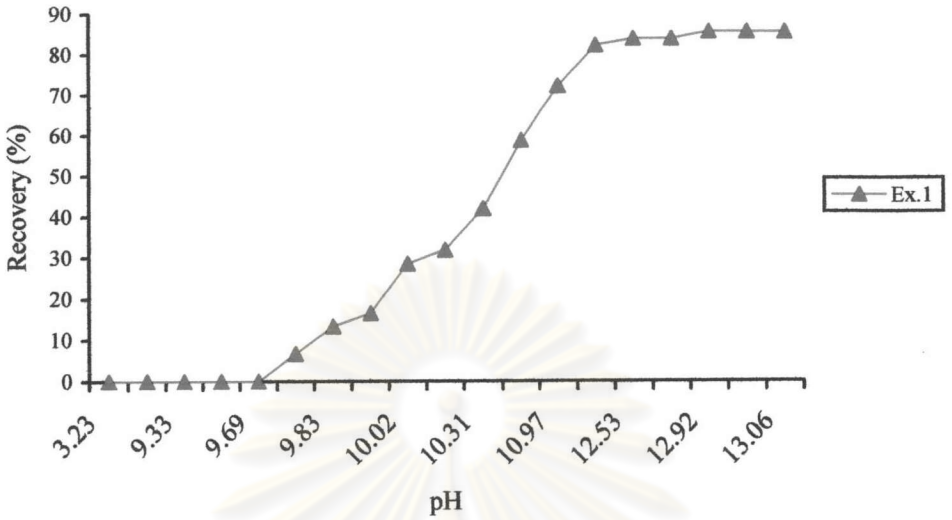


Figure 4.5 Effect of pH on the percentage recovery of triethylamine separation.

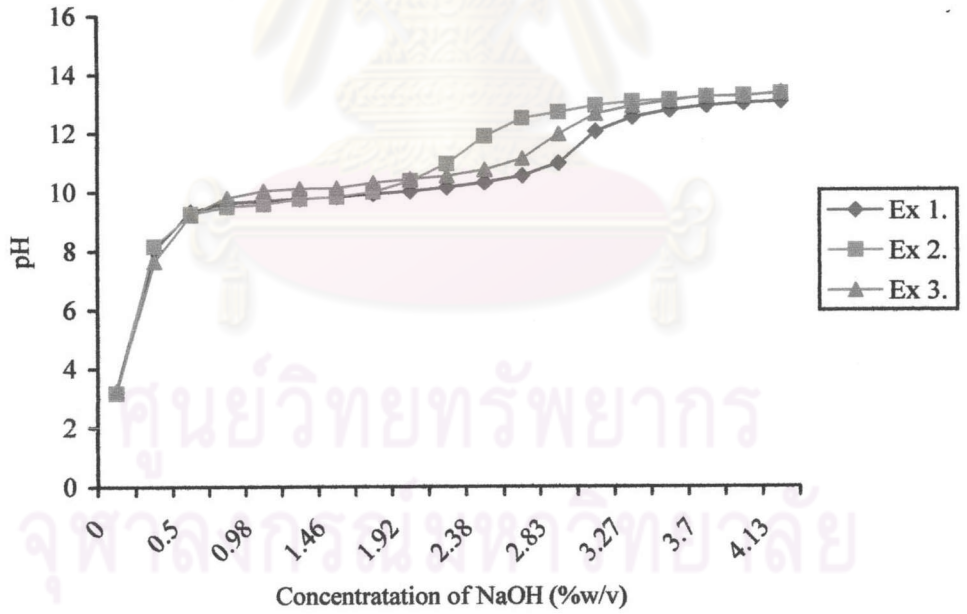


Figure 4.6 Effect of pH and the NaOH (%w/v) concentration of triethylamine separation.

4.2.3 Effect of Reaction Temperature on Triethylamine Separation

The effect of temperature on triethylamine separation from residual of mother liquor is shown in Table 4.4 and Figures 4.7-4.10. The details of all data and recovery percentage calculations are shown in Appendix D.The parameters of separation are as follows:

- Residual of mother liquor : 75 ml
  (Volume decrease 55% of residual mother after solvents separation)
- Reaction temperature : 25 ^{O}C to 75 ^{O}C
- Reaction time : 20 mins
- pH : 13
- NaOH concentration : 3.9 %w/v

Figures 4.7-4.8, the percentage recovery and purity of triethylamine increase with increasing temperature but the temperature must be less than 89°C because triethylamine could be partially evaporated. Figure 4.9 shows the decrease in percentage of water in triethylamine with increasing temperature.

Figure 4.10 shows the decrease in percentage of impurity in triethylamine with increasing temperature. When temperature increased from 25°C to 75°C, reaction enhanced the efficiency of separation phase. The triethylamine recovery of 81.61% and triethylamine concentration of 94.21% was obtained at reaction temperature of 75°C. The water content in triethylamine was 2.04% at high temperature of 75°C.

The key to the success of triethylamine separation is the property of inverse miscibility. At temperatures below 15^{O}C, triethylamine is completely miscible with water, above 15^{O}C triethylamine and water were only slightly miscible. A triethlamine chilled below 15°C could mixed with water [1]. Triethylamine was separated from the water-triethylamine mixture by heating the mixtures to temperatures above the miscibility point (about 55 °C) [18].

Table 4.4 Effect of reaction temperature on triethylamine separation from residual mother liquor.

Temp.	(%)															
	Experiment 1.				Experiment 2.				Experiment 3.				Average			
(^{O}C)	Recovery	TEA	H_2O	Impurity	Recovery	TEA	H_2O	Impurity	TEA	Purity	H_2O	Impurity	Recovery	TEA	H_2O	Impurity
25	78.50	94.00	2.05	3.95	75.70	94.04	2.64	3.35	74.77	94.92	2.10	2.98	76.32	94.32	2.26	3.43
35	76.64	94.00	2.02	3.98	76.64	94.19	2.57	3.24	72.90	94.29	2.03	3.68	75.39	94.16	2.21	3.63
45	80.37	93.58	2.01	4.41	79.44	94.21	2.46	3.33	76.64	94.83	1.94	3.23	78.82	94.21	2.14	3.66
55	80.37	93.68	2.03	4.29	79.44	94.38	2.24	3.38	76.64	94.95	2.00	3.05	78.82	94.34	2.09	3.57
65	82.24	94.02	2.05	3.93	82.24	94.35	2.11	3.54	78.50	95.00	1.93	3.07	80.99	94.46	2.03	3.51
75	82.24	93.58	2.01	4.41	82.24	94.02	2.18	3.51	80.37	95.04	1.93	3.03	81.61	94.21	2.04	3.65

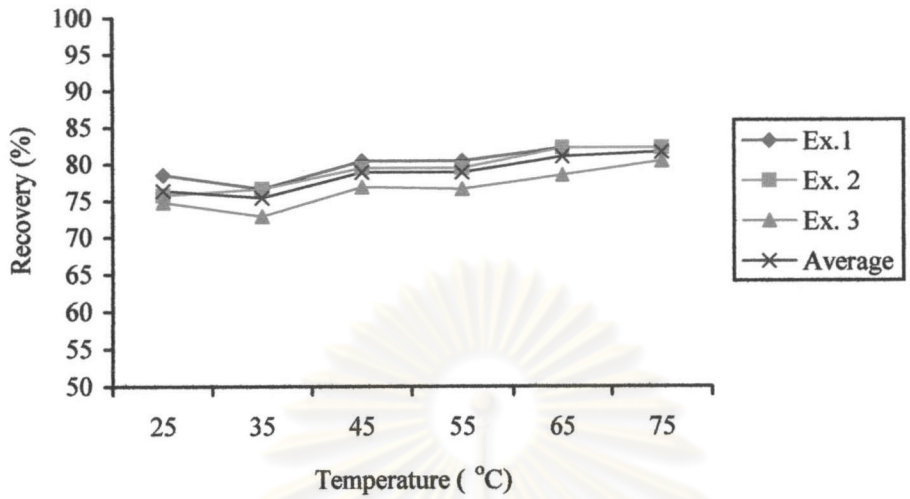


Figure 4.7 Effect of reaction temperature on the percentage recovery of triethylamine separation.

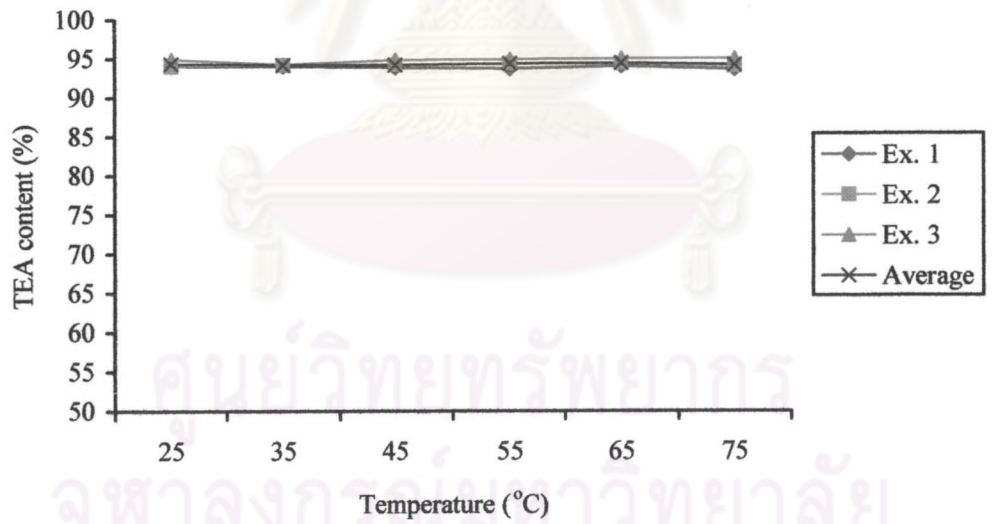


Figure 4.8 Effect of reaction temperature on the percentage TEA content of triethylamine separation.

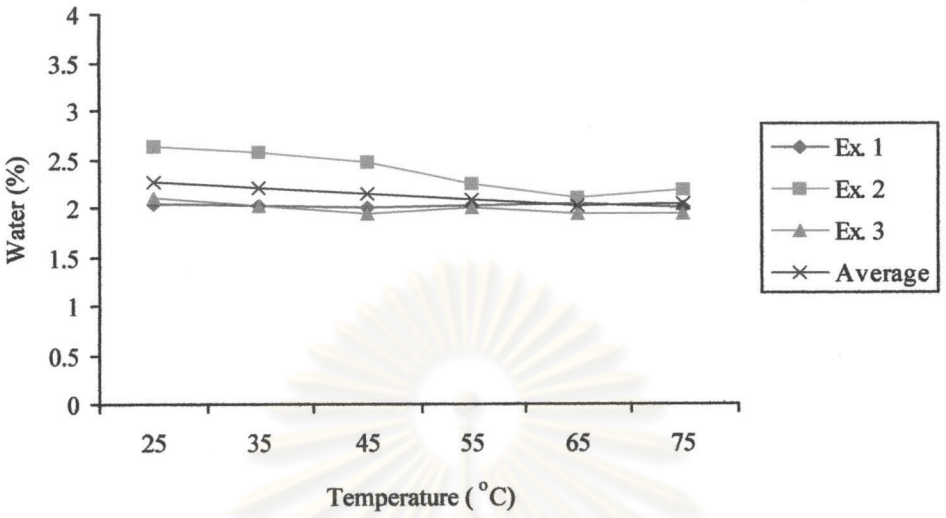


Figure 4.9 Effect of reaction temperature on the percentage water of triethylamine separation.

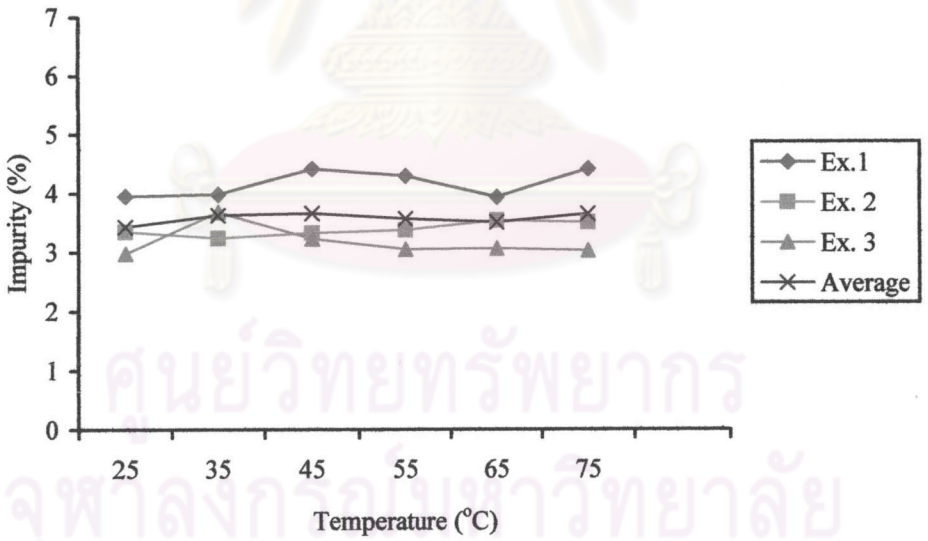


Figure 4.10 Effect of reaction temperature on the percentage impurity of triethylamine separation.

4.2.4 Effect of Reaction time on Triethylamine Separation

The effect of reaction time on triethylamine separation from residual of mother liquor is shown in Table 4.5 and Figures 4.11-4.14. The details of all data and recovery percentage calculations are shown in Appendix D. The parameters of separation are follows:

- Residual of mother liquor : 75 ml
(Volume decrease 55% of residual mother after solvents separation)
- Reaction temperature : 75 °C
- Reaction time : 5 to 30 mins
- pH : 13
- NaOH concentration : 3.9 %w/v

Figures 4.11-4.13 show that the percentage of recovery and the percentage of water in triethylamine increased with increasing time from 5 minutes to 30 minutes. Figures 4.12 shows that the percentage of purity of triethylamine decreased with time when reaction time was more than 15 minutes. The approximate reaction time was 15 minutes for separation and temperature was not less than 55°C, because triethylamine and water were only slightly miscible at low temperature.

Table 4.5 Effect of reaction time on triethylamine separation from residual of mother liquor.

Time	(%)															
	Experiment 1.				Experiment 2.				Experiment 3.				Average			
(min)	Recovery	TEA	H_2O	Impurity	Recovery	TEA	H_2O	Impurity	Recovery	TEA	H_2O	Impurity	Recovery	TEA	H_2O	Impurity
5	78.50	94.05	2.28	3.66	72.90	93.66	2.04	4.30	78.50	94.06	2.18	3.76	76.63	93.92	2.17	3.91
10	82.24	94.01	2.25	3.74	75.70	93.85	2.18	4.97	82.24	93.84	2.32	3.83	80.06	93.90	2.25	4.18
15	83.18	93.81	2.48	3.71	77.57	93.9	2.13	3.92	82.24	93.94	2.32	3.73	81.00	93.65	2.31	3.79
20	83.18	93.78	2.38	3.84	78.50	93.87	2.17	3.95	82.24	93.80	2.41	3.78	81.31	93.82	2.32	3.86
25	83.18	93.87	2.42	3.71	79.44	93.84	2.29	3.87	82.24	93.74	2.43	3.80	81.62	93.82	2.38	3.39
30	83.18	93.54	2.62	4.84	79.44	93.80	2.31	3.86	83.18	93.71	2.62	3.71	81.93	93.68	2.52	4.14

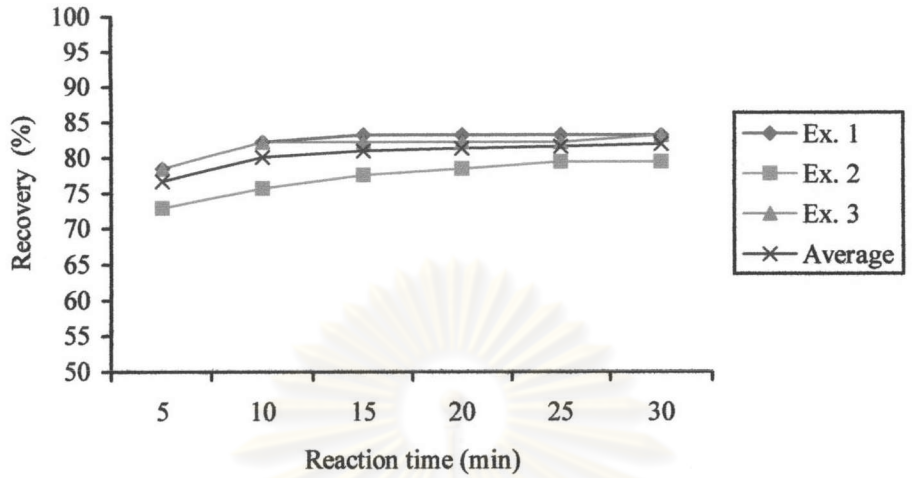


Figure 4.11 Effect of reaction time on the percentage recovery of triethylamine separation.

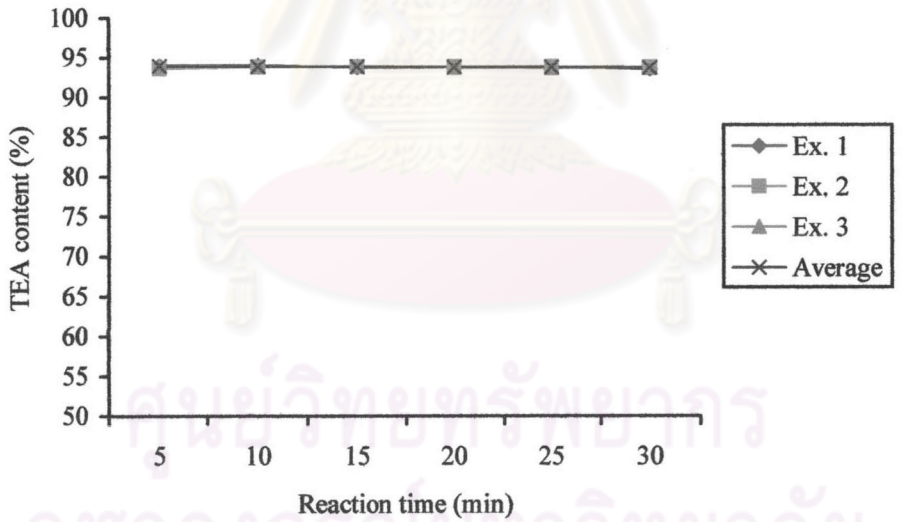


Figure 4.12 Effect of reaction time on the percentage TEA content of triethylamine separation.

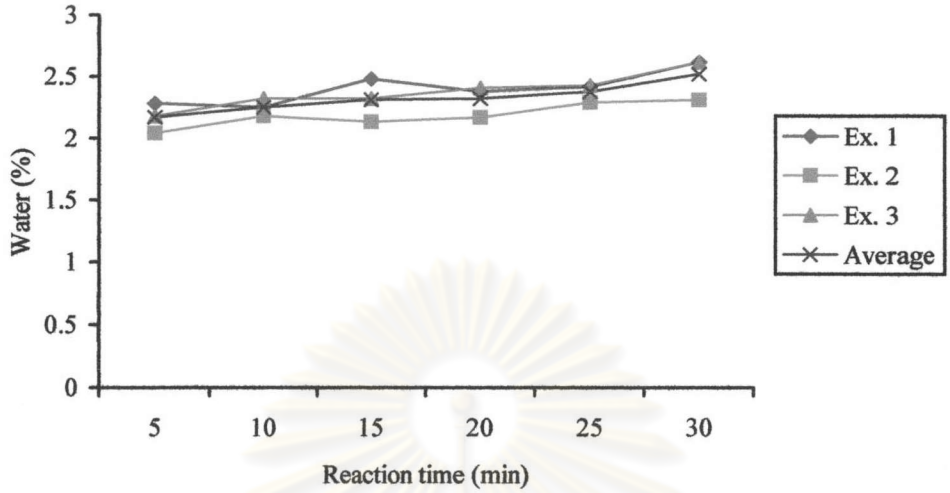


Figure 4.13 Effect of reaction time on the percentage water of triethylamine separation.

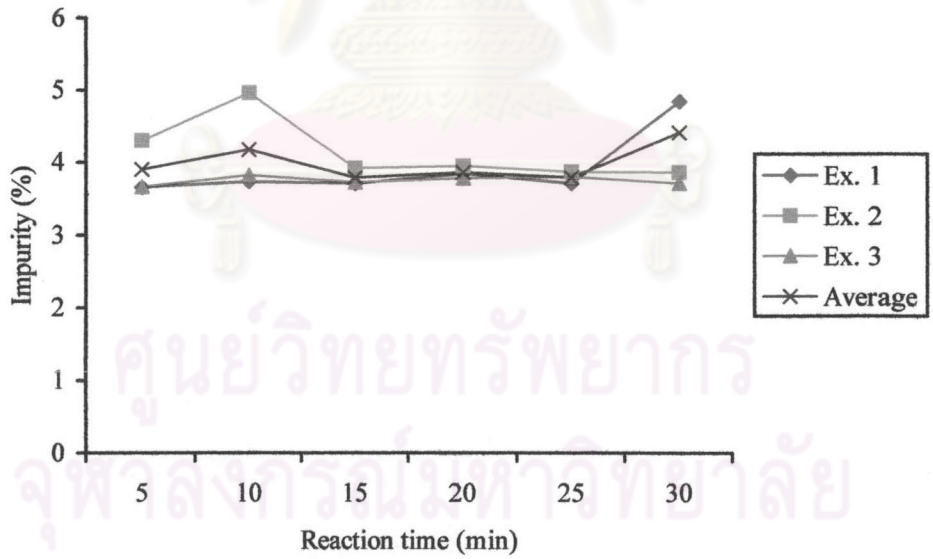


Figure 4.14 Effect of reaction time on the percentage impurity of triethylamine separation.

4.3 Analysis of Impurity of Triethylamine

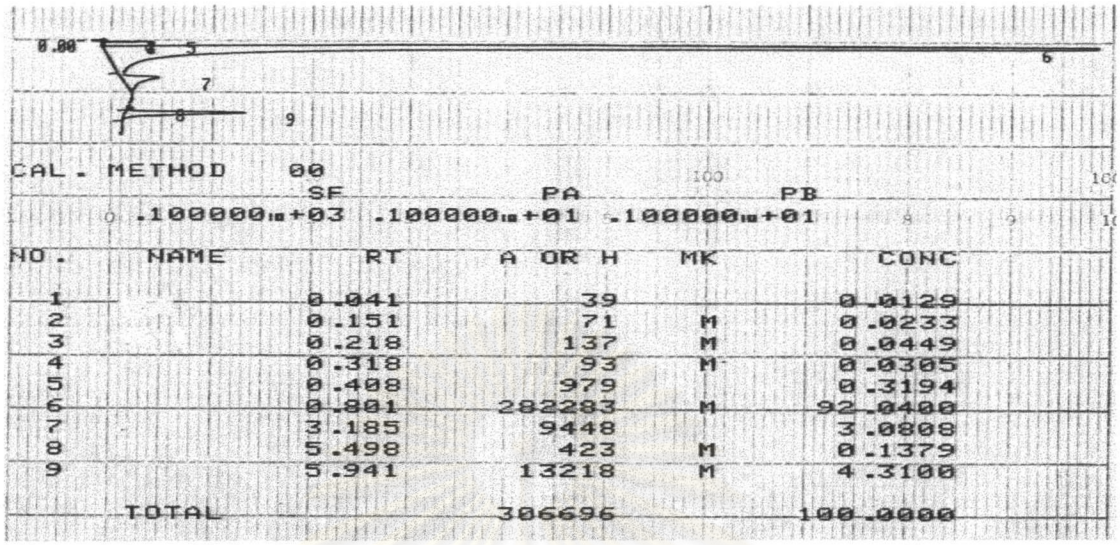
The results of the impurities of triethylamine analyzed by GC chromatography are shown in Figures 4.15-4.16 the recovered triethylamine product (See Figure G-3 in Appendix G) had the clear color.

Figure 4.15 shows GC chromatogram of recovered triethylamine, the retention time at 3.1 of the GC chromatographic peak was the water but the retention time at 5.9 was impurity in product.

Figure 4.16 shows GC chromatogram of recovered triethylamine mixed with 2,6-lutidine (addition of 2,6-lutidine 0.5 ml in 20 ml of recovered triethylamine), the peak at retention time of 5.9 increase. Therefore, peak of the impurity of triethylamine in Figure 4.15 was 2,6-lutidine.

In mother liquor, 2,6- lutidine was in the form of an amine salt. When 50%w/v NaOH was added, 2,6-lutidine could be separated in a free amine form, same as triethylamine. The reaction of 2,6-lutidine is shown in the following equation,

$$C_7H_9N^+HCl^- + NaOH \longrightarrow C_7H_9N: + NaCl + H_2O \quad \text{.....(4.2)}$$

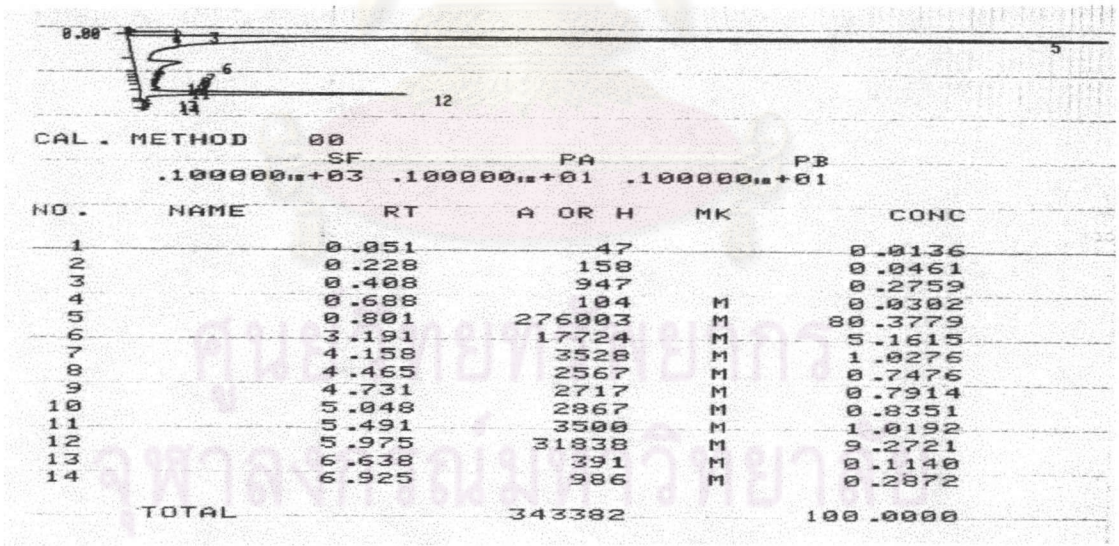


CAL. METHOD 00

	SF	PA	PB
	.100000E+03	.100000E+01	.100000E+01

NO.	NAME	RT	A OR H	MK	CONC
1		0.041	39		0.0129
2		0.151	71	M	0.0233
3		0.218	137	M	0.0449
4		0.318	93	M	0.0305
5		0.408	979		0.3194
6		0.801	282283	M	92.0400
7		3.185	9448		3.0808
8		5.498	423	M	0.1379
9		5.941	13218	M	4.3100
	TOTAL		306696		100.0000

Figure 4.15 GC chromatogram of recovered triethylamine.



CAL. METHOD 00

	SF	PA	PB
	.100000E+03	.100000E+01	.100000E+01

NO.	NAME	RT	A OR H	MK	CONC
1		0.051	47		0.0136
2		0.228	158		0.0461
3		0.408	947		0.2759
4		0.688	104	M	0.0302
5		0.801	276003	M	80.3779
6		3.191	17724	M	5.1615
7		4.158	3528	M	1.0276
8		4.465	2567	M	0.7476
9		4.731	2717	M	0.7914
10		5.048	2867	M	0.8351
11		5.491	3500	M	1.0192
12		5.975	31838	M	9.2721
13		6.638	391	M	0.1140
14		6.925	986	M	0.2872
	TOTAL		343382		100.0000

Figure 4.16 GC chromatogram of recovered triethylamine mixed with 2,6-lutidine.

4.4 Purification of Triethylamine

4.4.1 Fractional Distillation of Triethylamine

The impurity in triethylamine, 2,6-lutidine, was separated from triethylamine by distillation.

Tables 4.6-4.7 show the effect of distillation temperature on the separation of the triethylamine from 2,6-lutidine. Triethylamine recovery was about 92% at temperature ranged was 60°C-140°C (See Table 4.6). At higher temperature (120-140°C), more 2,6-lutidine separated and mixed with triethylamine. Therefore the temperature at the top of the column would be remained constant at 60°C-92°C during the distillation, triethylamine recovery was about 96-98.7% as shown in Table 4.7. The system water-triethylamine is the case of positive deviation of Raoult's law (Deviation Raoult's law, the boiling point of such a mixture is depressed, even below the normal boiling point of the more volatile of the two components) [19]. But triethylamine had approximately 2% moisture content, which could be separated by using drying agent.

Table 4.6 Purification of triethylamine by distillation, (temperature 60 °C to140°C), original volume of triethylamine = 300 ml.

Temp.-Top column	Time	Volume TEA	Composition		
			TEA	H_2O	Impurity
(°C)	(min)	(ml.)	(%)	(%)	(%)
60-92	120	275	96.51	2.33	1.16
92-120	15	10	87.28	0.32	12.40
120-140	20	9	0.07	0.30	99.63

Table 4.7 Purification of triethylamine by distillation, (temperature 60°C to 92°C), original volume of triethylamine = 150 ml.

Experiment	Temp.-Top column	Time	Volume TEA	Yield	Composition		
					TEA	H_2O	Impurity
	(°C)	(min)	(ml.)	(%)	(%)	(%)	(%)
1	60-87	45	37.5	25.00	94.81	4.64	0.55
	87-92	10	99.9	66.60	98.72	0.17	1.05
Total	60-92	55	137.4	91.60	97.65	1.40	0.96
2	60-87	50	36.0	24.00	-	5.59	-
	87-92	10	103.4	68.93	98.02	0.23	1.75
Total	60-92	60	139.4	92.93	-	2.33	-
3	60-87	45	21.0	14.00	-	5.54	-
	87-92	10	118.5	79.00	98.04	0.65	1.31
Total	60-92	55	138.5	93.00	-	1.39	-
Average	60-92	57	138	92.00	-	1.705	-

4.4.2 Demoisturization of Triethylamine by Adsorption and Absorption

Demoisturization of triethylamine by adsorption and absorption is shown in Tables 4.8-4.12 and Figures 4.17-4.23.

Figures 4.17-4.19 and Table 4.8 show the purity of triethylamine after adsorption with various drying agents, Figure 4.17 shows that the percentage purity of triethylamine is greater than 98.7% by adsorption and absorption using different drying agents. Figure 4.18 and Table 4.8 show that the impurity of triethylamine is less than 1.1%. Figure 4.19 shows that the water of triethylamine is less than 0.2%.

Tables 4.9-4.12 and Figure 4.20, show that when sodium hydroxide is used more than one time, the percentage water loss of triethylamine is only 2.2% which is less than other drying agents which is about 3.0-7.5%. Figure 4.21 shows the comparison of water absorption at the first time to third time of triethylamine by using CaH_2, K_2CO_3 and BaO, they were absorbed the water of triethylamine less at the time and high price.

When the water of triethylamine was adsorbed by molecular sieve, triethylamine was yellowish (impurity) because of the existence of little impurity could which not detected by gas chromatography. Therefore, molecular sieve could not be used to adsorb the water of triethylamine because the impurity of molecular sieve might mix in amoxicillin trihydrate and the price of molecular sieve is high.

Thus, solid sodium hydroxide was good absorbent for water and the price is low, sodium hydroxide (solid) absorbed water from residual mother liquor of amoxicillin trihydrate and formed sodium hydroxide solution accord to the following equation.

$$NaOH\ (solid) + nH_2O \longrightarrow NaOH\ (solution) \quad \text{.....(4.3)}$$

Thus, the purified triethylamine product obtained from this work has the following composition :

- Triethylamine content : not less than 97.5 %
- Water (moisture content) : not more than 0.2%

Table 4.8 Demoisturization of triethylamine by drying agents.

Drying Agents	Loss	Composition			Color
		TEA	H_2O	Impurity	
	(%)	(%)	(%)	(%)	
Original	-	96.51	2.33	1.16	Colorless
NaOH (solid)	2.50	98.78	0.12	1.10	Colorless
Molecular sieve	7.50	98.83	0.10	1.07	Yellowish
BaO	4.50	98.89	0.08	1.03	Colorless
CaH_2	4.50	98.96	0.07	0.97	Colorless
50%w/v NaOH	3.00	98.73	0.17	1.09	Colorless
K_2CO_3	5.50	98.86	0.12	1.01	Colorless

Table 4.9 Demoisturization of triethylamine by solid NaOH absorption.

Absorption at the time	Loss	Composition			Color
		TEA	H_2O	Impurity	
	(%)	(%)	(%)	(%)	
1st	2.50	98.78	0.12	1.10	Colorless
2nd	2.23	98.75	0.19	1.06	Colorless
3rd	2.23	98.79	0.18	1.03	Colorless
4th	2.23	98.89	0.18	0.93	Colorless
5th	2.23	98.87	0.17	0.96	Colorless
6th	2.23	98.77	0.20	1.03	Colorless
7th	2.20	98.78	0.16	1.06	Colorless
8th	2.23	98.85	0.17	0.98	Colorless

Table 4.10 Demoisturization of triethylamine by CaH_2 absorption.

Absorption at the time	Loss	Composition			Color
		TEA	H_2O	Impurity	
	(%)	(%)	(%)	(%)	
1st	4.50	99.00	0.05	0.95	Colorless
2nd	3.50	98.96	0.07	0.97	Colorless
3rd	2.23	98.63	0.17	1.20	Yellowish

Table 4.11 Demoisturization of triethylamine by K_2CO_3 absorption.

Absorption at the time	Loss	Composition			Color
		TEA	H_2O	Impurity	
	(%)	(%)	(%)	(%)	
1st	5.50	98.87	0.13	1.00	Colorless
2nd	3.00	98.79	0.25	0.96	Colorless
3rd	2.00	98.58	0.42	1.00	Colorless

Table 4.12 Demoisturization of triethylamine by BaO absorption.

Absorption At the time	Loss	Composition			Color
		TEA	H_2O	Impurity	
	(%)	(%)	(%)	(%)	
1st	4.50	98.88	0.11	1.01	Colorless
2nd	3.50	98.78	0.17	1.05	Colorless
3rd	3.00	98.61	0.23	1.16	Yellowish

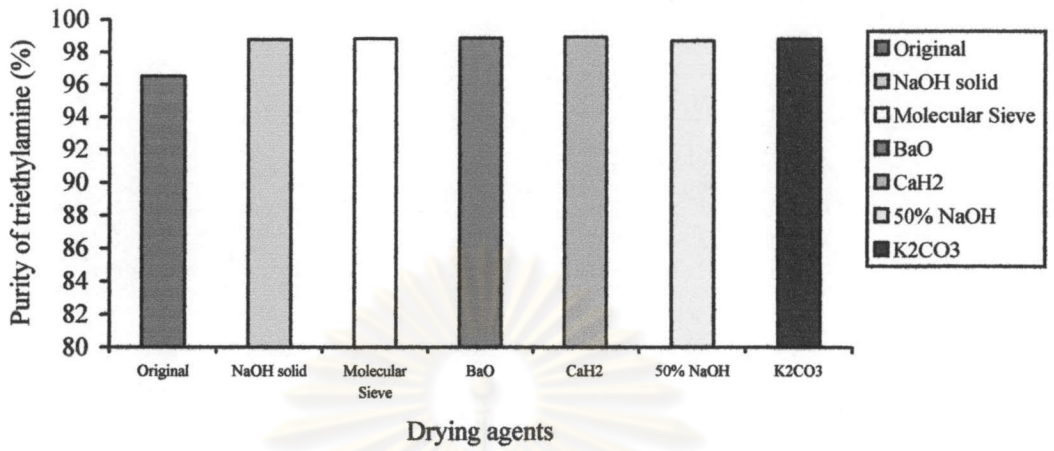


Figure 4.17 Comparison of the purity of triethylamine by using drying agents.

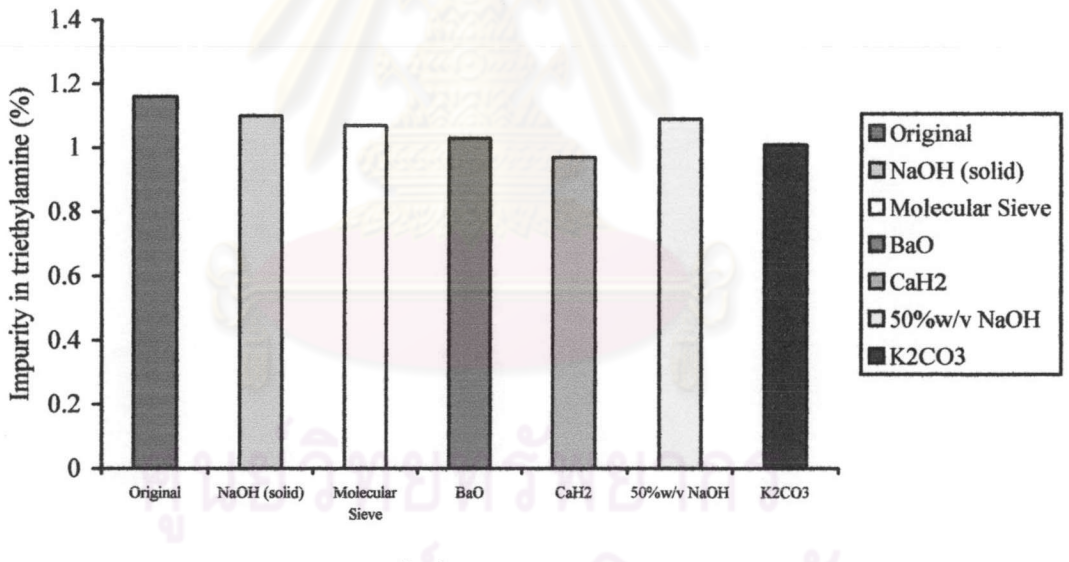


Fig.4.18 Comparison of the impurity of triethylamine by using drying agents.

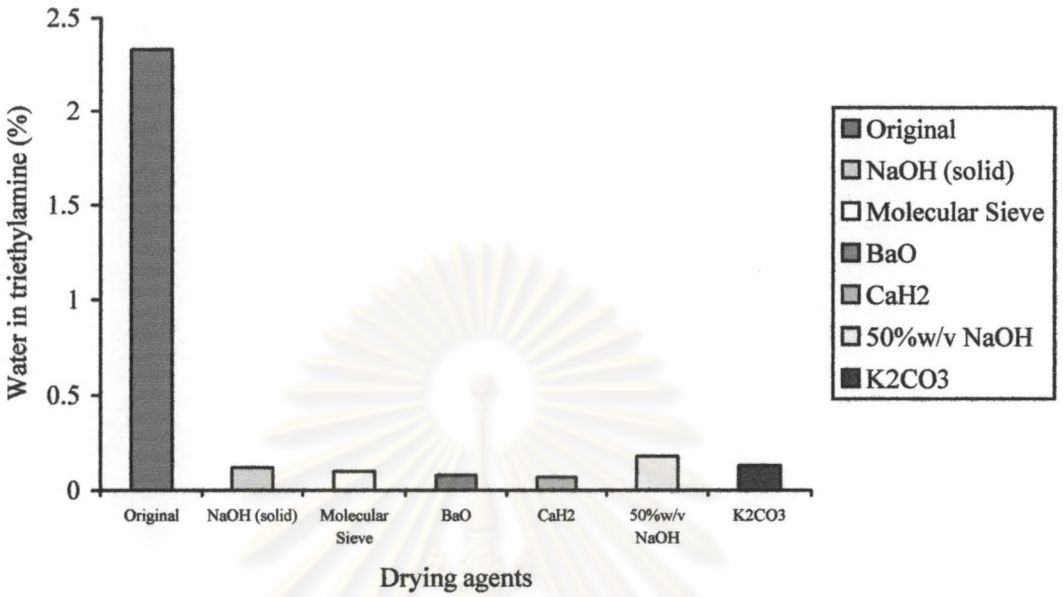


Figure 4.19 Comparison of the percentage water of triethylamine by using drying agents.

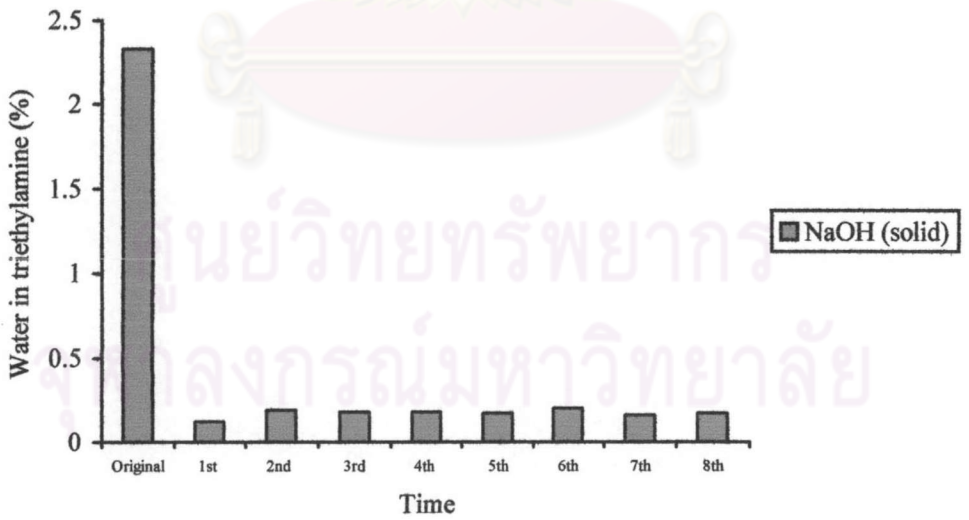


Figure 4.20 Comparison of triethylamine by using solid of NaOH in absorption.

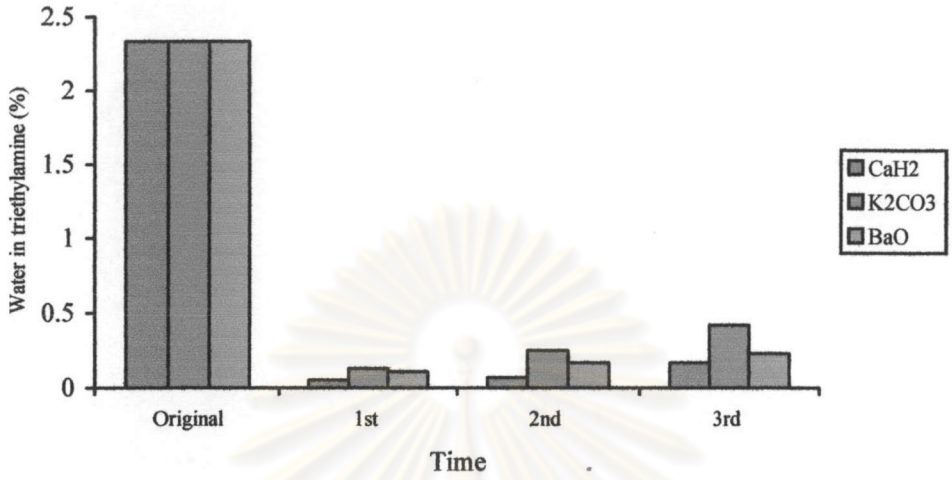


Figure 4.21 Comparison of triethylamine by using CaH_2, K_2CO_3 and BaO in absorption.

4.5 COD Analysis

The organic content of the wastewater can be estimated in several ways. The most common method is the oxygen demand method (COD), although the organic carbon measurement may also be used. The oxygen demand is the amount of oxygen that will be needed to stabilize the organic content of the effluent. The other two most common methods are the biochemical oxygen demand (BOD) and the chemical oxygen demand. In this work, the oxygen demand was used. The results of COD analysis of feed mother liquor and treated mother liquor are shown in Table 4.13 and Figures 4.22-4.24. An example of calculation is shown in Appendix E.

Figure 4.22 and Figure 4.24, show the comparison of COD values in the mother liquor between before and after solvent separation. The COD values of mother liquor were reduced to the range of appoximately11 % to 16 %.

From Figure 4.23 and Figure 4.24, the comparison of COD values in the mother liquor before and after separation of triethylamine using NaOH solution. The COD of mother liquor was reduced to the range of approximately 35 % to 50 %. Therefore, the separation of organic compound from mother liquor caused the decrease in COD approximately 50% to 60% from feed mother liquor.

Table 4.13 Comparison the COD of mother liquor before and after treatment.

Experiment	COD					
	Mother liquor fresh (mg/l)	After distillation of solvent (mg/l)	Reduction after distillation solvents (%)	Before separation using NaOH (mg/l)	After separation using NaOH (mg/l)	Reduction after NaOH treatment (%)
1	153,280	132,069	13.84	171,674	85,837	50.00
2	198,003	177,909	10.15	207,584	124,550	40.00
3	194,810	172,936	11.23	204,390	127,744	37.50
Average	182,031	160,971	11.57	194,549	75,377	42.07

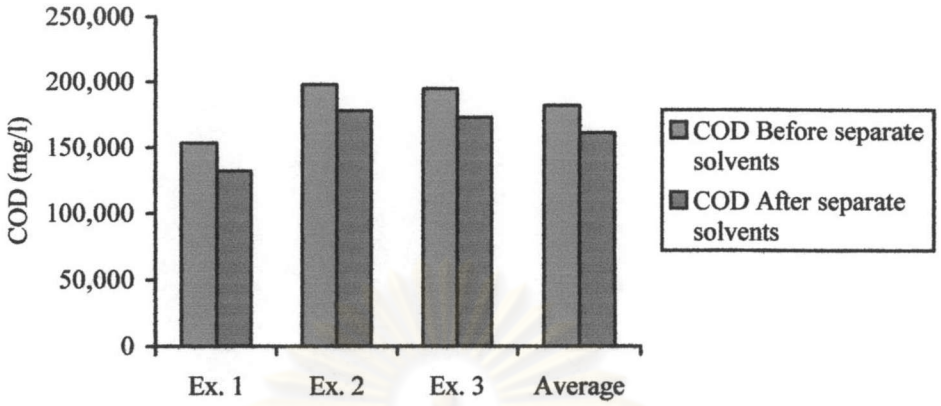


Figure 4.22 Comparison of the COD values between before and after the solvent separation.

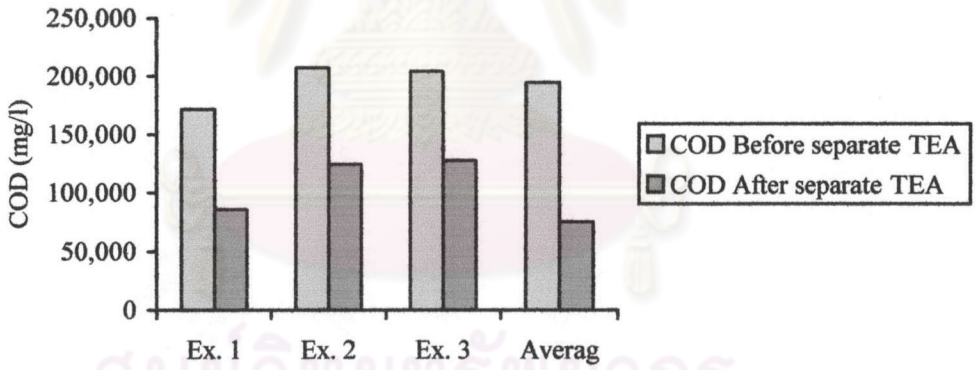


Figure 4.23 Comparison of the COD values between before and after the triethylamine separation.

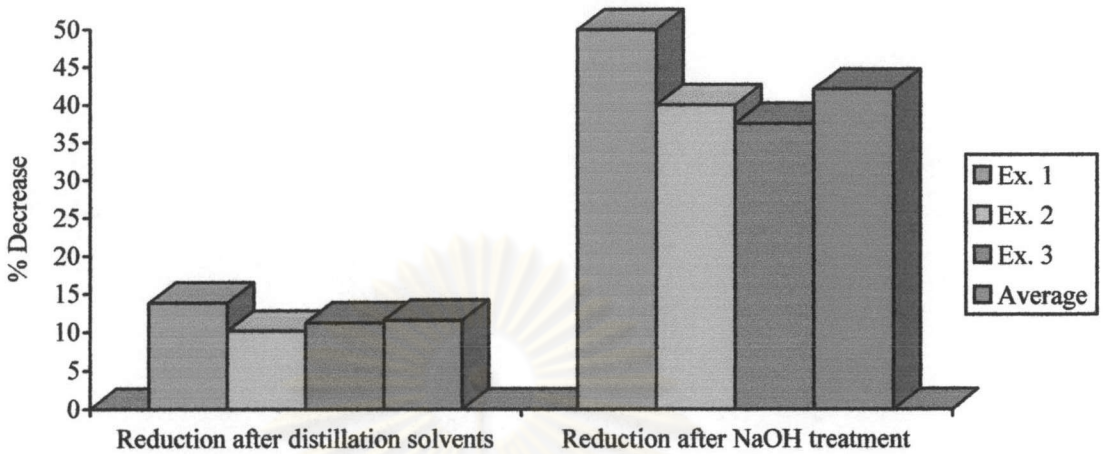


Figure 4.24 Comparison of the percentage COD values between reduction after distillation of solvents and NaOH treatment.

4.6 Preparation of Amoxicillin Trihydrate

The amoxicillin trihydrate was prepared from fresh triethylamine and recovered triethylamine (98.5% purity) and the results are compared in Tables 4.14-4.16. An example of calculation is shown in Appendix F. and Figures G-4 - G-6 in Appendix G.

Tables 4.14-4.16, show the comparison of amoxicillin trihydrate yield and percentage potency between using fresh triethylamine and recovered triethylamine. The yield and percentage potency of amoxicillin trihydrate of amoxicillin trihydrate prepared from fresh triethylamine and recovered triethylamine are the same and the peak of amoxicillin trihydrate from analysis (recovery triethylamine) not impurity peak. The yield of amoxillin trihydrate from recovered triethylamine was 1.736-1.739 and percentage potency was 98.8-99.4.

Therefore, the recovered triethylamine from residual mother liquor could be used in the production of amoxicillin trihydrate.

Table 4.14 Comparison of amoxicillin trihydrate prepared from fresh triethylamine and recovered triethylamine (triethylamine was demoistured by solid of NaOH).

	TEA (recovered) (solid of NaOH absorption)		TEA (fresh)	
	Yield	%Potency	Yield	%Potency
1	1.751	99.4	1.739	99.3
2	1.726	99.5	1.741	99.1
Average	1.739	99.4	1.740	99.2

Table 4.15 Comparison of amoxicillin trihydrate prepared from fresh triethylamine and recovered triethylamine (triethylamine was demoistured by 50%w/v NaOH).

Experiment	TEA (recovered) (50%w/v NaOH Absorption)		TEA (fresh)	
	Yield	%Potency	Yield	%Potency
1	1.739	99.0	1.744	99.6
2	1.745	98.6	1.746	98.6
Average	1.742	98.8	1.745	99.1

Table 4.16 Comparison of amoxicillin trihydrate prepared from fresh triethylamine and recovered triethylamine (triethylamine was demoistured by CaH_2).

Experiment	TEA (recovered) (CaH_2 Absorption)		TEA (fresh)	
	Yield	%Potency	Yield	%Potency
1	1.736	99.1	1.739	99.1