

CHAPTER III

EXPERIMENTAL

3.1 Materials

1. Isopropyl alcohol (IPA), AR grade : Labscan.
2. Cyclohexane (CHX), AR grade : Labscan.
3. High purity grade, air, hydrogen, : Thai Industrial Gas Co., Ltd.
and helium gases
4. Coulometric pyridine-free reagent : Merck

3.2 Apparatus and Instruments

1. Separatory funnel Witeg (250 ml)
2. Microsyringe 10 μ l capacity
3. Thermometer Temperature range from -10°C to $+110^{\circ}\text{C}$
4. Gas chromatography Hewlett Packard, model 6890 Series, detector type FID, manual injection.
5. GC capillary column HP-5 crosslinked 5% phenyl methyl siloxane.
6. Karl Fisher coulometer Mettler Toledo, model DL 37

3.2.1 Gas Chromatography.

1. Column HP-5 crosslinked 5% phenyl methyl siloxane.
2. Column length 30 m.
3. Column ID. 0.32 mm.
4. Carrier gas Helium gas, flow rate 1 ml/min.
5. Back pressure 5.1 psi

6. Column temperature	Initial temperature 40 °C with holding time 5 min., temperature program rate 20 °C/min., final temperature 100 °C with holding time 2 min.
7. Injector temperature	200 °C
8. Detector temperature	250 °C
9. Detector type	FID
10. Sample size	02 μ l splitless.

3.3 Study of the Phase Behavior of Ternary Liquid System for IPA – Water – CHX.

For determination of the solubility curve and plait point composition, two sets of experiments were used. For the first set, water and CHX were mixed according to Table 3.1 for determining the solubility curve. For the second set, IPA, water and CHX were mixed according to Table 3.2 for determining the plait point composition. Each sets of experiments were carried out at 26°C and 30°C, and then the results were compared.

3.3.1 Determination of Solubility Curve.

The volume of water and CHX were mixed according to Table 3.1. Each mixture was added with IPA and shaken until the turbidity produced. The volume of IPA was recorded. Percents by weight of IPA, water and CHX were calculated in each mixture.

Table 3.1 Mixing volume of water and CHX.

Sample No.	Mixing volume (ml)	
	Water	CHX
1	3.0	27.0
2	6.0	24.0
3	9.0	21.0
4	12.0	18.0
5	15.0	15.0
6	18.0	12.0
7	21.0	9.0
8	24.0	6.0

3.3.2 Determination of Plait Point Compositions.

The volume of CHX, water and IPA were mixed according to Table 3.2. Each mixture was shaken in separatory funnel about 1 min. and leave to separate. The compositions of each sample (upper layer and lower layer) were analyzed by using KF coulometer and GC.

Table 3.2 Mixing volume of CHX, water and IPA.

Sample No.	Mixing volume (ml)			Total volume (ml)
	CHX	Water	IPA	
1	45.0	45.0	10.0	100.0
2	45.0	35.0	20.0	100.0
3	45.0	25.0	30.0	100.0
4	45.0	10.0	40.0	100.0

3.4 Study of Operating Conditions by Heterogeneous Azeotropic Distillation.

The experiments were carried out in the production plant of nitrocellulose. A flow diagram for the dehydration of IPA is presented in Figure 3.1.

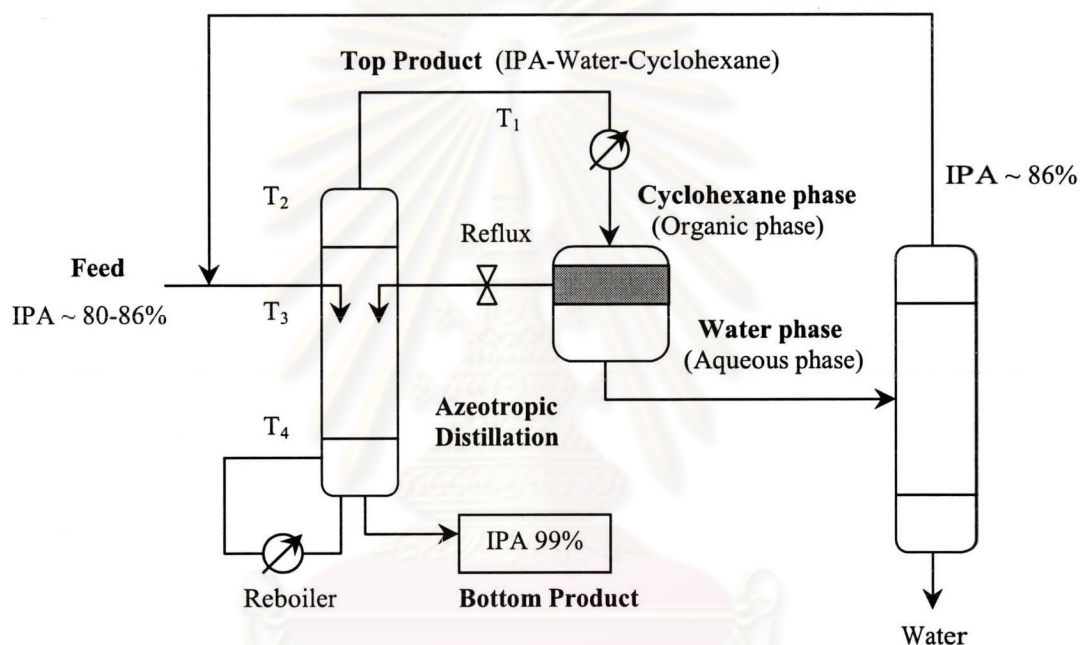


Figure 3.1 Azeotropic distillation process for separating IPA from water by using CHX as the entrainer.

To find a suitable operating condition, in this work two parameters were studied : feed rate and reflux rate. The effect of these two parameters on composition of bottom product, organic phase (CHX phase) and aqueous phase (water phase) were investigated.

3.4.1 Experiment by Varying Feed Rate.

The feed rate that used in this work were 8.0, 9.0, 10.5, 11.0 and 12.0 L/min. For the constant feed rate, the sample from feed, bottom product, CHX phase and water phase were taken every two hours during each experiment. The temperature at T_1 , T_2 , T_3 , T_4 (see Figure 3.1) and product rate were also recorded. For a constant feed rate, each experiment took 8 hours at constant reflux rate. The compositions of each sample were analyzed by using KF coulometer and GC.

3.4.2 Experiment by Varying Reflux Rate.

The reflux rates were varied by varying the valve, which controlled the volume of CHX to the distillation column, from 94 – 99% at a constant feed rate. When the system was in steady state; the sample from feed, bottom product, CHX phase and water phase were taken every one hour during each experiment. The temperature at T_1 , T_2 , T_3 , T_4 (see Figure 3.1) and product rate were also recorded. The compositions of each sample were analyzed by using KF coulometer and GC. The procedure was repeated by changing feed rate.

3.4.3 Experiment for Determination of Optimum Feed Rate and Reflux Rate.

From 3.4.1 and 3.4.2, the suitable feed rate and reflux rate were chosen to give the optimum results of azeotropic distillation. In this case, purity of product (IPA) was required to be 99%. When the condition was kept at optimum feed and reflux rate, the samples from feed, bottom product, CHX phase and water phase were taken every two hours. The temperature at T_1 , T_2 , T_3 , T_4 (see Figure 3.1) and product rate were also recorded. The compositions of each sample were analyzed by using KF coulometer and GC.

3.5 Determination of Vapor Composition by Material Balance.

In this work, the sample of top product composition or vapor composition (see Figure 3.1) cannot be taken for analysis. But the composition of vapor could be calculated by using material balance. Comparison of the results of vapor composition and plait point composition was made.



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