



Chapter 4

Experimental Work

4.1 Chemicals

1. n-Butanol : Carlo Erba Co. Ltd., Italy.
2. Acetone : Carlo Erba Co. Ltd., Italy.
3. Ethanol : Carlo Erba Co. Ltd., Italy.
4. Acetic acid : Carlo Erba Co. Ltd., Italy.
5. Butyric acid : Ajax chemicals Co. Ltd., Australia.
6. Demineral Water

4.2 Equipments

1. Peristaltic Pump : MP tube pump-3, EYELA Rikakikai Co. Ltd., Japan.
2. Silicone Rubber Tubing : Silex Co. Ltd., U.K.
inside diameter 2 mm., thickness 0.25 mm.
inside diameter 2 mm., thickness 0.50 mm.
inside diameter 2 mm., thickness 1.00 mm.
3. Rotary Vacuum Pump : E2M1.5, 220/240V, 50-60Hz, Edwards, U.K.
4. Pirani Vacuum Gauge : PRH10K, Edwards, U.K.
5. Vacuum Measurement : Pirani 501 (Pre-set to 200/240V), Edwards, U.K.
6. Condenser Unit : dip-type cold trap which dipped in dry ice
7. Thermocouple : Pt100 type MT201, Hanyoung Co. Ltd., Korea.
8. Temperature Indicator : type Hy850S, Hanyoung Co. Ltd., Korea.



4.3 Experimental Procedures

4.3.1 Sorption Process Experiment

This experiments were designed to determine the ability of solvents and acids sorption in the silicone pieces at various temperatures (40, 50, 60, and 70 °c) Therefore, sorption experiments were carried out by using a piece of 1 gram of silicone tubing, which was the same as what was used in the pervaporation process experiments. Firstly, the initial weights of the tubing, and mixtures were measured separately. Afterthat, the silicone piece was immersed in the mixtures which was kept in a constant temperature bath. Concentration in g/L of acetone, butanol, ethanol, acetic acid, and butyric acid in one liter of mixtures were 0.6, 0.7, 0.25, 0.5, and 0.2, respectively. The mixtures were periodically (for 24 hours) agitated by hand to ensure that equilibrium was reached. Next, the silicone pieces taken from the mixtures were dried at 120 °c and weighted. The residual mixtures were weighted and analyzed for the concentrations of each components.

For the acetone-butanol fermentation broth (see the preparation in Appendix A), the sorption process procedures were the same as mentioned above processes.

The experimental results of distribution coefficient in water-acetone-butanol-ethanol-acetic acid-butyric acid mixtures and acetone-butanol fermentation broth are shown in Appendix A.

4.3.2 Pervaporation Process Experiments.

Figures 4.1, 4.2 and 4.3 show experimental apparatus silicone hollow fiber membrane module, schematic diagram and silicone hollow fiber membrane module for pervaporation, respectively. Feed solutions were circulated by a peristaltic pump between a reservoir of feed solution (immersed in a constant temperature bath) and the inside of the hollow fiber

membrane module at a constant flow rate of 1 lh.^{-1} . The shell side of the hollow fiber module was kept at reduced pressure by the vacuum pump. The feed solution permeated from the inside to the outside of hollow fiber membrane by pervaporation. The operation time of pervaporation was 4 hours.

The permeation vapor outside the membrane was then condensed by a condenser unit. The condensation volumes were collected after 2 hours of operation at steady state. The steady state was checked by permeate measuring (See Appendix B). The temperature of feed solution was kept constant. The pressure on the permeation side was maintained at 2, 10, and 30 torr by regulating valve which was connected to a vacuum pump. The permeate pressure was measured by a pirani vacuum measurement. The thickness of silicone rubber membrane were 0.25, 0.5 and 1.0 mm.

The feed solution were water-acetone-butanol-ethanol-acetic acid-butyric acid mixtures and acetone-butanol fermentation broth which obtained with Clostridium acetobutylicum. These solutions were the same as what used in sorption process (See Appendix B).

Mass balances at steady state were checked to ensure that there was no experimental error(See Appendix B). The experimental results of pervaporation in water-acetone-butanol-ethanol-acetic acid-butyric acid mixtures and acetone-butanol fermentation broth are shown in Appendix A.

4.3.3 Analysis

Acetone, n-Butanol, ethanol, acetic acid and butyric acid were determined by gas chromatography using Shimadzu Model Gc 14 Ag equipped with a flame ionization detection. Separation took place in a $2 \text{ m.} \times 0.125$ in stainless steel column (pack with Parapak Q 80-100 mesh) at 210°C ,and N_2 was used as a carrier gas. The injection temperature was 185°C ,and the

detector temperature was 210°C. The analysis of chromatography data was recorded by a Chromatopac CR 1 A record integrator. Flow rate of carrier gas was 50 g/l, retention time of butanol, acetone, ethanol, acetic acid, butyric acid were 5.76 ,2.11 ,1.36 ,2.91 ,and 12.89 mins, respectively.



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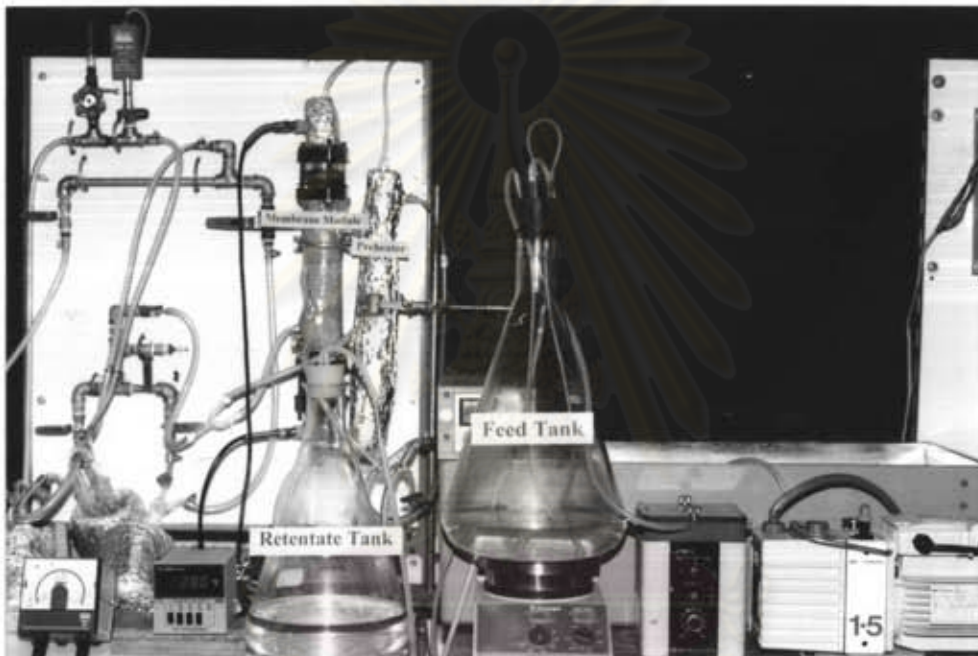


Figure 4.1 Experimental apparatus assembly

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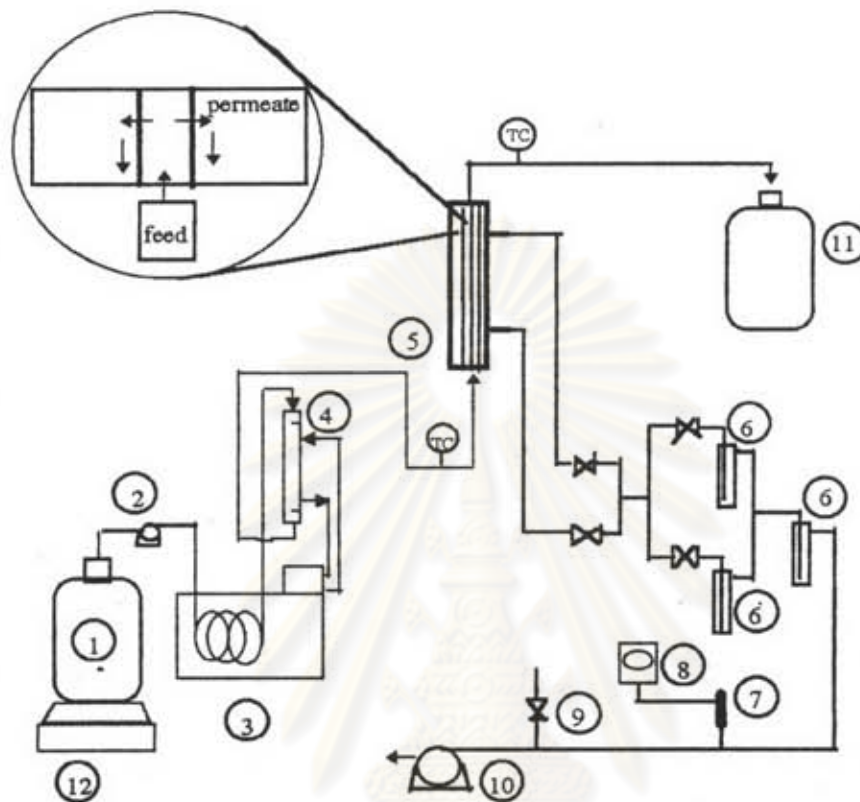


Figure 4.2 Schematic diagram of apparatus for pervaporation by hollow fiber membrane module.

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|---------------------------------|------------------------------|
| 1. Feed Reservoir | 7. Pirani Vacuum Measurement |
| 2. Feed Pump | 8. Pirani Vacuum Gauge |
| 3. Constant Temperature Bath | 9. Regulating Valve |
| 4. Preheater | 10. Vacuum Pump |
| 5. Silicone Hollow Fiber Module | 11. Retentate Tank |
| 6. Cold Trap | 12. Magnetic Stirrer |
| TC Thermocouple | |



Figure 4.3 Silicone Hollow Fiber Membrane Module.

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