# CHAPTER III

# EXPERIMENTAL

## 3.1 Chemicals

Methanol (commercial grade) and isopropanol (analytical grade) obtained from the Merck Co., Ltd. are used without further purification.

Whatman poly(tetrafluoroethylene) (PTFE) (type WTP) membrane was purchased from the Bang Trading Co., Ltd. The membrane is laminated onto a non-woven polypropylene (PP) support web for improved strength and facilitate handling. It has a pore size of 0.2 μm, porosity of 72 %, and thickness of 130 μm.

## 3.2 Instruments and apparatus

The circulating pump (Piston pump, Model QSY, FMILAB) and the vacuum pump (Oil rotary pump, Type RP-S 100) were obtained from Fluid Metering Co., Ltd. and Makashi Co., Ltd., respectively. A vacuum gauge has the pressure range of 0-76 cmHg and a thermometer has the temperature rang of 0-100 °C were selected for this study. Two three-necked vessels, having capacity of 500 $cm^3$, were used as the feed vessel and permeate vessel. The compositions of feed and retentate were analyzed by FID gas chromatography (GC) on a HP 5890 gas chromatography (Hewlett Packard) using DB-Wax column (diameter of 0.25 mm, length of 40 m, film thickness of 0.25 μm)

## 3.3 Experimental procedure

### 3.3.1 Design and installation the pervaporation apparatus in laboratory scale

The pervaporation apparatus was performed by employing PVC pervaporation cell as shown in Figure 3.1. The pervaporation cell is composed of two PVC disc compartments which are packed together using of four coupling bolts and nuts. Between the compartments, two mating of rubber rings were used

to seal the cell. The PTFE/PP membrane, with 4 cm in a diameter and 12.57 $cm^2$ in an effective area, was fixed in a specially designed pervaporation cell. Then, the membrane was backed with filter fabric support for improved strength. Figure 3.2 shows a laboratory scale of the pervaporation test apparatus used in this research. A feed solution was flowed through the upper compartment and the retentate was recirculated to a feed vessel. Vacuum was applied to the lower compartment of the cell connected with a permeate vessel.

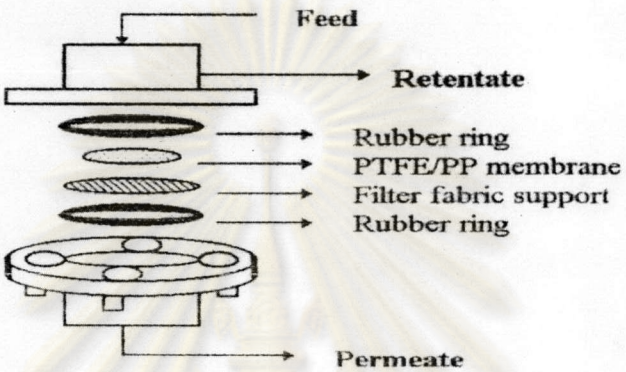


**Figure 3.1** Configuration of the pervaporation cell used in this study.

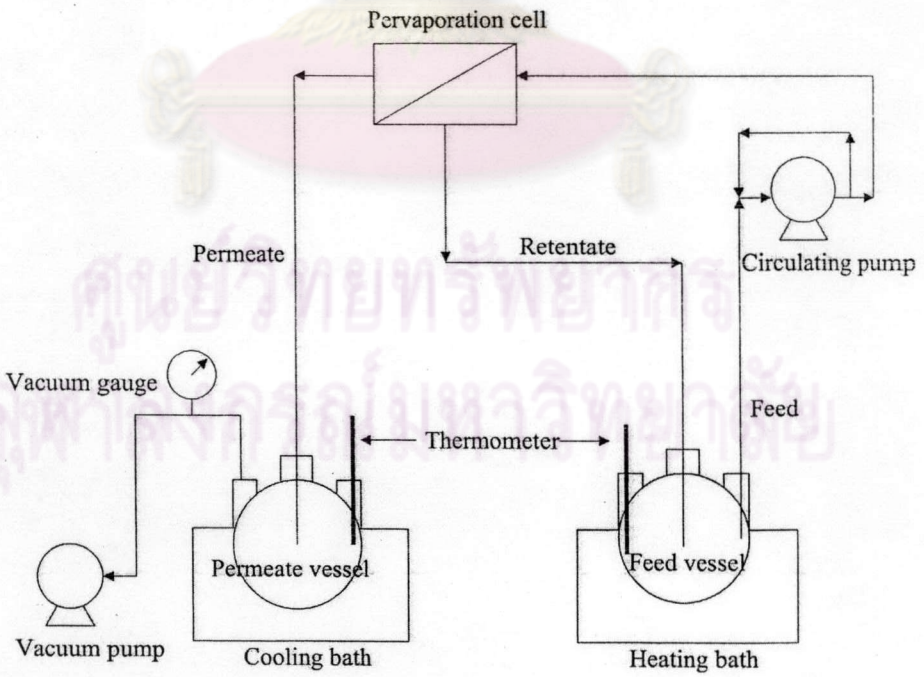


**Figure 3.2** Schematic representation of pervaporation test apparatus used in this work.

3.3.2 Operation of pervaporation system

3.3.2.1 Startup the pervaporation system

The methanol aqueous solution was prepared as feed solution. It was weighed and poured into the feed vessel. Then, the solution was heated until the desired temperature using a water bath. The permeate vessel was cooled using a ice bath. The vacuum pump and the circulating pump were opened to evacuate the permeate on downstream side and to circulate the feed solution through the pervaporation cell, respectively.

3.3.2.2 Measurement between the operating of pervaporation system

When the feed solution began to flow into the pervaporation cell, the operating time was counted. After 1 h, the system was shutdown. The permeate and retentate samples were weighed and collected using a 5 ml syringe into the glass bottle for GC analysis. The fluxes and selectivity were calculated to evaluate the performance of the pervaporation as shown in Appendix B.

3.3.2.3 Shutdown the pervaporation system

In case of high temperature, the feed solution was cooled to the room temperature. Then, the vacuum pump and the circulating pump were closed. The solution which retained in lines and cell was brought out from the system. After that, the pressure inside the pervaporation cell was returned to the normal condition.

3.3.3 The influence of operating parameters on the pervaporation performance

3.3.3.1 The effect of feed concentration

200 g of an aqueous solution containing 10 wt % methanol was poured into the feed vessel and circulated through the pervaporation cell by the circulating pump. The temperature of feed solution was maintained at 30 °C using a water bath. The flow rate of feed solution was adjusted at 17 ml $min^{-1}$. The system was evacuated to the pressure of about 34 cmHg by the vacuum pump. When the system was operated for 1 hour under these conditions, the permeate and retentate were weighed. The feed and retentate were analyzed for their composition by GC. The tests were then performed using 20 to 60 wt % methanol in water. Each experiment was carried out in duplicate.

3.3.3.2 The effect of downstream pressure

The feed solutions were bringed into the pervaporation system at 30 °C, and the feed flow rate of 17 ml $min^{-1}$. The pervaporation experiments were carried out with the values of downstream pressure of 34, 20, and 12 cmHg at constant of feed concentration, temperature, and feed flow rate. After finished the experiments, the permeate and retentate were weighed. The feed and retentate were analyzed for their composition by GC. Each experiment was carried out in duplicate.

3.3.3.3 The effect of feed temperature

The feed solutions were bringed into the pervaporation system at the downstream pressure of 34 cmHg, and the feed flow rate of 17 ml $min^{-1}$. The pervaporation experiments were carried out with the temperatures of 30, 40, and 50 °C at a constant of feed concentration, downstream pressure, and feed flow rate. After finished the experiments, the permeate and retentate were weighed. The feed and

retentate were analyzed for their composition by GC. Each experiment was carried out in duplicate.

3.3.3.4 The effect of feed flow rate

The feed solutions were bringed into the pervaporation system at the temperature of 30 °C, and the downstream pressure of 34 cmHg. The pervaporation experiments were carried out with the feed flow rates of 17, 10, and 3.5 ml $min^{-1}$ at a constant of feed concentration, temperature and downstream pressure. After finished the experiments, the permeate and retentate were weighed. The feed and retentate were analyzed for their composition by GC. Each experiment was carried out in duplicate.

3.3.4 Determination of methanol concentrations

The compositions of feed and retentate were analyzed by gas chromatography (GC) on a HP 5890 gas chromatography (Hewlett Packard), fitted with DB-Wax column which has a inside diameter of 0.25 mm, length of 40 m, film thickness of 0.25 μm, and equipped with a flame ionization detector (FID). The following GC conditions were used for the analyses.

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|---|---|
| Column initial temperature | 30 °C |
| Column initial time | 1 minute |
| Column parameter rate | 20 °C $min^{-1}$ |
| Column final temperature | 110 °C |
| Column final time | 1 minute |
| Injector temperature | 220 °C |
| Detector temperature | 220 °C |

This study used the internal standard method to calculate the concentrations of methanol in feed and retentate as shown the detail in Appendix A.