CHAPTER 3 EXPERIMENT

Experiments were performed to observe permeability of organic compounds through a carbon membrane. Experiments were carried out in pervaporation and vapor permeation modes of operations. The carbon membranes were prepared by carbonizing Kapton® polyimide at 600 °C. The chemicals, apparatus and experimental procedures are described in this chapter.

3.1 Equipment and Materials

3.1.1 Experimental apparatuses

3.1.1.1 Tubular Furnace
3.1.1.2 Hot air Oven (Binder FD 115)
3.1.1.3 Gas Chromatograph equipped with FID detector (Agilent 6890)
3.1.1.4 Gas tight syringe volume 5 mL
3.1.1.5 10 µL-Micro syringe
3.1.1.6 Stopwatch (Casio HS-5)
3.1.1.7 Stainless steel cylindrical chamber
3.1.1.8 Glassware such as graduated cylinder, beaker, pipettes, volumetric flask etc.
3.1.1.9 Wash bottle

3.1.2 Pervaporation and Vapor permeation Apparatus

A schematic diagram of the pervaporation apparatus is illustrated in Figure 3.1

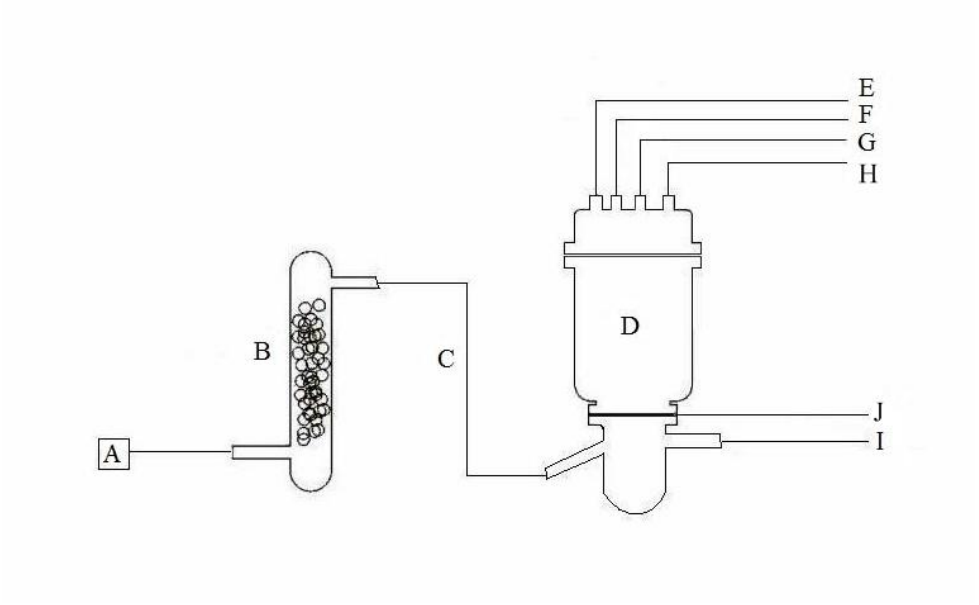

Figure 3.1 Schematic diagrams of the pervaporation apparatus

The pervaporation apparatus consists of following parts

A. Air pump
B. Silica gel
C. Air flow line
D. Feed container
E. Heater
F. Stirrer
G. Thermostat
H. Thermometer
I. Permeate outlet
J. Membrane

A schematic diagram of the vapor permeation apparatus is illustrated in Figure 3.2

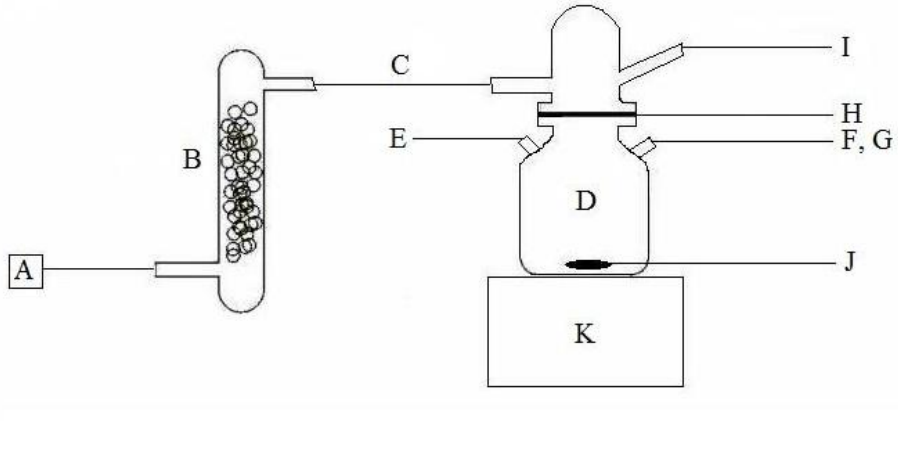

Figure 3.2 Schematic diagrams of the vapor permeation apparatus

The vapor permeation apparatus consists of following parts

A. Air pump
B. Silica gel
C. Air flow line
D. Feed container
E. Heater
F. Thermostat
G. Thermometer
H. Membrane
I. Permeate outlet
J. Magnetic bar
K. Magnetic stirrer

3.1.3 Chemicals and Materials

3.1.3.1 Kapton® polyimide
3.1.3.2 Methanol purity 100 %
3.1.3.3 Ethanol purity 99.5 %
3.1.3.4 Acetone purity 99.99 %
3.1.3.5 Isopropanol purity 99.8 %
3.1.3.6 High-purity nitrogen gas
3.1.3.7 Silica gel

3.2 Experimental Procedures

This research was divided into two parts. For the first part, carbon membranes were prepared by carbonizing Kapton® polyimide at 600 °C. In the second part, the membranes were tested on the pervaporation and vapor permeation of organic compounds under a constant temperature of 55 ° C. The experiments were carried out using a single-component and bi-component feeds. The experimental procedures were as follows:

3.2.1 Membrane Preparation

As received Kapton® polyimide, approximately 46 μm thick, was cut into circular pieces of 4.8 cm in diameter. The film was sandwiched between two round stainless steel disks, 5 cm in diameter, and placed inside a stainless steel cylindrical chamber whose dimensions were 6.5 cm in length and 6 cm in diameter. After placing the chamber in a tubular furnace, nitrogen was flown through at the rate of 60 ml/min for 30 min. Temperature of the furnace was first increased from room temperature to 100 °C and then increased stepwise, 50 °C for each step until the temperature reached 600 °C. The heating rate was approximately 10.67 °C/min and there was a 12 minute interval between each heating step. After the temperature was 600 °C, the furnace was switched off and allowed to naturally cool to room temperature. The thickness of the polyimide film remained essentially unchanged after carbonization.

3.2.2 Pervaporation and Vapor permeation Experiments

The apparatus for pervaporation (PV) and vapor permeation (VP) experiments were carried out with apparatus shown in Figure 3.1 and 3.2 respectively. Effective membrane area in contact with the feed solution was 5.31×10^{-2} cm^2. The feed volume and temperature were 150 ml and 55 °C, respectively. The permeate side of the membrane was swept by dry air at the flow rate of 50 ml/min. For each test, both process were allowed to proceed for 1 hour before the permeated was collected every 20 min and analyzed for its concentration by a gas chromatograph equipped with FID. Every experiment was repeated at least 3 times.

The experimental procedures were as follows:

3.2.2.1 Preparation of 150 mL mixture containing ethanol 80 wt % with methanol 20 wt %

3.2.2.2 Place a carbon membrane between the o-rings by using joint clips to hold the permeation cell.

3.2.2.3 Fill the feed container with 150 mL of mixture. Then, install the stirrer blade, thermostat, heater bar, and thermometer into test equipment. After that, switch on the stirrer blade and set the temperature at 55 °C.

3.2.2.4 After the temperature of the feed reached 55 ^{o}C, the process was allowed to proceed for 1 hour before the permeate was collected from the sampling port (position I in figures 3.1 and 3.2).

3.2.2.5 Analyze for vapor permeate concentration at every 20 min by a gas chromatograph equipped with FID. The permeability (P) was calculated by equation 2.2 and 2.3 for PV and VP respectively.

$$P_i = \frac{J_i \; l}{(x_i^F \gamma_i P_i^{sat} - \; y_i^P P^P)} \tag{2.2}$$

$$P_i = \frac{J_i \; l}{(P_i^F - P_i^P)} \tag{2.3}$$

3.2.2.6 The experiment is repeated from step 3.2.2.1 to 3.2.2.5 using following organic compounds and their mixtures as feeds

The single-component feeds were methanol, ethanol, isopropanol and acetone.

The bi-component feed were as follows:

Acetone:Isopropanol (IPA)	(20:80 wt. %)
Methanol:Acetone	(20:80 wt. %)
Methanol:Ethanol	(20:80 wt. %)
Acetone:Ethanol	(50:50 wt. %)