CHAPTER III
EXPERIMENTAL

3.1 Test Materials and Coating Procedure

3.1.1 Test Materials

The properties of test materials are presented in Appendix A.

a) 0.15 mm Teflon

b) Fluorodyn Solution made with Viton FKM (Viton)

c) Fluorodyn Caulk made with Viton FKM (Viton)

d) 0.70 mm Fluorodyn Sheet (Viton Sheet)

e) Epoxy Vinyl Ester Resin (Derakane Resin)

3.1.2 Coating Procedure

Teflon should be cleaned before coated with test material.

a) Fluorodyn Solution Coating

Multiple coats of Fluorodyn solution could be applied to obtain the desired thickness. Let the coated Fluorodyn solution dry, approximately an hour, before applying another coat and allow drying for, at least, 48 hours after the last coat. The curing of the coating was done in the oven at 121.1°C for one hour.

b) Epoxy Vinyl Ester Resin Coating

Epoxy vinyl ester resin was mixed with the solution named MEKP to form the polymer chain before coating on Teflon. Allowed the coated epoxy vinyl ester resin to dry for few days depending on the thickness.

3.1.3 Coated Material Thickness Determination

Since the density of test material and the area the gas mixture diffuses through are known the test cell using in ASTM F739-91 are as shown in Table 3.1 and 20.4 cm^3 respectively. The coated material thickness was determined directly from the weight measurement, i.e., Eq.3.1.

$$\text{Test material thickness} = \frac{\text{weight of test material}}{\text{density of test material} \times \text{area of diffusion}} \quad (3.1)$$

Table 3.1 The density of test materials used in the present work.

Test material	Density (g cm^{-3})
Fluorodyn[a]	2.000
Epoxy vinyl ester resin[b]	0.957

[a] Goodfellow company catalog 1998/1999

[b] Dow chemical company product information form no. 125-00270-398X SMG

3.2 Terminology

ASTM F739-91 (1991) defines important terms used in the method of gas permeability determination.

3.2.1 Permeation

This is the process by which a chemical moves through a test material on a molecular level.

3.2.2 Steady State Permeation

This process has a constant rate of permeation that occurs after the breakthrough when the chemical contact is continuous and all forces affecting permeation have reached equilibrium.

3.2.3 Challenge Gas

This is the gas that is used to challenge the test material specimen.

3.2.4 Collection Medium

This is a liquid or gas that does not have an effect on the measured permeation and in which the test chemical is freely soluble or adsorbed to a saturation concentration grater than 0.5 weight or volume percent.

3.2.5 Elapsed Time

This is the time at which the challenge gas first passes through the test material and the time at which the collection gas is withdrawn to the analytical instrument.

3.2.6 Breakthrough Time

This is the elapsed time measured from the start of the test to the sampling time that immediately proceeds the sampling at which the test chemical is first detected.

3.3 Apparatus for Hydrogen and Chlorine Permeability Study

The apparatus for determining hydrogen and chlorine permeability was assembled as shown in Figure 3.1 and 3.2, respectively. The test cell consists of two identical chambers which are made of Pyrex glass. These sections have a nominal size of 51-mm (2.0 in) diameter as indicated in the ASTM Standard issued under the fixed destination F739-91. The test material is mounted in between the two-sections. The gas mixture needed to be

analyzed continuously flows through the first section, known as 'a challenge side' and that gas mixture is referred to 'a challenge gas'. The collection gas, argon, which flows continuously through the other section, known as 'a collection side', carries the challenge gas permeated through the test material to determine the gas composition in an analytical instrument.

Two analytical devices were used to analyze the collection gas. The gas chromatograph (GC Varian 3600) was used to detect and measure the hydrogen concentration in the collection gas. Another device, the Detector tube, was used to detect and measure the concentration of chlorine in the collection gas for determining chlorine permeability. The method of using the detector tube in conjunction with ASTM 739-91 has been described by Sarner and Henry (1989). The detector tube used in the experiment was a Kitagawa precision gas detector tube for chlorine (Tube No. 109SB) with the detection limit from 0-10 ppm. Other standard equipment used in this experiment included a flow meter, mass flow controllers and a rotameter for controlling gas flow rates. In addition, an oven was used to allow the tests to be conducted at elevated temperature (80°C). The temperature of the test cell is measured by a thermocouple with an associated data acquisition system.

3.4 Methodology

3.4.1 Determination of Hydrogen Permeability

A 5% hydrogen gas mixture with 95% argon and pure argon were used as a challenge gas and a collection gas respectively.

1. The test material was installed within the test cell.

2. In Figure 3.1, the set up of the experimental apparatus was shown.

3. After installing the test cell into the oven, the temperature was held constant at 80°C.

4. A continuous flow of the collection gas to the collection side was maintained at the flow rate of 10 cm^3/min.

5. GC Varian 3600 was set to initiate sampling and analyze the collection gas every 10 minutes in order to establish the base line values of hydrogen concentration before changing to the challenge gas.

6. The challenge gas flowed continuously to the challenge side by passing the equivalent of five challenge chamber volumes (volume of a challenge chamber is 100 cm^3) in one minute. The challenge gas flow rate was reduced to 10 cm^3/min

7. The hydrogen concentration determined by GC Varian 3600 and the elapsed time were recorded.

8. Until a constant hydrogen concentration in the challenge gas was achieved, testing was continued. (See Figure 4 for examples of the constant gas concentration with time in ASTM F739-91, 1991).

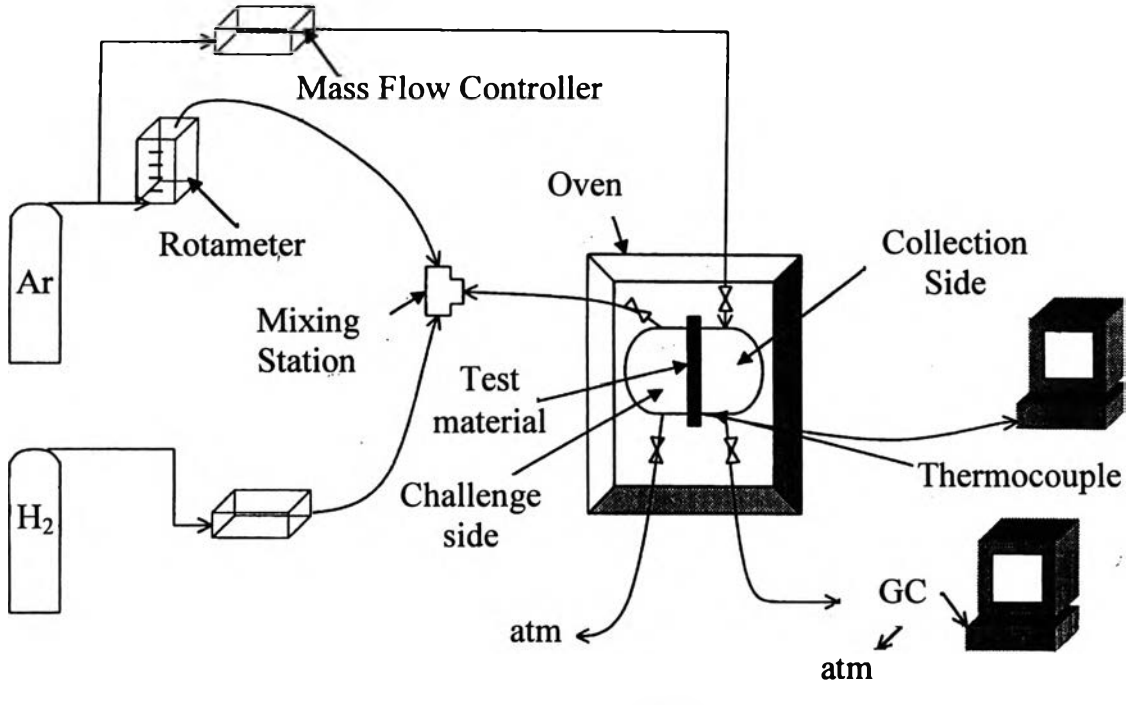

Figure 3.1 The apparatus for the determination of the hydrogen permeability.

3.4.2 Determination of Chlorine Permeability

Test materials were tested with pure dry chlorine gas.

1. The solution of 20% NaOH solution was prepared by using 400 g of sodium hydroxide solid and 1600 cm^3 of distilled water.

2. Before the dry chlorine gas apparatus was started, the following procedures had been done.

- ❖ The fume hood was cleaned.
- ❖ The rotameter was calibrated by using argon gas by the flow meter.
- ❖ A Calculation was made for the corresponding flow rate of chlorine.

3. The test material was mounted within the test cell.

4. Figure 3.2 showed the experimental apparatus with pure dry chlorine and argon as a challenge and collection gas respectively. Before installing the test cell in the oven, gas leaking was checked by flowing argon into the chamber.

5. When no leaks were found, the flow of chlorine and the toxic monitor for safety were started.

6. Previous procedures from step 3-6 in section 3.4.1 were carried on.

7. The elapsed time was recorded until the Breakthrough Time (B/T) in the detector tube was observed (Breakthrough Time in the detector tube tests was determined as the first indication of stain).

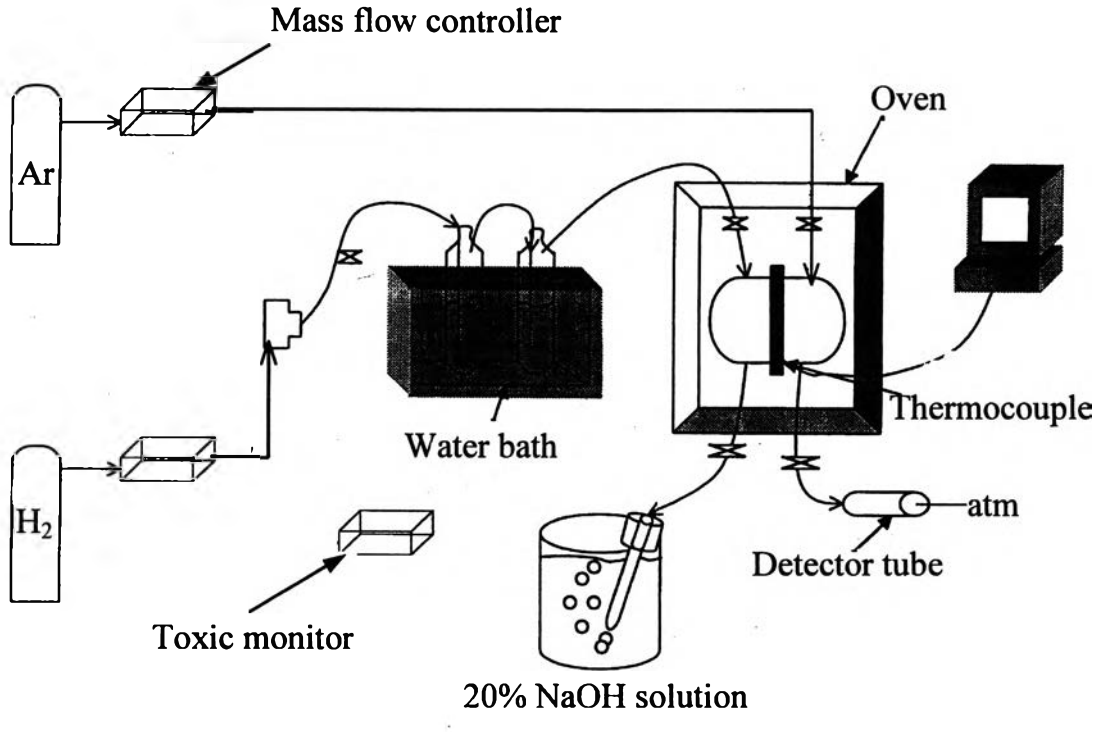

Figure 3.2 The apparatus for the determination of the chlorine permeability.

3.5 Calculation Based on Experimental Data

3.5.1 Determination of Hydrogen Permeation Rate

Calculation for systems using a continuous flow of collection gas in an open-loop are applicable to a system where fresh argon gas transports hydrogen from the collection side to the GC Varian 3600 as shown in Figure 3.3.

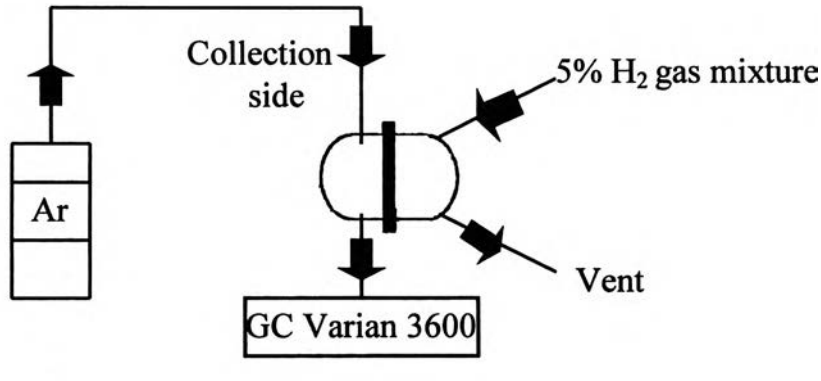

Figure 3.3 Example of the set-up for continuous flow of the collection gas.

The permeation rate of hydrogen in the collection gas at any time is

$$P_i(t_i) = \frac{C_{Hi} F_{Ar}}{A} \qquad (3.2)$$

where:

$P_i(t_i)$ = permeation rate, $\mu g\ m^{-2}\ min^{-1}$

t_i = time at which concentration of hydrogen in collection gas was C_{Hi}, min

C_{Hi} = concentration of hydrogen at time t_i, $\mu g\ m^{-3}$

F_{Ar} = flow rate of fresh argon through the test cell, $m^3\ min^{-1}$

A = cross sectional area to flow, m^2.

3.5.2 Determination of the Chlorine Permeation Rate

According to the use of the detector tube as the analytical method in ASTM F739-85 (1986), the detector tube measures the absolute amount of the permeant. It is necessary to determine the absolute amount of the permeant, which is hydrogen, in the tube at any one time by,

$$P_M = C_b(\text{conversion factor})(\frac{1\ m^3}{1000\ L})\ V_a \qquad (3.3)$$

where:

P_M = absolute amount of permeant at any one time, mg

C_b = tube calibration concentrations as scale unit on the tube, ppm

conversion factor = $\frac{\text{molecular weight of challenge gas}}{\text{gas molecular volume}}$, the molecular volume of a gas using an ideal gas law is 24.06 L per mol at 20°C

V_a = volume of gas that is used to generate the calibrations, equals to 100 mL for Kitagawa detector tube.

Then the permeation in $\frac{mg}{m^2}$ unit is determined using Eq. 3.4,

$$\text{permeation} = \frac{P_M}{A_T} \qquad (3.4)$$

where A_T represents test cell exposed area, which is 0.00204 m^2.

A graph of the permeation versus time is constructed as show in Figure 3.4. The slope of the linear portion of the graph is the permeation rate. At low times, the transition region before steady state occurs and the sharp curve is plotted. The permeation rate is calculated from the first region after the transition period.

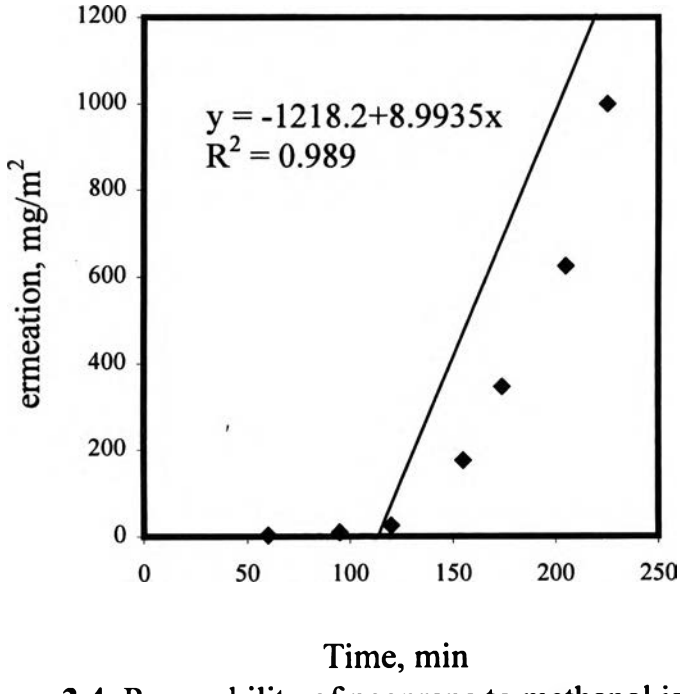

Figure 3.4 Permeability of neoprene to methanol is an example of the relationship between the permeation (in $\frac{mg}{m^2}$) versus time.

Thus, the permeation rate of chlorine can be determined from the slope of the linear portion, which is presented in the graph of permeation of chlorine versus time.

3.5.2 Determination of Permeation Coefficient

After the value of the permeation rate is obtained using either method in section 3.5.1 or 3.5.2, the permeability coefficient of the gas is calculated from,

$$\phi_i = \frac{P_i L_j}{A \Delta p_i} \qquad (3.5)$$

where:

ϕ_i = permeability coefficient of permeant i in the material, $\frac{\text{mol}}{\text{m Pa sec}}$

P_i = permeation rate of permeant i through the material, $\frac{\text{mol}}{\text{sec}}$

L = thickness of the material, m

A = cross sectional area to flow, m^2

Δp_i = partial pressure difference of permeant i on either side of material, Pa.