



## CHAPTER III EXPERIMENTAL

### 3.1 Materials

Poly(vinyl alcohol) 99+% hydrolyzed with a molecular weight of 89,000-98,000 and poly(acrylic acid) with an average molecular weight of 450,000 were supplied from Sigma-Aldrich Company. All chemicals were used without further purification.

### 3.2 Instruments and Equipment

#### 3.2.1 Viscometer

Polymer blended solution (polymer concentration of 10 wt%) viscosity was measured by Brookfield viscometer Model DV-III using spindle no. 21 at room temperature (25°C). Approximately 0.5 mL of the mixture was placed in the center of the small sample adapter. This sample was sheared for 5 min to ensure through contact between the solution and the spindle. The viscosity of the mixture was then measured at desired shear rates. The viscosity was reported as centi-Poise (cP) at the highest %torque.

#### 3.2.2 BET Surface Area Analyzer

The surface area, pore volume, and pore size were investigated using an Autosorp-1 gas sorption system (Quantachrome Corporation).

#### 3.2.3 Fourier transform infrared spectrophotometer (FTIR)

The structural characterization was done by a Nicolet/Nexus 670 with a 32 scans at a resolution of 4  $\text{cm}^{-1}$ . and recorded in frequency range of 4000-400  $\text{cm}^{-1}$ . Thin films of homopolymers and interpolymer complexes (5 wt%) were cast on ZnSe windows, and then the films were dried by hot-air blower for 5 min.

#### 3.2.4 Differential scanning calorimeter (DSC)

Thermal characteristics of aerogel, including the glass transition temperature ( $T_g$ ) and melting temperature ( $T_m$ ) were determined by a Perkin-Elmer DSC-7 instrument and analyzed in one scan. The instrument was calibrated with an Indium standard. The sample (7-10 mg.) was sealed in an aluminum pan. The heating was

carried out from 30°C to 300°C with a heating rate of 10°C/min in a N<sub>2</sub> atmosphere. The calorimetric melting temperature and heat of fusion ( $\Delta H_f$ ) of each sample were determined from the maximum and the area of the melting peak, respectively.

#### 3.2.5 Thermal Gravimetric Analysis (TGA)

Thermal stability of the aerogel was determined by a Dupont thermo gravimetric analyzer under N<sub>2</sub> atmosphere. The degradation temperature (T<sub>d</sub>) was reported at the differential thermal analysis peak. The temperature range between 30°C to 550°C and heating rate of 10°C/min.

#### 3.2.6 Scanning Electron Microscope (SEM)

The morphology of the inperpolymer complex aerogels was studied by a JEOL/JSM 5200 at 15 kV. The aerogel samples were torn and coated with a thin layer of gold.

#### 3.2.7 Wide-Angle X-ray Diffraction (XRD)

X-ray diffraction pattern of the aerogel were recorded at 2 $\theta$ / $\theta$  range of 5 degree to 25 degree by a RIGAKU RINT 2000 with CuK $\alpha$  as an X-ray source. The operating condition is 40 kV and 30 mA with 5 degree/min scanning speed and scan step 0.2 degree using Ni filter and 1 degree, 1 degree, 0.3 mm slits.

#### 3.2.8 Mechanical Testing

Tensile strength, Young's modulus and percentage elongation at break were measured by using a LLOYD Model LRX Mechanical Universal Testing Machine with a 500 N load cell, a crosshead speed of 10 mm/min and a 15 mm gauge length. The aerogel test samples were cut into a rectangular shape with a size of 10×50 mm and the thickness of 2 mm. The results are the average of five specimens.

### **3.3 Methodology**

#### 3.3.1 PVA/PAA interpolymer complex solution preparation

A 5 wt% aqueous solution of PVA was prepared by dissolving PVA in distilled water at 100°C in a round bottom flask equipped with a reflux condenser. The dissolution of PVA was completed in 4 h. The solution was left at room temperature before further mixing. A 5 wt% aqueous solution of PAA was prepared

by dissolving PAA with distilled water in a glass beaker with a cover glass. PAA was stirred at room temperature by a magnetic stirrer until the homogeneous solution was obtained. PAA solution was left overnight to eliminate the bubbles generated during stirring.

### 3.3.2 Hydrogel Preparation by Freezing-Thawing

PVA/PAA blends with 50/50, 60/40, 70/30, 80/20, 90/10, and 100/0 (v/v) ratios were prepared. Each blend was poured into polystyrene mold. Samples were submitted to seven freezing-thawing cycles to obtain hydrogels. With the exception of the first cycle, each cycle involved lowering the temperature to  $-40^{\circ}\text{C}$ , standing at this temperature for 2 h, and then rising the temperature to room temperature ( $25^{\circ}\text{C}$ ) for 1 h. The first cycle differed from the others because of a longer standing time at  $-40^{\circ}\text{C}$  (12 h). The obtained hydrogels were kept in the refrigerator. For the reference samples, freezing-thawing method is not applied.

### 3.3.3 Aerogel Preparation by Freeze-Drying

For the reference samples, the PVA/PAA blends were suddenly frozen at  $-40^{\circ}\text{C}$  then freeze-drying directly without applying freezing-thawing method. The aerogels were kept in desiccator before characterizations.

### 3.3.4 Enhancement Aerogel Properties by Heat-treatment

Aerogel obtained from freezing-thawing technique was selected to study effect from heat-treatment. The aerogels were heated in a vacuum oven at various temperature range from 100, 130, 150, and  $180^{\circ}\text{C}$  and the time is varied from 10, 20, 30, 40, 50, 60 min. Thermal characteristic such as melting temperature,  $T_m$  and decomposition temperature,  $T_d$  was investigated by DSC and TGA.

### 3.3.5 Water Absorption

The water absorption of the prepared aerogels was analyzed by casting polymer solution in a  $2.5 \times 2.5 \text{ mm}^2$  polystyrene mold with a thickness of 0.5 mm. The samples were then placed in distilled water, allowed to swell for 1 week at room temperature. Water absorption can be calculated from below equations;

$$\text{Water absorption (\%)} = \frac{W_{wet} - W_{dry}}{W_{dry}} \times 100\% \quad (1)$$