



CHAPTER III EXPERIMENTAL

3.1 Materials

3.1.1 Chemicals

3.1.1.1 Bisphenol-A (Sigma Aldrich), (100% purity), 500 g

3.1.1.2 Para-Formaldehyde (Merck), (99%wt.), 500g

3.1.1.3 Aniline (Fluka), (99.8% purity), 100g

3.1.2 Solvents

3.1.2.1 Xylene (Lab-Scan), 4L

3.2 Measurements

3.2.1 Fourier Transform Infrared Spectroscopy (FT-IR)

A Fourier Transform Infrared Spectroscopy (FT-IR); Nicolet Nexus 670, was used to identify structural characteristics of polybenzoxazine in absorption mode with 64 scans and a resolution of $\pm 2 \text{ cm}^{-1}$ and wave numbers range of 4000-650 cm^{-1} . ZnSe-Diamond composite crystal was used as the background material. Polybenzoxazine was mixed with KBr.

3.2.2 Scanning Electron Microscopy (SEM)

The morphology of polybenzoxazine xerogel and polybenzoxazine xerogel-based carbon membrane was investigated by using a scanning electron microscope, Hitachi S-4800, with an accelerating voltage of 2 kV. Samples were coated with Platinum under vacuum before observation.

3.2.3 Surface Area Analyzer (SAA)

The surface area, average pore size, total pore volume and micropore volume of polybenzoxazine xerogel-based carbon membrane was investigated by using Surface area Analyzer; Quantachrome, with outgas time 12 hours.

3.2.4 Thermogravimetric Analyzer (TGA)

The thermal properties such as curing temperature of polybenzoxazine xerogel was investigated by using Thermo gravimetric Analyzer; Perkin Elmer with heating rate 20 °C/min

3.2.5 Gas permeability measurement

The gas flow pass the membrane will be measure by universal gas flow meter, Agilent Technologies ADM1000

3.2.6 X-Ray Diffractometer (XRD)

The X-ray powder diffraction pattern of the carbon foam was obtained using an XRD (Bruker AXS D8 ADVANCE) spectrometer with Cu K α irradiation ($\lambda = 0.15406$ nm) at 40 kV and 30 mA to examine the graphitization of the carbon membrane.

3.2.7 Differential Scanning Calorimetry (DSC)

Differential Scanning Calorimeter (DSC), Perkin-Elmer DSC , was used to study the thermal behavior of partially-cured and fully-cured polybenzoxazine. The samples were heated from 30 °C to 280 °C at a heating rate of 10 °C/min under N₂ atmosphere with a flow rate of 10 ml/min

3.3 Methodology

3.3.1 Preparation of Benzoxazine Monomer

The benzoxazine monomer was synthesized by mixing bisphenol-A, aniline and para-formaldehyde together at a 1:2:4 molar ratio and heated at 110 for 60 min until the mixture appeared a transparent pale yellow color.

3.3.2 Preparation of Polybenzoxazine Xerogel Membrane

Benzoxazine solutions were prepared from the benzoxazine monomer using xylene as a solvent at a concentration of 40 wt%. After that, the mixture were transferred into vial and seal at room temperature then slowly raised the temperature to 130 °C for 96 hours in an oven. The attained products were partially cured by benzoxazine xerogels witch were cut into diskshape with a thickness of approximately 4 mm. by diamond saw before drying at ambient temperature and pressure for 2 days to remove xylene from the matrices. The xerogels were step-cured in

an oven at 160 and 180 °C for an hour, and 200 °C for 2 hours. As shown in figure 3.1

3.3.3 Preparation of Polybenzoxazine Xerogel-based Carbon Membrane 800°C

Polybenzoxazine membranes were pyrolyzed under a nitrogen flow rate of 500cm³/min. using the following ramp cycle: 30-250°C in 1 hour, 250-600°C in 5 hours, 600-800°C in 1 hour, and holding at 800°C for 1 hour to obtained carbon membrane 800°C .As shown in Figure 3.2

3.3.4 Preparation of Activated Polybenzoxazine Xerogel-based Carbon Membrane

Activated carbon membrane was obtained by pyrolysis of carbon xerogel from 3.3.3 in a quartz reactor using the following ramp cycle: 900 °C for 180 min. The system was carried out in CO₂ flow at 550 cm³/min. As shown in Figure 3.2

Experimental Procedures

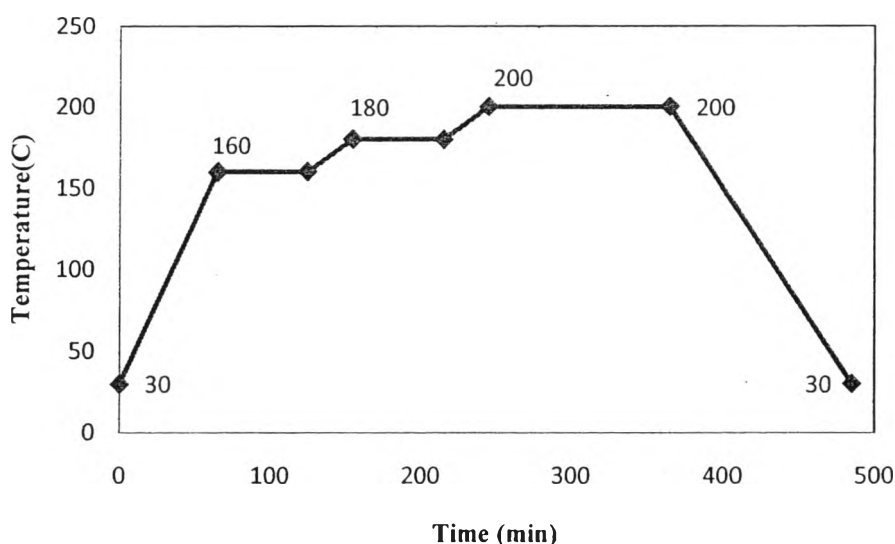


Figure 3.1 Schematic of a step of curing.

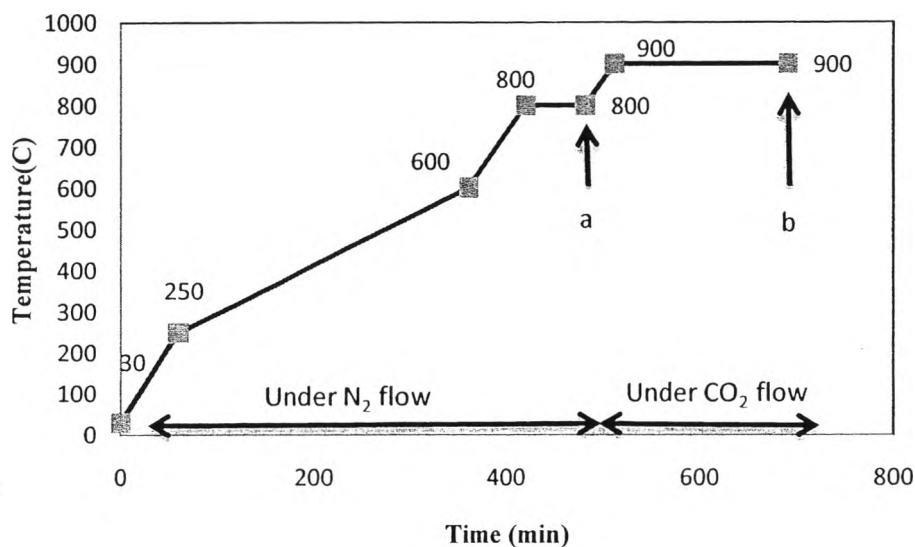


Figure 3.2 Schematic of a step of pyrolysis (a) carbon xerogel membrane 800 °C, (b) activated carbon xerogel membrane.

3.3.5 Preparation of polybenzoxazine xerogel-based carbon membrane 900°C

Polybenzoxazine membranes were pyrolyzed under a nitrogen flow rate of 500cm³/min. using the following ramp cycle: 30-250 °C in 1 hour, 250-600 °C in 5 hours, 600-900 °C in 1.5 hour, and holding at 900 °C for 1 hour to obtained carbon membrane 900 °C .As shown in Figure 3.3

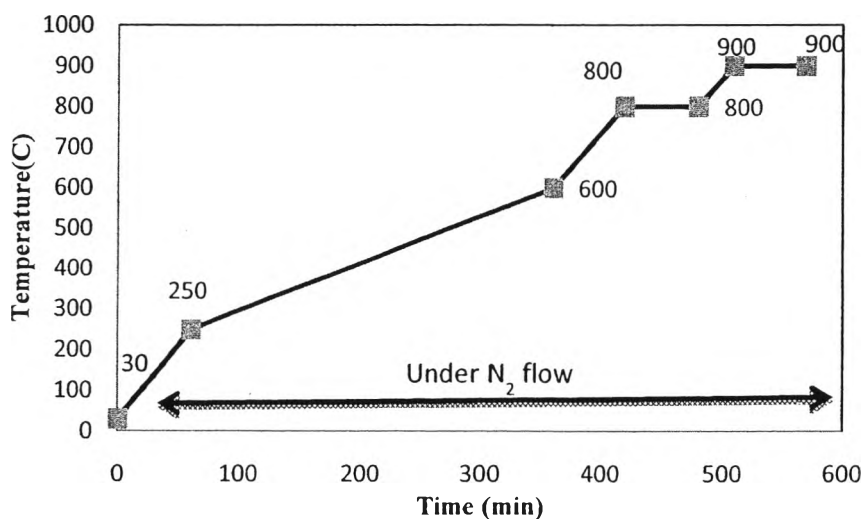


Figure 3.3 Schematic of a step of pyrolysis carbon xerogel membrane 900 °C