

## CHAPTER III EXPERIMENTAL

### 3.1 Materials

#### 3.1.1 Chemicals:

##### Polymer Chemicals

- Bisphenol-A (BPA)
- Formaldehyde
- Tetraethylenepentaamine (TEPA)
- Poly(ethylene glycol)-poly(propylene glycol)-poly(ethylene glycol) (Pluronic P123)

##### Solvents

- Dimethylformamide (DMF)
- Isopropanol
- Dioxane

### 3.2 Equipment

#### 3.2.1 Fourier Transform Infrared Spectroscopy (FT-IR)

The functional groups related to structure of carbon precursors investigated by using FT-IR technique. The FT-IR spectra of fully-polymerized benzoxazine xerogel and as synthesized benzoxazine xerogel were obtained by using a Nicolet Nexus 670 FT-IR spectrometer in the frequency range of 400-4000  $\text{cm}^{-1}$ . KBr pellet technique was applied in the preparation of powder samples.

#### 3.2.2 Thermogravimetric Analysis (TGA)

TG-DTA curves were collected on a Perkin-Elmer Pyris Daimond TG/DTA instrument. The 5 mg of sample was loaded on the platinum pan and heated from 30 to 900°C at a heating rate of 10 °C/min under  $\text{N}_2$  flow of 50 ml/min.

### 3.2.3 Differential Scanning Calorimetry (DSC)

DSC analysis were carried out using a Perkin-Elmer DSC 7 instrument. The sample was first heated from 30°C to 300 °C by heating rate 10°C/min under a N<sub>2</sub> atmosphere with a flow rate of 20 ml/min.

### 3.2.4 Scanning Electron Microscope (SEM)

Microstructure and surface morphology of porous carbon material was observed by a Scanning electron microscope (SEM; HITACHI TM3000). The specimens were coated with platinum under vacuum before observation to make them electrically conductive.

### 3.2.5 Surface Area Analyzer (SAA)

BET surface area and pore size distribution of all carbon xerogel were calculated from the nitrogen adsorption isotherms at 77K using a Quantachrome/Autosorb-1. Surface area analyzer based on the Brunauer-Emmett-Teller (BET) and Barret-Joyner-Halenda (BJH) methods, respectively.

### 3.2.6 Wide Angle X-Ray Diffraction (WAXD)

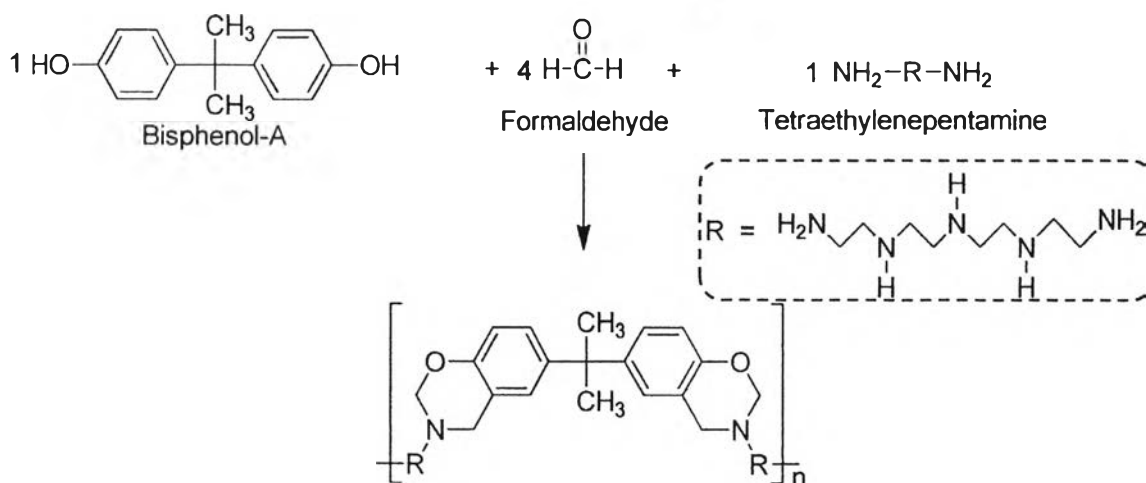
Wide Angle X-Ray Diffraction (WAXD) was performed on a Rigaku Smartlab<sup>®</sup> with a scanning speed of 2 °/min and CuK $\alpha$  source ( $\lambda = 0.154 \text{ \AA}$ ) in a range of  $2\theta = 5\text{--}80^\circ$  to determine the XRD pattern of carbon in variation parameters .

## 3.3 Experimental Procedures

### 3.3.1 Synthesis of Polybenzoxazine Precursor

Polybenzoxazine precursors were synthesized by dissolving Pluronic P123 in which the concentration was varied in different types of solvent (N, N,-dimethylformamide, Isopropanol, 1, 4-Dioxane (see in table 3.1, 3.2, 3.3)) in glass bottles and stirred until the clear solution was obtained. Bisphenol-A (2.28 g.) was then added into the P123 solution and stirred until the clear solution was obtained. Next formaldehyde solution (3.24g) was then added into the bisphenol-A solution.

The solution was kept under low temperature by using ice bath. After that tetraethylenepentamine (1.89 g) was added dropwise into the mixture and stirred continuously for approximately 1 hour while the reaction was cooled with the ice bath until transparent yellow viscous liquid was obtained. The molar ratio of bisphenol-A: formaldehyde: TEPA was 1:4:1. The synthetic reaction is shown in Figure 3.1. Then, the precursor was filled in a vial and placed in an oil bath at 80°C for 24 hr. to get benzoxazine gel. The benzoxazine gels were cut into small pieces and then dried at ambient temperature follow by placing in an oven at 80°C for 2 hr., 100°C for 1 hr., 200°C for 1.30 hr., and 220°C for 15 min.



**Figure 3.1** Preparation of polybenzoxazine precursor.

**Table 3.1** Template loading content of each sample in N, N-dimethylformamine

Concentration of PBZ precursor	Sample name	Concentration of surfactant (Pluronic P123)	Molar ratio P123:BPA
20 %	20%PBZ/DMF	-	-
	20%PBZ/1%P123/DMF	1 %	0.0032:0.323
	20%PBZ/3%P123/DMF	3 %	0.0096:0.323
	20%PBZ/5%P123/DMF	5 %	0.0161:0.323
	20%PBZ/6%P123/DMF	6 %	0.0194:0.323
25 %	25%PBZ /DMF	-	-
	25%PBZ/1%P123/DMF	1 %	0.0043:0.0430
	25%PBZ/3%P123/DMF	3 %	0.0129:0.0430
	25%PBZ/5%P123/DMF	5 %	0.0215:0.0430
	25%PBZ/6%P123/DMF	6 %	0.0258:0.0430
	25%PBZ/7%P123/DMF	7 %	0.0301:0.0430
	25%PBZ/8%P123/DMF	8 %	0.0344:0.0430
	25%PBZ/9%P123/DMF	9 %	0.0387:0.0430
	25%PBZ/10%P123/DMF	10 %	0.0430:0.0430
	25%PBZ/12%P123/DMF	12 %	0.0516:0.0430
	25%PBZ/15%P123/DMF	15 %	0.0645:0.0430
	25%PBZ/18%P123/DMF	18 %	0.0774:0.0430
	25%PBZ/20%P123/DMF	20 %	0.0860:0.0430
	25%PBZ/22%P123/DMF	23 %	0.0989:0.0430
25%PBZ/25%P123/DMF	25 %	0.1075:0.0430	
30 %	30%PBZ/DMF	-	-
	30%PBZ/3%P123/DMF	3 %	0.0166:0.054
	30%PBZ/6%P123/DMF	6 %	0.0326:0.054

**Table 3.2** Template loading content of each sample in Isopropanol

Concentration of PBZ precursor	Sample name	Concentration of surfactant (Pluronic P123)	Molar ratio P123:BPA
20 %	20%PBZ / IP	-	-
25 %	25%PBZ/ IP	-	-
	25%PBZ/3%P123/ IP	3 %	0.0107:0.359
	25%PBZ/6%P123/IP	6 %	0.0215:0.359
	25%PBZ/10%P123/ IP	10 %	0.0359:0.359
	25%PBZ/12%P123/ IP	12 %	0.0431:0.359
	25%PBZ/15%P123/ IP	15 %	0.0538:0.359
30 %	30%PBZ/ IP	-	-
	30%PBZ/6%P123/ IP	6 %	0.0276:0.461
	30%PBZ/10%P123/ IP	10 %	0.0461:0.461
35 %	35%PBZ/ IP	-	-

**Table 3.3** Template loading content of each sample in 1, 4-dioxane

Concentration of PBZ precursor	Sample name	Concentration of surfactant (Pluronic P123)	Molar ratio P123:BPA
35 %	35%PBZ/Dioxane	-	-
	35%PBZ/6%P123/Dioxane	6 %	0.0444:0.740
	35%PBZ/7%P123/Dioxane	7 %	0.0518:0.740
	35%PBZ/8%P123/Dioxane	8 %	0.0592:0.740
	35%PBZ/12%P123/Dioxane	12 %	0.0888:0.740
	35%PBZ/15%P123/Dioxane	15 %	0.1110:0.740

### 3.3.2 Preparation of Carbon Xerogel

Polybenzoxazines were pyrolyzed under nitrogen flow rate of 500 cm<sup>3</sup>/min in different final temperature (800 °C, 850 °C, 900 °C, 1000 °C). The heating profile was as follows: heating from room temperature to 200°C in 60 min, 200 to 600°C in 360 min, 600 to final temperature in 120 min, and holding at final temperature for 120 min and then cooling down to room temperature.

### 3.3.3 Preparation of Activated Carbon Xerogel

Activated carbon xerogel was prepared by carbonization of carbon xerogel under carbon dioxide atmosphere at 900 °C for 180 mins.

### 3.3.4 CO<sub>2</sub> Adsorption

Activated polybenzoxazine porous carbon with highest surface area was test the adsorption application by loading 0.1 g. of sample into the stainless steel adsorption chamber which was heated by the furnace in order to reach the adsorption temperatures. He gas was used as a purge gas in this study. The adsorption processes were carried out by using high purity CO<sub>2</sub> gas.