CHAPTER III
EXPERIMENTAL

3.1 Materials

3.1.1 Chemicals:

Polymer Chemicals

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

Solvents

- Dimethylformamide (DMF)

3.2 Equipments

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 using a Nicolet Nexus 670 FT-IR spectrometer in the frequency range of 4000-400 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 sample was loaded ~5 mg on the platinum pan and heated from 30 to 900°C a heating rate of 20 °C/min under 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 and cooled down at a rate of 5°C/min under a N_2 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 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.

3.3 Experimental Procedures

3.3.1 Synthesis of Polybenzoxazine Precursor

Polybenzoxazine precursors were synthesized by dissolving Pluronic P123 and bisphenol-A (varied the concentration of P123 in four conditions,(see in Table 5.1)) in *N,N,*-dimethylformamide (10 g) in glass bottle and stirred until the clear solution was obtained. Formaldehyde solution (1.20 g) 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 5.1. Then, the precursor was filled in a vial and placed in an oil bath at 80°C

for 24 hr to generate benzoxazine gel. The benzoxazine gels were cut into a small pieces and then dried at ambient temperature follow by placing in an oven at 100 °C in 1 hr, 200 °C in 1.30 hr, and 220 in 15 min.

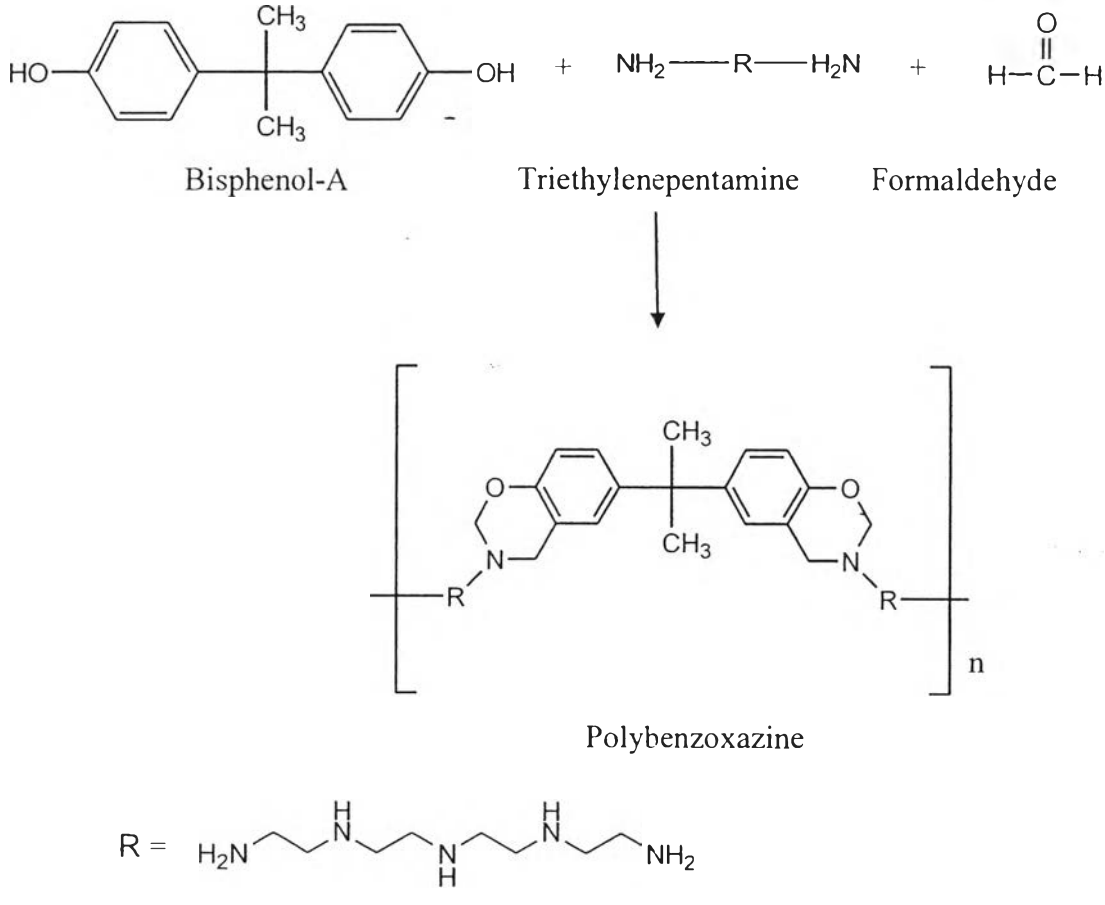

Figure 3.1 Preparation of polybenzoxazine precursor.

Table 3.1 Template loading content of each sample

Sample name	Molar ratios
0.188% P123	0.0013 P123 : 0.695 bisphenol A
0.375% P123	0.0026 P123 : 0.695 bisphenol A
0.750% P123	0.0052 P123 : 0.695 bisphenol A
1.500% P123	0.0104 P123 : 0.695 bisphenol A
3.000% P123	0.0208 P123 : 0.695 bisphenol A
6.000% P123	0.0416 P123 : 0.695 bisphenol A

3.3.2 Preparation of Carbon Xerogel

Polybenzoxazines were pyrolyzed under nitrogen flow rate of 500 cm^3/min. 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 800 °C in 120 min, and holding at 800 °C for 120 min and finally cooling down to room temperature.

Activated carbon xerogel was prepared by carbonization of carbon xerogel with carbon dioxide at 900 °C for 180 °C