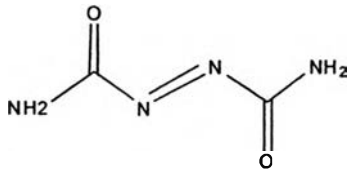
CHAPTER III
EXPERIMENTAL

3.1 Materials

Benzoxazine precursors was prepared by using phenol, ethylenediamine (EDA) and paraformaldehyde as reactants. Phenol (C_6H_5OH, analytical grade, 99% purity) was purchased from Panreac Company. EDA ($C_2H_8N_2$, analytical grade, 99% purity) was purchased from Fluka Company. Paraformaldehyde (CH_2O, reagent grade, 95% purity) was purchased from Fluka Company. Azodicarbonamide (ADC or AZD, $C_2H_4O_2N_4$) used as blowing agent was purchased from A.F Supercell Co, Ltd.. The structure of AZD is shown in Scheme I.



Azodiacarbonamide (AZD)

Scheme I

3.2 Equipments

3.2.1 Fourier Transform Infared (FT-IR) Spectroscopy

FT-IR spectra of P-eda and Poly(P-eda) obtained in the absorbace mode in arange of 400-4000 cm^{-1}. Spectra grade KBr, Carlo Erba®, was used as a background for P-eda and Poly(P-eda) (using a hydraulic press under 8 kg/cm^2 for 2 minutes, with 1 cm in diameter and about 0.005 cm in palletized thickness).

3.2.2 Different Scanning Calorimeteric (DSC) Analysis

Thermal analysis was carried out using a differential scannig calorimeter, Perkin-Elmer DSC 7. All scans were made under nitrogen atmosphere to minimize oxidative degradation. The temperature of indium. About 10 mg of samples were exposed to the following condition : the specimens, encapsulated in aluminum pans, were heated from 30°C to 300°C at a heating rate of 0.50-3.00°C/min.

3.2.3 Scaning Electron Microscope (SEM)

SEM, JEOL 5200-2AE (MP152001) was used to study phase morphology of the blends. The specimens were then coated with gold under vacuum. All SEM studies were characterized using magnification of 1500 times at 15-20 KV. The specimens were then coated with gold, under vacuum, to make them elecrically conductive.

3.2.4 Mechanical and Physical Properties Testing

An Instron Universed testing machine was used to measure the compressive strength of organic foams. The tests were conducted according to ASTM D-1621-00 test produre, using a crosshead speed of 2.50 mm min^{-1}.

3.2.5 Thermogravimatic Analysis (TGA)

TGA curves were collected on a TA Instrument TGA Q50 instrument. The samples were loaded on the platinum pan and heated from 30° to 850°C at a heating rate of 10°C/min under N_2 flow of 90 ml/min

3.2.6 Bruuauer-Emmett-Teller (BET)

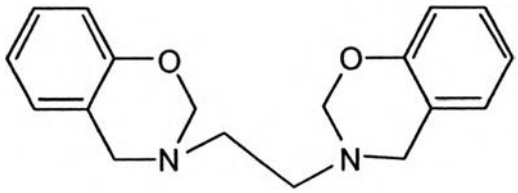
A surface area and pore size of carbon foam was determined using the BET method. These characterizations are based on the physical adsorption of nitrogen gas through the Autosorb-1 Gas Sorption System (Quantachrome Coporation) at liquid nirogen temperature of 77 K. Before starting characterization, Carbon foam samples were shaped cubic samples and was weighted for about 50 mg each samples. The specific surface area and pore size of carbon foam were obtained from twenty-point nitrogen adsorption and desorption isotherm plot.

3.3 Methodology

3.3.1 Preparation of Phenol-ethylenediamine (P-eda) based Benzoxazine Monomer.

P-eda based benzoxazine monomer used in study was synthesized from phenol, ethylenediamine and para-formaldehyde with the mole ratio 2:1:4, respectively. The reactants were mixed and heated at 110°C for one hour until viscous yellowish liquid was obtained. The structure of 1,2-di-(2H-benzo[e][1,3]oxazine-

3(4)-yl)ethane or Phenol-ethylenediamine (P-eda) benzoxazine monomer is shown in scheme II



Scheme II

3.3.2 Preparation of Organic Foam

P-eda monomer was mixed with various AZD contents (0-40 wt%) through dry-mixing method. The mixtures were cured in an oven at 30 to 210°C. In order to study the effect of heating rate on the morphology fo organic and carbon foams, the heating rate varied from 0.50-3.00°C/min were employed.

3.3.3 Preparation of Carbon Foam

Carbon foam was prepared by pyrolysis of the organic foam in a quartz reactor. The pyrolysis took place in a furnace under the nitrogen flow at 600 ml/min using the following ramp cycle : 30 to 250°C in 60 min, 250 to 600°C in 300 min, 600 to 800°C in 60 min, and hold at 800 °C in 60 min. Then the furnace was cooled down to room temperature under nitrogen atmosphere