CHAPTER III
EXPERIMENTAL

3.1 Materials

All chemicals were used without further purification. Bisphenol-A ($C_{15}H_{16}O_2$) was purchased from Aldrich, Germany. Formaldehyde solution (CH_2O) (37% by weight) was purchased from Merck, Germany. Triethylenetetramine (TETA, $C_6H_{18}N_4$) was purchased from Fluka, Switzerland. Dimethylformamide (DMF, C_3H_7NO) was purchased from Labscan, asia Co., Ltd., Thailand. Poly(ethylene oxide) (PEO, $C_{2n+2}H_{4n+6}O_{n+2}$) was purchased from Aldrich, Germany. Poly(ethylene oxide) was used without further.

3.2 Measurements

3.2.1 Fourier Transform Infrared Spectroscopy (FT-IR)

A Fourier Transform Infrared Spectroscope (FT-IR), Nicolet Nexus 670, was used to identify structural characteristics of polybenzoxazine (PBZ), poly(ethylene oxide) (PEO) and polybenzoxazine/poly(ethylene oxide) blended fibers in absorption mode with 64 scans and a resolution of $\pm$ 4 cm^{-1} and wave numbers range of 4000-400 cm^{-1}. Optical grade KBr (Carlo Erba Reagent) was used as the background material.

3.2.2 Field Emission Scanning Electron Microscopy (FE-SEM)

Fiber morphology of polybenzoxazine/poly(ethylene oxide) blended fibers was investigated by using 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 Thermogravimetric Analyzer (TGA)

The thermogravimetric analyzer (TGA) was used to determine the thermal behavior of polymers. The experiment was carried out by weighting a powder sample of 5-10 mg and placed it in a platinum pan, and then heated it under nitrogen flow with the heating rate 10°C/min from 30-800°C.

3.3 Experimental

3.3.1 Preparation of Benzoxazine Precursor

Benzoxazine precursor was prepared by using bisphenol-A, formaldehyde and triethylenetetramine with mole ratio of 1:4:1, respectively. The synthesis procedure included dissolving bisphenol-A (2.283 g) in dimethylformamide (20.964 g) in a glass bottle and stirred until the clear solution was obtained. Formaldehyde solution (3.243 g) was then added into the bisphenol-A solution and stirred continuously for approximately 20 min while the temperature was kept under 10°C using an ice bath. After that triethylenetetramine (1.723 g) was added dropwise into the mixture and stirred continuously for approximately 15 min while the reaction was cooled with the ice bath until transparent yellow viscous liquid was obtained.

3.3.2 Preparation of Poly(ethylene oxide) Solution

Poly(ethylene oxide) solution was prepared by dissolving poly(ethylene oxide) (M.W. = 900,000) in DMF at a concentration of 5 %w/v and stirred continuously for approximately 3 hours while the temperature was kept under 50°C.

3.3.3 Preparation of Polybenzoxazine/Poly(ethylene oxide) Blended Fibers

The polybenzoxazine/poly(ethylene oxide) blended fibers were prepared by mixing benzoxazine precursor and poly(ethylene oxide) solution with various weight compositions; 100:0, 90:10, 80:20, 70:30 and 60:40 and stirred continuously for 15 min. Electrospinning was done at a voltage ranging from 4 to 8 kV, with distance between the tip of the needle and the grounded aluminium from 10 to 25 cm, and using needles with diameter varied from 0.8 and 0.5 mm.

3.3.4 Characterizations of Benzoxazine Precursor, Poly(ethylene oxide) Solution and Polybenzoxazine/Poly(ethylene oxide) Blended Fibers

Proton Nuclear magnetic Resonance (^{1}H NMR) was used to identify the chemical composition of benzoxazine precursor. The structural characteristics, the fiber morphology and the thermal stability of polybenzoxazine, poly(ethylene oxide) and polybenzoxazine/poly(ethylene oxide) blended fibers were investigated by using Fourier Transform Infrared Spectroscope (FT-IR), Field Emission Scanning Electron Microscopy (FE-SEM) and Thermogravimetric Analyzer (TGA).