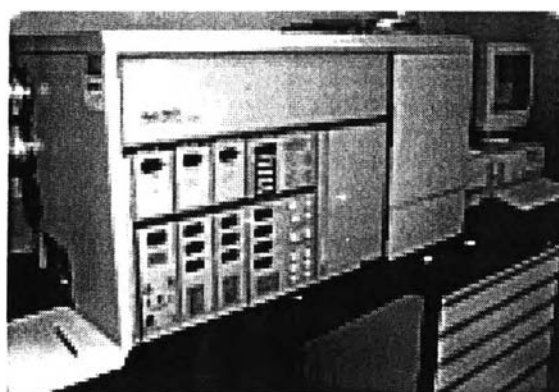


## CHAPTER III EXPERIMENTAL

### 3.1 Materials

The polystyrene (PS) resin used in this work was a general purpose grade (685, Dow Plastics, USA). A molecular weight characteristic of the resin was investigated using a Waters 150 CV size-exclusion chromatograph (see Figure 3.1). The operating conditions were as follows: diluents = tetrahydrofuran (THF), injection flow rate = 1.0 ml/min, injection volume = 100  $\mu$ l, and temperature = 30°C. Table 3.1 shows molecular characteristics of the resin:  $M_w = 3.00 \times 10^5$  Da,  $M_n = 1.19 \times 10^5$  Da, and polydispersity = 2.51.

The four solvents used in this work were 1,2-dichloroethane [DCE; Labscan (Asia), Thailand], *N,N*-dimethylformamide [DMF; Labscan (Asia), Thailand], ethylacetate [EA; Labscan (Asia), Thailand], methylethylketone [MEK; Carlo Erba, Italy]. All of these solvents were analytical research grade and used without further purification. Some important properties of these solvents (i.e. chemical formula, molecular weight, boiling point, density, dipole moment, and solubility parameter) are summarized in Table 3.2. Lithium chloride [LiCl; Ajax Chemicals, Australia], and potassium chloride [KCl; Ajax Chemicals, Australia] were used as inorganic salts.



**Figure 3.1** Waters Division of MILLIPORE (solvent system), Waters 150CV.

**Table 3.1** Chromatogram of PS pellet (685D)

<b>Sample Name</b>	PS685D	<b>Sample Type</b>	Broad Unknown
<b>Vial</b>	2	<b>Date Acquired</b>	3/23/03 10:48:54 AM
<b>Injection</b>	1	<b>Acq Method Set</b>	MethR_THF_30C_8
<b>Injection Volume</b>	100.00 $\mu$ l	<b>Processing Method</b>	Proc_THF_30C_8
<b>Channel</b>	SATIN	<b>Date Processed</b>	9/23/03 12:25:34 PM
<b>Run Time</b>	22.0 Minute		

## Peak Results

Name	Mn	Mw	MP	Mz	Mz+1	Polydispersity
PS685D	118604	298614	225326	561326	882796	2.518

**Table 3.2** Properties of solvents used in this work

Solvents	Chemical formula	Molecular weight (g/mol)	Boiling point ( $^{\circ}$ C)	Density ( $\text{g}/\text{cm}^3$ )	Dipole moment (Debye)	Dielectric constant	Solubility parameter ( $\text{MPa})^{1/2}$
DCE	$\text{ClCH}_2\text{CH}_2\text{Cl}$	99	83.5	1.239	2.94	10.19	20.2
DMF	$(\text{CH}_3)_2\text{NCHO}$	73.1	153	0.94	3.82	38.3	24
EA	$\text{CH}_3\text{COOCH}_2\text{CH}_3$	88.1	77.1	0.888	1.78	6.02	18.3
MEK	$\text{CH}_3\text{CH}_2\text{COCH}_3$	72.1	79.6	0.794	2.76	18.5	18.8

**3.2 Preparation of Spinning Solutions**

The PS solutions were prepared in either single or mixed solvent system at room temperature (i.e.  $\approx 26 \pm 2^{\circ}\text{C}$ ). In either case, a measured amount of PS pellets was dissolved in a measured quantity of DCE, DMF, EA, MEK, DMF/DCE, DMF/EA, or DMF/MEK, with the volumetric weight ratio of the solvent mixture being 100/0, 75/25, 50/50, 25/75, and 0/100 to produce PS solutions with

concentrations of 10%, 20%, and 30% (w/v). These solutions were characterized for their viscosity using a Brookfield DV-III programmable viscometer (see Figure 3.2), conductivity using an Orion 160 conductivity meter (see Figure 3.3), and surface tension using a Krüss DSA10 Mk2 drop shape analyzer (see Figure 3.4.). To investigate the effect of salt addition, about 1% (w/v) of salt was dissolved in the as-prepared PS solutions.



**Figure 3.2** Brookfield DV-III programmable viscometer (model LVTDCP).



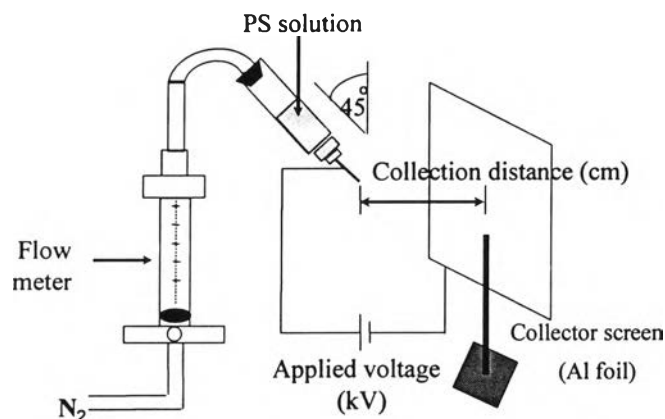
**Figure 3.3** Orion 160 Conductivity meter.



**Figure 3.4** Drop shape analysis system Krüss model number DSA10 Mk2.

### **3.3 Electrospinning Set-up**

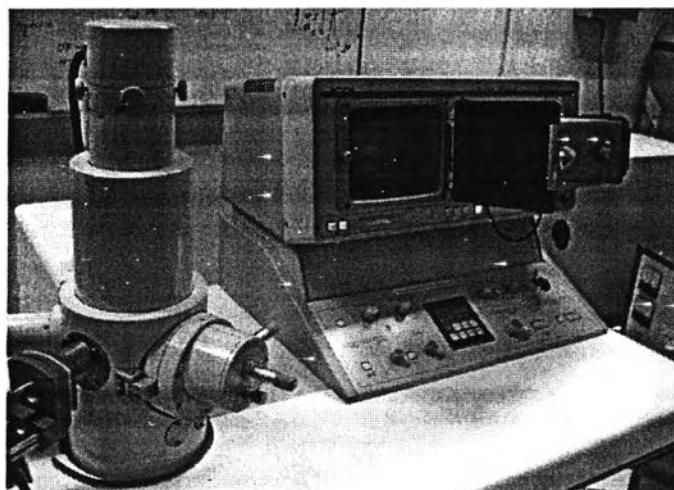
In the electrospinning set-up as shown in Figure 3.5, a glass syringe was used to stock each of the as-prepared PS solutions. A 1-cm long, blunt-end stainless steel needle with a flat tip, a cone angle of  $90^\circ$ , and 1.2 mm outside diameter (Gauge 18) was used as a needle. The tilt angle of the syringe was  $45^\circ$  from a horizontal baseline. The angle of  $45^\circ$  was to maintain constant presence of a droplet at the needle tip during an experiment. The feed rate of the solution was controlled by pressurized nitrogen gas through a flow meter. A piece of thick aluminum (Al) sheet was used as a collector screen. A Gamma High Voltage Research D-ES30PN/M692 power supply was used to charge the spinning PS solution by connecting the emitting electrode of positive' polarity to the needle and the grounding electrode to the collector screen. Unless otherwise noted, the electrical field strength was varied (i.e. 1:1, 2:1, or 3:1), either by fixing the collection distance (i.e. 7 kV/7 cm, 14 kV/7 cm, or 21 kV/7 cm) or by fixing the applied voltage (i.e. 25 kV/25 cm, 25 kV/12.5 cm, or 25 kV/8.3 cm). In order to compare the result obtained from these experimental conditions, the collection time for each spinning condition was fixed at 15 seconds.



**Figure 3.5** Experimental set-up for the electrospinning process of PS solution.

### 3.4 Morphological Observation

The morphological appearance of the as-spun PS fibers was investigated using a JEOL JSM-5200 scanning electron microscope (SEM) (see Figure 3.6). Each sample was coated with a thin layer of gold using a JEOL JFC-1100E ion sputtering device prior to observation under SEM. The average fiber diameter of the electrospun fibers was measured by Semafore 4.0 software from SEM images. The electrospinnability (e.g. the area of the as-spun fibers in two dimensions per unit area per unit time) of the as-spun PS fibers was investigated by using the Adobe Photoshop<sup>®</sup> CS software. SEM micrographs of PS fibers were converted to black and white images (threshold) by adjusting the pixels until obtained the certain images (Hongrojjanawiwat *et al.*, submitted). A thermo Nicolet Nexus 670 Fourier-transformed infrared spectroscope (FT-IR) was used to characterize the as-spun PS fibers whether they contained appreciable amount of solvent molecules using the KBr-pellet technique.



**Figure 3.6** Scanning Electron Microscope JEOL JSM-5200.



**Figure 3.7** Thermo Nicolet Nexus 670 Fourier-transformed infrared spectroscope (FT-IR).