CHAPTER III
EXPERIMENTAL

3.1 Materials

3.1.1 Polymer

Commercial grade PS (685D) was characterized for determine molecular weight averages by a Waters 150CV, Waters Division of MILLIPORE (solvent system) Gel permeable chromatograph, in which they were found to be Mn $=1.2x10^5$, Mw $=3.0\times10^5$, polydispersity 2.5. Tetrahydrofuran was used as the eluent, flowrate was 1.0 ml/min, injection volume was 100µl, and the measuring temperature was 30°C.

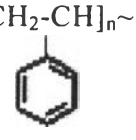

Figure 3.1 Chemical structure of polystyrene

3.1.2 Solvents

Various solvents were used to study the effect of solvents on morphological appearance of the as-spun fibers. They are benzene, butyl acetate, carbon tetrachloride, chlorobenzene, chloroform, cyclohexane, decalin, 1,2-dichloroethane, 1,4-dioxane, n-hexane, methylethylketone, tetrahydrofuran, tetralin, toluene, dimethylformamide, ethyl benzene, ethyl acetate and nitrobenzene.

3.2 Experimental Equipment

3.2.1 High Voltage Power Supply

A high voltage power supply (Gammar High Voltage Research model no. D-ES30PN/M692, Ormond Beach, Florida) was used to supply electrostatic field between a needle tip and a collection screen.

3.2.2 GPC chromatogram Waters 150CV, Waters Division of MILLIPORE (solvent system)

3.2.3 Brookfield digital viscometer (model LVTDCP)

3.2.4 Drop shape analysis system KRUSS model number DSA10 MK2

3.2.5 Conductivity Meter (ORION)

3.2.6 Scanning Electron Microscope JEOL,model JSM6301F

3.3 Experimental Methods

3.3.1 Sample preparation

PS solutions were prepared by dissolving PS pellets in various solvent at a concentration of 10, 20 or 30 %(w/v). Each mixture was stirred until the pellets completely dissolved.

3.3.2 Electrospinning process

The experimental set up for study is shown in Figure 3.2. Each polystyrene solution was placed in a 50-ml glass syringe. The syringe was clamped (45 degree to a horizontal baseline) to a PVC stand. Stationary collection screen(an aluminum foil sheet) was grounded. The polymer solution was subjected to external electrical field by attaching a positive electrode to the nozzle. The constant pressure of nitrogen gas was applied into the syringe to suspend the polymer drop at the tip of the nozzle. The electrospun fiber was kept in vaccuum for 24 h prior to characterization to ensure a complete drying of the sample.

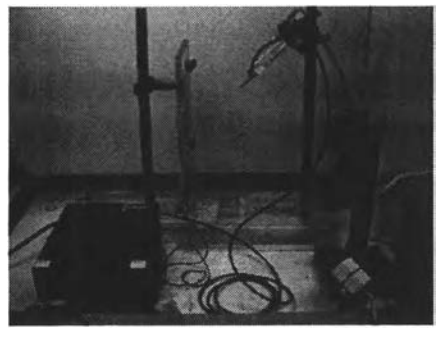

Figure 3.2 Experimental set up

3.3.2.1 Effect of applied voltage

To study the effect of applied voltage on various solvent, the Electrospun fibers obtained, applied voltage was varied among 15, 20, and 25kV, while the collection distance was fixed at 10 cm.

3.3.2.2 Effect of distance between tip of nozzle and screen

Distance between tip of nozzle and screen was varied from 7,10 and 15cm and the applied voltage of 20kV was used.

3.3.3 Characterization

3.3.3.1 GPC chromatography

Molecular weight and polydispersity of polymer pellets are determining by using GPC chromatogram from Waters Division of MILLIPORE (solvent system) model Waters 150CV

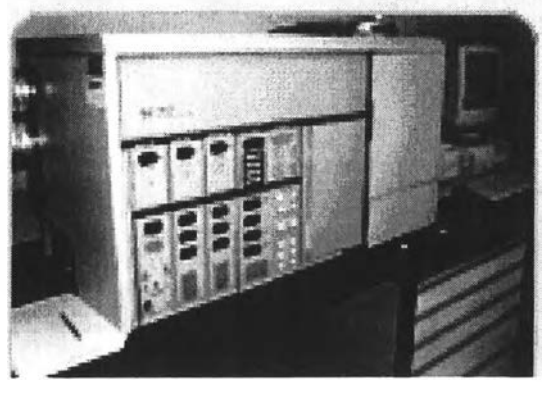

Figure 3.3 Waters Division of MILLIPORE (solvent system),Waters 150CV

3.3.3.2 Viscometer

The viscosity of polymer solution was determined by using a Brookfield digital viscometer (model LVTDCP) at 30±0.1°C

Figure 3.4 Brookfield digital viscometer (model LVTDCP)

3.3.3.2 Tensiometer

The surface tension was measured by calculate drop shape of solution by using KRUSS model number DSA10 MK2 and average surfacetention will calculate 5 times in second and we obtained this average value for 3 times to find surface tension of polymer solution

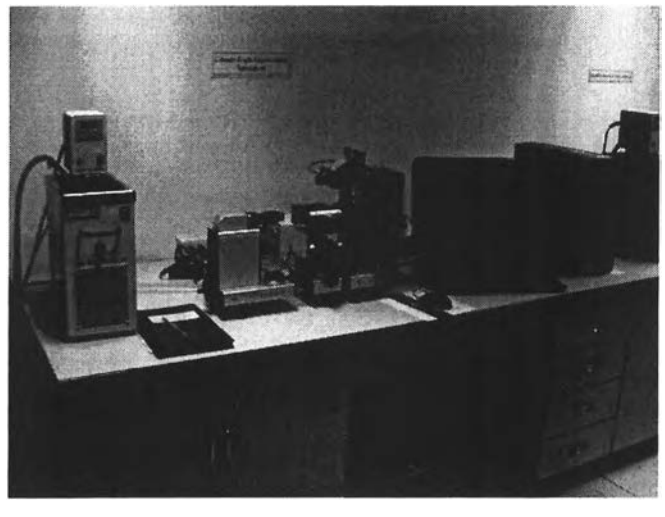

Figure 3.5 Drop shape analysis system KRUSS model number DSA10 MK2

3.3.3.3 Conductivity meter

The conductivity of PS solutions were measured at 30.0±0.1°C by using conductivity meter.

Figure 3.6 Conductivity meter

3.3.3.4 Scanning Electron Microscopy (SEM)

The surface morphology and diameter of the resulting electrospun PS fiber were observed by SEM (JEOL JSM6301F). They were coated with thin film of gold for 5 min prior to analysis. The average fiber diameter and diameter distribution were obtained from the diameter of 100 fibers taken from a number of SEM micrographs. Fiber diameter was measured by using SemAfore program.

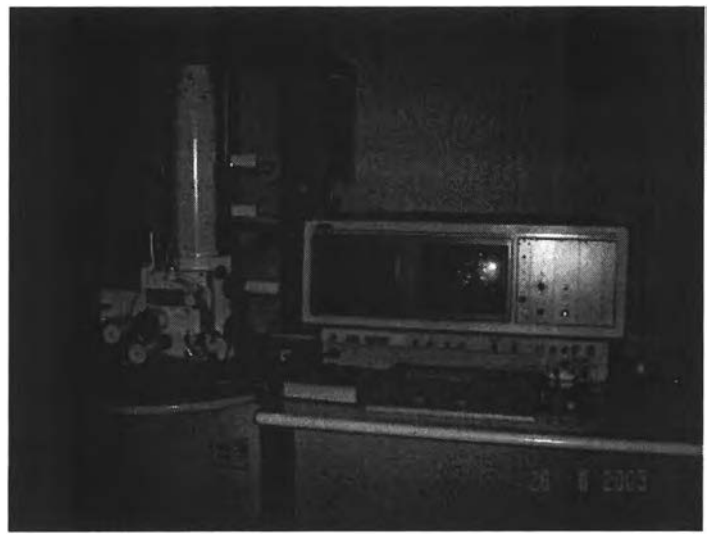

Fig 3.7 Scanning Electron Microscope JEOL JSM6301F