CHAPTER III

EXPERIMENTAL

3.1 Materials

All operations were carried out with careful exclusion of moisture and air because the silatrane complexes are slightly sensitive to moisture and air. The starting materials are fumed silica powder, ethylene glycol, and triethanolamine.

Amorphous, precipitated silicon dioxide (SiO_2), with a multi-point BET surface area of 1500 m^2/g (average particle size of 0.007 μm), was purchased from Aldrich Chemical Company and used as received. It was kept in a dry environment prior to use to prevent moisture adsorption.

Ethylene glycol (EG) and triethanolamine (TEA) were purchased from Merck Chemical Company and used as received.

Acetonitrile was purchased from Baker Analyzed Company and purified by distillation with calcium hydride, acting as a dehydrating agent.

The glassware was cleaned by soaking in an NaOH/iPrOH cleaning solution bath, followed by rinsing with water and acetone, respectively, and finally dried in an oven at 125-150 °C prior to use.

3.2 Characterisation Instruments

3.2.1 Thermogravimetric Analyser (TGA)

Thermogravimetric analysis was carried out on a Netzsch TG 209 thermogravimetric analyser. Samples (10-20 mg) were loaded in a platinum pan and heated under Nitrogen or air using a flow rate of 15 mL/min. The balance flow metre was set at 40 mL/min. Heating rate of 10 °C/min to 900 °C were used. TGA data were be used to determine the decomposition temperature, product purity and experimental ceramic yields.

3.2.2 Fourier Transform Infrared Spectrophotometre (FTIR)

It was recorded on a FT-45A Biorad spectrometre with a resolution of ± 4 cm^{-1}. Sample preparation was done by putting a powder of 1 mg in the mortar. It was then smeared on a potassium bromide (KBr) disc to get a thin film of the substance, which are then placed in the infrared beam. Another method was the compressed disc technique. A sample was thoroughly ground using 1.0% sample to 99 wt% pure and dry crystalline potassium bromide (KBr) and then hydraulically pressed. The liquid product was detected by directly smearing on KBr disk. The sample was placed in the chamber with nitrogen atmosphere purging for 20 min. before data acquisition was detected.

3.2.3 Scanning Electron Microscope (SEM)

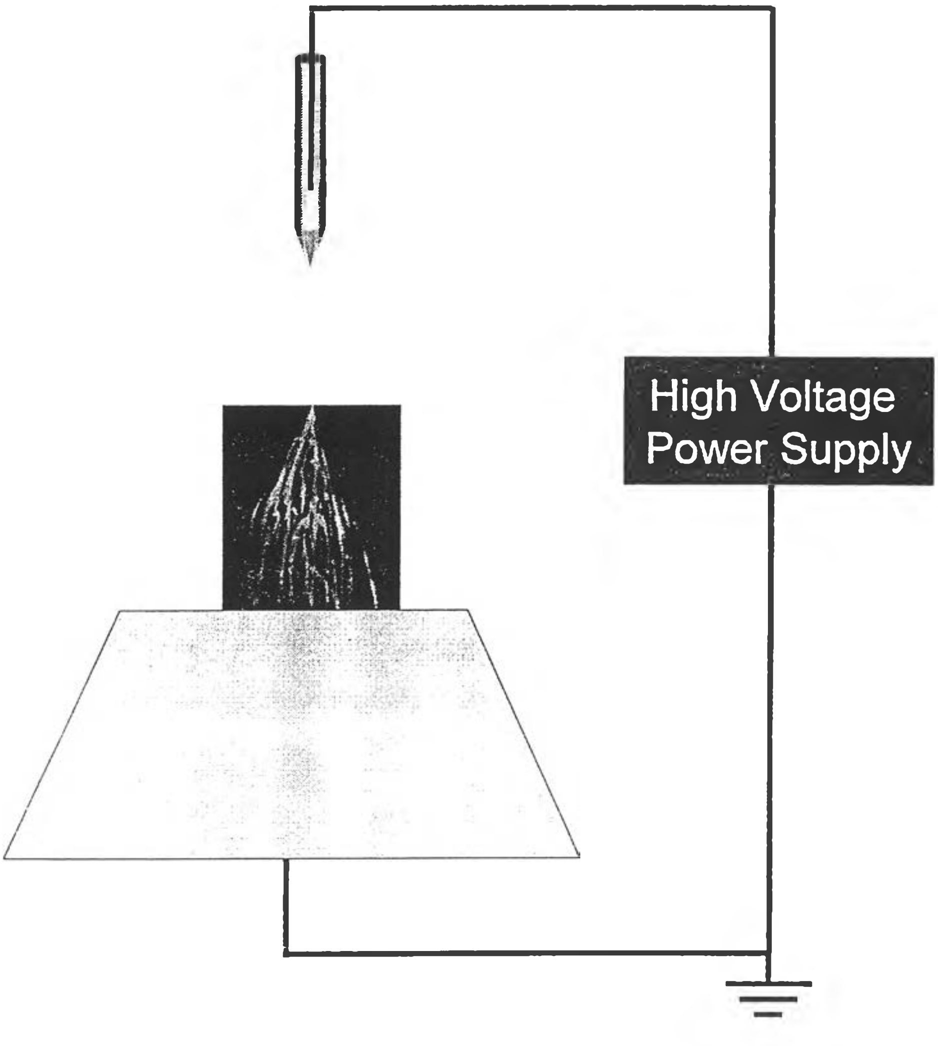
The surface morphology of the resulting electrospun PVA/silatrane composite fibres and silica fibres were observed by SEM (JEOL LS002). They were coated by thin film of gold for 4 min prior to analysis.

3.2.4 Wide-angle X-ray Diffractometre (WAXS)

XRD spectra were recorded by using a D/MAX-2000 series of Rigaku/X-ray diffractometre that provides X-rays of Cu K-alpha at 40 kV/30mA. The glass sample holders were applied to both ground samples and PVA/silatrane fibre mats. The experiment was operated in the 2θ range of 3-60 degree at the scan speed of 5 degree/min and 0.02 degree of scan step.

3.3 Electrospinning Apparatus

The schematic of the electrospinning apparatus that is used in this work is shown in scheme 3.1. The components of the apparatus and their functions are described as follows.



Scheme 3.1

- A high voltage power supply (ES30PN, Gamma High Voltage Research Inc., Ormond Beach, Florida) is used to generate either positive or negative DC voltage upto 30 kV, with very low electrical current of 166 microamperes.
- A 20 ml syringe is used as a container for polymer solutions. The syringe is built by a plastic structure and is set at a vertical direction.
- A stainless steel needle (guage number 20 and the outside diameter of 0.90 mm) is used as a nozzle and as an electrode to conduct the electrical energy from the power supply to the solutions. The tip of the needle is cut into a flat shape and the length of the needle is 1 cm.
- Aluminum foil is used as a ground collector which is covered on the plastic stand.

3.4 Calcination Apparatus

A Carbolite furnace (type STF 15/75/450, maximum temperature of 1500°C) was used to convert PVA/silatrane composite fibres into silica fibres. The electrospun PVA/silatrane composite fibres were subjected to heat treatment at the temperature of 500, 600, 700, and 800°C, heating rate of 4°C/min.

3.5 Procedure

3.5.1 Silatrane Synthesis and Characterisation

Silatrane is synthesised by reacting 0.125 mol triethanolamine with 0.1 mol silicon dioxide using ethylene glycol as solvent at 200 °C under nitrogen atmosphere. The reaction is completed within 10 hr., and the resulting mixture is cooled down to room temperature, before removing solvent by distilling under vacuum (10^{-2} torr) at 110 °C overnight. The resulting brownish white solid is washed three times with dried acetonitrile to obtain a fine white powder. Then, it is introduced to TGA and FTIR to observe its thermal stability and functional groups respectively.

3.5.2 Spinning of the PVA/silatrane composite fibres

A silatrane solution is prepared by mixing 1 g of silatrane in 7.5 and 9 g of a weighed amount of 6 wt % solution of poly(vinyl alcohol) (molecular weight = 115,000 amu) in water under vigorous stirring at room temperature for 2 hours to obtain a spinning mixture. The spinning mixture is spun under applied potentials of 11.25, 15, 18.75, and 22.5 kV. A collecting screen (i.e. aluminum foil) is set 10 cm from the tip of the stainless steel needle. The collecting time is fixed at 20 min.

3.5.3 Calcination of the PVA/silatrane composite fibres

The electrospun PVA/silatrane composite fibres are subjected to heat treatment at the temperature of 500, 600, 700, and 800°C, heating rate of 4°C/min by using Carbolite furnace (type STF 15/75/450)

3.5.4 Characterisation of the PVA/silatrane composite fibres and the calcined silica fibres

Both the PVA/silatrane composite fibres and the calcined silica fibres are introduced to FTIR, SEM, and XRD to study their functional groups, microstructure, and crystallinity respectively.