

## **CHAPTER II**

### **EXPERIMENTAL PROCEDURE**

#### **2.1 Materials**

1.  $\gamma$ -Methacryloxypropyltrimethoxysilane ( $\gamma$ -MPS) is used as silane coupling agent in this work. It was provided by Asia Glass Fiber Co.,Ltd. and purchased from United Chemical Technologies, Inc.,USA. It was used as received.

2. Polyvinylacetate (PVAc) is used as an example of a film forming agent. It was provided by Asia Glass Fiber Co.,Ltd. and was used as received.

3. Polyethyleneglycol (PEG) is used as an example of a lubricant. It was purchased from United Chemical Technologies, Inc., USA. and was used as recieved.

## 2.2 Equipment

Fourier transform infrared spectroscopy (Bomem Michelson MB) with liquid cooled mercury-cadmium telluride (MCT) detector and N<sub>2</sub> purge was used to study the mixture system. Typically, it was used at the resolution of 4 cm<sup>-1</sup> with coaddition of 100 scans. The mixture samples of  $\gamma$ -MPS hydrolyzate and PEG were cast as a thin film on KBr plates after evaporating the solvent. The sum of Lorentzian and Gaussian curve fitting method was applied to resolve the overlapped carbonyl bands.

Gel permeation chromatography consisting of a metering pump (Waters HPLC Pump model 510), one series of packed columns, and ultraviolet detector at 254 nm (Waters Associates, Turnable Absorbance Detector model 484) was used to separate the polymethacryloxypropylsiloxane oligomeric fractions and the polymeric lubricating sizing agent,  $\mu$  Styragel<sup>TM</sup> 10<sup>3</sup> nm (Waters Associates),  $\mu$  Styragel<sup>TM</sup> 10<sup>2</sup> nm (Waters Associates), and  $\mu$  Styragel<sup>TM</sup> 50 nm (Waters Associates) columns were used. Tetrahydrofuran (HPLC grade) was used as the mobile phase at the elution rate of 1 ml/min. The molecular weight distribution of the  $\gamma$ -MPS hydrolyzate fraction and PEG sizing agent was measured relative to linear monodisperse polystyrene standards (TOSOH Corporations).

### **2.3 Preparation of model treating system consisting of film forming sizing agent**

$\gamma$ -MPS was hydrolyzed in distilled, deionized water which was adjusted to pH 3.5 by diluted solution of acetic acid.  $\gamma$ -MPS was allowed to hydrolyze at room temperature by vigorous stirring for 1 hour. After that PVAc solution was added and it was allowed to further hydrolyze in the presence of PVAc for 1 hour. Finally the solvent was removed in the desiccator under reduced pressure at room temperature for 2 hours. The %wt of PVAc in the system was varied from 0, 10, 20, 50 to 80%wt.

### **2.4 Preparation of model treating system consisting of lubricating sizing agent.**

The alcoholic aqueous solution (92% ethanol) was used as the solvent for this system. PEG was dissolved in the solvent and stirred vigorously for 30 minutes. The solution was adjusted to pH 3.0 by HCl (aq) solution, followed by the addition of 0.5 grams  $\gamma$ -MPS. The  $\gamma$ -MPS was hydrolyzed in this solution in the presence of PEG for 1 hour at room temperature. The amount of PEG was varied from 10, 20, 50 to 80 % mol.

After  $\gamma$ -MPS was hydrolyzed for 1 hour, 8 grams of the solution was spread on a petri dish and the solvent was removed in a desiccator under reduced pressure at room temperature. The drying time was varied from 1.5, 3.5, 7.5, 22.5, 44 and 400 hours.

## 2.5 Measurements

### 2.5.1 Fourier transform infrared spectroscopy (FTIR)

The viscous sample of the mixture between polymethacryloxypropyl silsesquioxane and the sizing agents was cast as a thin film on KBr plate and was studied at the different drying time by FTIR technique. Curve fitting method was applied to analyze the overlapped band of carbonyl stretching peaks. Since the carbonyl groups which settled at different environments will appear at different positions. The expected positions of carbonyl stretching bands were fixed manually instead of allowing the program to run automatically to get the physical curve fitting meaning.

### 2.5.2 Gel permeation chromatography (GPC)

Tetrahydrofuran (HPLC grade) which was filtered by membrane filter (pore size  $0.45\ \mu\text{m}$ ) was used as the solvent for dissolving the viscous sample of the mixture between polymethacryloxypropylsilsesquioxane and the sizing agents. This solution was filtered again by using NYPOR membrane filter (pore size  $0.45\ \mu\text{m}$ ). The filtrate was injected into the GPC.