CHAPTER II

EXPERIMENTAL SECTION

2.1 Materials

γ-methacryloxypropyltrimethoxysilane (γ-MPS) was the silane coupling agent used in this work.Vinyl trimethyl quaternary ammonium chloride(VTAC) and polyvinylacetate(PVAc) were used as an antistatic sizing agent and a film forming agent respectively. All the three chemicals were supplied by Asia Glass Fiber Co.,Ltd and used as received. Polyethyleneglycol (PEG), used as an example of a lubricant, was purchased from Fluka Chemika- Biovhemika, Switzerland and was used as received.

2.2 Equipment

Fourior transform infrared spectroscopy (BIORAD Model FT-45S)with DTGS detector and dry air purge was used to study the chemical structure of γ - MPS in the mixture system. It was used at a resolution of 4 $cm^{-1}$and coaddition of 100 scans. The mixture system of γ - MPS hydrolyzate with VTAC, and with polyvinylacetate and polyethyleneglycol was cast as a thin film on KBr plate after evaporating the solvent.

Size exclusion chromatography, consisting of a metering pump (water HPLC pump Model 600 E), one series of packed column and Ultraviolet detector at 254 nm (Water Division of Millipore, Turnable Absorbance

Detector Model 486) was used to separate the polyMPS oligomeric fraction, the polymeric lubricant, and film forming sizing agent. Ultrastyragel $10^3$and 10^2 nm and Water styragel HR2(Water Associates) columns were used. Tetrahydrofuran (HPLC grade) was used as the mobile phase at the elution flow rate of 1 ml/min. The molecular weight distribution of γ-MPS hydrolyzates, antistatic agent, lubricant and film forming sizing agent was measured relative to linear monodisperse polystyrene standards.

The pH meter used was the ORION pH/mv/temperature meter Model 420A.

2.3 Preparation of model treating system consisting of antistatic sizing agent

2.3.1 Preparation of the mixture system to measure pH

The alcoholic aqueous solution (92% ethanol) was used as the solvent for the model treating system. VTAC was dissolved in the solvent and stirred vigorously for 30 minutes, followed by the addition of 0.5 grams of γ-MPS. The mixture was then stirred for 1 hour at room temperature. The concentration of VTAC in the system was 0, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0 to 4.0 % by weight.

2.3.2 Preparation of system with γ-MPS and VTAC

The alcoholic aqueous solution (92% ethanol) was used as the solvent for the model treating system and was adjusted to pH 3.0 by HCl (aq) solution. In the preparation of system with only γ-MPS, 0.5 grams of γ -MPS was dissolved in 10 ml of deionized water and stirred vigorously using a magnetic strirrer for 1 hour. After this, 8 grams of the sample solution was

spread on a petri dish and dried in a desiccator under reduced pressure at room temperature for 5 hours. The petri dish was then placed in a closed container where the relative humidity was controlled at 84%with saturated KCl aqueous solution at room temperature before drying. The drying was carried out in an oven at 30°C and the drying time used was 1.5, 3.5, 7.5, 22.5, 44 and 110 hours.

In the preparation of systems with VTAC, the same procedure as described above was used except that the VTAC was added to the alcoholic aqueous solution (92% ethanol) and stirred vigorously for 1 hour before γ-MPS was added. The concentration of VTAC in the system was 5, 10, 20, 50 and 80% by weight.

2.3.3 Preparation of model treating system with VTAC at fixed pH

The alcoholic aqueous solution (92% ethanol) was used as the solvent for the model treating system. VTAC was dissolved in the solvent and stirred vigorously for 30 minutes, followed by the addition of 0.5 grams of γ - MPS. It was then hydrolyzed in this solution in the presence of VTAC for 1 hour at room temperature. After this, the pH of the mixture system was adjusted to 7.0 by 0.1 N HCl and 0.1 N NaOH (aq) solution, 8 grams of the sample solution was spread on aluminium foil and the solvent was driven off 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 110 hours. The concentration of VTAC in the system was 0, 5, 10, 20, to 50 % by weight.

2.4 Preparation of model treating system consisting of lubricant and film forming sizing agent

Deionized water was used as the solvent for the model treating system and was adjusted to pH 3.5 by acetic acid. In the preparation of the system with only γ-MPS, 0.5 grams of γ -MPS was dissolved in 10 ml of deionized water and stirred vigorously with a magnetic strirrer for 1 hour. After this, 8 grams of the sample solution was spread on a petri dish and dried in a desiccator under reduced pressure at room temperature for 5 hours. The petri dish was then placed in a closed container where the relative humidity was controlled at 84%with saturated KCl aqueous solution at room temperature before drying. The drying was carried out in an oven at 30 ^{o} C and the drying time used was 1.5, 3.5, 7.5, 22.5, 44 and 110 hours.

In the preparation of systems with PEG and PVAc, the same procedure as described above was used except that the PEG and /or PVAc was added to the deionized water and stirred vigorously for 1 hour before γ-MPS was added. The amount of PEG in the system is 0.2 grams whereas the concentration of PVAc in the system was 5, 10, 20, 50 and 80% by weight.

For samples to be used in FTIR analysis, the drying time was fixed at 1.5, 3.5, 7.5, 22.5, 44 and 110 hours.

2.5 Measurements

2.5.1 Fourior transform infrared spectroscopy (FTIR)

The sample obtained from 2.3, 2.4 was cast as a thin film on KBr, ZnSe plate, respectively and before measurements were taken. In case where the absorbanec at a specific wavenumber was required, the value was read directly off the instrument.

2.5.2 Size exclusion chromatography (SEC)

Tetrahydrofuran(THF, HPLC grade) was used as the solvent in this work. The solvent was filtered by membrane filter (pore size 0.45 μm) and used to dissolve the sample. After that, the solution was filtered again by using membrane filter (pore size 0.45 μm). The filtrate was then injected into the SEC to obtain the chromatograms.

2.5.3 pH measurements

The pH meter was calibrated by using buffer solution at pH 4.01 and 10.01. After this, the samples obtained from 2.3.1 were measured. For the samples in section 2.3.3, the pH value was adjusted to 7.00 by 0.1 N HCl for sample with pH higher than 7.00, and by 0.1 N NaOH (aq) solution for sample with pH lower than 7.00.