

## CHAPTER III

### EXPERIMENTAL

#### 3.1 Materials

Deoxycholic acid, DCA (AR grade), was purchased from Nacalai Chemical, Japan and used without purification. Dioxane (AR grade) was purchased from Lab Scan (Thailand). Ethyl acetate (AR grade), *o*-xylene (AR grade), and *p*-xylene (AR grade) were purchased from Ajax Chemical and used without purification. Methyl alcohol anhydrous (AR grade) was purchased from Mallinckrodt. Vinylchloride monomer and commercial PVC were supplied by Thai Plastic and Chemicals Public Co., Ltd. and used without purification.

#### 3.2 Equipments

##### **Infrared Spectrum**

Infrared spectrum of the DCA-solvent adducts, guest free DCA, DCA-polymer adduct and the obtained polymer were obtained with Bruker Equinox 55/S Fourier transform infrared spectrometer (FT-IR) by using KBr pellet technique. The DCA-monomer adduct was characterized by microscope FTIR.

##### **DSC Measurement**

Differential scanning calorimetry (DSC) was used to determine the guest-releasing temperature, melting point of crystal, and T<sub>g</sub> of the obtained polymer on Nertzsch DSC 200 at a scan rate of 10°C/min in the range of 25-200°C under nitrogen atmosphere. The measurement of the DCA-VCM guest releasing temperature was done by Perkin Elmer DSC7 at a scan rate of 20°C/min in the 20-200 °C range under nitrogen atmosphere.

### **TGA/DTA Measurement**

Thermal gravimetric analysis/ differential thermal analysis (TGA/DTA) measurement was used to determine thermal stability of the guest in DCA host and obtained polymer on RigakuThermoplus Thermogravimetric and Differential Thermal Analyzer at a scan rate of 10 °C /min in the scan range of 30-200 °C under nitrogen atmosphere.

### **TGA Measurement**

TGA measurement was made with Nertzsch TG 209 at a scan rate of 10°C/min in the scan range of 25-700°C under nitrogen atmosphere to determine the host guest ratio and thermal stability of the compounds.

### **Powder X-ray Diffraction.**

The Horizontal X-ray diffractometer Rigaku D/MAX-2000 was used for analyzing crystal structure at room temperature at the angle range ( $2\theta$ ) of 3-25°.

### **ESR Measurement**

Electron spin resonance (ESR) measurement was performed by a Varian E-109 spectrometer equipped with an automatic temperature control. The measurement was achieved at X-frequency band to observe the difference spectra between commercial PVC and inclusion PVC after irradiated with  $\gamma$ -ray. ESR JEOL ESPRIT-425 was used for propagating radical observation at mode frequency 100 kHz and mode width of 0.5 mT. One-tenth gram of the guest free DCA and 1 ml. of liquefied VCM were added into an ESR tube. ESR tube was evacuated for 5 min and sealed under vacuum and irradiated at 20 kGy at -78 °C.

### **SEM Measurement**

A scanning electron microscopy (SEM), type JEOL 5200, was used to examine the aggregation of the obtained polymer in comparison with the commercial PVC resin. The aggregated PVC was kept in the original form in

methanol solution and vacuum dried. In the case of the commercial PVC resin, the powder was analyzed without purification.

#### **NMR Measurement**

The solid state  $^{13}\text{C}$  nuclear magnetic resonance spectroscopy ( $^{13}\text{C}$  NMR) was performed with Bruker 300 MHz spectrometer with cross polarization (CP) and magic angle spinning (MAS) compatibility in order to study the structure of the obtained PVC and the commercial PVC. The sample was washed with methanol for several times and vacuum dried for 1 day.

### **3.3 Methodology**

#### **Preparation of Inclusion Compound with Various Solvents.**

DCA was dissolved in a certain solvent to saturation at the boiling point of each solvent until a clear solution was obtained. The solution was slowly cooled to room temperature to obtain DCA-solvent adduct as a needle-like crystal.

#### **Preparation of Guest Free Inclusion Compound**

The guest free DCA was subsequently prepared by heating the DCA-solvent adducts at  $140\text{ }^{\circ}\text{C}$  for 12 hours, under reduced pressure around 1020 mbar to remove the solvent.

#### **Preparation of the DCA-monomer Adduct**

Vinylchloride monomer was collected into a tube containing guest free DCA (1 g) at low temperature ( $-196^{\circ}\text{C}$ ). The tube was allowed to stand at  $-15\text{ }^{\circ}\text{C}$  for 2 days to obtain DCA-VCM adducts.

#### **Polymerization of the DCA-monomer Adduct.**

The tube which contains DCA-monomer adduct was irradiated by  $\gamma$ -ray at dose rate of 2 Mrad/h for 1 hour at  $-78\text{ }^{\circ}\text{C}$  to initiate polymerization. The post polymerization reaction was continued at  $-15\text{ }^{\circ}\text{C}$  for 4 days.

**Separation of Polymer after Postpolymerization.**

After the postpolymerization, the DCA-polymer adduct was poured into an excess methanol to dissolve DCA. The separated polymer was filtered off and washed with absolute methanol for five times to exclude DCA completely. The obtained product was dried under vacuum.