CHAPTER III
EXPERIMENTAL

3.1 Materials

Cholic acid (AR grade) was purchased from TCI Co., Ltd. Japan. Methylmethacrylate (AR grade) was the product of Fluka, Switzerland. Absolute methanol was purchased from Mallinkrodt, Germany, tetrahydrofuran (HPLC grade) from Lab Scan (Thailand). Ethanol was purchased from BDH Laboratory Supplies, England. Vinyl chloride monomer (VCM) and commercial PVC were supplied by Thai Plastic and Chemicals Public Co., Ltd.. The chemical reagents were used without further purification

3.2 Measurement

3.2.1 Structural Analysis

Infrared spectra of CA-VCM adduct, CA-PVC adduct, the obtained PVC and the commercial PVC were measured using a Bruker Equinox 55/S FT-IR spectrometer using KBr pellet technique. The structure of the obtained PVC and commercial PVC were characterized by ^{1}H-NMR using JNM-GSX 270 JEOL 270 MHz.

3.2.2 Microstructure Analysis

Powder X-ray analysis was performed by Rigaku D/MAX 2200, at room temperature, to study the microstructure of the obtained crystal by using wide angle measurement at 2θ angle 5°-40°.

3.2.3 Thermal Analysis

Thermogravimetric analysis, (DuPont, model TGA 2950) and

Differential Scaning Calorimeter, (Nertzsch Differential Scanning Calorimeter 200) were used to analyze CA-VCM adduct and CA-PVC. The guest releasing temperature of the adducts were observed under nitrogen atmosphere with the flow rate 20 mL/min and heating rate 10°C/min from 25-200°C. The thermal stability of the VCM guest in CA channel, was studied using the low temperature DSC equipped with the liquid nitrogen (Perkin Elmer Differential Scanning Calorimeter Pyris1 with Perkin Elmer Liquid Nitrogen Cooling System CryoFill). The sample preparation was conducted in the cold room (5°C). The adduct was heated from -20°C to 120°C by the heating rate 10°C/min, under nitrogen at the flow rate of 20 mL/min.

3.3 Methodology

3.3.1 Preparation of cholic acid-VCM (CA-VCM) adduct

Inclusion compound of cholic acid was prepared by recrystallization with methylmethacrylate (MMA) to be CA-MMA. Cholic acid guest free crystal was obtained from evacuation CA-MMA adduct at 80°C for one day to remove all MMA guest. The obtained crystal was collected and kept in a tube. The liquidified VCM was gradually added into the tube which was cooled by liquid nitrogen. The tube was sealed in vacuo and left at –15°C for two days to let VCM adsorbed into guest free crystal CA before using as CA-VCM adduct.

3.3.2 Inclusion polymerization of CA-VCM adduct

The CA-VCM adduct was carried out in a γ-cell (Co-60) by a courtesy of Office of Atomic Energy for Peace, Ministry of Science and Technology, Thailand. The amount of γ-ray was varied from 10 to 40 kGy, at dose rate 15 kGy/h in order to evaluate the inclusion polymerization. The

postpolymerization reaction was continued at -15°C for 2 days. After the postpolymerization, the crystal was taken out from the tube and washed by excess methanol for 20 times to exclude polymer from CA host completely. The obtained product was dried under vacuum.

3.3.3 Polymerization of VCM on Silica Surface

Silica gel was applied as a reference substrate for physisorption of monomer. Silica gel was collected in the tube and VCM was poured into the tube under liquid nitrogen atmosphere. The inclusion polymerization was performed by γ-irradiation at the dose rate of 30 kGy at -78°C to initiate polymerization. The polymerization was operated as in the case of CA-VCM adduct.