

CHAPTER III

EXPERIMENTAL

3.1 Materials

3.1.1) Copolymerization Materials

Commercial grade vinyl chloride monomer (VCM) was used, and vinylidene chloride (VDC), reagent grade, purchased from Aldrich Chemical Co.Ltd., was used as comonomer. Peroxide initiators, ethyl hexyl peroxide and cumyl peroxide which have half-life of 10 hours at 44°C and 36°C were employed. Hydroxy propyl methyl cellulose (MHPC) that has viscosity of 40-60 centipoise (2% solution at 20°C) , methoxy content of 28-30% and hydroxy propyl content of 7-12% was used as suspending agent. Poly(vinyl alcohol) (PVA) which has degree of hydrolysis of 69-75 mole percent and viscosity of 4-6 centipoise (4 % solution at 20°C) was also employed as suspending agent.

All of these chemicals except vinylidene chloride were kindly supplied by Thai Plastic and Chemicals Public Co.Ltd.

3.1.2) Compound Materials

Barium-zinc stearate was used as the stabilizer, dioctyl pathalate (DOP) and epoxidized soybean oil were used as primary and secondary plasticizers. Calcium carbonate was used as filler. These chemicals were also supplied by Thai Plastic and Chemicals Public Co.Ltd.

3.2 Equipments

3.2.1) Differential Scanning Calorimetry (DSC)

Thermograms were obtained using DSC carried out on Du Pont Instrument model 910s. Hermetically sealed aluminum pans were used. Sample sizes were typically 5-25 mg and experiments were carried out under N₂. The N₂ flow rate was set at 40 mL/ min. The temperature analysis range was from 30-100°C with a heating ramp rate of 15°C/ min.

3.2.2) Fourier Transform Infrared Spectroscopy (FTIR)

FTIR analysis were performed on a Bruker equinox 55 instrument. The film samples were prepared by casting from tetrahydrofuran. The FTIR chamber was purged with N₂ for 20 min. to remove CO₂ before initiating data acquisition. The resolution for this method was $\pm 4 \text{ cm}^{-1}$.

3.2.3) Nuclear Magnetic Resonance Spectroscopy (NMR)

¹³C-NMR were performed by courtesy of the Department of Macro Molecular Science, CWRU, USA.

3.2.4) Size Exclusion Chromatography

GPC analysis were carried out on Water model 510c. Tetrahydrofuran was used as the mobile phase at the elusion flow rate of 1 ml/min.

3.2.5) Particle Size Analyzer

Particle Size Analyzer, Malvern instrument model master sizer x, with the lens size of 30 mm was employed. Water was used as a medium.

3.2.6) Universal Testing Machine

Tensile properties were measured using Instron model 4206 according to the JIS 6723 test procedure at a cross head speed of 5 mm/min.

3.3 Methodology

3.3.1) Preparation of PVA Suspending Agent

7% PVA solution was prepared as follow. PVA powder was weighed and slowly added into pure water at room temperature. Sprinkle process was needed while the solution was being stirred to avoid lump formation. The stirring speed was then reduced to prevent foaming. The solution was then heated to 70-80°C and stirring for 1 hour. Finally it was cooled down to room temperature.

3.3.2) Preparation of MHPC Suspending Agent

2% MHPC solution was prepared as follow. Demineral water, one third of total volume, was prepared and heated to 80-90°C. MHPC powder was weighed and added with stirring vigorously until the vortex occurred to avoid lump formation. After the powder was dissolved, the rest of water was added while the stirring speed was lowered and the solution was then cooled down.

The actual concentrations of PVA and MHPC solutions were obtained by observing the weight loss after drying in an oven at 100°C for half an hour.

3.3.3) VDC Purification

Inhibitor in VDC was removed by washing with caustic solution. The monomer was shaken with about one-half of its volume by 13% sodium hydroxide solution in a large separatory funnel for several minutes. After the monomer layer had settled, it was separated and washed with two portions of distilled or deionized water and added immediately to the reactor.

3.3.4) Copolymerization

The pressure polymerization vessel of approximately 2 liter capacity was charged with deionized water, suspending agents, and catalysts. It was then evacuated with slow stirring. Vinylidene chloride was sucked into the reactor after removing the polymerization inhibitor, followed by vinyl chloride under nitrogen pressure. The suspension was stirred and then brought to the polymerization temperature.

3.3.5) Specimen Preparation for Tensile Property Testing

The copolymer resins and compound ingredients were weighed (copolymer resin 100 parts/ Ba-Zn 3 parts/ DOP 60 parts/ EPO 5 parts/ CaCO₃ 5 parts) and premixed by physical mixing and fed into 2 roll mill. The mixer was blended at 150°C for 7 min. The sheets were then placed in a picture frame and molded by Wabash V50H compression molder at 150°C for 5 min. under a force of 10 tons for 1 min. The compression molded sheet was then

cooled down to 40°C. Test specimens of appropriate form were finally cut out from the compression - molded sheet.