



CHAPTER III

EXPERIMENTAL

3.1 Materials

Isotactic-polypropylene (iPP) and iPP/glass fiber composites having melt flow index (MFI) of 10.8 and 3.5 g/10 min, respectively, were obtained from IRPC Public Co., Ltd. (Thailand). Organoclay (BAMP15) synthesized by ion-exchanged process [18] was supplied by Metallurgy and Material Science Research Institute, Chulalongkorn University. Briefly, sodium bentonite (BLH) obtained from Southern Clay Products, was reacted with octadecyl trimethyl ammonium chloride ($(\text{CH}_3)_3\text{C}_{18}\text{H}_{37}\text{N}^+\text{Cl}^-$) (MW = 348.05 g/mol). The structure is shown in Figure 3.1. It was supplied by Akzo Nobel (Thailand).

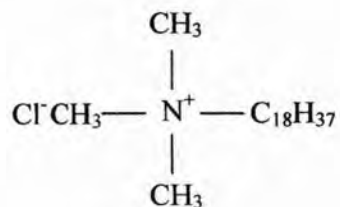


Figure 3.1 Molecular structure of octadecyl trimethyl ammonium chloride

3.2 Preparation of organoclay

Sodium bentonite is hydrophilic pristine clay. To obtain organophilic clays, sodium bentonite must be treated with surfactant to convert the hydrophilic clay to hydrophobic or organophilic clay. The procedure of modification of sodium bentonite is shown as follows:

- 1) 20L distilled water was poured into a tank.
- 2) 400g sodium bentonite was put into a tank and stirred intensively for 3 hours to disperse sodium bentonite into homogeneous phase. A tank was then heated up to 70° C.
- 3) 20L distilled water was added into another tank and heated up to 70°C. After that octadecyl trimethyl ammonium chloride was put into a tank and stirred intensively

to dissolve it.

- 4) The solution of ammonium salt of octadecyl trimethyl ammonium chloride was added into dispersed sodium bentonite and stirred vigorously at 70°C for a hour.
- 5) The solid part was filtered and washed by distilled water for several times.
- 6) The solid part was dried in an oven at 110°C and was then mortared and sieved respectively.

3.3 Preparation of iPP/organoclay nanocomposite by melt processing method

Organoclay powder and iPP were dried at 80°C for at least 24 h in an oven to remove the absorbed water. Organoclay powder and iPP were thoroughly mixed with maleic anhydride-grafted polypropylene (PP-g-MA) used as a compatibilizer and then placed into the hopper of a Betol, BTS40L twin-screw extruder. The compositions of PP-g-MA and organoclay were 1 and 3 wt%. Melt blended iPP/organoclay nanocomposite was formed by using a Betol, BTS40L twin screw extruder with a barrel temperature set at 220°C and screw speed of 50 rpm.

3.4 Preparation of iPP/glass fiber composite blended with iPP/organoclay nanocomposite.

iPP/glass fiber composite was thoroughly mixed with iPP/organoclay nanocomposite at the content 10, 30 and 50 wt% and then placed into the hopper of screw feeder 0.8%. Melt blends were formed by using a Betol, BTS40L twin screw extruder with a barrel temperature set at 220°C and screw speed of 160 rpm before injection molded into dumbbell-shaped with a temperature profile from die to feed zone of 200, 190, 180, 170 and 160°C with pressure 1,000 psi.

Table 3.1 Composition percentage of composites.

Sample	iPP (%)	GF (%)	Organoclay (%)	PP-g-MA (%)
neat iPP	100	0	0	0
iPP/GF	90	10	0	0
iPP/organoclay with compatibilizer	96	0	3	1
iPP/GF : iPP/organoclay 90 : 10	89.7	9	0.3	1
iPP/GF : iPP/organoclay 70 : 30	91.1	7	0.9	1
iPP/GF : iPP/organoclay 50 : 50	92.5	5	1.5	1

3.5 Characterization techniques

3.5.1 X-ray Diffraction (XRD)

XRD patterns of pristine clay, organoclay powder and iPP/organoclay nanocomposite were obtained by X-Ray diffractometer model PW3710, with CuK α radiation of wavelength 1.542 Å. An angle value used was in the range of $2\theta = 3-25^\circ$ with a scanning rate of 0.0125 degree/min. The voltage and current used in this analysis were 40 kV and 30 mA, respectively. The interlayer spacing of pristine clay, organoclay powder and the crystalline phase of iPP matrix was determined by means of XRD according to Bragg's equation (see Appendix A).

3.5.2 Scanning Electron Microscopy (SEM)

Dispersion of glass fibers and surface morphology of the composites were examined using a scanning electron microscopy (SEM), model JSM6400, at an accelerating voltage of 10 keV. The samples were coated with gold before analysis.

3.5.3 Transmission Electron Microscopy (TEM)

Dispersion of layered silicates and surface morphology of the composites were examined using a transmission electron microscopy (TEM), Jeol model JEM2010, at an accelerating voltage of 200 keV.

3.5.4 Differential Scanning Calorimetry (DSC)

Thermal analysis of composites were determined using a Perkin Elmer differential scanning calorimeter (DSC), model DSC-7. All of the composites were heated from 30 to 200°C at heating rate 10°C/min and then maintained at this temperature (200°C) for at least a minute to remove the thermal history. After that, the composites were cooled at the same scanning rate of 10°C/min until the temperature approached 30°C. And then, all composites were immediately subjected to a heating scan at a rate of 10°C/min to 200°C. In this process, all composites were analyzed under nitrogen atmosphere. The values of crystallization temperature (T_c), melting temperature (T_m), melting enthalpies (ΔH) and percent crystallinity (see Appendix B) were measured over the same temperature range of 30 to 200°C.

3.5.5 Tensile measurement

Tensile properties of isotactic-polypropylene (iPP) and iPP composites were performed by using Universal Testing Machine (Instron 5565) with a load cell of 5 kN, a crosshead speed of 50 mm/min and length between grips was 115 mm. which was used to determine tensile modulus, percentage of elongation at break and yield strength of all materials according to ASTM D638. The results were reported as averages of the data taken from at least ten specimens.