CHAPTER III
EXPERIMENTAL

3.1 Materials

A commercial general purpose grade (Moplen HP400 K) and an impact copolymer grade (Moplen CS 42 HEXP) of isotactic polypropylene (iPP) used in this study were supplied by HMC polymer Co., Ltd. (Thailand). Certain properties of the resins, provided by the manufacturer, are shown in Table 3.1. Two grades of $CaCO_3$ nanoparticles (NPCC 111 and NPCC 201, respectively) were supplied by Nano Materials Technology Co, Ltd. (Singapore). Certain characteristics of these two grades are similar. But, the main difference between both types was that the surface of NPCC 201 grade was modified with stearic acid to facilitate particle dispersion and distribution within the matrix, while that of NPCC 111 grade was uncoated $CaCO_3$ nanoparticles. The specific properties of both grades are shown in Table 3.2. TiO_2 nanoparticles used are CYU 201, CYU202, and CYU 203 and were supplied by Advance Nanotechnology Co., Ltd. (Thailand). These grades are different in their surface modification. Certain specific properties of these grades are shown in Table 3.3.

Table 3.1 Specific properties of the PP matrix

Item	CS 42 HEXP	HP 400K
Melt flow rate (dg/min)	12	4
Density (g/cm^3)	0.9	0.9
Tensile strength at yield (MPa)	24	33
Elongation at yield (%)	6	10
Flexural modulus (MPa)	1240	1400
Notched izod impact strength (J/m^2)	100	30
Deflection temperature at 455 kPa (^oC)	97	94

Table 3.2 Specific properties of the $CaCO_3$ nanoparticles

Typical valves	Samples	
	NPCC 111	**NPCC 201**
Specific gravity	2.5-2.6	2.5-2.6
Particle size (avg),nm	40	40
Surface area (BET) m^2/gm	40	40
Whiteness %	> 90	> 90
Particle shape	cubic	cubic
$CaCO_3$ %(wt) dry basis (modified)	>94.5	>94.5
Surface treatment agent	-	Stearic acid

Table 3.3 Specific properties of the TiO_2 nanoparticles

Item	CYU 201	CYU 202	CYU 203
Average particle size (nm)	50	50	50
Crystal type	Rutile	Rutile	Rutile
Content of TiO_2 (%)	98	95	92
Specific surface area (m^2/g)	>35	>35	>35
Surface properties	Hydrophilic	Hydrophilic	Hydrophilic
Surface treatment agent	-	SiO_2	Fatty acid

3.3 Methodology

3.3.1 Nanocomposites Preparation

The nanoparticulate fillers were first dried in an oven at 90°C for 24 hours and then pre-mixed with iPP pellets in a tumble mixer for 15 minutes in various compositional ratios. The pre-mixed compounds were then fed into a Collin ZK25 self-wiping, co-rotating twin-screw extruder operating at a screw speed of 55 rpm and a temperature profile of 190 (die), 190 (zone 4), 180 (zone 3), 170 (zone 2),

160 (zone 1), and 70°C (feed zone). The extrudate was cooled in water and cut into pellet form by a Planetrol 075D2 pelletizer.

3.3.2 Rheological Testing

A CEAST Rheologic 5000 twin-bore capillary rheometer was used to measure shear viscosity and extrudate swell of the PP Polymer and nanoparticle compounds. The inner diameter and the length of the barrel used were 9.95 and 250 mm, respectively, while the inner diameter and the length of the die were 1 and 2 mm in various lengths of the capillary. The testing temperature was calibrated at 190 ± 0.5°C. The hold-on time and hold-on pressure for stages 1, 2, and 3 were 200 seconds and 10, 20, 30 Pa, respectively. An automatic data collection system was used to collect and analyze the test results. The extrudate swell was measured using the attached KEYENCE VG laser analytical device, which comprises a laser light source and a light sensor. The equipment provides very accurate measurement of the extrudate diameters, hence vary accurate extrudate swell values.

3.3.3 Melt Fracture Testing

A high-resolution CCD camera was setup beside the exit of the capillary die. It was used to observe real-time occurrence of the melt fracture, if existed. The data in analog were transferred to an analog-to-digital converter, from which the digital signals were further analyzed in a personal computer. An image analytical software was used to analyzing the captured images.