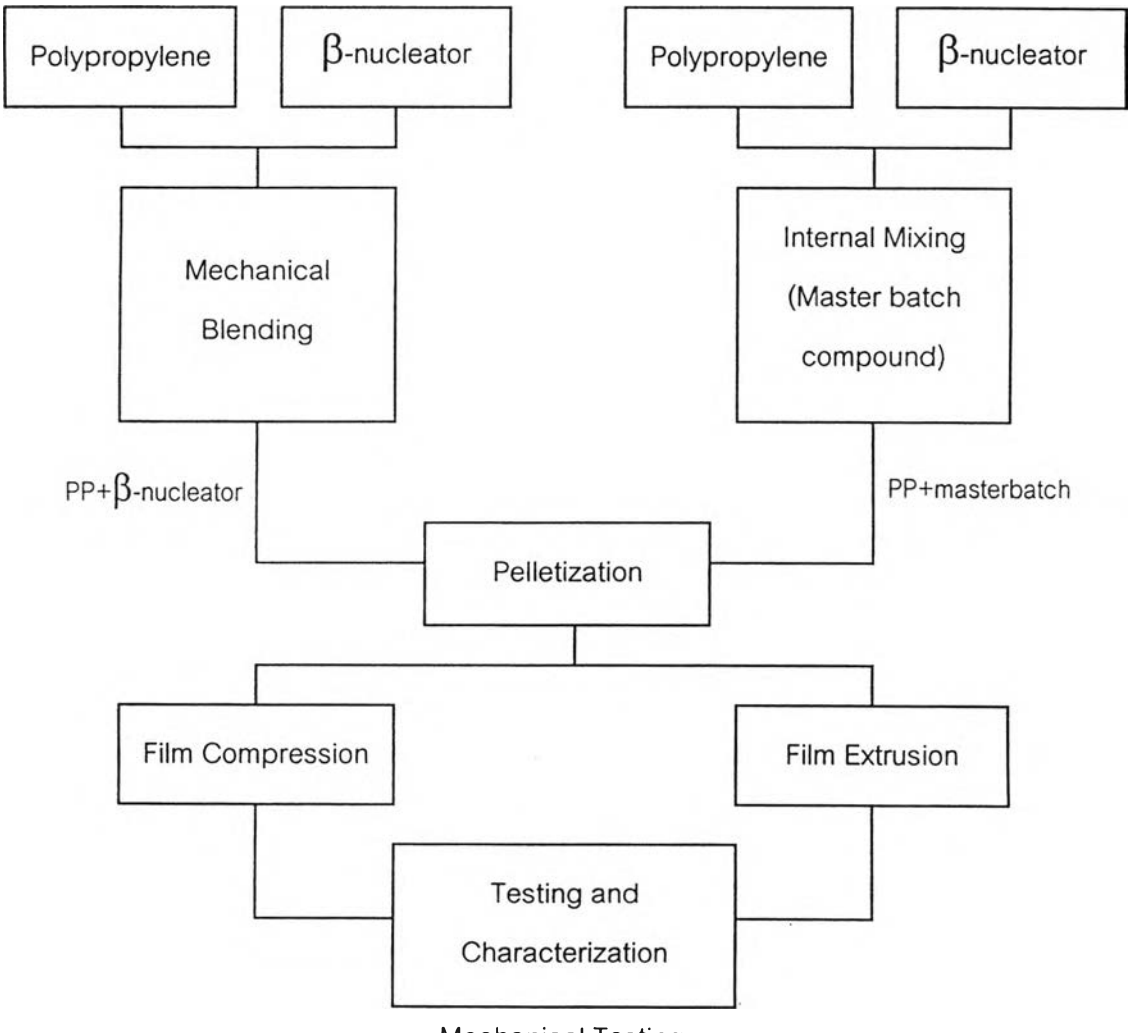
CHAPTER 3

MATERIALS AND METHODS

In this chapter, materials and sample preparation are first described, followed by processing techniques used in this study including pelletization, film compression moulding and cast film extrusion, mechanical testing and methods of characterization are also discussed. Figure 3.1 is a schematic overview of the experimental approach used in this study.



- Mechanical Testing
- WAXD
- DSC
- Polarizing Optical Microscope

Figure 3.1 Flow chart of experimental approach

3.1 Materials

The polypropylene (iPP) used was extrusion grade(Pro-fax 6531) resin supplied by HMC Polymer Marketing Co. Ltd. It has a melt flow index 4 dg/min, density 0.90 g/cm^3. The select β-nucleator was quinacridone (Permanent Red E3B) supplied from Clairiat Co. Ltd. This nucleator will be referred to as " E3B" in this study. This β-nucleator has a chemical structure as illustrated in Figure 3.2

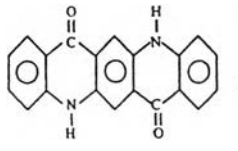


Figure 3.2 chemical structure of quinacridone

3.2 Processing

Films of β-nucleated iPP were produced using a two-pass process, namely pelletization followed by either compression or extrusion.

3.2.1 Mixing and Blending

The mixture of β-nucleator (Permanent Red E3B) and iPP was prepared prior to pelletization: 1) masterbatch preparation by internal mixing of iPP with E3B and 2) mechanical blending of iPP and E3B. The permanent Red E3B-iPP masterbatches were mixed by Brabrender Plastic corder, PI-2100 with mixing temperature of 180°C, speed of 50 rpm. The permanent Red E3B content used were 0.0001%, 0.001%, 0.01% by weight of the masterbatches. Another set of experiment involved mechanical dry blends of permanent Red E3B with iPP resin. These blends were charged directly to the extruder for further mixing and pelletizing.

3.2.2 Pelletization

Pelletization of the polypropylene with β-nucleator was performed on a small scale a co-rotating twin – screw extruder (PRISM TSE-16TC) with a screw diameter of 16 mm, L/D 25, intermeshing, at a fixed screw speed of 150 rpm. The temperature profile was 165/175/185/190/190 °C, respectively, temperature at the hopper zone, three barrel zones, and die head, respectively. Strand exiting the extruder was immediately quenched in a water batch and subsequently pelletized.

3.2.3 Compressed film preparation

Compressed films of the pelletized β- nucleator iPP compounds were produced by a lab scale Scientific-Labtech hot press. Optimum molding temperature and pressure were found to be 210°C and pressure 15 tons, respectively . Films were heated at the molding temperature of 210 °C under pressure for 3 minutes and then cooled by inserted cooling plates for 5 minutes. The insert system provides control over cooling by having water run through the separate platen assembly that press on the samples after heating cycle.

3.2.4 Extruded film preparation

Extruded films were produced on a 16-mm miniature extruder from Randcastle. Films were prepared by extruding pelletized resins (iPP-E3B)) through a 15.3 cm wide, 0.6 mm die gap slit die. Beyond the die extruded sheet was air cooled, uniaxially drawn and collected by a take-up assembly with adjustable hual-off speed. The extruder temperature profile was 190/195/200/220°C for the feed zone, two barrel zones, compression and zone in the barrel and slit-die zone, respectively. The constant screw speed of 35 rpm and take-up speed of 15 rpm were used. Resulting films had widths and thickness in the range of 12 cm and 0.05 mm, respectively.

3.3 Testing and characterization.

3.3.1 Mechanical testing

Mechanical properties of films were tested by a universal testing machine LLOYD 100K with a computer controlled measurement system. The initial grip seperation is 100 mm. The rate of grip seperation is 50 mm/min Tensile tests were performed in accordance with ASTM D 882. The sample is rectangular shape, sample size of 5x20 $mm.^2$ Young's modulus, tensile strength, yield strength, elongation at break, toughness were collected for each specimen tested and data were averaged from at least 5 specimens.

3.3.2 Wide Angle X-ray Diffraction (WAXD)

Wide Angle X-ray diffraction (WAXD) was used to investigate the crystal structure. The k-value corresponding to the relative amount of the β-form was calculated using following formula.

$$K = \frac{H\beta(300)}{H\beta(300) + H\alpha(110) + H\alpha(040) + H\alpha(130)}$$

Where Hα(110) (at 2θ=14.20), Hα(040) (at 2θ=17.00), and Hα(130) (at 2θ=18.8) are the intensities of the X-ray diffraction of the α-reflections and Hβ(300) (at 2θ=16.2) is the β-reflection. The intensity of the β-reflection measured as the height of peak. The k-value tend to be zero if no β-form is present and to be unity in the case when a lot of pure β-form is present. Diffractometer was used in this thesis is JOEL, JDX-3540.

3.3.3 Differential Scanning Calorimetry (DSC)

DSC studies were carried out on β-nucleated PP to obtain information on the thermal transition including melting endotherm of specimen and crystallization exotherm. Sample was scanned on a Perkin-Elmer DSC7 at heating and cooling rate

of 20°C/min. Chopped granules of β-nucleated PP pellets of approximately 10 mg were used. Resulting thermograms were normalized to constant weight and analyzed using the software provided in the instrument.

3.3.4 Microscopy study

Morphologies of β-nucleated PP were determined using a polarizing optical microscope equipped with a hot stage. According to the set temperature program , samples were heated from 30°C to 170°C with a heating rate of 20°C/min and hold at 170°C for 2 min. Sample were then cooled to 125°C with a rate of 20°C/min and hold at 125°C for 3 min in order to observe crystallization or spherulite growth. All micrographs were then taken with magnification of 200X and 500X.

3.3.5 Oxygen permeation study

Oxygen permeation of β-nucleated PP of extruded film and compressed films were tested by using Gas permeability tester GDP-C. Test were performed on a sample area of 78.4 cm^2 at a temperature of 24°C, gas stream feed of 60 cm^3/min and under 47% relative humidity, in accordance with ASTM D 1434-82.