

## CHAPTER II

### EXPERIMENTAL DETAILS

#### 2.1 Materials

##### 2.1.1 Polyacetal

The polyacetal (polyoxymethylene, POM) used was Delrin 500 (acetal resin) from DuPont. The density was  $1.42 \text{ g / cm}^3$  and melt flow index at  $190 \text{ }^\circ\text{C}$  and  $1.05 \text{ kg}$  load was about  $6.0 \text{ g / 10 min}$ .

Polyacetal resins or polyoxymethylene (POM) are a family of polymers derived primarily from formaldehyde. The linear unbranched molecular chain (Figure 2.1) allows the polymer chains to be closely packed into a highly crystalline structure. The reported degree of crystallinity is between 60-77 %, measured either by x-ray or from density data [McCrum et al., 1994].

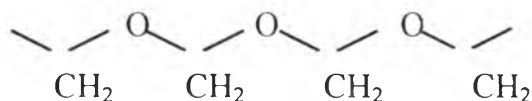


Figure 2.1 Chemical structure of POM.

The highly crystalline structure provides good creep resistance, hardness, toughness, high tensile strength and heat resistance. But POM can be adversely affected by UV radiation. It loses much of its tensile strength and becomes embrittled [Domininghans, 1993].

### 2.1.2 Polyamide

The polyamide (nylon) used was Zytel 101 (nylon 6,6 resin) from DuPont with density  $1.14 \text{ g / cm}^3$ .

Nylon 6,6 is a product of the condensation polymerization of hexamethylene diamine and adipic acid. The crystallization of nylon 6,6 is facilitated by the essentially linear character of polymer chain and the hydrogen bonds from the amide group on the backbone (Figure 2.2).

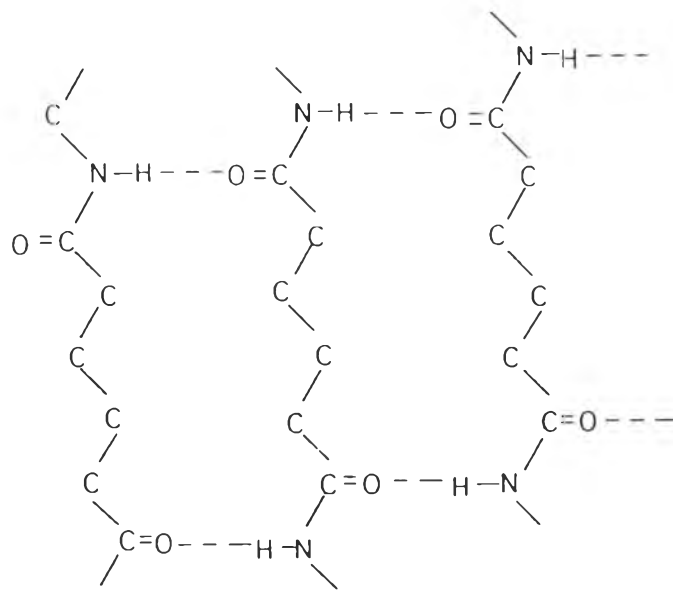


Figure 2.2 Chemical structure of nylon 6,6.

Nylon 6,6 crystallizes with the strong cohesive forces exerted between molecules [Margolis, 1985; Rubin, 1990; McCrum, et al., 1994]. This structure leads to excellent strength, toughness, thermal stability and

resistance to most solvents. A limitation of nylon is its high sensitivity to moisture [Whelan, 1994]. Moisture causes dimensional changes, discoloration and loss of mechanical properties. Moisture control is the primary concern when reprocessing nylon. It has to be kept dry and be handled properly [Kroschwitz, 1991].

### 2.1.3 Polyetherimide

The polyetherimide (PEI) used was Ultem 1000 from GE. PEI consists of imide groups linked by ether groups and thus have good mechanical and thermal properties. The regular repeating structure (Figure 2.3) with a meta-substituted bulky unit and a flexibility of chain provided by the ether linkage inhibits chain-chain packing and allow less interactions to occur. This particular PEI has a completely amorphous structure [Wilson et al., 1990].

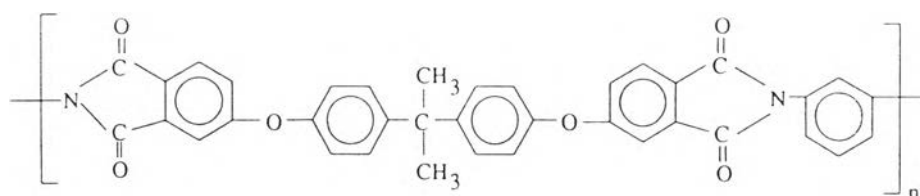


Figure 2.3 Chemical structure of PEI.

The PEI structure provides stiffness, and excellent mechanical and thermal properties. It also withstands UV and gamma radiation, so it can be used in medical applications. A disadvantage of PEI is poor solvent resistance from aliphatic and ether components on their backbone.

## 2.2 Processing

Virgin POM and nylon were reprocessed for 10 passes in a COLLIN co-rotating twin screw kneader ZK-25 (25 x 30D). The PEI was reprocessed by a single screw Brabender plasticorder PL 2000 (19 x 25D). All processes were carried out with the conditions for each polymer as shown in Figure 2.4.

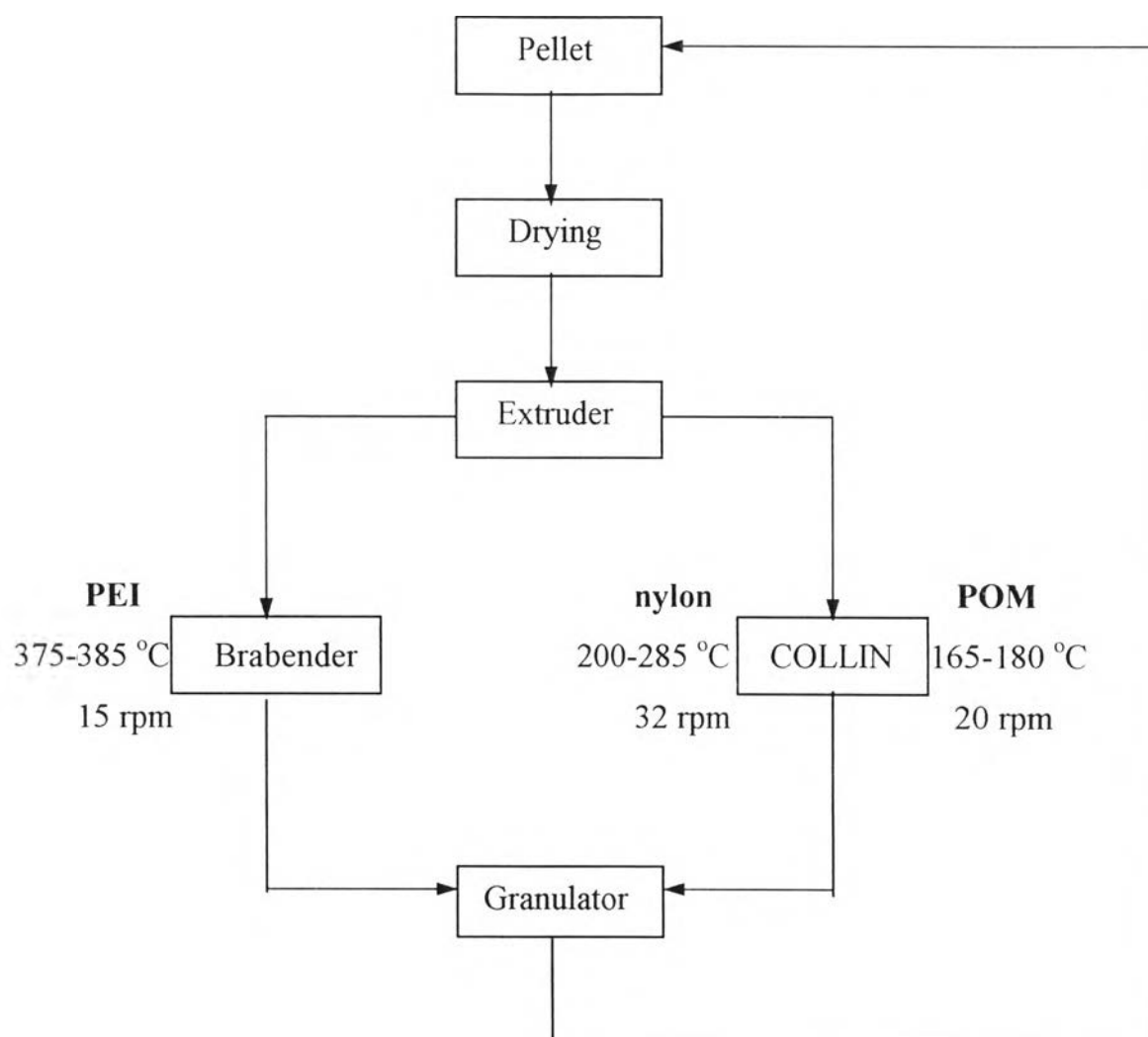


Figure 2.4 Processing flow chart.

Drying before processing was needed at 80 °C 30 hrs. for nylon and at 150 °C 4 hrs. for PEI in order to get rid of moisture.

After passing through the extrusion machine the extrudate was then run through room temperature water and cut into pellet form by a granulator. Samples from the first, third, fifth and tenth passes were collected for characterization. The processed pellets were dried and taken back to the hopper for the next reprocessing pass with the same condition. The extruder was purged sufficiently to make sure that good material for each pass was attained.

## 2.3 Characterization

### 2.3.1 Thermal Analysis

The thermal behavior of the reprocessed materials were determined by both Differential Scanning Calorimetry (DSC) and Thermogravimetric analysis (TGA).

Differential Scanning Calorimetry (DSC) was carried out on a DuPont 910S. Samples of 8-12 mg were prepared in aluminum sample pans. The temperature was programmed at a heating rate of 10 °C / min from room temperature to 300 °C for POM and nylon and 400 °C for PEI. The chamber was purged with dry nitrogen at flow rate of 25 ml / min. The melting point ( $T_m$ ), heat of fusion ( $H_f$ ) and glass transition temperature ( $T_g$ ) were determined from the thermogram by using DSC standard data analysis V.4.0 (DSC-4.0) software. The corresponding degree of crystallinity of the samples were calculated from the  $H_f$  by using the following equation :

$$\text{Percentage crystallinity} = \frac{H_f}{H_{fc}} \times 100, \quad (2.1)$$

where  $H_f$  is the heat of fusion of sample from thermogram and  $H_{fc}$  is the theoretical heat of fusion of 100 percent crystallinity of the same polymer.  $H_{fc}$  of nylon 6,6 and POM [Brandrup and Immergut, 1989] are 196 and 316 J/g respectively.

Thermogravimetric Analysis (TGA) of the samples were determined on a Netzsch TG 209 Thermo Microbalance. Samples of 8-12 mg were placed into aluminum pans and heated from room temperature to 700 °C for nylon and PEI and 350 °C for POM in O<sub>2</sub> with a ramp rate of 10 °C / min. A Netzsch TG 209 evaluation program was used to find the percentage of mass loss and decomposition temperature profile.

### 2.3.2 Density Measurement

The density of the reprocessed materials from compression molding were measured by the water-replacement technique, according to ASTM D792-91, at 25 °C ± 1 °C. The molding materials were cut into 1-2 g sample size. The determination of the mass of samples in air and the mass upon immersion in water were measured by a Sartorius MC1 balance and density kit with a precision of 0.1 mg. The densities of the samples were calculated as follows:

$$D_s = \frac{a - b}{b} \cdot D_w, \quad (2.2)$$

when  $D_s$  = density of sample  
 $D_w$  = density of water  
a = weight of sample in air  
b = weight of sample immersed in water

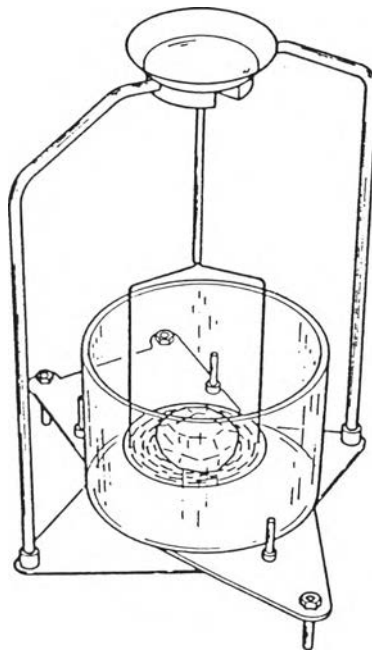


Figure 2.5 Sartorius density kit.