CHAPTER III
EXPERIMENTAL

3.1 Materials

The materials used for this study include

1. ESCOR ® terpolymer; grade 310, 320 and 325.

2. EAA copolymer; grade 1, 2, 4 and 5.

Both polymers were supplied by Exxon Chemical Co., Ltd. (U.S.A.). The characteristics of the materials used are given in Table 1.

Table 1 The characteristics of ESCOR® terpolymer.

Physical Properties	ESCOR® 310	ESCOR® 320	ESCOR® 325
Melt Index (g/10 min)*	6.0	5.0	20
Density (g/cm^3)*	0.941	0.953	0.950
Acid Number (mg KOH/ g polymer)*	45	45	45
Peak Melting Temperature (°F (°C))*	201(94)	169 (76)	163 (73)
Peak Crystallization Temperature (°F (°C))*	165 (74)	-	120 (49)

* using EXXON method

Table 2 The composition of ESCOR® terpolymer.

	Ethylene (%)	Methyl acrylate (%)	Acrylic acid (%)
ESCOR®310	87	6.5	6.5
ESCOR®320	76	18	6.0
ESCOR®325	74	20	6.0

3.2 Experimental Procedure

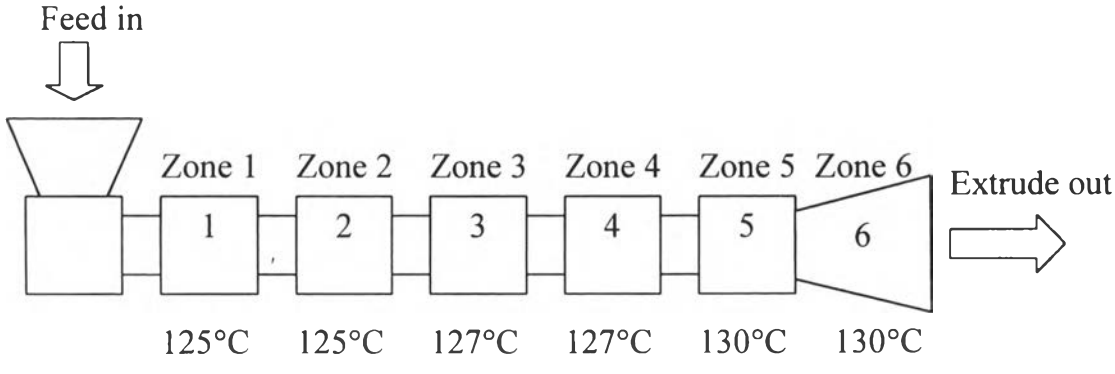
3.2.1 Polymer Blend Preparation

Blends of an ESCOR® terpolymer (ESCOR® 310, 320 and 325) and an EAA copolymer (EAA 1, 2, 4 and 5) were prepared in different compositions (see table 2) by initially mixing the materials in a tumbler mixer for 10 minutes and then melt-blended in a Collin co-rotating twin screw extruder, KNEADER ZK-25 with 6 zones of temperatures, at temperature settings ranging from 125°C to 130°C with screw speed of 50 rpm. The extrudate was then cooled in water at approximately 27°C and pelletized by Planetrol 075D2 palletizer. All processing passes were carried out using the processing condition as presented in figure 2.

Table 3 Composition of ESCOR® terpolymer and EAA copolymer.

ESCOR® terpolymer	EAA	% wt. of ESCOR® /EAA
ESCOR® 310	EAA 1	0/100, 20/80, 40/60, 60/40, 80/20, 100/0
	EAA 2	0/100, 20/80, 40/60, 60/40, 80/20, 100/0
	EAA 4	0/100, 20/80, 40/60, 60/40, 80/20, 100/0
	EAA 5	0/100, 20/80, 40/60, 60/40, 80/20, 100/0
ESCOR® 320	EAA 1	0/100, 20/80, 40/60, 60/40, 80/20, 100/0
	EAA 2	0/100, 5/95, 10/90, 15/85, 20/80, 40/60, 50/50, 60/40, 80/20, 100/0
	EAA 4	0/100, 20/80, 40/60, 60/40, 80/20, 100/0
	EAA 5	0/100, 20/80, 40/60, 60/40, 80/20, 100/0
ESCOR® 325	EAA 1	0/100, 20/80, 40/60, 60/40, 80/20, 100/0
	EAA 2	0/100, 20/80, 40/60, 60/40, 80/20, 100/0
	EAA 4	0/100, 20/80, 40/60, 60/40, 80/20, 100/0
	EAA 5	0/100, 20/80, 40/60, 60/40, 80/20, 100/0

The polymer blends obtained were filled into a picture frame mold and the mold was preheated at 160°C for 5 minutes in a Wabash V 50 H Compression Press machine. The mold was then compressed under a pressure of 15 tons for 3 minutes, followed by cooling to 30°C under pressure. Finally, the compressed sheets were cut to the required shape according to ASTM standard of each subsequent testing methods.



Screw speed = 50 rpm.

Figure 2 Processing conditions of Twin Screw Extruder.

3.2.2 Characterization

3.2.2.1 *Thermal Analysis*

In order to determine the melting temperature, glass transition temperature and crystallization temperature of the blends, thermal analysis was conducted by heating the polymer blends from -100°C to 250°C and then cooling them down to 25°C and heating them again to 250°C to remove any thermal history on the polymers. The heating rate and cooling rate used were 10°C/min. using a Netzch differential scanning calorimeter.

3.2.2.2 *Thermogravimetric Analysis*

To determining the decomposition temperature of the polymer blends, TGA was employed. This was conducted by heating the blends from 25°C to 600°C at a heating rate of 10°C/min using a DuPont Instrument model 2950.

3.2.3 Mechanical Properties of Polymer blends

3.2.3.1 Tensile Properties Testing

Tensile strength and tensile modulus of the blends were determined as per ASTM D638-91 using an Instron Universal Testing Machine at room temperature on a three mm. thick dumbbell-shaped specimen that was cut by a pneumatic punch. The tests were carried out using a 100 kN load cell at cross-head speed of 200 mm min^{-1} with gauge length of 50 mm. The results were obtained from a mean value of five specimens.

3.2.3.2 Hardness

Hardness of the blends was measured using Durometer (shore D) on a six mm. thick specimen. The tests were carried out according to ASTM D 2240. Results of the tests were reported from a mean value of five measurements on each sample.

3.2.3.3 Gloss

Glossiness of the blends was measured by Gloss-Haze Tester at both 20° and 60°. Results for each sample were obtained from a mean value of five-measurement on each specimen.

3.2.4 Rheological Properties Measurement

An ARES Rheometric Scientific Instrument under the dynamic condition with cone-and-plate geometry was used for measuring rheological properties, storage modulus, and loss modulus and tan δ. All experiments were carried out at 130°C on a one-mm thick circular shaped specimen. To make sure that the behavior of the test specimens were in the linear viscoelastic range, frequency and strain amplitude were varied between 0.1 and 100 to obtain a suitable range of the torque. This value, higher than the melting temperature of each polymer blends, was chosen to allow adequate duration of the experiments without noticeable degradation.

3.2.5 Dynamic Mechanical Properties

The storage modulus (G'), loss modulus (G") and tan δ were measured as a function of temperature at a frequency of 6.28 rad s^{-1} and strain rate of 0.025%. The temperature range studied was from -120°C to 200°C and the sample was heated at rate of 10°C min^{-1}. Dimension of the rectangular testing specimens were 45 x 10 x 1 mm.