CHAPTER III
EXPERIMENTAL

3.1 Materials

3.1.1 ESCOR® Terpolymer

ESCOR® 310 terpolymer is a random terpolymer that consists of 87 %wt of ethylene, 6.5 %wt of acrylic acid and 6.5 %wt of methyl acrylate. It provides excellent adhesion to a variety of polar and non-polar materials. For example, ESCOR® 310 can be used as an adhesive film for industrial laminations or to bond elastomers to fabrics and metals. It can also be used for extrusion coating/laminating and co-extrusion applications where bonding of polar to non-polar materials is critical. It delivers the highest service temperature and modulus of all the ESCOR® acid terpolymer. It is used as heat-activated adhesive sealant compounds, TPOs with improved adhesion to metal parts, engineering thermoplastic impact modifier, and compatibilizer for polar and non-polar polymers.

ESCOR® 320 terpolymer consists of 76 %wt of ethylene, 6 %wt of acrylic acid and 18 %wt of methyl acrylate. It provides excellent adhesion to a variety of substances and other film components, such as aluminum foil, polyolefin, polycarbonate, PET, etc. Applications for these terpolymers are tie resin for blown film, tie resin for extrusion coating and laminating, OPP/ESCOR®320/nylon laminates, and PE/ESCOR®320/aluminium foil laminates.

The characteristics of the ESCOR® terpolymer used are given in Table 1.

Table 1 Characteristics of ESCOR® terpolymer

Properties	ESCOR® 310	ESCOR® 320
Melt Index (g/10min)*	6.0	5.0
Density (g/cm^3)*	0.941	0.953
Acid Number (mg KOH/ g polymer)*	45	45
Peak Melting Temperature (°F(°C))*	201(94)	169(76)
Peak Crystallinity Temperature (°F(°C))*	165(74)	-
Tensile strength at break (ASTM D-638)(Psi(MPa))	2100(14)	-
Elongation at break (ASTM D-638) (%)	570	-
Hardness, 15s Shore D	41	-

* using EXXON method

3.1.2 Ethylene Acrylic Acid Copolymer

Acrylic acid contains a carbonyl group that will easily give up a hydrogen ion (H^-) to a chemical that will accept hydrogen ions. In the bulk material, a substantial amount of hydrogen bonding is present, which helps to increase toughness. This acid group is also responsible for the high adhesion of these copolymers to a variety of surfaces. As would be expected from the presence of a polar pendent group, the bondability (i.e. the ability to join two pieces of EAA film together with heat) of EAA is high. Crystalinity is lower than in PE and hence film clarity is higher. EAA films are more resistant to oils and greases than PE but is more permeable to water vapor. They are, therefore, another choice for food packaging films and shrink-wrap.

EAA copolymers used in this study are EAA1, EAA2, EAA4, and EAA5 that they have different amount of acrylic acid.

Both of ESCOR®310 terpolymer and EAA copolymers were supplied by Exxon Chemical Co., Ltd. (USA).

3.2 Experimental Procedure

3.2.1 Polymer Blend Preparation

3.2.1.1 Blending

Blends of ESCOR® 310 terpolymer and EAA copolymers (EAA1, EAA2, EAA4, and EAA5) were prepared at ratio of 0/100, 5/95, 10/90, 20/80, 30/70, 40/60, 50/50, 60/40, 70/30, 80/20, 90/10, 95/5, 100/0 using a Collin co-rotating twin screw kneader ZK-25 (25mmx30D). Screw speed of 50 rpm was employed. All processing passes were carried out using the processing condition as shown in Figure 3.1.

Zone 1	Zone 2	Zone 3	Zone 4	Zone 5	Zone 6
125 °C	125 °C	127 °C	127 °C	130 °C	130 °C

Figure 3.1 Processing condition.

The extrudate was cooled in the water (~25°C) and cut into pellet form by a Planetrol 075D2 pelletizer.

3.2.2 Specimen Preparation

A sample of 75 g was pressed on a Wabash V50H compression molding machine. The steps used in the process for this study were 160 °C without pressure to preheat 5 minutes and 160 °C with 15 tons force for 3 minutes. Mold was cooled down under pressure to 32 °C. The mold used was a picture-frame type mode stainless steel and 3 mm of thickness of the mold cavity.

3.3 Characterization of Polymer-Polymer blends

3.3.1 Mechanical Properties

3.3.1.1 Tensile properties

Tensile properties such as Young's modulus, Tensile strength at break and Elongation at break were measured using an Instron Universal Testing Machine, Model 4206 following different American Standard Test Method (ASTM). Results of these tests were obtained from an average of five specimens for each sample.

A. Young's modulus and tensile strength at break

Young's Modulus and Tensile strength at break were measured according to the ASTM D638-91 test procedure. The load cell of 100 kN and 200 mm/min cross-head speed were used for all tests. Dumbbell shape specimens were cut from compressed sheets using a pneumatic punch, and the specimen dimensions, were as follow: width of narrow section was 13 mm and the gauge length was 50 mm.

B. Elongation at break

Elongations at break were measured according to ASTM D-1708 test procedure. The load cell of 100 kN and 1.3 mm/min cross-head speed were used for all test specimens. Dumbbell shape specimens were cut from compressed sheets using a pneumatic punch, and the specimen dimensions, were as follow: width of narrow section was 4.75 mm and the gauge length was 22.25 mm.

3.3.1.2 Hardness

Hardness of the blends were measured using Durometer (ShoreD), according to ASTM D-2240. Thickness of test specimens were 6 mm. The results were obtained from mean value of five measurements carried out on each sample.

3.3.1.3 Gloss

Gloss of the blends was measured using BYK Gardner Gloss-Haze Reflectometer at 20° and 60°, according to ASTM D-521. The results were averaged from five measurements on each sample to obtain a mean value.

3.3.2 Thermal Analysis

Differential Scanning Calorimetry (DSC)

The thermal behavior of the ESCOR®310/EAAs blend samples was determined on a Perkin-Elmer DSC7. A sample of 9-12 mg was sealed in clamped closed aluminum sample pans. Nitrogen gas was used as a purge gas. The heating and cooling program are as follow:

The sample was heated from 30 °C to 140 °C at heating rate of 5 ° C/min and cooled down to 30 °C at cooling rate of 5 °C/min and reheated it again to 140 °C. The first heating scans were carried out to remove any thermal history of the sample and the melting temperature (T_m), Enthalpy of fusion (ΔH_f) were collected from thermogram of the second scan.

3.3.3 X-ray Diffraction (XRD)

X-ray diffraction patterns of all ESCOR®310/EAAs blends were obtained using a Rigaku Rint2000 diffractometer equipped with a graphite monochromator and a Cu tube for generating a CuKα radiation (1.5046 °A). First, the sheet samples were erased their history of polymers using DSC at heating rate 5° C/min. Then, the sheet sample was put on a glass slide specimen holder using vasaline as binder between sample and glass holder. The sample was examined between 5°-35° 2θ at a scanning rate of 2° 2θ /min in 0.02° 2θ increments. CuKα radiation with $\lambda = 0.154$ nm. was used as the X-ray source and operated at 40 kV and 30 mA. The digital output of the proportional X-ray detector and the gonimeter angle measurement was sent to an online microcomputer for storing the data.

The XRD patterns of the crystalline and amorphous scattering in the diffraction pattern were separated from each other and were used for determination

the degree of crystallinity. The degree of crystallinity (χ_c) is equal to the ratio of the crystalline scattering area to the total scattering area, both crystalline and amorphous.

3.3.4 Rheological Measurement

Pellets of polymer blends of ESCOR® 310 and EAA1-5 were compressed into circular disk of 1-mm thickness. An ARES Rheometrics Dynamic Analyzer RDA-II with cone-and-plate geometry was used for measuring rheological properties, storage modulus, loss modulus and tan δ. Measurements were carried out at 130° C and to make sure that the behavior of the test specimens were in linear viscoelastic range, frequency and strain amplitude were varied in the range between 0.1 to 100 rad/s to obtain a suitable range of the torque. This value, higher than the melting temperature of each polymer blends, was chosen a allow adequate duration of the experiments without noticeable degradation.

3.3.5 Dynamic Mechanical Properties

The storage modulus (E'), loss modulus (E") and tan δ were measured by A Solid Analyzer RSAII (Rheometric Scientific). Film and fiber fixture was used to mount the samples and 3K temperature steps were used. All experiments were measured with a frequency of 10 rad/s and a strain rate of 0.1% and with static force tracing dynamic force. The temperature range studied was from −150°C to 150°C and the sample was heated at rate of 10°C/min.