CHAPTER 3

MATERIALS AND METHODS

3.1 POLYMERS

1. POLYPROPYLENE HOMOPOLYMER (PP)

REPOL H200MA, with a melt flow index (MFI) of 20 g/10 min, was supplied by Reliance Industries Limited, Mumbai, India.

2. HIGH-DENSITY POLYETHYLENE (HDEPE)

HDPE grade Indothene HD 50 MA 180 was supplied by Indian Petro Chemicals Limited, Baroda, India, with density 0.950 g/cm³ and MFI 18 g/10 min.

3. POLYSTYRENE (PS)

PS grade LGG104 was supplied by LG Plastics with MFI 18 gm/10 min.

4. HIGH IMPACT POLYSTYRENE (HIPS)

HIPS grade LGH 302 was supplied by LG Plastics with MFI 18 gm / 10 min.

3.2 MODIFIERS

1. CALCIUM CARBONATE

Calcium Carbonate used in this study was commercial grade used as filler in polymers.
2. ETHYLENE PROPYLENE DIENE MONOMER (EPDM)

EPDM used in this study was grade 301T supplied by Herdelia Unimers

3.3 POLYMER BLEND PREPARATION

HDPE and PP granules were placed in an air oven set at 100 °C for 4 hours to remove any moisture present and allowed to cool to room temperature in a desiccatior. Six blend compositions were selected namely 100% HDPE/0% PP; 80% HDPE/20% PP; 60% HDPE/40% PP; 40% HDPE/60% PP; 20% HDPE/80% PP and 0% HDPE/100% PP and the granules were weighed out. Each mixture was melt blended either with or without the modifiers. The modifiers Calcium Carbonate and EPDM were added in 2%, 4% and 6% by weight of the neat blends. The granules were fed into the mixing chamber of a Thermo Haake Rheomix 600P blender set at 180° C. The blender is fitted with Roller Rotor blades counter rotating at 3:2 speed ratio. They were set to rotate at 30 rpm. Blending was continued till the mixing torque stabilized to constant values in all the cases [4]. Afterwards, the modifiers (either CaCO₃ or EPDM) were added. A mixing time of 5 minutes was allowed to complete the blending, during which time the torque would become steady.
Thermo Haake has a mother unit Thermo Haake Rheocord 300P. It is fitted with a motor of 4kW capacity and can work at a speed up to 250 rpm. The blending can be done up to a torque of 300 Nm. The blender has a mixing chamber of capacity 120 cm$^3$ without the rotors and 69 cm$^3$ with the rotors. The rotors are Roller Rotor type and work counter rotating. The mixing chamber is provided with three separate heaters and can be heated to a temperature of 450°C and the rotors can handle a torque of 160 Nm. The best mixing efficiency is obtained when the mixing chamber is about 70% filled. If the melt density of the test substance is known we can determine the sample weight as follows:

$$\text{Sample weight} = \text{melt density} \times \text{chamber volume} \times 0.7$$

$$= \text{melt density} \times 69 \times 0.7$$

A sample weight of 40g was chosen for each mixing.
3.4 PREPARATION OF TEST SPECIMENS

The hot polymer blend taken out from the mixing chamber was passed through a laboratory size two roll mill. The sheet form so obtained was cut to small pieces and subjected to injection moulding at 180° C in a semiautomatic plunger type injection moulding machine (Texair JIM –1H). Dumbell specimens were prepared (according to ASTM D 638 specification). Specimens for conducting the notch sensitivity test were notched to 1 mm depth before testing.

3.4.1 MOULDS FOR SAMPLE PREPARATION

Moulds for injection moulding of the samples were of low carbon steel. Single gating system was employed. Mould cavities were made for the specimens as per ASTM – D – 638 specification. Moulds for Flexure and Impact tests were of similar configuration.

*Figure 3.2:* Injection mould for preparing test samples
3.5 MEASUREMENTS

3.5.1 TENSILE TEST

The tensile properties of the specimens were determined using dumb bell shaped specimens according to ASTM – D – 638. The length between the jaws at the start of each test was fixed to 40 mm and at least six concordant measurements are taken to represent each data point. A computerised Universal Testing Machine (Schimadzu AG1).

The tensile test was conducted at different crosshead speeds for obtaining various strain rates.

To conduct notch sensitivity test, the specimens were centrally notched to 1 mm depth before testing and the test was conducted at different crosshead speeds for studying the notch sensitivity at various strain rates.

3.5.2 IMPACT TEST

The resistance to impact is one of the key properties of materials. The ability of a material to withstand accidental knocks can decide its success or failure in a particular application. When the design of a component is being considered, knowledge of the impact strength of a prospective material is important even though it cannot be used quantitatively in the design calculations. Instead it serves as a qualitative check that the material is in some desired condition, thought to be appropriate to the envisaged service conditions. Unfortunately, however, it is not possible to attach a unique value to the impact strength of a thermoplastic material. This depends on a wide range of variables.
including temperature, straining rate, stress system, anisotropy, geometry of component, fabrication conditions, environment and so on. This is a formidable list and certainly enough to discourage any designer who is looking for precise information on which to base a design. Experience has shown that the best the designer can do to avoid premature service failures is to relate, as closely as possible, the conditions pertaining in the specific application to the impact data which is available. This task will be reduced slightly when it is realized that most of the complaints regarding unsatisfactory impact performance arise from sporadic brittle fractures which are uncharacteristic of that material. The key to the problem is then to avoid conditions which are likely to promote brittleness in the material. In general the factors likely to increase the tendency towards brittle fractures are low temperatures, stress concentrations, high strain rates and internal stresses.

For normal use, if a material does not fail in a brittle manner in tests under all conditions likely to be found in service then it may be regarded as tough. In a few applications, impact loading may be the primary stress system experienced by the component in which case the relevant factors need to be studied in greater detail.

3.5.2.1 MODES OF FAILURE UNDER IMPACT

The most convenient classification of failure of thermoplastics materials under impact is due to two classes - brittle or ductile. A brittle failure is a low energy process in which a crack initiates and propagates before any yielding of the material occurs. In a ductile fracture there is yielding, probably localized in the failure region and considerable energy is absorbed.

It is important to realize that both types of failure may be observed in the one material depending on the service conditions. As temperature is reduced, for
example, there is often a marked transition from ductile to brittle fractures. The appearance of a failure in service is frequently the only method of classifying the type of failure. Brittle failures usually exhibit smooth, glassy or possibly splintered fracture surfaces. Ductile failures on the other hand normally leave evidence of appreciable deformation and yielding. Identification of the latter is usually aided by stress whitening of material in the failure zone. In practice intermediate failure may also occur. In such cases there will be a change from one type of failure to the other across the fracture surface.

The impact characteristics of the specimens were investigated for the various blend compositions using an impact Testing Machine.
Make and model: Resil Impact Tester (Izod and Charpy) CEAST, Italy.

![Resil Impact Tester](image)

*Figure 3.3: Resil Impact Tester*

For applying impact load, a 4J hammer was used.

Hammer striking velocity was maintained at 3.46 m/second.
The test was conducted in the Izod configuration.

The impact characteristics obtained were,

1. Energy absorbed by the test specimen (J)
2. Resilience (KJ/m²)
3. Impact Strength (J/m)

3.5.3 FLEXURE TEST

The flexural properties of the specimens were determined using a computerised universal testing machine (Schimadzu UTM). The simply supported beam configuration was used with a span of 50 mm.

3.5.4 PHOTO ELASTIC INVESTIGATIONS

Photoelasticity is an experimental method to determine stress distribution in a material. The method is mostly used in cases where mathematical methods become quite cumbersome. Unlike the analytical methods of stress determination, photoelasticity gives a fairly accurate picture of stress distribution even around abrupt discontinuities in a material. The method serves as an important tool for determining the critical stress points in a material and is often used for determining stress concentration factors in irregular geometries.

3.5.4.1 PRINCIPLES OF PHOTOELASTICITY

The method is based on the property of birefringence, which is exhibited by certain transparent materials. Birefringence is a property by virtue of which a ray of light passing through a birefringent material experiences two refractive indices. The property of birefringence or double refraction is exhibited by many optical
crystals. But photoelastic materials exhibit the property of birefringence only on the application of stress and the magnitude of the refractive indices at each point in the material is directly related to the state of stress at that point. Thus, the first task is to develop a model made out of such materials. The model has a similar geometry to that of the structure on which stress analysis is to be performed. This ensures that the state of stress in the model is similar to state of stress in the structure.

When a ray of plane polarized light is passed through a photoelastic material, it gets resolved along two principal stress directions and each of these components experiences different refractive indices. The difference in the refractive indices leads to a relative phase retardation between the two component waves. The magnitude of the relative retardation is given by the Stress Optic Law:

$$R = Ct (\sigma_{11} - \sigma_{22})$$

Where $R$ is the induced retardation, $C$ is the stress optic coefficient, $t$ is the specimen thickness, $\sigma_{11}$ is the first principal stress, and $\sigma_{22}$ is the second principal stress.

![Figure 3.4 Principle of plane Polariscope.](image-url)
The two waves are then brought together in a polariscope. The phenomena of optical interference take place and we get a fringe pattern, which depends on relative retardation. Thus, by studying the fringe pattern, one can determine the state of stress at various points in the material.

Two types of fringes are formed in a plane polariscope. They are isochromatics and isoclinics.

Isochromatics are the locus of points along which the difference in the first and second principal stress remains the same. Thus, they are the lines which join the points with equal maximum shear stress magnitude. Isochromatic fringes are colourful and the fringe colour is related to the difference in principal stresses through the Stress – Optic Law. From the photoelastic stress pattern, isochromatics provide qualitative information regarding stress distribution within the component.

Isoclinics are locus of points in the specimen along which the principal stresses are in the same direction. They are black in colour and are superimposed on the Isochromatic pattern. They occur whenever either principal stress direction coincides with the axis of polarization of the polarizer. Isoclinics provide information about the directions of principal stresses in the model and provide necessary information for solution of two – dimensional stress problem.

A standard plane polaiscope shows both Isochromatic and Isoclonic fringes and this makes quantitative stress analysis difficult.
3.5.4.2 EXPERIMENTAL PROCEDURE

PS / HIPS mix was prepared at various blend compositions in Thermo – Haake rheomix by melt mixing. Specimens were injection moulded in Texair (JIM - 1H) injection moulding machine at 180° Celsius.

A plane polariscope was used for conducting the investigations. The test specimens were placed between the polarisr and analyzer. A broad source of light was used for illuminating the system. The fringe patterns were observed through the analyzer. Isochromatic fringes with distinct colours were visible for each PS/HIPS blend composition. The fringe patterns were photographed for qualitative analysis.

Figure 3.5: Plane polariscope
3.5.5 MELT FLOW STUDIES

The Melt Flow Index (MFI) of polymer blends was determined using a melt flow index apparatus (make CEAST, Italy).

For HDPE/PP blends, the temperature was set at 190°C and a weight of 2.15 kg was applied. For PS/HIPS blends, the temperature was set at 200°C and the weight applied was 5 kg.

![Figure 3.6: Melt Flow Index apparatus](image-url)