Chapter 1

Introduction to Blends

CONTENTS

Most of the materials be either natural or synthetic, have limited utility. However, technical ingenuity increases the utility of these materials beyond imagination. The wide range of steel, which is produced by just varying carbon or other elements in iron, gives the required balance of properties, such as strength, hardness and corrosion resistance. Since ages, mixing has been preferred for widening the performance range of various materials. This is evident in the history of any type of material, including polymers. Polymers, on their own have very limited utility. In fact in many cases their processing is very difficult e.g. PVC. In order to make them useful, certain additives are incorporated to give ease of processibility, and useful mechanical and load bearing properties[1-3].

Up to 1846 only natural rubber and Gutta - Percha were known and were blended. Once nitrocellulose was invented, it was also mixed with natural rubber. Soon after when cellulose acetate became available it was blended with nitrocellulose. From the dates of commercialization of new polymers and their use in blends, one can notice the surprising closeness of these events. Many early blends were prepared from natural elastomers. In the beginning of the 20th century the thermoset resins and their mixtures were extensively used for blending. However, in recent times use of thermoplastic polymer blends is dominating the thermosets[4-8].

Mixing or otherwise blending of polymers is the most versatile way for producing new materials with tailor-made properties, which are not possible otherwise via copolymerization, or by developing a new monomer.

The polymer blending is one of the major areas of thrust in science and technology and is a convenient route for the development of newer polymeric materials, which combine the excellent properties of more than one existing polymers. This strategy is usually cheaper and less-time consuming, than the development of new monomers and/or new polymerization routes for the development of entirely new polymeric material[6, 8].

Different levels of improved physical properties can be achieved in polymer blends by proper selection of blend compositions, processing techniques, and use of a suitable compatibilizers[9].

Today, demand of polymer blends is over 30 % of total polymer consumption, and its demand is increasing with 9 % annual growth rate. Considering worldwide scenario in the field of polymer blends and their growing commercial applicability, this field has become most popular choice for raw material manufacturers, polymer educators, scientists, and technologists[10].

The blends have some unique properties, which are different from the basic polymers from which they have been derived. The exploitation of certain unique sets of properties of the individual polymer for the benefit of overall properties of a multi component system is the main concept behind the polymer blends.

Polymer blends are subdivided into two groups

a. homogeneous blends, and
b. heterogeneous blends.

In homogeneous blends both blend components lose part of their identity and the final properties usually are the arithmetical average of the properties of the blend components.

In heterogeneous blends, the properties of all the blend components are present and weaknesses of one of the polymer to a certain extent are camouflaged by the strength of the other one. However, in a very few exceptional cases, properties of the either homogeneous or heterogeneous blends can be better than those of the individual components. This synergism is unfortunately hard to explain. Heterogeneous blends appear in a variety of morphologies. Frequently observed morphologies are

- a dispersion of one polymer in the matrix of the other polymer; and
- a co-continuous two-phase morphology.

The type of morphology obtained is dependent on the nature of the components, the viscosity and the ratio of the two polymers at the blending temperature and the presence of third component as compatibilizers.

1.2 The Reasons for Blending[4, 5]

Polymer blending is desirable because

(i) it gives products with tailor made properties.

(ii) it reduces the cost of an engineering plastics by diluting it with commodity one

(iii) it can develop the products with improved mechanical performance.

(iv) it helps to quicker formulation changes, plant flexibility and high productivity.

(v) it results in savings in space and capital investment as it reduces the number of grades to be manufactured and stored.

(vi) it helps in recycling of industrial and municipal waste.

1.3 Some Important Terminologies

- **Polymer blends**: mixture of chemically different polymers or copolymers with no covalent bonding between them.
- **Polymer alloys**: a class of polymer blends, heterogeneous in nature with modified controlled interfacial properties or morphology.
- **Compatibilization:** a process of modification of interfacial properties of an immiscible polymer blend, leading to the creation of a polymer alloy.
- **Homologous polymer blends**: a subclass of polymer blends limited to mixtures of chemically identical polymers differing in molecular mass.

- **Miscible polymer blends**: polymer blends which form uniform single phase structure, for which $\Delta G \approx \Delta H_{mix} \leq 0$
- **Immiscible polymer blends**: polymer blends which exhibit two or more phases at all compositions and temperature, for which $\Delta G \approx \Delta H_{mix} > 0$
- **Compatible polymer blends**: a term indicating commercially important materials with enhanced physical properties over the constituent polymers and homogenous to the eye.
- **Interpenetrating polymer network (IPN)**: a subclass of polymer blends reserved for the mixture of two polymers where both components form a continuous phase and at least one is synthesized or crosslinked in the presence of the other.
- **Miscibility**: the term miscibility of polymer blends is being defined in terms of the equilibrium thermodynamics which must be considered only within the range of dependent variables e.g. temperature, pressure, molecular weight and chain structure, under which free energy of mixing is negative. The miscibility concept of given polymer phase is insufficient in generalization of such behavior for the other pairs of the similar polymers or at other conditions of mixing.

The general relation between blends and alloys is shown in **Fig.1.1.** which indicates, polymer alloy as a specific sub-class of polymer blend. The polymer alloys can be further subdivided into two classes

(i) alloys containing sufficient amount of compatibilizer which leads to very fine usually sub-micron level dispersion of the phases. As a result molded part does not show streaking and joint weakening.

(ii) alloys containing some amount of compatibilizer which facilitates the formation of the desired morphology in a successive processing steps. Most of the commercial polymer alloys belong to the first type.

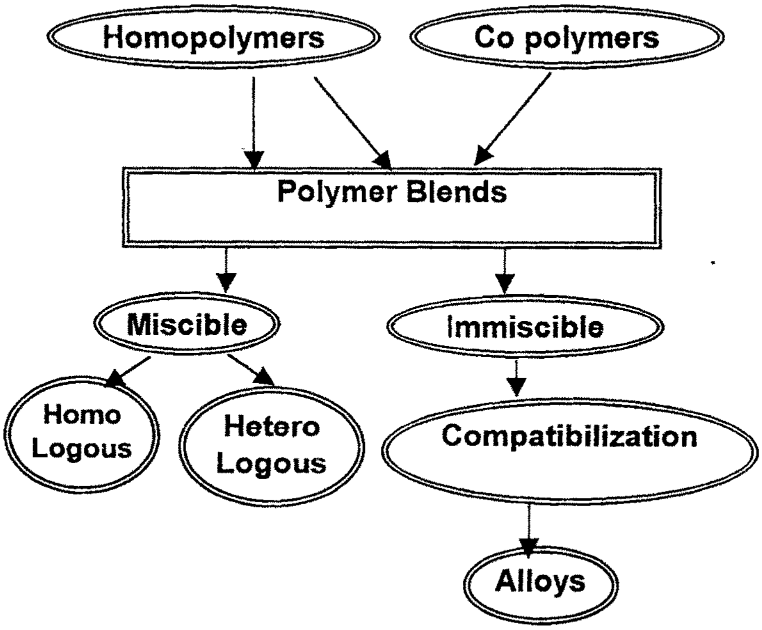

Fig. 1.1 General relation between blends and alloys

1.4 Phase Behavior of the Polymer Blends

Like small molecules the polymer blends also exhibit varied phase behaviors. However, encountering of one or the other kind of phase behavior in a particular mixture is quite different for a pair of polymers than for small molecules[10-12].

The kind of phase diagrams for mixtures of small molecules such as metal alloys, in which the components are soluble at high temperatures and phases separate below a temperature, known as critical temperature are illustrated in **Fig. 1.2.** The critical temperature for such a situation is called as upper critical solution temperature or **UCST**, because it is the highest temperature at which there is some range of composition where the blend is immiscible. Above the **UCST** the blend is miscible at all compositions. This is represented as **Fig 1.2 a.**

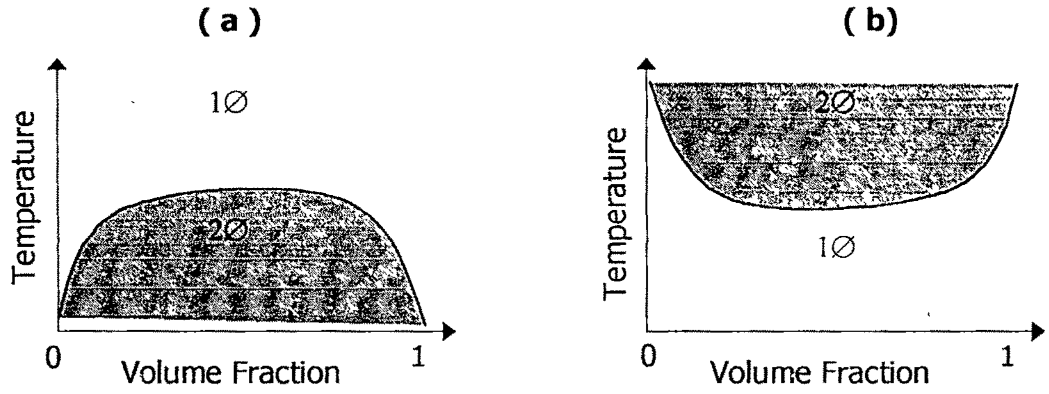

Fig. 1.2 Polymer Blends phase diagram types

(a) UCST, (b) LCST

The opposite case is where the mixture is miscible at low temperatures, but begins to show phase separation as it is heated. This critical temperature at which phase separation begins is called as a lower critical solution temperature, or **LCST**. Such behavior is quite rare for small molecules, but is often found in polymer solutions and blends. (**Fig.1.2 b**)

1.5 Flory-Huggins-Staverman Theory of Thermodynamics

The concept of miscibility of polymer blends was explained by Flory in 1941 through thermodynamics, Which is discussed in brief here.

Homogeneous miscibility in polymer blends requires a negative free energy of mixing

$$\Delta G_{mix} = \Delta H_{mix} - T\Delta S_{mix} \qquad (1)$$

However, if two high molecular weight polymers are blended, the gain in entropy, ΔS_{mix} is negligible, and the free energy of mixing can only be negative if the heat of mixing ΔH_{mix} is negative. In other words, one can say that the mixing must be exothermic. This requires specific interactions between the blend components. These interactions may range from strongly ionic to weak Van der waals' forces, which explain why polymer miscibility is the exception rather than the rule. Using this simple thermodynamics, Flory-Huggins-Staverman in 1941 developed a theory based on the lattice model for polymer mixture[13]. Lattice model is based on the assumption that each monomer repeat unit of the chains occupies a single lattice site.

The free energy of mixing of two polymers as per this theory is given by

$$\frac{Gm}{VRT} = \frac{\varnothing_1}{v_1 N_1} \ln\varnothing_1 + \frac{\varnothing_2}{v_2 N_2} \ln\varnothing_2 + \varnothing_1\varnothing_2 \frac{\chi}{v} \qquad (2)$$

where **V** is the total volume of the sample, **R** is the gas constant, **T** is the absolute temperature, **N_i** is the degree of polymerization of components, $\varnothing_i$ is the volume fraction of the components, **v_i** is the molar volume of its units, and **v** is an arbitrary volume expressed as $\sqrt{v_1 v_2}$. The first two terms of right hand side of equation represent the combinatorial entropy of mixing, and the last term comes from the interaction enthalpy, χ is called the 'Flory interaction parameter'. This theory has been quite successful in describing many of the qualitative features of polymer blend thermodynamics.

This theory also explains why most of the polymer blends are immiscible. Since polymers with commercial value have degree of polymerization more than 1000, the first two terms representing the entropy of mixing become very small. This low combinatorial entropy of mixing can be considered as a direct result of the high configurational entropy which is a characteristics of polymer chains. The entropy of mixing depends on the mole fraction of chains, rather than the volume or weight fraction. This is why the first two terms of the **equation 2** are divided by the degrees of polymerization of the two components. Since these forces are quite local, extending only over a range of the order of a repeating unit, the interactions between the two polymers are quite similar to those of their small analogs. Thus the enthalpy of mixing will not depend significantly on the molecular weight of the components, but it is positive for most of the mixtures[14, 15].

The entropy of mixing of small molecules can be so large that it overwhelms the positive mixing enthalpy and thus causes the components to mix under a wide range of temperature and composition. This explains why polyethylene and polypropylene are immiscible at any condition whereas octane and iso- octane are miscible at any composition in their phase diagram. Thus the miscibility of polymers is largely determined by the interaction parameter χ.

Further χ helps to estimate the interfacial tension γ at the interface of two phases. The size of domains produced during mixing is also controlled by γ. Thus both χ and γ are crucial determinants of the physical properties of the blends.

Through FHS theory, miscibility in blends can be predicted by following ways.

(i) In polymers of low molecular weights the entropy of mixing is large enough to balance the enthalpy of mixing. From FHS model, one can derive the following expressions for the critical composition and temperature of the blend.

$$\varnothing_{1,crit} = \frac{1}{1 + \left[\frac{N_2 v_2}{N_1 v_1} \right]^{1/2}} \qquad (3)$$

$$\chi_{crit} = \frac{v}{2} \left(\frac{1}{N_1 v_1} + \frac{1}{N_2 v_2} \right)^2 \qquad (4)$$

Blend is symmetric if both components have similar parameters, i.e. $N_1 = N_2 = N$ and $v_1 = v_2 = v$ resulting into $\varnothing_{1,crit} = 0.5$ and $(\chi N)_{crit} = 2$.

In most cases the components of polymer blend do not differ widely in molecular weight or density. So even if χ is large for a particular blend, if N is small enough, the blend can be miscible. However, as polymer properties depend on its molecular weight, many properties will be lost at small N[16].

(ii) Alternately blends in which χ as well as enthalpy of mixing are negative due to the presence of strong inter molecular interactions between the blend components. Such as intermolecular interactions can induce the miscibility in blends through hydrogen bonding[17-19].

(iii) Thirdly in the blends where χ is negative but there are no inter and intramolecular attractive interactions between the components, rather one or both polymers are statistical copolymers and the balance of the forces among the several monomers results in miscibility. This has been termed as copolymer effect which was explained by Brogly et.al.[20].

Thus the FHS theory explains many of the general observations for the immiscibility in blends. However, it neither explains miscibility quantitatively nor the behavior of blends in **LCST** phase.

1.6 Blending Process

Quality blends can be prepared by controlling rheology and morphology of the blends.

Rheology

Rheology controls the viscoelastic behavior of polymer blends, its processing conditions, flow mechanism and its effect on phase morphology and mechanical properties. For polymers the viscosity η increases as the shear rate Υ decreases and reaches the Newtonian plateau. Thus the nonlinear part of this viscosity dependence may be approximated by

$$\eta = K_1 \Upsilon^{m-1} \qquad (5)$$

where K_1 is a viscosity constant, and **m** is the rheofluidity index [21].

The thermal dependence of melt viscosity, which is measured at a constant shear rate, can be expressed by a modified Arrhenius law

$$\eta = K_2 \exp(\Upsilon E / RT) \qquad (6)$$

Where E is the activation energy and K_2 is a viscosity constant[22].

Considering these classical relationships of polymers, the melt viscosities of immiscible polymer blends show dependence on the interactions at the interface and the phase morphology, which is strongly depended on the addition of third component as a compatibilizer.

Log additivity rule also depends on the interaction of two polymers when blended. The interactions between two polymers govern the viscosity of blends. The log-additivity rule can be expressed as

$$\log \eta = \Sigma_i \varnothing_i \log \eta_i \qquad (7)$$

where $\varnothing_i$ and η_i are volume fraction and viscosity of component i, respectively.

On the basis of viscosity - composition dependence with respect to the log-additivity rule, polymer blends can be classified in four categories[23].

(i) additive blends, where melt viscosity follows Eqn.7,

(ii) blends showing positive deviation from the log additivity rule Eqn. 7. Some blends particularly immiscible show a positive deviation from the log-additivity rule

(iii) blends showing negative deviation from the log-additivity rule due to weaker interfacial interaction . and,

(iv) blends showing both positive and negative deviation from eqn. 7 due to the phase structur changes with composition.

In the case of immiscible blends, two mechanisms govern rheology

- The 'emulsion effect' where an increase in the relative blend viscosity is observed as a result of the addition of the second phase. The effect is maximum (η_{max}) at phase inversion composition.
- The 'interlayer slip', which increases with temperature and with shear stress[21]

An easy and qualitative way to prove the efficiency of an interfacial agent (block or graft copolymer) is to compare the viscosities of the ternary and the binary blends. An increase in viscosity would be an indication of the interfacial activity of the compatibilizer. The interfacial adhesion and thus blend viscosity, usually increases with increasing amounts of interfacial agent, until the interface saturates.

Morphology

Control of the phase morphology during blend processing is very important for the improvement of the performance of the blends. The shape, size and distribution of the phases depends on the viscosity of the phases, interfacial properties, blend composition and processing parameters.

Fig.1.3 shows the processes that occur when two polymers are blended in the melt condition, particularly when one polymer is dispersed in another one.

In a first step of morphology development the deformation of dispersed drop in the flow field takes place and then the interfacial area increases accordingly and volume of drop decreases perpendicular to the flow direction.

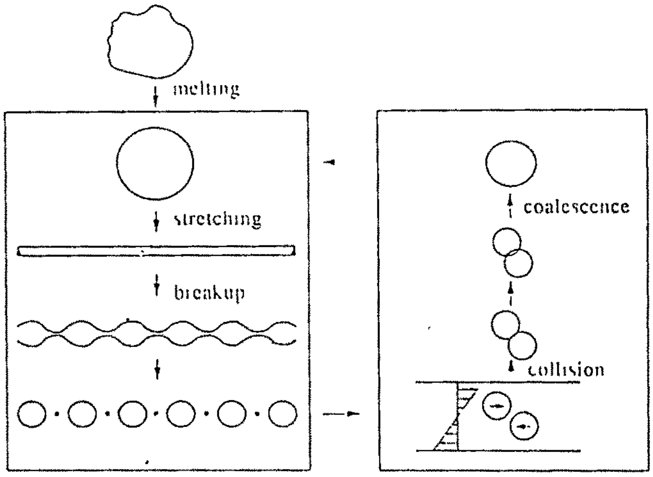

Fig.1.3 Schematic representation of the processes occurring during the melt blending of two polymers

This deformation is mainly governed by the capillary number, C_a, which is given as a ratio of the viscous force to the surface force [24, 25].

$$C_a = (\eta_m Ga) / \gamma \qquad (8)$$

where η_m is the matrix viscosity, **G** is the shear rate **a** is the average particle size and γ is shear rate. The critical value of C_a at which the particles break depends on the ratio of the particle viscosity to that of the matrix, which is minimum at ratio 1. At a given shear rate, only **a** can vary and so the steady state size of the particles varies with the viscosity ratio. Typically in the initial stage of mixing when the dispersed domains are large, the shear stress dominates the interfacial stress and drops are stretched affinely with the matrix into long thin threads. Now, when the local radius of the threads becomes sufficiently small, interfacial (Raleigh) disturbances grow on the thread and breakup threads into small droplets[26]. These drops again break into smaller drops following the same process till the shear stress exerted on drop becomes negligible.

1.7 Compatibilization

Understanding of compatibilization at a submicron level is a current research interest[27]. There has been an increasing interest in understanding the arrangement of polymer chains at and near interface and surfaces. Efforts have been made in understanding how these compatibilizers migrate towards the interface and affect the morphology and physical properties[28]. Polymer - polymer compatibility can be defined in many ways[29-31].

(i) polymer mixtures exhibiting desirable physical properties when blended are said to be a compatible polymers

(ii) polymer mixtures which do not show phase separation when blended are also said to be compatible blends.

(iii) the most widely used definitions of polymer compatibility is the miscibility of the blend components at the molecular level.

1.8 The Role of Compatibilizers in Blending

Compatibilizers are polymers exhibiting interfacial activity in heterogeneous polymer blends. The role of compatibilizer is similar to the high molecular weight surfactant molecules in oil / water or water / oil emulsions. The only difference between surfactant and compatibilizer is, the surfactant is simple organic molecule where as compatibilizer is a macromolecule with a block or graft type structure.

Like surfactant one part of the compatibilizer is miscible with one and the other part is miscible with other polymer phase. The block or graft or branched structured copolymers can be added as compatibilizers or they can also be generated in-situ during the blending process. The later one is called reactive compatibilization[32]. **Fig 1.4** represents a schematic picture of the supposed conformation of some compatibilizer molecules at the interface of a heterogeneous polymer blend. The role of compatibilizer in blending process is to retard the formation of the Raleigh disturbances generated on the threads of dispersed phase. This increases deformation tension leading to the lowering interfacial tension. As a result thread will be stretched more and becomes thinner and ultimately collapses in to small size droplets.

Moreover, the presence of compatibilizer molecules at the interface prevents the coalescence of particles during processing. Thus compatibilizers generate and stabilize a finer morphology[7].

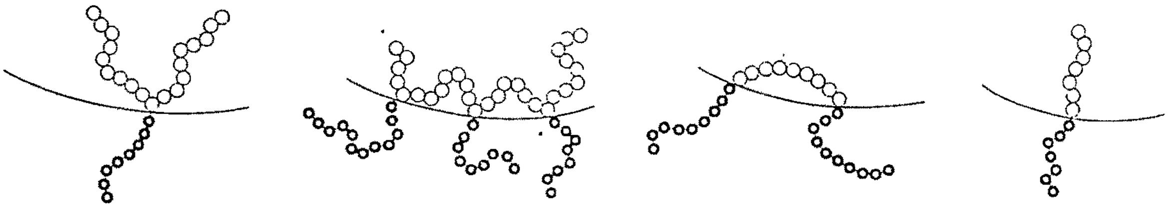

Fig.1.4 Schematic picture of conformation for diblock (a), triblock (b), multi block (c) and graft copolymer (d) at the interface of a heterogeneous polymer blends

1.9 Strategies in Compatibilization of Polymer Blends

The compatibilization of two immiscible polymers can be achieved through following routes.

By using graft or block copolymers

The emulsification of polymer blends using block or graft copolymers as compatibilizers has been proposed as the most efficient tool for obtaining a fine phase morphology and good mechanical properties[33]. Blends prepared by using block copolymers with segments identical or chemically similar to the homopolymers used in blending are designated as ' A / B / AB ' type. The use of graft copolymer is another possible route for the control of the phase morphology and the mechanical properties of immiscible polymer blends[34, 35]. Synthesis of graft copolymer is relatively easy compared to block copolymers.

By using reactive polymers

The addition of a reactive polymer, miscible with one blend component and reacting with the functional groups of the second blend component results in the *insitu* formation of a block or graft copolymers. This technique has been reported to be more advantageous compared to the addition of a block or graft copolymers to the blends[36]. For the successful application of reactive polymers as block or graft precursors, the functionality in compatibilizer must have a suitable reactivity, which can induce suitable chemical reactions e.g. use of PP-g-maleic anhydride for PP/ Nylon-6, where PP segment of compatibilizer is miscible with PP and anhydride group reacts with Nylon–6. Thus at the interface PP-g-maleic anhydride-nylon-6 is formed (**Fig. 1.5**).

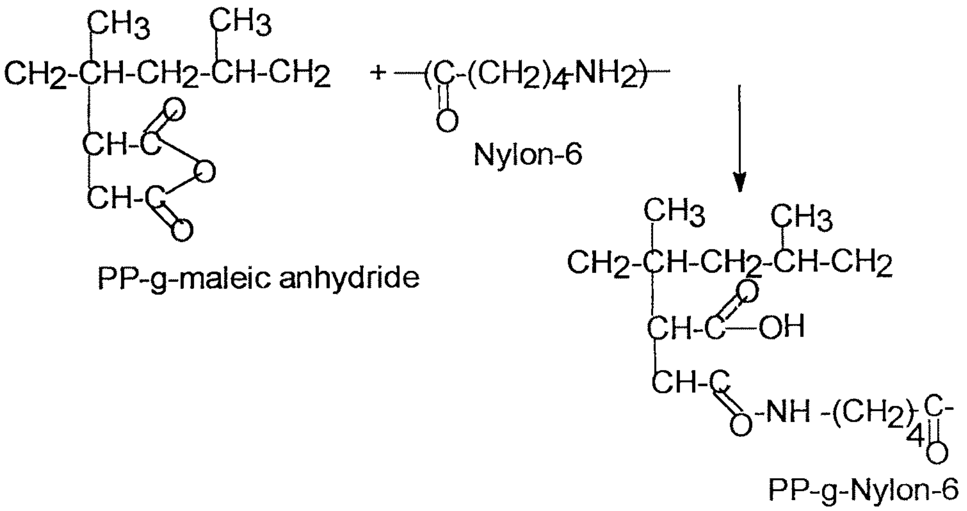

Fig.1.5 Possible reactions of reactive polymers during blending

By addition of low molecular weight chemicals

Compatibilization in blends can also be achieved by generating insitu compatibilizer using low molecular weight substance such as peroxides, anhydrides, diacids etc[36,37]. In the PE and PP blends addition of dicumyl peroxide during melt mixing results into insitu formation of PE - PP graft copolymer which brings compatibilization in PE – PP (**Fig. 1.6**).

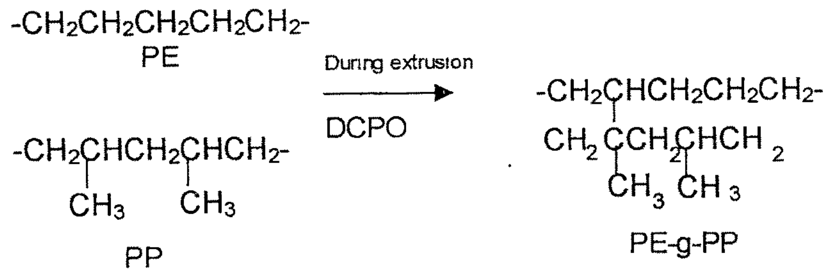

Fig.1.6 Insitu graft copolymer formation during blending

By interchange reactions

When two or more polycondensates are melt blended, several interchange reactions can take place, extent of which depends on the type of polymers, nature and concentration of the reactive groups, blending temperature, moisture content, residence time in the melt and the concentration of an interchange catalyst. e.g. interchange reaction taking place in PET / PBT during melt blending[38-40] (**Fig.1.7**).

All these above mentioned mechanisms for blend compatibilization involve either addition or insitu generation of compatibilizer. Which leads to the decreased interfacial tension and increased adhesion between the phases leading to the improvement in mechanical performance.

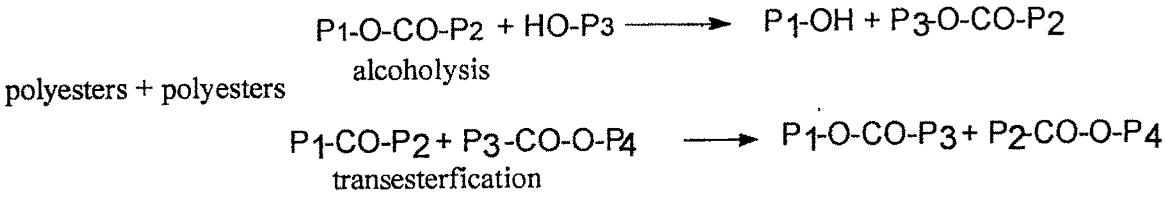

Fig. 1.7 Possible interchange reactions during the melt blending of Polycondensates (' P ' represents various condensates)

1.10 Evaluation of Compatibilization

An important thing in the development of any compatibilizer is a method of measurement of its efficiency. This involves several aspects such as the location of the compatibilizer, its effect on blend morphology and the growth of phase domains and the resulting physical properties of the blend. This can be done through various characterization techniques[41-44]. Evaluation of compatibility can be done through microscopic studies and mechanical properties.

- Optical or light microscopy,
- Scanning electron microscopy, and
- Transmission electron microscopy

are widely used, among the microscopic studies.

In most cases some mode of sample preparation has to be used such as staining by reagent, extraction of selective components, etching or phase contrast. One of the most important aspects is the resolution needed to see the domains. TEM generally gives highest resolution.

The most common reason for compatibilization is to toughen the plastics by controlling the size of a dispersed phase, which is usually an elastomer. Improvement in mechanical properties upon addition of compatibilizer can be taken as evidence of compatibilization of two phases.

1.11 The methods of Blending

Polymer Blends can be prepared through melt mixing, latex mixing and solution mixing. Details of which are given by Kienzle[45] and Xanthos and Dagli[46].

Melt blending

In this technique, the melting of constituent polymers is of utmost importance for the generation of a uniform phase morphology, which finally controls the performance of the blended materials. In general, melt mixing is used for a system in which thermal degradation does not occur at the processing temperatures. This method involves the preparation of a master batch of the compatibilizer and the dispersed phase, which is then blended with the matrix through the melt-mixing technique. Most of the blends have been prepared by this technique, using single screw or twin screw extruders.

Latex mixing

Latex mixing is also an important technique for the preparation of the commercial polymer blends. In this process two important factors pH and particle size are to be taken into consideration. For better mixing of blend components it is desirable to have the latexes of approximately similar pH. If the latex particle sizes of the blend components differ considerably, stabilizer is needed to be added to the latex with the larger size prior to blending the two latexes. Blending results in a random suspension of dissimilar particles in the mixture of latexes. This suspension is then coagulated to form solid mass in mixture.

Solution mixing

This method is useful only if polymers show degradation at melt processing conditions. In this method both blend components are either dissolved in the same solvent individually and then mixed uniformly or dissolved in two different solvents separately and mixed uniformly. The major disadvantage of this technique is the use of solvent, which needs to be removed and may lead to a change in the domain sizes in the blend, and some times may lead to a phase separation. In addition it is not economically viable as well as eco friendly process.

1.12 References

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Proposed Work

The rapid increase in the use of blends has become one of the most prominent areas of the polymer chemistry over the last few decades. The driving forces behind the increased utility of blends can be related to the high cost indeed in the production of entirely new polymer molecule relative to that for blends of existing materials and the ease and ability of blending to produce materials with combinations of properties superior to those of single polymers. However, achievement of desired properties of such products developed by blending of a particular pair of polymers is a challenge for the polymer scientists, engineers, technologists and manufacturers due to low compatibility between polymer pairs. As a result, there is a strong need to enhance this compatibility, and the compatibilization of polymer blends by the addition of block or graft copolymers, which has become the source of inspiration for researchers since last few decades. A block copolymer is very effective in reducing the interfacial tension and improving the interfacial properties by entanglement or bridging different polymer chains near the interface. However, difficult and costlier synthetic route and almost impossible to prepare at industrial scale put question for its industrial implication. The hope remains with graft copolymers, as they can be prepared easily at industrial scale. However, like block they can not be prepared with well defined molecular weights.

Hence we have undertaken development of graft copolymers as compatibilizers and testing of their efficiency in PP / ABS blends.

The question arises, why to develop PP/ABS blends?

PP being commodity semicrystalline thermoplastic offers useful balance of heat and chemical resistance, good mechanical and electrical properties, and processing ease.

Whereas ABS is a versatile family of thermoplastics that contains three monomeric constituents of acrylonitrile, butadiene and styrene. Each of these major components bring a different set of useful properties to the ABS. Acrylonitrile brings chemical resistance and heat stability. Where as butadiene provides impact resistance and toughness and styrene provides rigidity and processing ease.

Thus resulting PP/ ABS blends can have the properties of ABS (tough ness and modulus) and PP, which makes the blend more cost effective, and with chemical resistivity and improved tensile properties.

It has been proposed to carry out the work in following steps

(i) Synthesis of PP-g-2-HEMA, PP-g-acrylic acid and graft copolymers of hydroperoxidised PP as compatibilizers.

(ii) Characterization of the graft copolymers by FTIR, ^{13}C NMR, SEM, contact angle measurement, TGA, DSC and gravimetry.

(iii) Use of graft copolymers in the preparation of PP / ABS blends. Development of binary and ternary blends of different compositions. Testing of their thermal, mechanical and morphological properties.

(iv) Development of the mathematical models for the prediction of tensile modulus of the blends.