Prediction of the Behavior for Polymer Blends Using Thermodynamic Model

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Abstract

Two polymer blends were prepared. The first one was Poly methyl methacrylate (PMMA) and Poly vinyl acetate (PVAc), and the second was Poly vinyl chloride (PVC) and Polystyrene (PS). The first blend prepared by mixing two polymers (PMMA, PVAc) with co-solvent (Chloroform) at different weight percentages (0/100, 20/80, 40/60, 50/50, 60/40, 80/20, 100/0). The second blend prepared by mixing two polymers (PVC, PS) with co-solvent N,N dimethylacetamide (DMAC) at different weight percentages (0/100,20/80,40/60,50/50,60/40,80/20,100/0). Two processes were used for preparation; dissolution process was used at a room temperature (25°C) and (1atm.) pressure using co-solvent. Viscosity test was performed for stock solutions (1%wt/v=1g/dL) and other concentrations (0.75, 0.6, 0.5, 0.4, 0.3 g/dL) these concentrations are obtained by dilution of stock solution. The obtained data were collected and substituted accordance to equations (Huggins Equation) and certain criteria (Interaction parameters ΔB,μ) to identify the miscibility (or compatibiliIty).To get polymer blends in solid state, The same procedure to prepare the stock solution of diluted solutions followed but the stock solutions of polymer blends were at concentration (10%wt/v) for all different compositions and then followed solvent casting (evaporation then drying). The solid state samples were taken to hold tests of DSC and FTIR. The aim of the present work was to study the thermodynamic behavior of polymer blend via determination of miscibility. Miscibility (or immiscibility) tests are performed in three techniques: viscometry method, DSC analyzer and FTIR to test polymer blend in solid state. It was found from the viscosity method that for first blend (PMMA/PVAc), the interaction parameters (ΔB,μ) are positive for all compositions therefore the system is miscible whilst the second system (PVC/PS) is immiscible where the interaction parameters (ΔB,μ) are negative.

Differential scanning colarimetry (DSC) analyzer gave thermogram curves revealing one glass transition temperature for PMMA/PVAc blends except at (20/80) composition, this indicates that the system is miscible except at (20/80) composition. But, PVC/PS blends showed two glass transition temperatures so the system is immiscible. FTIR analysis for PMMA/PVAc blends depicted a weak interaction between the two polymers and solvent due to the miscibility establishment, whilst for PVC/PS blends, there was no interaction between two polymers to establish the miscibility, this indicates immiscible system. Calculations of Flory –Huggins interaction parameter for two systems was performed and the results pointed to miscible blend for PMMA/PVAc blends and immiscible blend for PVC/PS blends. So there is a good agreement between the experimental results and calculations of interaction parameter based on Flory – Huggins theory.

Keywords: Polymer blends; Flory –Huggins interaction parameter; Miscibility; DSC

Introduction and Theoretical Framework

Polymeric materials find growing applications in various fields of everyday life because they offer a wide range of application relevant properties. Blending of polymers is a technological way for providing materials with full set of desired specific properties at the lowest price, e.g. a combination of strength and toughness, strength and solvent resistance, etc. Blending also benefits the manufacturer by offering improved processability, product uniformity, quick formulation changes, plant flexibility and high productivity.

Polymer blends have received much attention since blending is a simple, effective approach to develop new materials exhibiting combinations of properties that cannot be obtained by individual polymers. Miscibility between two components is governed by the thermodynamics represented by the Gibbs free energy of mixing ($\Delta G_{mix} = \Delta H_{mix}$ - $T\Delta S_{mix}$). The mixtures are miscible when the value of ΔG_{mix} is negative, i.e., low value of ΔH_{mix} and high value of ΔS_{mix}. Immiscibility is a rule in polymer blends because both the ΔH_{mix} and the ΔS_{mix} are unfavorable. The ΔS_{mix} is unfavorable because there are few molecules of large molecular weight per unit volume. The ΔH_{mix} is also unfavorable because the van der Waals dispersion force is always positive. However, when the specific interactions such as hydrogen bonding, dipole-dipole interaction, or ionic interaction are established, the miscibility between two polymer mixtures can be achieved [1].

If two polymers are mixed, the most frequent result is a system that exhibits a complete phase separation due to the repulsive interaction between the components (i.e. the chemical incompatibility between the polymers).

Complete miscibility in a mixture of two polymers requires that the following condition is fulfilled:

$$\Delta G_m = \Delta H_m - T\Delta S_m < 0 \ (1)$$

where ΔG_m, ΔH_m, and ΔS_m are the Gibb's free energy, the enthalpy and entropy of mixing at temperature T, respectively.

The most important characteristic of a polymer blend of two (or more) polymers is the phase behavior. Polymer blends (like low molecular weight solvents) can exhibit miscibility or phase separation and various levels of mixing in between the extremes (e.g., partial miscibility).

For a stable one-phase system, criteria for phase stability of binary mixtures of composition ϕ at fixed temperature T and pressure p are:

$$\Delta G_m < 0, \left(\frac{\partial^2 \Delta G_m}{\partial \phi^2}\right)_{p,T} > 0 \ (2)$$

Miscible polymer blend is a polymer blend which is homogeneous down to the molecular level and associated with the negative value of the free energy of mixing and the domain size is comparable to the dimensions of the macromolecular statistical segment. The value of $T\Delta S_m$ is always positive since there is an increase in the entropy on mixing. Therefore, the sign of ΔG_m always depends on the value of the enthalpy of mixing ΔH_m. The polymer pairs mix to form a single phase only if the entropic contribution to free energy exceeds the enthalpic contribution, i.e.,
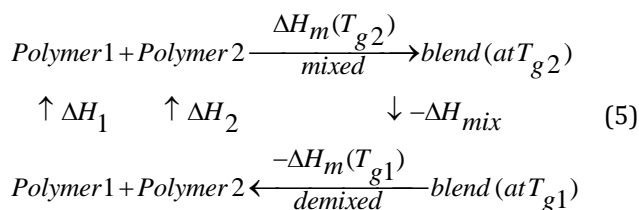
$$\Delta H_m < T\Delta S_m \quad (3)$$

For most polymer blends the miscibility increases with increasing the pressure. The effect depends on the magnitude of the heat of mixing ΔH_m. For $\Delta H_m < 0$ the miscibility is enhanced by compression, whereas for those with $\Delta H_m > 0$ it is reduced.

For low molecular weight materials, increasing temperature generally leads to increasing miscibility as the $T\Delta S_m$ term increases, thus driving ΔG_m to more negative values. For higher molecular weight components, the $T\Delta S_m$ term is small and other factors (such as non-combinatorial entropy contributions and temperature dependant ΔH_m values) can dominate and lead to the reverse behavior, namely, decreasing miscibility with increasing temperature [2].

Consider two polymers having glass transition temperatures T_{g1} and T_{g2} and respective molar enthalpies H_1 and H_2. The molar enthalpy of a mixture of the two polymers is:

$$H_{mix} = x_1 H_1 + x_2 H_2 + \Delta H_m \quad (4)$$

where x_i represents the mole fraction of polymer i in the blend and ΔH_m is the excess enthalpy of mixing.

$$Polymer1 + Polymer2 \xrightarrow[\text{mixed}]{\Delta H_m(T_{g2})} blend\,(at\,T_{g2})$$

$$\uparrow \Delta H_1 \qquad \uparrow \Delta H_2 \qquad\qquad \downarrow -\Delta H_{mix} \qquad (5)$$

$$Polymer1 + Polymer2 \xleftarrow[\text{demixed}]{-\Delta H_m(T_{g1})} blend\,(at\,T_{g1})$$

The thermodynamic cycle for the mixing process is represented by Equation 5, where ΔH_1, ΔH_2, and ΔH_{mix} are the changes of enthalpies when the temperature is increased from T_{g1} to T_{g2} at constant pressure for polymer 1, polymer 2, and their blend, respectively. $\Delta H_m(T_{g1})$ and $\Delta H_m(T_{g2})$ are the excess enthalpies of mixing at T_{g1} and T_{g2}. Solving Equation 5 for ΔH_{mix} yields

$$\Delta H_{mix} = x_1 \Delta H_1 + x_2 \Delta H_2 + \Delta H_m(T_{g2}) - \Delta H_m(T_{g1}) \quad (6)$$

The enthalpy changes corresponding to heating the individual components and the mixture from T_{g1} to T_{g2} are [3]:

$$\Delta H_1 = \int_{T_{g1}}^{T_{g2}} c_{p1}^1 \, dT$$

$$\Delta H_2 = \int_{T_{g1}}^{T_{g2}} c_{p2}^g \, dT \qquad (7)$$

Knowledge of the glass transition temperatures (T_gs) as function of composition reflects miscibility (or lack of it) and is decisive for virtually all properties of polymer-based materials. Immiscible polymers (not an infrequent case) clearly demonstrate two T_g values for the respective pure components that are independent of composition.

The glass transition temperature (T_g) reflects the molecular rearrangement rate in supercooled liquid and thus is one of the most important factors among the many transitions and relaxations in polymers. Measurement of T_g is one of the easiest determinations of whether they are miscible or immiscible in polymer mixtures. A single T_g between individual two polymers appears for miscible blends whereas two T_gs are shown for immiscible blends. When T_g of miscible polymer blends shows the linear relationship against the polymer composition, the value of T_g is adequately expressed by the widely used Fox equation.

$$\frac{1}{T_g} = \frac{W_1}{T_{g1}} + \frac{W_2}{T_{g2}} \qquad (8)$$

where w_1 and w_2 are the weight fractions of components 1 and 2 with T_{g1} and T_{g2}, respectively. This equation is also used to predict the T_g of copolymers.

The change in the glass transition temperature produced by blending polymer 1 and the polymer 2 can be described by equation 9 which provide a configurational entropy theory for the glass transitions of binary polymer blends. From this model, we can predict T_g for the polymer blends with various compositions.

$$In\left(\frac{T_{g12}}{T_{g1}}\right) = \beta(1 - \gamma_{spe} In\left(\frac{z-1}{e}\right))\left(\frac{\phi_1}{r_1} In\varphi_1 + \frac{\phi_2}{r_2} In\varphi_2\right) + \phi_2 In\left(\frac{T_{g2}}{T_{g1}}\right) \quad (9)$$

where T_{g1} and T_{g12} are the T_gs of the pure polymer 1 and of the blend of polymer 1 and polymer 2, respectively. ϕ_1 and ϕ_2 are the volume fractions of components 1 and 2, respectively. z is the lattice coordination number. γ_{spe} is a proportionality constant representing the specific interaction such as hydrogen bonding, dipole-dipole interaction, or ionic interaction between two polymers,

and $\beta = zR / (M_{1u}\Delta C_{pp})$. R, M_{1u}, and ΔC_{pp} are the gas constant, the molecular weight of the repeat unit, and the isobaric specific heat of polymer 1, respectively [4].

A schematic phase diagram is shown in Figure 1. There are three regions of different degree of miscibility: 1. The single-phase miscible region between the two binodals, 2. The four fragmented metastable regions between binodals and spinodals, and 3. The two-phase separated regions of immiscibility, bordered by the spinodals. The diagram also shows two critical solution temperatures, the lower, LCST (at higher temperature), and the upper, UCST (at lower temperature). The phase diagram with two critical points is a rule for mixtures of low molar mass components, whereas the polymer blends usually show either LCST (most) or UCST [5].
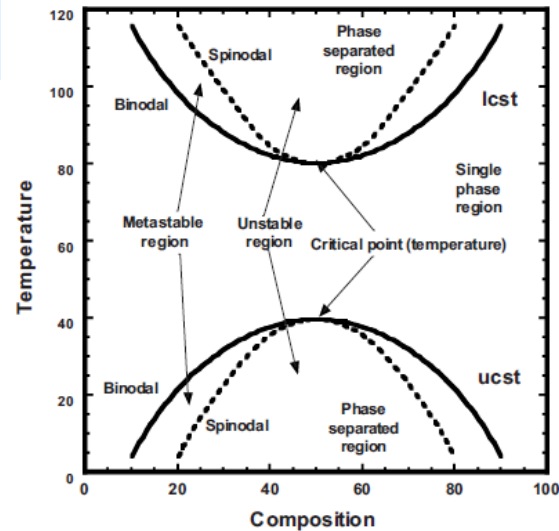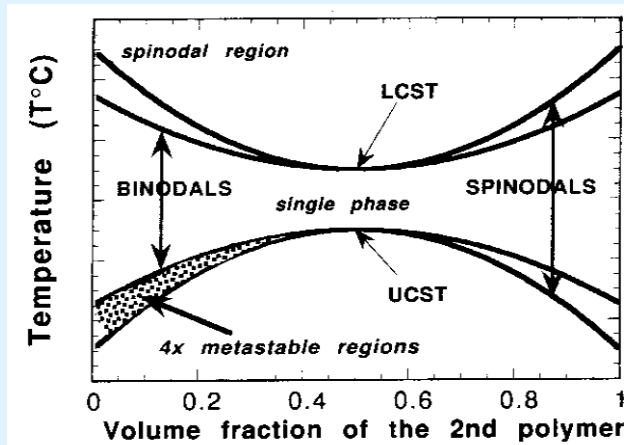

Figure 1: Phase diagram for liquid mixtures with the upper and the lower critical solution temperature, UCST and LCST, respectively.

The binodals (Figure 1) separate miscible (one-phase) and metastable region, the spinodals separate metastable and two-phase region. The thermodynamic conditions for phase separations are given by:

Binodal:
$$\left[\frac{\partial \Delta G_m}{\partial \phi}\right]_{p,T} = 0 \qquad (10)$$

Spinodal:
$$\left[\frac{\partial^2 \Delta G_m}{\partial \phi^2}\right]_{p,T} = 0 \qquad (11)$$

Critical point:
$$\left[\frac{\partial^3 \Delta G_m}{\partial \phi^3}\right]_{p,T} = 0 \qquad (12)$$

The experimental phase diagrams are often not symmetrical, unless the molecular weights of the components are similar, and in the case of large differences in molecular weights, they can be highly non-symmetric. With phase separation, the binodal defines the composition of the component 1 rich phase and component 2 rich phase (Figure 2). The tie line noting temperature T_2 between the binodal points can be employed to determine the relative amounts of each phase. The tie line is illustrated in Figure 3. The volume fraction of component 1 rich phase, ϕ_{1r}, and component 2 rich phase, ϕ_{2r}, can be determined from the expression, with ϕ representing the overall composition of the component noted in Figure 3:

$$\frac{\phi_{1r}}{\phi_{2r}} = \frac{\phi_b - \phi}{\phi - \phi_a} \qquad (13)$$
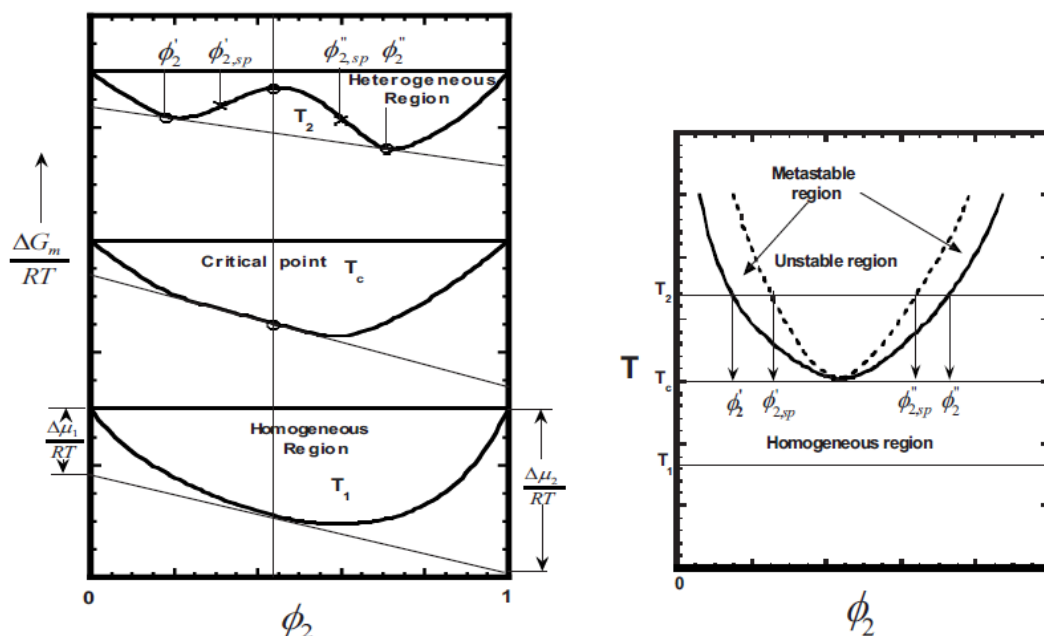
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Figure 2: Free energy of mixing versus volume fraction generalized behavior for various positions on the phase diagram.
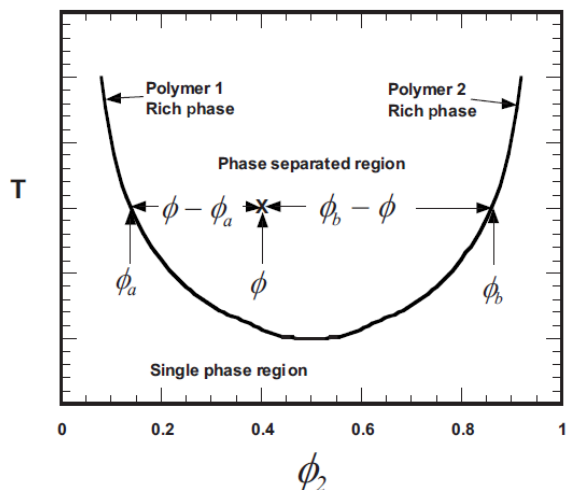

Figure 3: Tie-line calculation of phase compositions.

Highly miscible polymers exhibit single phase behavior over the entire temperature-volume fraction space available for experimental verification. If *ucst* or *lcst* behavior exists, it cannot be determined. At low temperatures, the *ucst* cannot be determined due to the glassy state restricting molecular motion (phase separation); and at higher temperatures, polymer degradation occurs before phase separation can be observed. With highly immiscible polymer blends, the phase diagram is virtually all in the two phase region with the binodal curves virtually overlapping the y axis at 0 and 1.0 volume fraction.

The phase separation takes place when a single-phase system suffers a change of either composition, temperature or pressure that forces it to enter either the metastable or the spinodal region. When the system enters from single-phase region into the metastable region, the phase separation occurs by the mechanism resembling crystallization – slow nucleation followed by growth of the phase separated domains. By contrast, when the system is forced to jump from a single-phase into the spinodal region of immiscibility the phases separate spontaneously by a mechanism called spinodal decomposition [6].

Starting point for most of the theoretical interpretations of polymer solutions and blends is the Flory-Huggins lattice theory. It is basically an extension of the concept of regular solutions on polymer solutions. Thus the model restrictions are no change of volume

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during mixing (incompressible model), the entropy of mixing is entirely given by the number of rearrangements during mixing (combinatorial entropy) and the enthalpy of mixing is caused by interactions of different segments after the dissolution of interactions of the same type of segments. It is a mean-field model, i.e. only average interactions are taken into consideration. The main problem was to find an expression for the entropy of mixing because it was found experimentally that polymer solutions show significant deviations from values expected for ideal solutions. Assuming a rigid cubic lattice model, this problem was independently solved for polymer solutions by Huggins and Flory [3].

The lattice theory for the enthalpy of mixing in polymer solutions, developed by Flory and Huggins, can be formally applied to polymer mixtures, which provides a rough estimation of the miscibility of the polymers. Assuming random mixing of two polymers and $\Delta V_m = 0$ yields the well-known expression for the combinatorial entropy of mixing ΔS_m of the Flory-Huggins theory:

$$\Delta S_m = -R\left[\frac{\phi_1}{r_1}In\phi_1 + \frac{\phi_2}{r_2}In\phi_2\right] \tag{14}$$

where ϕ_i is the volume fraction of the component i and r_i is the number of polymer segments, R is the gas constant. It can be seen that the entropy of mixing decreases with increasing molar mass (r_i is proportional to the degree of polymerization) and vanishes for infinite molar masses. Applying the concept of regular solutions and assuming all pair interactions in the framework of a mean-field theory yields for the enthalpy of mixing ΔH_m :

$$\Delta H_m = RT\chi\phi_1\phi_2 \tag{15}$$

For binary systems the Flory-Huggins equation can be expressed in the following form:

$$\Delta G_m = RT\left[\frac{\phi_1}{r_1}In\phi_1 + \frac{\phi_2}{r_2}In\phi_2 + \chi\phi_1\phi_2\right] \tag{16}$$

where χ is the so called Flory-Huggins binary interaction parameter. R is the universal gas constant, and T is the absolute temperature. The first two terms of the right hand side in Equation 16 are related to the entropy of mixing and the third term is originally assigned to the enthalpy of mixing.

For polymers having infinite molar mass (i.e. r_i is infinite) the entropic contribution is very small and the miscibility or immiscibility of the system mainly depends on the value of the enthalpy of mixing (Equation 15). Miscibility can only be achieved when χ is negative.

The term 'parameter' is widely used to describe χ but it is definitively better characterized by the term 'function', because χ depends on such quantities as temperature, concentration, pressure, molar mass, molar mass distribution and even on model parameters as the coordination number of the lattice and segment length.

For polymers, the miscibility can only be achieved when $\chi < \chi_{cr}$. The χ parameter at the critical point χ_{cr} can be obtained from the definition of the critical point (Figure 1) and Equation 16 as follows:

$$\chi_{cr} = \frac{1}{2}\left[\frac{1}{\sqrt{r_1}} + \frac{1}{\sqrt{r_2}}\right]^2 \tag{17}$$

where r_i is the number of polymer segments (which is proportional to the degree of polymerization).
It should be mentioned that the Equations 16 and 17 are based on the assumption that χ is not a function of composition, χ_{cr} is only a function of the molar masses [7].

Statement of the problem

The factors affecting the miscibility of polymer blends will be examined experimentally and predicted thermodynamically. The miscibility and dynamics of phase separation will be investigated experimentally for different polymer blends concentrations to determine both the binodal and the spinodal curves. The relationship between the binodal curve and the phase transition, such as melting and glass transitions, will be studied to discuss the compatibility of blend system including solid component. The glass transition temperature will be experimentally determined and predicted thermodynamically to establish polymer miscibility.

Purpose of the Study

The miscibility of the blends through glass transition temperature T_g analysis by DSC will be evaluated. T_g will be correlated with a measure of the intermolecular interactions, such as the Flory-Huggins interaction parameter, χ.

The effect of blend ratios on the mechanical properties and the probability to creating compatible system for the polymer blend will be studied.

Methods of polymer blends preparation, and the best methods for detecting miscibility will be studied.

The thermodynamic relationships will be applied to determine the free energy of mixing, heat of mixing, the binodal and spinodal curves, the critical points and thus the phase behavior.

Phase behavior and phase separation kinetics of polymer solutions will be investigated experimentally.

The miscibility and dynamics of phase separation will be investigated in solutions of different polymer concentrations to determine both the binodal and the spinodal

Review of the Literature

The kinetics of phase separation in "polymer + solvent" systems is of continuing interest. The phase separation in polymer solutions can be induced by changes in the temperature, the solvent (concentration or composition) or the pressure. Binder, et al. [8-9] studied the kinetics of spinodal decomposition of polystyrene solutions in cyclohexane induced by a temperature jump into the unstable region. Phase separation process was monitored by time- and angle-resolved light scattering at ambient pressures. Berghmans, et al. [10] recently carried out a numerical study of temperature-induced phase separation kinetics of polymer solutions subjected to a linear spatial temperature gradient. Their results showed that anisotropic structures and morphologies can be induced by a gradient temperature jump. In addition to temperature quench, phase separation can also be induced by exposing the solution to a nonsolvent vapor as antisolvent. Binder, et al. [11] reported the kinetics of phase separation in a polymer solution (polysulfone in N-methyl-2-pyrrolidinone) film induced by a nonsolvent vapor, water. The data were analyzed based on Cahn-Hilliard linear theory in the initial stages of phase separation.

Much attention has been paid to the phase behavior of polymers in compressed fluids at high pressures [12-14]. However, these have mostly focused on the liquid-liquid (L-L) phase boundary, with limited data on the solid-fluid (S-F) boundary. For crystallizable polymer-solvents systems, it is possible to observe the S-F boundary as well as L-L phase boundary in a phase diagram.

Question and/or Hypotheses

1. It is reasonable to believe that the changes of thermal enthalpy are caused by the occurrence of phase separation during annealing. Therefore, the phenomenon is indicative of the occurrence of phase separation.
2. The changes of thermal properties of blends composed of polymer can provide information about the occurrence of phase separation, and thermal analysis can be used to determine the phase boundary of such blends.
3. The specific interactions such as hydrogen bonding, dipole-dipole interaction, or ionic interaction between two polymers reduce the overall entropy

Method and Procedures

The binary blends will be made by mixing solutions (approximately 3% w/w) of each polymer in 98% formic acid. The solutions will be stirred for 2h at ambient temperature. The films for DSC and FTIR measurements will be prepared by casting the polymer solutions into shallow soda-glass dishes and allowing the solvent to evaporate slowly. The thin films will be dried at 60 °C and placed under vacuum at 70-80 °C for at least 3 days to constant weight. Blend compositions will be 80/20, 60/40, 50/50, 40/60 and 20/80 by weight.

Sampling

Differential Scanning Calorimetry (DSC): DSC measurements will be performed on a DuPont 910 DSC Differential Scanning Calorimeter equipped with a Thermal Analysis Data System (TA-2000). The samples will be first heated from room temperature to 250 °C and maintained for 2 minutes, and then the samples will be quenched cooled to 0 °C to prevent crystallization before thermograms will be taken. The scanning rate was 10 °C /min. In the isothermal crystallization experiments blends samples of 9-11 mg will be used. Starting at room temperature, the samples will be rapidly heated to melting temperature to remove any previous crystallinity. The samples will be held for 6h at a certain crystallization temperature (T_C = 50, 100 and 150 °C), and then will be heated to 250 °C at a rate of 10 °C /min under dry nitrogen atmosphere.

Fourier Transform Infrared Spectroscopy (FTIR): The FTIR spectra will be collected on dry thin film polymer blend samples using a UNICAM (Mattson 5000)

spectrophotometer by sandwiching the film samples between two KBr disks. All of the films will be sufficiently thin to be within a range where the Beer-Lambert law is obeyed. Films will be annealed at 50, 100, and 150 $^{\circ}$C for 6h, then 32 scans will be made for each T_C at a resolution of 8 cm^{-1} .

Instrumentation

The most common method to establish polymer miscibility is Differential Scanning Calorimetry (DSC), with which determination of the glass transition temperature (T_g)

IR spectroscopy has proved to be an excellent tool to study the hydrogen-bonding behavior in polymer blends. If the blend is immiscible, the absorption spectrum of the blend will be the sum of those for the components. If the blend is miscible because of the specific interactions, then differences will be noted in the spectrum of the blend relative to the sum of those for the components. The FTIR investigation of a miscible blend will not only reveal the presence of such an interaction, but will provide information on which groups are involved

Data collection and Analysis

Several approaches based on kinetic or thermodynamic features of the glass transition phenomenon will be proposed to provide a theoretic foundation for the equations currently used to predict the compositional dependence of the T_g of blends of miscible polymers. Information on the experimental details and any special procedures used for preparation of the mixtures (temperature of mixing, type of solvent, drying processes, etc) can be found in the original references [1-7]. The experimental data analyzed will be obtained using conventional DSC and FTIR.

Limitation and Delimitation

The most important factor leading to miscibility in low molecular weight materials is the combinatorial entropy contribution which is very large compared to high molecular weight polymers. This contribution is the reason that solvent-solvent mixtures offer a much broader range of miscibility than polymer-solvent combinations. The range of miscible combinations involving polymer-polymer mixtures is even much smaller.

The Flory-Huggins approach is not directly capable of predicting *lcst* behavior unless a temperature dependent

χ_{12} value exhibiting increasing values (negative to positive) with increasing temperature is employed.

The Flory-Huggins lattice model theory is an incompressible model that does not allow for the compressibility effects on the system thermodynamics.

For high molecular weight polymers, ΔS_m is negligibly small and the sign of ΔG_m, is dominated by ΔH_m. In general, ΔH_m, is negative only if there are specific associative interactions between the two polymers. Therefore, the formation of miscible polymer blends depends on the occurrence of exothermic interactions such as hydrogen bonding, dipole-dipole interactions, acid-base interactions, or transition metal complexation.

The thermodynamic functions, entropy, enthalpy, and volume, are continuous at T_g, but the first derivatives of these functions undergo discontinuities at T_g. In principle, any of these thermodynamic functions could be used to derive the relation for the compositional variation of T_g, but, in practice, the dependence of these variables on composition is not usually known. As a result, approximations of the thermodynamic functions are usually used.

The value of χ_{12} for any polymer blend is usually small and representative of the overall interaction of two different polymer molecules

Many polymer pairs are not only immiscible but also incompatible, Compatibility arises from thermodynamic interaction between the blend constituents, which is a function of their physical and chemical structure

Significance of the Study

Blending of friendly environmental polymers with synthetic polymers has proven to be a suitable tool to produce novel materials with combined characteristics in having both improved application properties and low cost advantages in material performance.

Polymer blend represents very important field in processing of new materials, which has better properties in comparison with the net polymers. They are significant also from ecological and economical viewpoint.

The relationship between the binodal curve and the phase transitions, such as melting and glass transitions, become important to discuss the compatibility of binary blend system including solid component.

Considerable interest in the study of polymer blends because of their importance in academic and technical aspects. Particularly, much attention has been paid to miscibility and phase behavior in polymer blends

Glass transition temperature T_g values are useful indeed for a variety of purposes. Particularly needed are T_g values as a function of composition for binary polymer blends; they tell us whether the blends are miscible, semimiscible (called compatible) or not miscible at all.

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