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1986

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Classifications of Experimental Methods on Polymer Compatibility

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Recent experimental studies have revealed us an establishment of molecular order miscibility 1)- 4) on polymer blends. In addition, there are several applicable accumulations 6)- 8) concerning to compatibility of polymer blends.

The properties of polymer blends may depend largely on their dispersion structures such as whether they have homogeneous or micro-heterogeneous phase structures. It is important to make sure of these phase structures for the polymer alloys including paint and adhesive materials. In this reports, we classify the experimental methods for determining polymer compatibility, and describe properties measured by the methods and their characteristics to be noted.

CLASSIFICATIONS OF EXPERIMENTAL METHODS ON COMPATIBILITY

The classifications of experimental methods are shown in Tables 1,2 and 3, which are interpreted as follows.

(A) Methods based on Morphology: The methods are based on direct observation of dispersed polymer particles of average sizes from visible order to segmental order. As to the segmental order observation, SAXS and SANS have revealed a molecular miscible polymer blends 1) at ordinary temperature.

(B) Methods based on Physical Properties: The glass transitions of blends are most common and effective to show the segmental miscibility with listed methods.

(C) Methods based on Thermodynamics: It is important to determine whether blends are miscible from thermodynamic point of view. Listed methods can identify the miscibility, for example, from χ parameters of the blends.

As found in Tables 1,2 and 3, each method has its own character in the order to be measured and has its own limiting. It will be convenient to distinguish three regions corresponding to the average size of particles at room temperature. The first is about 400 nm, visible wave length order, beyond which the phase separation can be easily observed by optical microscopy. The second is about 15 nm, below which a single Tg is observed in compatible blends. The third is about 5 nm, below which the segment order polymer miscibility plays a major role.

Table 1 (A) Methods based on Morphology

Methods	Properties measured	Notes
Transparency	turbidity	particle diameters must be greater than 0.4 μm difficult to determine if the components have the same refractive index
Refractive Index	refractive indices	
Phase Contrast Microscopy	refractive index differences phase homogeneity	
Optical Microscopy	translucence morphology of dispersion scattered light intensity	
Light Scattering	chord length radius of gyration(R_g) correlation function parameter of heterogeneity	statistical description of structures including laser scattering
Electron Microscopy	electron scattering differences in the density	μg sample be used changeable in structure by beams 10 nm detectable
Small-angle X ray Scattering	electron density differences chord length, and R_g	segment order miscibility
Small-angle Neutron Scattering	R_g of isolated chain correlation function	deuterium sample must be prepared segment order
X ray Microanalyzer (XMA)	interface thickness distribution situation	detectable elements be prepared
E S C A	interface thickness intensity of electron kinetic energy	distribution can be detectable
S I M S (Secondary Ion Mass Spectroscopy)	interface thickness	direction of distribution
Fluorescence	fluorescence intensity intermolecular energy transfer	fluorescent probe be prepared segment order miscibility
Fourier Transform Infrared Spectroscopy (FT-IR)	spectral shift intermolecular interaction	including dichroism of IR spectrum

Table 2 (B) Methods based on Physical Properties

Methods	Properties measured	Notes
Additivity of Specific Volume	densities specific volumes	volume change in blend
Refractometry	glass transition temperature (Tg)	single Tg indicate less than 15nm of particle size difficult at low temp.
Dilatometry	Tg, thermal expansion coefficients	difficult at low temp.
Calorimetry (DTA, DSC)	Tg, heat capacities	mg sample required
Thermophotometry (TOA, TP ⁹)	Tg, transmitted light intensity	transparent film is necessary included birefringence
Mechanical Relaxation	α , β — dispersion E' , E'' (or $\tan \delta$)	10 deg. difference between Tg of polymers is necessary single dispersion indicate less than 15 nm in particle size
Dielectric Relaxation	dielectric α , β — dispersion ϵ' , ϵ'' ; contour maps ¹⁰ normalized curves	10 to 20 deg. difference between Tg of polymers necessary limited to polar polymers
Pulse NMR	T ₁ (spin-lattice relaxation time) T ₂ (spin-spin relaxation time)	local segmental motion of blend component indicate less than 5nm segment size order
Thermo Luminescence	Tg, luminescent intensity	small amount of luminescent probes segment order motion

Table 3 (C) Methods based on Thermodynamics

Methods	Properties measured	Notes
Heat of Mixing	heat of solution of blends & components	quantitative way difficulty in precise temp. controlling
Precipitation from Solution	precipitating concentration	solubility difference in components is necessary
Osmotic Pressure	interaction parameter (χ)	difficulty in accuracy of getting χ
Viscosity (Feldman way)	viscosities of blend and homopolymers	simple prediction experiential method
Polymer Solubility Parameters	solubility parameters of each polymers (δ_p)	many kinds of methods for determinating δ_p comparisons sometimes uncertain due to the interactions

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