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Compatibility Studies on Dehydrated Castor Oil Epoxy Blend with Poly(Methacrylic Acid)

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Blending is a useful technique to improve upon the physico-mechanical properties of the polymers. Synergies of the properties of the two polymers occur best when they are miscible or compatible with each other. Vegetable oil epoxy can be used for blending with polymers to improve upon their physical and mechanical properties. Poly(methacrylic acid) (PMAA) is a hard, brittle and water sensitive material. Dehydrated castor oil epoxy (DCOE), a product from a sustainable resource, has been chosen to improve upon the physical and mechanical properties of PMAA through solution blending. Blends of DCOE/PMAA were prepared in the weight ratios 80/20, 60/40 and 20/80 through a solution method by mixing in dimethyl sulphoxide. In the first instance, the miscibility of the two components was investigated using the techniques of viscosity and ultrasonic measurements. The study revealed that the two components showed semicompatibility/semimiscibility in solution. The compatibility in the solid phase was examined by differential scanning calorimetry and scanning electron microscopy which revealed that DCOE–PMAA pair were incompatible in solid phase.

Keywords dehydrated castor oil epoxy, polymethacrylic acid, blend

Introduction

Among the techniques available for improving upon the physical and mechanical properties of polymers, blending of a polymer with another polymer is the simplest and cost effective (1, 2). Blends of polymers are characterized as miscible/compatible partially miscible/semi-compatible, and immiscible/incompatible depending upon the degree of miscibility. The development of final properties of the blend depends upon the degree of miscibility/compatibility of the polymer pairs (3). Although even immiscible/incompatible blends have found commercial applications, miscible polymer pairs are the most desirable because of mixing at molecular scale and the maximum synergy of the properties of individual polymers (2–3). Blending of commercial polymers has therefore, been vigorously investigated for the past three decades (4–6).

Blending a polymer of high molar mass with another polymer of high molar mass is done by mixing them through different methods. Improvement in toughness, flexibility and tensile strength can also be achieved by blending a hard polymer with smaller

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molecule like plasticizers or additives (7). Petroleum based plasticizers like dioctyphthalate, dibutyl sebacate, tricresyl phosphate have been used for improving the properties of polyvinyl chloride (3) and other commercial polymers. Low molar mass materials from vegetable resource have also been investigated for improving the properties of commercial polymers (8). Lactose (9), starch acetate (3), starch cinnamate (10), have been used to modify the properties of polyvinyl chloride, poly(methylmethacrylate) and other polymers. These plasticizers and additives of low molar mass are also required to be miscible for being effective in modifying the properties of the commercial polymers and also for obviating the migration of the former.

World wide effort is in progress to develop polymers from a renewable resource to substitute those from petroleum in view of the exhaustion of petroleum stock in the near future (11). Vegetable oils, being a major resource of several polymers, viz, alkyd, polyesters, polyurethane epoxies and others (12, 13) with its vast production across the globe constitutes a significant renewable resource to the above end. In addition to their other applications, vegetable oil based products particularly epoxies, fatty acid esters can be used as plasticizer and additives to commercial polymers to improve upon their toughness and tensile strength (8). Soybean oil epoxy has notably been used in the processing of the polyvinyl chloride (14).

Polymethacrylic acid (PMAA) is a transparent, hard and brittle polymer of high molar mass, but it is too water sensitive to serve as plastic (15). An attempt has been made to improve upon its toughness and reduce its water sensitivity through complex formation (16) and copolymerization (17, 18). Interpolymer complex formation between PMAA and polyethylene glycol has been extensively investigated (19, 20). Likewise, complex formation with poly(ethyleneoxide) at different pH values has also been studied (21). The interpolymer complexes of PMAA have been mainly used for obtaining membranes for ultrafiltration (22), reverse osmosis (23) battery separators and other applications. Studies on blending of PMAA with other polymers to improve upon its undesirable characteristics are far less. Blending of PMAA with poly(2-hydroxy ethyl methacrylate) (PHEMA) has been studied (24). The system yields a completely miscible pair in solid phase on a scale of 5–10 nm for all compositions.

In order to improve upon the physico-mechanical properties of PMAA, we have thought it worthwhile to use an additive from a vegetable oil resource for blending with it. Vegetable oils are known to possess varying amounts of fatty acids as triglycerides with varying unsaturation in their chains. A vegetable oil based epoxy as an additive would, therefore, show different behavior in their interaction with commercial polymers of different chemical nature especially with regard to their compatibility with these polymers, as well as with regard to the physical and mechanical characteristics produced in their blends.

Keeping these facts in mind, we have chosen the epoxy synthesized from the commercial available dehydrated castor oil (DCO) of medium unsaturation for blending with PMAA in different ratios. In the first instance, we have attempted to investigate the miscibility/compatibility of the two components, both in solution and solid phase, using the techniques of viscosity, ultrasonic velocity measurements, differential scanning calorimetry (DSC) and scanning electron microscopy (SEM).

Experimental

Materials

The linseed oil was procured from M/s Atul Chemicals Pvt. Ltd. (Delhi). The oil was dewaxed by keeping it in a refrigerator at 15°C and filtering before use. PMAA was synthesized from methacrylic acid monomer [Aldrich] in the laboratory. The polymer was purified by a solvent-nonsolvent method. Molecular weight of the polymer was determined by viscosity measurement. Molecular weight of PMAA was found to be 2.3×10^5 Dehydrated castor oil epoxy (DCOE) was prepared after a reported method (25), which yielded epoxidixed oil of epoxy equivalent weight of 260.

Blending

Blends of DCOE with PMAA were prepared by mixing the DCOE and polymer in weight ratios DCOE : PMAA, 80/20, 60/40, 40/60, 20/80 by taking the requisite amounts of the two components to obtain 100 ml of 2 wt% and 4 wt% solutions of the blend in dimethyl sulphoxide [DMSO], (Merck, A.R.). All solutions were thoroughly mixed by agitation on a magnetic stirrer for 2 h. Selected samples were kept for over a week to observe the appearance of turbidity or precipitation, which was not noticed in any case.

Film Preparation

6 wt% solutions of selected composition of the blend in DMSO were cast on transparency sheet and were allowed to dry under ambient conditions. In 10 days, free-standing films were obtained. The films were further dried in vacuum oven kept at 60°C for 24 h.

Investigations of the Blends

DCOE-PMAA blends were studied experimentally by viscosity measurements, ultrasonic velocity measurements, differential scanning calorimeter (DSC), and scanning electron microscopy (SEM). DSC thermograms were recorded on a differential scanning calorimeter Dupont 910, TA Instruments, USA in N_2 atmosphere at a heating rate of 10°C min $^{-1}$. Viscosity of the blend solutions was measured at temperatures 20, 30, and 40°C (accuracy $= \pm 0.05$°C) in a thermostatic bath using an Ubbelhode suspended level viscometer. The ultrasonic velocity of the blend solutions were measured by an ultrasonic interferometer Model MX-20 (Mittal Enterprises, New Delhi). The temperature in the sample cell was maintained at 20°C, 30°C, and 40°C by circulating water from a thermostatic bath through the outer jacket of the sample cell with a thermal stability of ±0.1°C. The densities of the solutions were measured at the above temperatures by a pycnometer. The morphology of a cross-section of the cryogenically fractured blend films were examined by scanning electron microscope (SEM, JEOL, JSM-840 scanning electron microscope) at different magnifications. SEM micrographs were taken after coating the fractured surface with a thin layer (10–20 nm) of gold.

Results and Discussion

Viscosity Measurements

The variations of relative viscosity with the change in blend composition at temperature 20°C, 30°C, and 40°C have been plotted in Figure 1. It shows a progressive increase in the relative viscosity with the increasing proportion of PMMA in the blends. Two linear portions in all the curves were observed with inflection at 60% PMMA in the blend. It has been observed by Y. N. Sharma et al. (26, 27) and Mamza. et al. (2) that

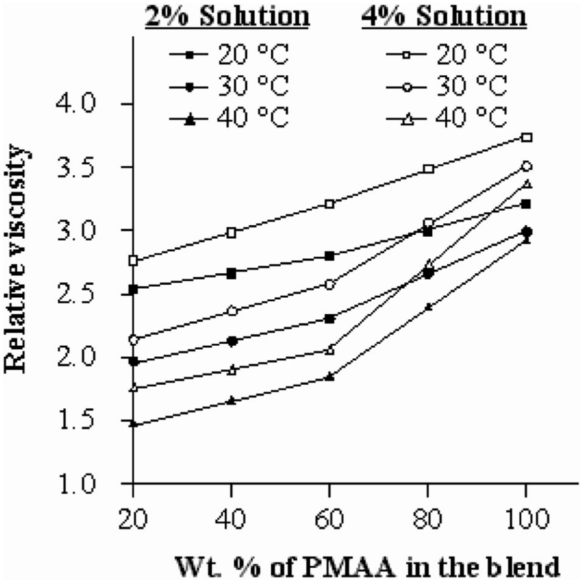

Figure 1. Effect of varying composition of DCOE : PMAA blend on the relative viscosity of 2% and 4% solution.

the plot of the relative viscosity of the blend solutions against their composition is linear at all concentrations and temperature when the components are compatible/miscible with each other, while if these plots are not linear at any of the concentrations and temperatures the components are incompatible or semicompatible depending upon the extent of nonlinearity. The curves in Figure 1 are neither S-shaped or distorted S-shaped indicating any immiscibility. They have distinctly two linear parts. We attribute this behavior to the semi-compatibility of the two components. Since the increase in relative viscosity is similar at the 20°C, 30°C, and 40°C, it can be inferred that association of the components is stable even at relatively higher temperatures. It has also been observed that the blend of all compositions in 2 wt% and 4 wt% solution were transparent and on keeping even for several months no turbidity or phase separation appeared. This shows that some phase separation that occurs in the solution because of semi-compatibility does so at sub-micrometer scale.

We have attempted to compare the observed reduced viscosities of the blend solutions of different composition with calculated reduced viscosities of the same. The calculated reduced viscosities was calculated by rule of mixture using the following:

$$(\eta_{\mathrm{spc/C}})_{\mathrm{AB}} = \mathrm{W_A}(\eta_{\mathrm{spc/C}})_{\mathrm{A}} + \mathrm{W_B}(\eta_{\mathrm{spc/C}})_{\mathrm{B}}$$

where $\mathrm{W_A}$ and $\mathrm{W_B}$ are the weight fractions of the components A and B in the blend of a given composition, $(\eta_{\mathrm{spc/C}})_{\mathrm{A}}$ and $(\eta_{\mathrm{spc/C}})_{\mathrm{B}}$ are the reduced viscosities of pure components A and B at calculated concentrations C and $(\eta_{\mathrm{spc/C}})_{\mathrm{AB}}$ are the calculated reduced viscosity of the blend AB of the same concentration.

On comparing the observed and calculated reduced viscosities of the blend solutions of different compositions at 2 wt% and 4 wt% concentrations (Figure 2a, a′, b, and b′), respectively. We observe that the calculated reduced viscosities are higher than the observed reduced viscosities for all compositions of the blend system. However, the

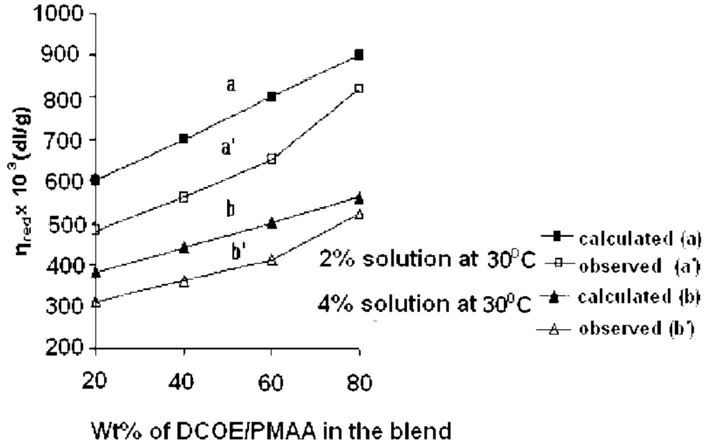

Figure 2. Variation of reduced viscosity with composition of the blend.

variation of observed reduced viscosities with composition is found to be nonlinear. Paladhi and Singh (28) have observed that the reduced viscosity of blend compositions follows the simple additive rule of mixture when the polymers are miscible. The lower values of observed reduced viscosity than the calculated ones and their slightly nonlinear nature can be attributed to semi-compatibility of the components. Singh et al. (29) have observed nonlinear variation of the observed values of the reduced viscosity in the case of poly(ethylene oxide) and poly(vinylalcohol) in water and also in the case of poly(acrylic acid) and poly(vinyl pyrrolidone/polyvinyl alcohol) and concluded for the immiscibility of these pairs. The lower values of the observed reduced viscosities than the calculated values for the same composition show interaction between DCOE and PMAA and consequently tight binding of the two components and enhanced compactness of the blend structure. The blend of composition 20 wt% PMMA and 80 wt% DCOE shows the lowest reduced viscosity or the most tightly bound structure while the blend of composition 80 wt% PMAA and 20 wt% DCOE shows the highest value of the reduced viscosities that indicates an expanded structure of the blend in solution.

Ultrasonic Velocity Measurements

We notice that the ultrasonic velocity changes linearly in two portions each with different slopes as the proportion of PMAA in the blend increases (Figure 3). An inflection is observed in all the curves at composition 60 wt% PMMA in the blend. The similar nature of nonlinearity is observed in 2 wt% and 4 wt% solutions at 20°C, 30°C, and 40°C. The inflection at 60 wt% PMMA in the blend can be attributed to phase separation. It is well established that for a compatible and miscible blend that the ultrasonic velocity varies linearly with compositions at all concentrations and temperature (28, 30, 31). While an S-shaped curve is observed for incompatible/immiscible blend pair. The ultrasonic plots do not show S-shaped curve or distorted S-shaped curve, instead two linear portions with an inflection is observed which can be attributed to semi-compatibility/semi-miscibility of the DCOE-PMMA pair in DMSO. This inference is further corroborated by the observation that blends of all composition in 2 wt% and 4 wt% solutions remained transparent and no turbidity appeared over several months. The similar

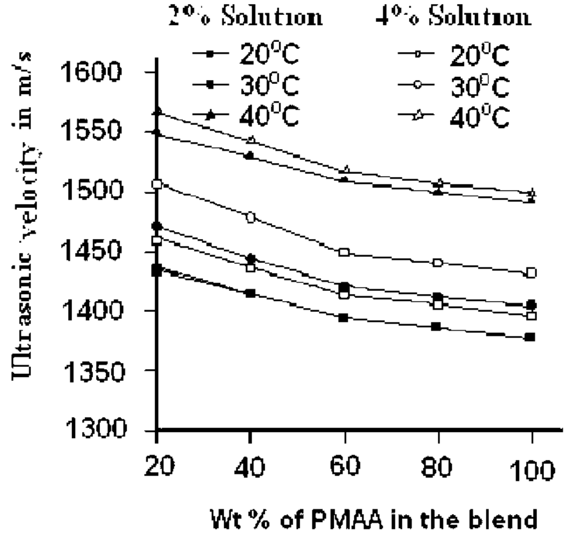

Figure 3. Effect of varying composition of DCOE : PMAA blend on the ultrasonic velocity of 2% and 4% solution.

variations in the ultrasonic velocity in 2 wt% and 4 wt% solutions at 20°C, 30°C, and 40°C indicates that the interaction between the two components is stable against the increase in temperature and may be occurring through electrostatic forces and hydrogen bonding.

Adiabatic Compressibility

Using ultrasound velocity, adiabatic compressibility of the system can be calculated by the following equation (32):

$$\beta = 1/\mathrm{v}^2\rho$$

where β is adiabatic compressibility of the medium, v is the velocity of the sound waves and ρ is the density of the medium. Adiabatic compressibility is inversely proportional to the cohesive energy of the polymer molecules (33). Figure 4 shows a slight nonlinear variation of adiabatic compressibility in 2 wt% and 4 wt% solutions of the blends of different composition at 20°C, 30°C, 40°C, which can be correlated to the semi-compatability of DCOE-PMMA pair in DMSO as has been inferred previously. The value of adiabatic compressibility was found to be the lowest for the composition 20 wt% PMAA and 60 wt% DCOE in the blend due to the compact structure of the blend resulting from the highest interaction between the components. Beyond 20 wt% PMAA in the blend, the adiabatic compressibility was found to increase showing loosening of the blend structure and the decrease in mutual interaction between the components. This observation about the blend structure matches with the inference derived from reduced viscosity measurements.

Density Measurements

The density of the blends of different compositions calculated on the basis of additivity of volumes and compared with corresponding observed densities provides evidence of

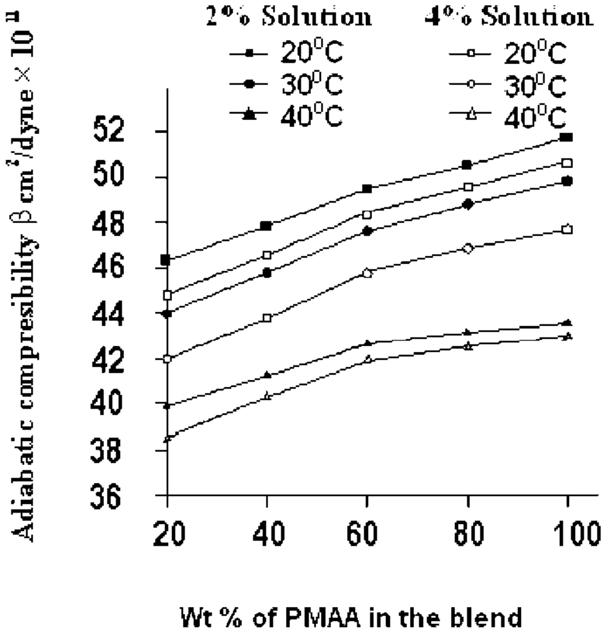

Figure 4. Effect of varying composition of DCOE : PMAA blend on the adiabatic compressibility of 2% and 4% solution

miscibility or immiscibility of the blend components. For immiscible blends, the observed densities have been reported to agree with the calculated densities or to be lower than the latter (8, 10). Those of miscible blends have been found to be larger up to 5% than the calculated densities (34). Figure 5 shows the plots of calculated and observed densities of DCOE : PMAA blends against composition in 2 wt% and 4 wt% solutions at 30°C, the two plots appear to be linear and parallel. From the insignificantly lower values of observed densities than the calculated values, as well as from the linearity of the plot, (Figure 5a, a′ and b, b′) it can be inferred that the two components are semi-compatible in solution phase.

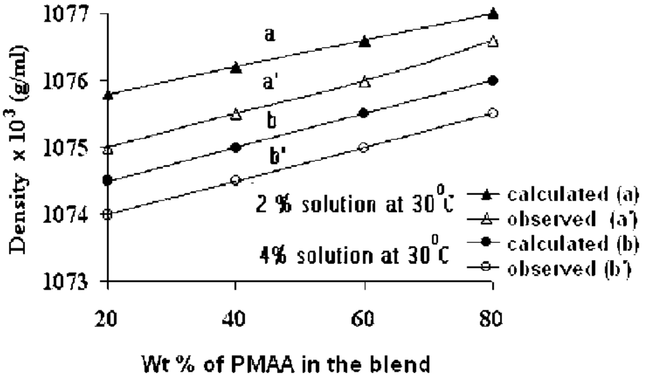

Figure 5. Variation of density with composition of the blend.

Film Study

The films of pure PMMA and varying compositions of PMAA-DCOE blends were found to be transparent and brittle. The films of blend containing 20 wt% PMAA were highly fragile and could not be recovered intact from the substrate. The films of blend with 40 wt% PMAA was toughest (qualitative estimate). The films of blend with 60 wt% PMAA was found to be less tough (qualitative estimate) than that of the blend with 40 wt% PMAA. The film of pure PMAA was transparent, hard and brittle. It appears that toughness depends upon the morphology and microstructure of the films which appears to be most favorable in the blend containing 40 wt% PMMA as compared to other compositions. The films of all blend compositions were found to be sticky; but the toughness and the integrity of the films were not affected. The stickiness of the films can be correlated to the phase separation and exudation of one of the phases. In the films, inter and intramolecular hydrogen bonding in PMAA molecules, which are presumably larger than the hydrogen bond formation between epoxy-PMAA molecules cause the loosening of the epoxy molecules which slowly exude out of the films. We can thus infer that blend is not stable in the solid phase and the components are incompatible.

Heat of Mixing, ΔH_{mix}

The two components in the blend will be thermodynamically miscible if ΔG_{mix} is negative. Since entropy of mixing of polymers has insignificant value, ΔG_{mixing}, therefore depends upon ΔH_{mixing}. The latter may therefore, be taken to approximate the former (35). The ΔH_{mixing} values are therefore indicative of miscibility of blend components. ΔH_{mix} can be calculated using Schneier's equation (36):

$$\Delta H_{mix} = \left\{ x_1 M_1 \rho_1 (\delta_1 - \delta_2)^2 \left[\frac{x_2}{(1 - x_2) M_2 \rho_2 + (1 - x_1) M_1 \rho_1} \right]^2 \right\}^{1/2}$$

where ΔH_{mix} is the heat of mixing, subscripts 1 and 2 represent the component polymers and x, M, ρ, and δ, respectively are the weight fraction, molar mass of one repeat unit, density and solubility parameter of the component polymers. Value of the solubility parameter δ for PMAA is 9.9, has been taken from Brandrupp and Immergut (37), that of DCOE has been calculated using cohesive energy values of different structural units as given by Hoy and produced by Krause (17) and was found to be 8.04. The densities of PMAA and DCOE were found to be 1.23 and 0.995 g/cm^3, respectively. The molar mass of DCOE was taken to be that of one epoxidized fatty acid chain, equal to 308.0 g. This normalizes the molar mass of DCOE with that of a repeat unit of PMAA. Using these values, the ΔH_{mix} was calculated with PMAA and DCOE alternately as component 1 for different composition of the blend and the same were plotted alternatively against increasing wt% of PMAA and DEOE as component 1 of the blend in Figure 6. It was observed by Schneier (36) that the components were miscible if ΔH_{mix} was near 10×10^{-3} cals uniformly for all compositions. Figure 6 shows that ΔH_{mix} values for different compositions of the blends are higher than 10×10^{-3} cals both for PMAA and DCOE taken as components alternatively. The above observations confirm incompatibility of the DCOE with PMAA in solid phase.

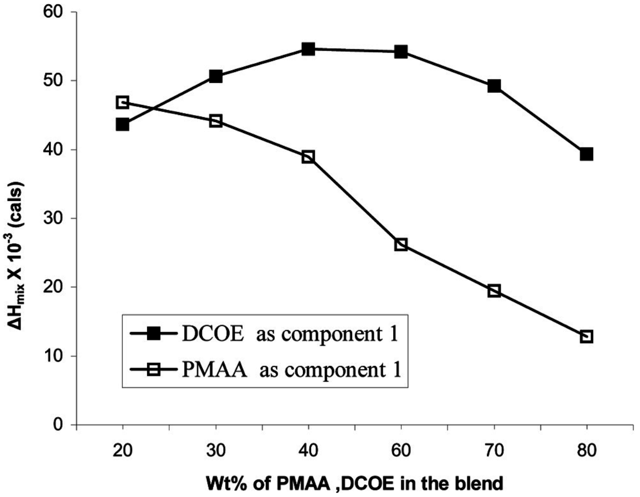

Figure 6. Variation of ΔH_{mix} with composition of the blend.

Thermal Analysis

The DSC thermogram of pure PMAA, (Figure 7(a)) shows a Tg at 175°C followed by an endothermic event spanning between 185°C–300°C. The PMAA-DCOE blend containing 60 wt% PMAA, (Figure 7b) gives Tg at 176°C. The endothermic peak in this case spans

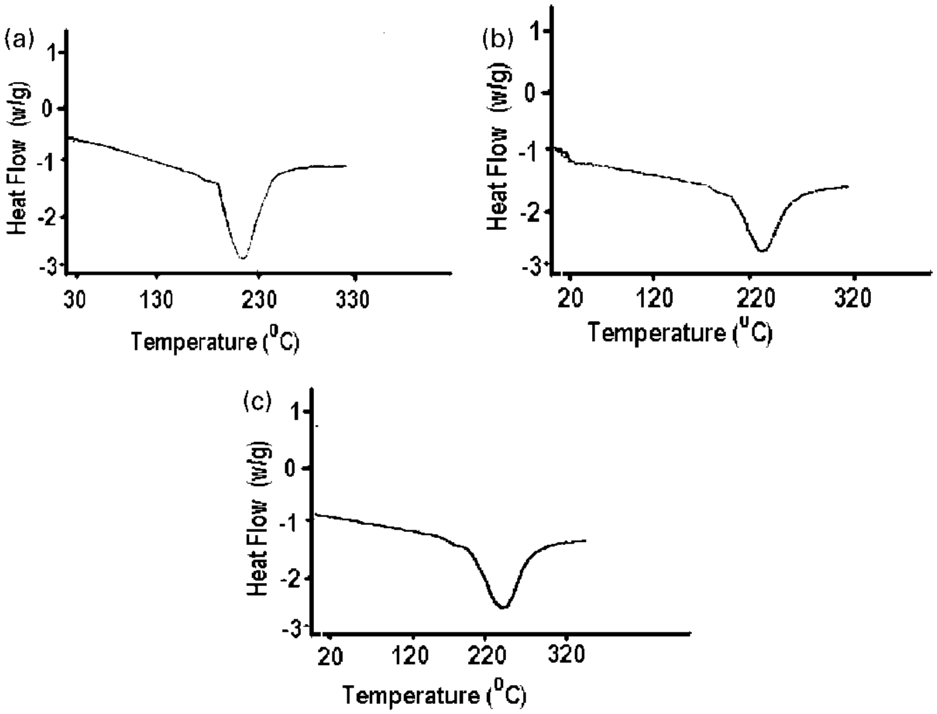

Figure 7. (a) DSC thermogram of pure PMAA; (b) DSC thermogram of blend of composition DCOE : PMAA 60 : 40; (c) DSC thermogram of blend of composition DCOE : PMAA 40 : 60.

over a similar temperature range. The Tg in DCOE blend of composition 40 wt% PMAA, (Figure7(c)) is observed at 174°C, while in this case also a similar endothermic peak is observed. It is thus observed that the glass transition temperature of PMAA does not change on mixing with DCOE showing that the two components are incompatible in the solid phase.

Morphological Studies

The SEM micrograph of the blend of compositions 40 wt% PMAA and 60 wt% PMAA were recorded. The SEM micrograph of the blend of composition 40 wt% PMAA and 60 wt% DCOE, (Figure 8(a)) clearly shows a continuous amorphous phase in which distinct white lamellae are emerging as a second phase. The white lamellae in the SEM micrograph can be correlated to the phase separation occurring in the blend indicating

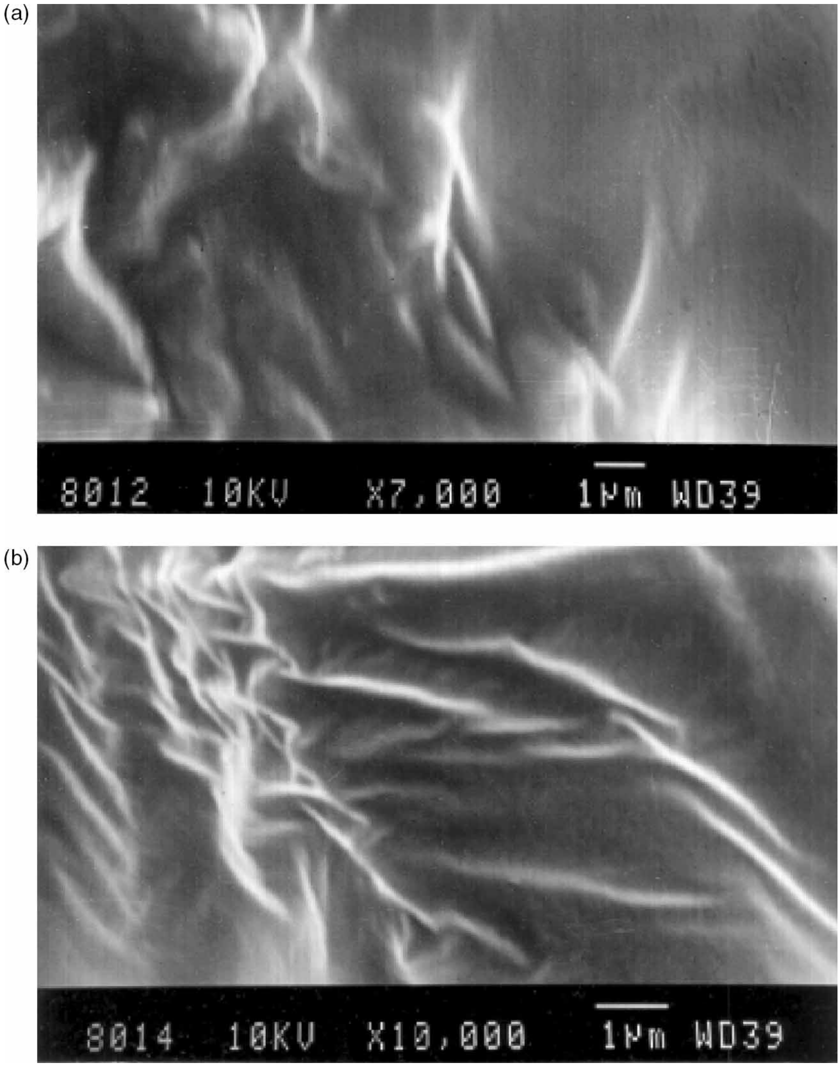

Figure 8. (a) SEM micrograph of DCOE : PMAA 60 : 40; (b) DCOE : PMAA 40 : 60.

the onset of incompatibility. This also explains the stickiness of the films. DCOE is therefore immiscible/incompatible with PMAA in solid phase.

The SEM micrograph of the blend containing 60 wt% PMAA and 40 wt% DCOE, (Figure 8(b)), is similar to the previous one except that the lamellar phase appears to be larger, and more widely distributed, presumably because of a higher proportion of PMAA in this blend causing greater phase separation and incompatibility. The white lamellae in this case also appear to be emerging in the continuous amorphous phase. We have earlier observed that films of the blend with 40 wt% PMAA were tougher than those of 60 wt% PMMA and 40 wt% DCOE blend which were presumably due to lesser phase separation in the former case as shown by SEM. On the basis of the above observations, we can conclude that these blends are heterogeneous and incompatible in solid phase, as was also inferred from the film study.

Conclusions

Blends of DCOE with PMAA were found to be semicompatible in solution phase by ultrasonic velocity, viscosity and density measurements. The films were transparent, flexible and sticky, having poor stiffness. Thermal analysis, as well as morphological investigations, indicate that the blends are incompatible in solid phase.

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