Miscibility Behavior of Poly(*n*-butyl Methacrylate) Latex Films Containing Alkali-Soluble Resin

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ABSTRACT: The dynamic mechanical properties of poly(*n*-butyl methacrylate) (PBMA) latex films postadded with alkali-soluble resin (ASR) have been studied and compared with those of latex films prepared by emulsion polymerization in the presence of ASR (ASR-fortified latex). The miscibility between PBMA and ASR, poly(styrene/alpha-methylstyrene/acrylic acid) (SAA), was found to influence the dynamic mechanical behavior of the films. The dynamic properties of PBMA latex films postadded with SAA show two distinct damping peaks, which correspond to those of PBMA and SAA, respectively, in the phase-separated state. The SAA migrates onto film surface during film formation and, as a result SAA preserved their domains in the matrix phase, showing two distinct relaxations in the dynamic mechanical spectrum. On the other hand, the ASR-fortified films exhibit single damping peak. SAA-fortified latex particles would be core/shell structured, and the miscibility between PBMA and SAA is clearly improved by the grafting reaction between PBMA and SAA. © 2000 John Wiley & Sons, Inc. J Appl Polym Sci 78: 639–649, 2000

Key words: dynamic mechanical properties; miscibility; alkali-soluble resin(ASR); SAA-fortified latex

INTRODUCTION

Synthetic latexes are important industrial products widely used in the papers, paints, and coatings, and adhesives industries. The essential feature of such application is the formation of continuous latex films. The interdiffusion of polymer chains across the particle–particle interface eventually leads to the formation of a coherent film. The nature of the interface has thus a profound influence on the mechanical strength of the film formed. Thus, viscoelasticity and polymer type of the latex particles are important considerations in the film formation process. The fundamental driving force behind the investigations on polymer blend latex films is the anticipation that in the blend one can obtain different properties than those of the individual components, and under some circumstances might even obtain unique properties. Over this period of time there has been much less attention paid to latex blends. Such blends are prepared by mixing two polymers where each is present in the form of polymeric microspheres dispersed in a fluid medium.[1] The miscibility between the blend constituents has been variously interpreted as being due to specific interaction between the components, such as hydrogen bonding, dipole–dipole interactions, ion–dipole interactions, ion–ion interactions, or repulsive interactions. However, some of the blends are

also reported to be miscible as a result of chemical reaction between the blend constituents.[2]

Recently, we have demonstrated that preparation of polystyrene (PS) and polymethyl methacrylate (PMMA) latex particles by emulsion polymerization using alkali-soluble resin (ASR) as a polymeric surfactant.[3,4] Very small latex particles were formed compared with conventional surfactant system. ASRs are low molecular weight carboxylated random copolymers that have been used in the preparation of durable emulsion polymers, prepared using these types of resins, on a variety of substrates such as floor tiles, wall, and shower tiles can be found in the literature.[5,6]

Another variation of this is to postadd the ASR to monodisperse surfactant-free poly(n-butyl methacrylate) (PBMA) latex prepared by emulsion polymerization in the absence of any surfactant. It has been recognized that amphiphilic polymers with both hydrophobic and hydrophilic groups can function as stabilizers for latex particles via intermolecular and/or intramolecular hydrophilic interactions.[7,8] The morphology and kinetics of film formation of monodisperse surfactant-free PBMA latex are strongly influenced by the presence of postadded ASR.[9–11]

In the case of polymer latex film prepared from acid comonomers, a separate phase of polar material acts as an interconnecting phase in the newly formed film.[12] The diffusion of polymer chains in latex films prepared from PBMA latex particles containing different amounts of the acid group at the particle surface had been reported by Winnik et al.[13]

In this study, the ASR, namely poly(styrene/alpha-methylstyrene/acrylic acid) (SAA) was used as a polymeric emulsifier in the emulsion polymerization of PBMA to investigate the latex film properties. We called such latexes ASR-fortified PBMA latexes, and we prepared the emulsifier-free monodisperse PBMA latex postadded with SAA and studied the film properties. The dynamic mechanical properties of SAA-fortified PBMA latex films were studied using DMTA and compared with those of the SAA-blended PBMA latex films. The system chosen consists of PBMA polymer latexes that contain various amounts of SAA in different two ways.

This article addressed the issue of polymer miscibility in films prepared from latex particles on whose surface acid groups are adsorbed or grafted. This SAA layer on the PBMA particle surface could have an influence on mechanical properties of films.

EXPERIMENTAL

Materials

Twice-distilled water was used throughout. n-Butyl methacrylate was purchased from Junsei Chemical Co., Japan. It was purified by vacuum distillation under reduced pressure and refrigerated at 4°C until needed. The alkali-soluble resin, poly(styrene/alpha-methylstyrene/acrylic acid) (SAA), with M_n = 4300 g mol^{-1}, M_w = 8600 g mol^{-1}, acid number = 190 and T_g = 115°C was purchased from Morton Inc., USA, and used as received. Potassium persulfate (KPS), from Samchun Pure Chemical Ind. Ltd., Korea, was recrystallized before used. Reagent grade sodium bicarbonate from Samchun Pure Chemical Ind. Ltd., Korea, was used as received.

Preparation and Characterization of PBMA Latex/Resin-Fortified Emulsions (RFE)

PBMA latexes were prepared by emulsion polymerization in different two ways. One was the emulsifier-free emulsion polymerization. In the other way the SAA was used as a polymeric surfactant. The SAA and NaOH were dissolved in distilled deionized water and the SAA solution was used throughout. The basic recipe of emulsion polymerization is given in Table I. The agitation rate was maintained at 300 rpm throughout the reaction. The number average particle size (D_n) and polydispersity (D_w/D_n) of the PBMA latex particles were determined by a particle size analyzer, namely the Capillary Hydrodynamic Fractionation (Model CHDF-1100, Matec Applied Sci., USA), and were found to be 473.9 nm and 1.007, respectively. The D of resin-fortified emulsion latex particles was found to be much smaller than that of PBMA. Table I compares the number average particle sizes of all latex samples. All latex samples were purified by ion exchange followed by a serum replacement technique until the final conductivity of the serum was less than 10^{-5}ohm^{-1}cm^{-1}.[14] The latex obtained was stable without the need of any postadded stabilizer.

Preparation of the Blends

The SAA resin was postadded to the PBMA latex under moderate magnetic stirring at room tem-

Table I Basic Recipe of Emulsion Polymerization of Butyl Methacrylate Using SAA

Components	Amounts (g)			
	PBMA	RFE-S10	RFE-S20	RFE-S30
D. D. I. Water	720	390	425	460
poly(styrene/alpha-methylstyrene/acrylic acid) (SAA)	—	10	20	30
Sodium Hydroxide (NaOH)	—	1.43	2.86	4.29
Sodium Bicarbonate (NaHCO$_3$)	0.25	—	—	—
Butyl methacrylate	80	100	100	100
Potassium persulfate	0.8	0.5	0.5	0.5
Particle Size (nm)	473.9	72.1	69.7	67.6

perature. The mixed dispersions initially contained SAA adsorbed on the PBMA particles, and unadsorbed SAA should be present in the aqueous phase before drying. However, during drying, it was found that the pH of the mixed dispersion dropped, and this would lead to a higher amount of SAA being adsorbed on the surface.[10] The mixture was kept for saturation prior to use.

Preparation of Latex Films

Homogeneous transparent solid films were prepared by dehydration of the dispersions according to the following procedure. First the dispersions were degassed at room temperature. Then they were cast on a nonsticking smooth teflon substrate. These liquid films were then dried in a convection oven for 1 day. The drying temperature was 60°C. Samples exhibiting a constant thickness ranging between 0.5 and 1 mm were then cut to dimensions of 1.5 (0.8 mm^2 for DMA experiments.

Dynamic Mechanical Analysis (DMA)

For DMA, we worked with the simple extension mode. The dynamic mechanical behavior of the films was investigated using a DMTA (Polymer Labotatories, Model MK-III, UK) equipped with an attachment for temperature control. This apparatus enabled us to measure the extension storage modulus (E'), the loss modulus (E'') and the loss tangent (tan δ) over a wide range of temperatures (−50–200°C). The isochronal temperature dependence of moduli and tan δ were obtained for a frequency of 1 Hz at a constant heating rate of 2°C min^{-1} under a nitrogen atmosphere. The accuracy of the data values was confirmed by repeating measurements twice in the same temper-

ature range and the reproducibility was found to be satisfactory.

Atomic Force Microscopy

All films were imaged in air at 25°C with a Nanoscope III AFM (Digital Instruments, Inc., Santa Barbara, CA) in the Tapping Mode. In the Tapping Mode, the cantilever on which the tip is mounted is oscillated at a frequency of ca. 250 kHz. The oscillation is driven by a constant driving force, and the amplitude of its oscillation is monitored. The scans were done under ambient condition without any sample surface treatment. Latex films were prepared by placing a few drops of the mixed dispersion of PBMA and SAA onto freshly cleaved mica surface (ca. 10 × 10 mm) and allowed to dry at 25°C in a desiccator. Aging of the film was carried out in a convection oven at 90°C. After annealing, the samples were returned to the desiccator before imaging by AFM.

Tensile Strength Measurement

Tensile strength measurements were done in a Universal Testing Machine (Instron Corporation Series IX Automated Materials Testing System, USA) according to ASTM D638M at a strain rate of 10 mm/min.

Thin-Layer Chromatographic Separation Technique

Thin layer chromatography (TLC) was used to separate the RFE into three components; that is, the ungrafted SAA, PBMA, and the SAA-grafted PBMA. Polymer solutions were prepared by dissolving the sample in tetrahydrofuran (THF) at a concentration of 0.5 g/L and applying them to the TLC plate with microsyringe. The detailed de-
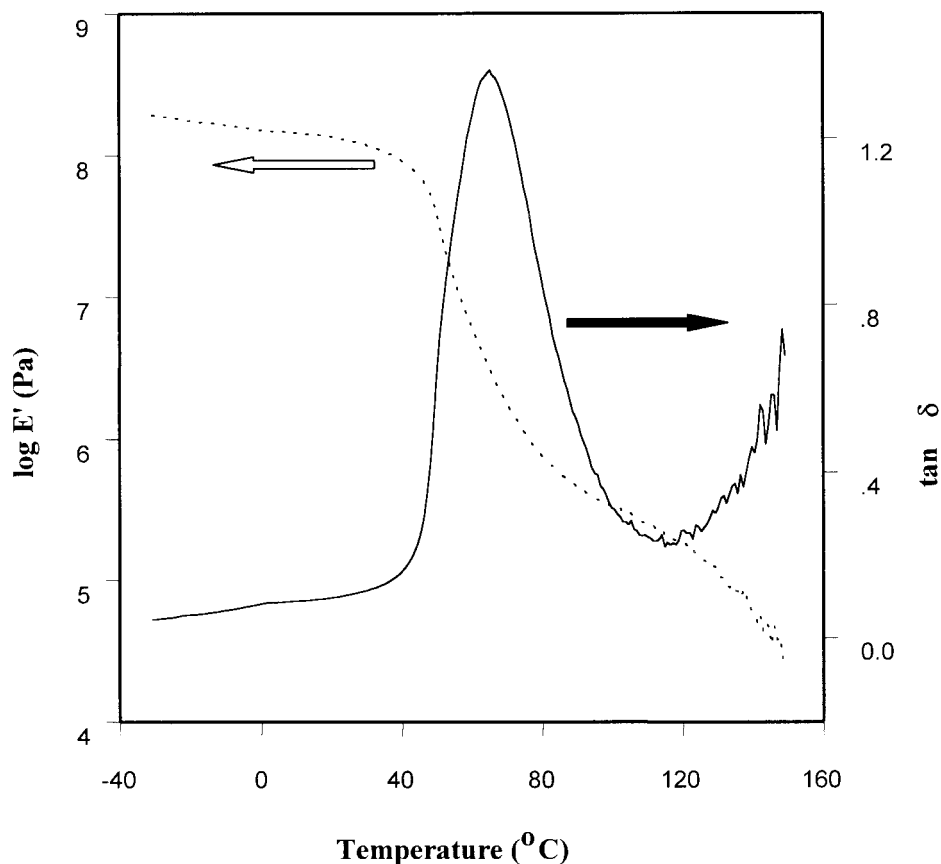
Figure 1 Dynamic mechanical properties of PBMA latex film as a function of temperature; storage modulus (E'); damping curve (tan δ).

RESULTS AND DISCUSSION

SAA-Blended System

Dynamic mechanical analysis (DMA) is often used to study polymer/polymer miscibility in polymer blends. The results of dynamic mechanical testing add information to the behavior of the blends and the phase morphology. Typical isochronal temperature dependence of E', E'', and tan δ recorded under the above conditions for PBMA latex film is plotted in Figure 1. These curves classically display three regions where the viscoelastic behavior of the polymers is well identified: the glassy region in the low-temperature range, the transition region, and the rubbery region in the higher temperature range. In this plot, three regions of viscoelastic behavior are defined.[15]

Only one main relaxation (α-relaxation) is found in the spectrum, which corresponds to PBMA. This is attributed to the initiation of micro-Brownian motions of the macromolecular chain segments in the molecules.

The miscibility of different polymer blends through various specific interactions can be predicted theoretically with the solubility parameters. The solubility parameter components may be predicted from group contribution, using the method of Hoftyzer and Van Krevelen.[16] The following equations are used;

scription of the TLC procedure has been reviewed elsewhere. The thin layer chromatography/flame ionization detector (TLC/FID) method was used for direct qualitative analysis. The separations were carried out on thin quartz rods 0.9 mm in diameter and 150 mm long and coated with silica gel 75 μm thick (Iatron Chromarod-SII). After development, the quartz rods were dried to vaporize the developer solvent. At the end of the run, the rods were placed in an apparatus equipped with a FID (Iatroscan MK-5 TLC/FID analyzer) for analysis.

Table II   Physical Parameters of the Pure Constituents of the Blend Obtained by Group Contribution

	Molar Volume (cm^3mol^{-1})	Molecular Weight ($g\ mol^{-1}$)	Solubility Parameter ($J^{1/2}cm^{-3/2}$)
Poly(*n*-butyl methacrylate)	86.79		17.9
Polystyrene	99	103.95	18.01
Poly(alpha-methylstyrene)	111	118.22	18.49
Poly(acrylic acid)	38	72.06	30.36
poly(styrene/alpha-methylstyrene/acrylic acid) (SAA)			20.91

$$\delta = \sqrt{\delta_d^2 + \delta_p^2 + \delta_h^2} \qquad (1)$$

$$\delta_d = \frac{\sum F_{di}}{V} \qquad (2)$$

$$\delta_p = \frac{\sqrt{\sum F_{pi}^2}}{V} \qquad (3)$$

$$\delta_h = \sqrt{\frac{\sum E_{hi}}{V}} \qquad (4)$$

where V is the molar volume and δ_d, δ_p, and δ_h are the solubility parameter components, dispersion, polar, hydrogen and bonding components, respectively, and δ is the solubility parameter.
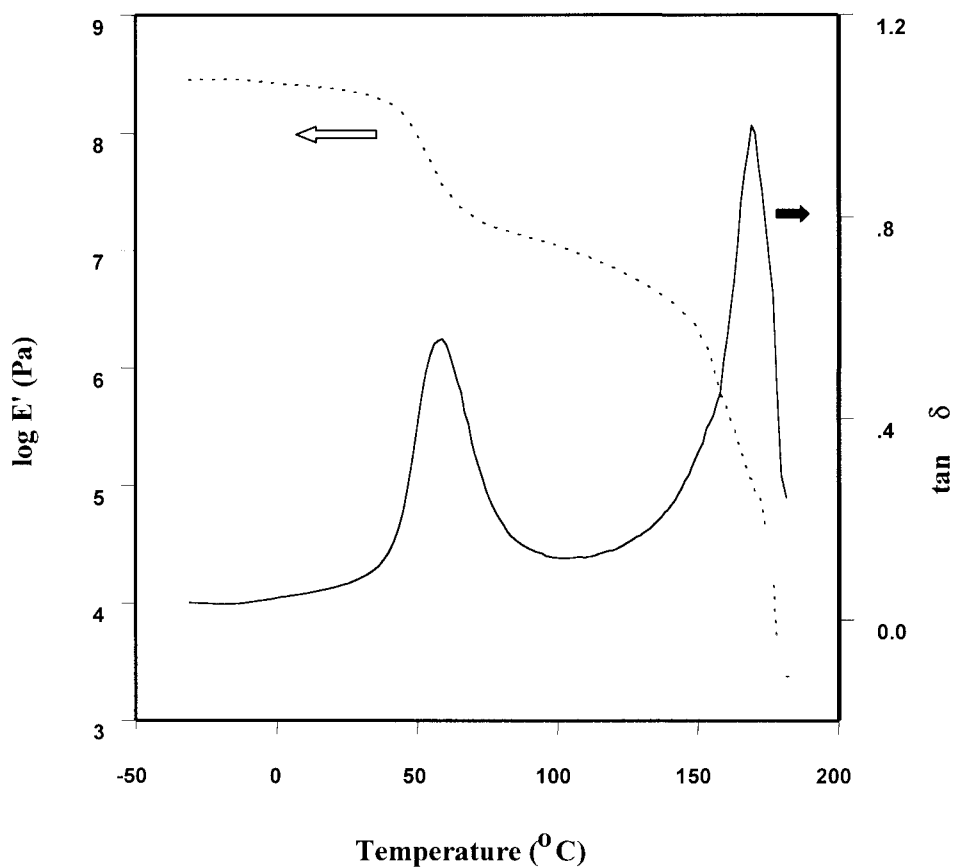

Figure 2   Dynamic mechanical properties of the 10 wt % SAA-blended PBMA latex film as a function of temperature; storage modulus (E'); damping curve (tan δ).

The solubility parameter for a random copolymer can be calculated by using the Hildebrand Equation, as follows;

$$\delta = \sum \delta_i \phi_i \quad (5)$$

where δ_i and ϕ_i are the solubility parameter and volume fraction, respectively, of the homopolymers in the copolymer.

To achieve molecular-level mixing of the blend constituents the value of the interaction parameter (χ) should be <0.002, which can be calculated by using the Hildebrand equation, as follows:

$$\chi = V_R/RT(\delta_A - \delta_B)^2 \quad (6)$$

where V_R is the reference volume, δ_A and δ_B are the solubility parameters of the two blend constituents, T is the temperature, and R is the molar gas constant.[2]

Using the data given in Table II, the miscibility of the binary blend system of PBMA and SAA has been predicted. With the solubility parameter, δ_A and δ_B obtained from group contribution and a reference volume of 86.79cm^3 mol^{-1} at 25°C, the interaction parameter (χ) has been calculated and found to be 0.32, which is well above 0.002 predicted by the Hildebrand approach. This value substantiates the fact that the blends of PBMA and SAA are completely immiscible.

The spectrum in Figure 2 shows two distinct relaxations, which correspond to those of PBMA and SAA resin, respectively, in the phase-separated state. These results were interpreted from immiscibility between PBMA and SAA as predicted theoretically. Figure 2 confirms the theoretical prediction. The presence of two well-defined peaks in the blends established the immiscibility of the two phases. The SAA resin was phase separated from PBMA during film formation and, as a result, SAA resin preserved their domains in the matrix phase, showing two distinct relaxations in the DMA spectrum.

The postadded SAA would be adsorbed on the PBMA particle surface in the mixed dispersion. The presence of this polar material would therefore form a separate phase in the interparticle spaces between the particles as the film dries. Our experimental results show that SAA was not compatible with PBMA. The two components would thus remain phase separated in the film as clearly seen in DMA spectra.
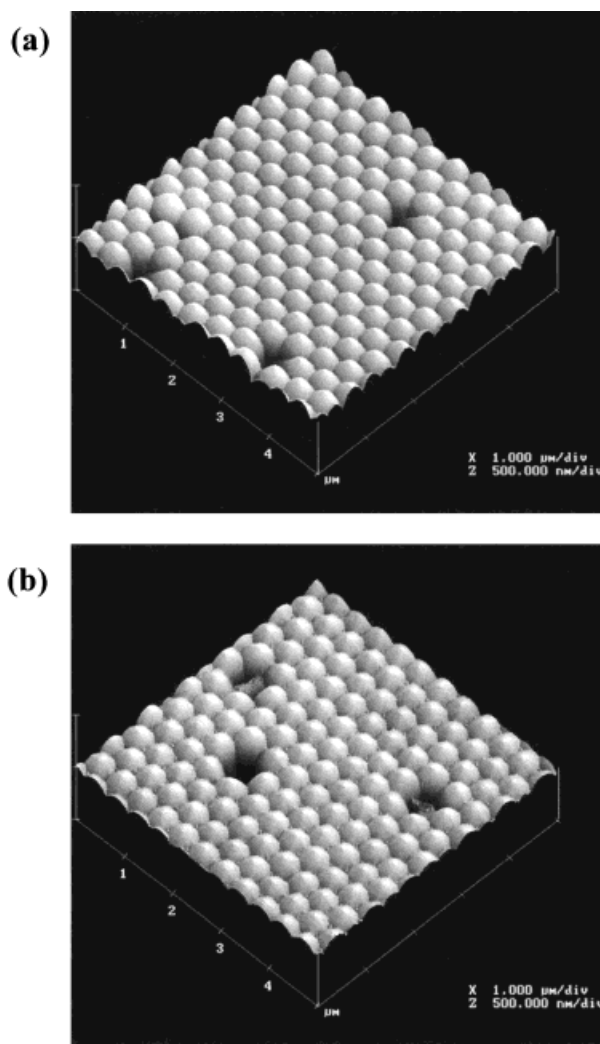

Figure 3 Atomic force micrographs of PBMA latex film containing 10 wt % SAA (a) before annealing, and (b) annealed for 10 min at 90°C. Interfaces of PBMA latex particles are filled with the migrated SAA.

The immiscibility between PBMA and SAA indicates that the two are incompatible. The intervening SAA among the PBMA particles hinders interdiffusion between the PBMA core particles during film formation.[11] In addition, the preferential accumulation of the polar SAA at the film surface (air/SAA interface) during annealing preserved their domains in the matrix phase. The accumulation of SAA at the interstices of the arrays of PBMA particles is clearly discernible from the three-dimensional surface image of PBMA latex film containing 10 wt % SAA annealed for 10 min at 90°C, respectively (Fig. 3). The deformations and eventual coalescence of the PBMA par-
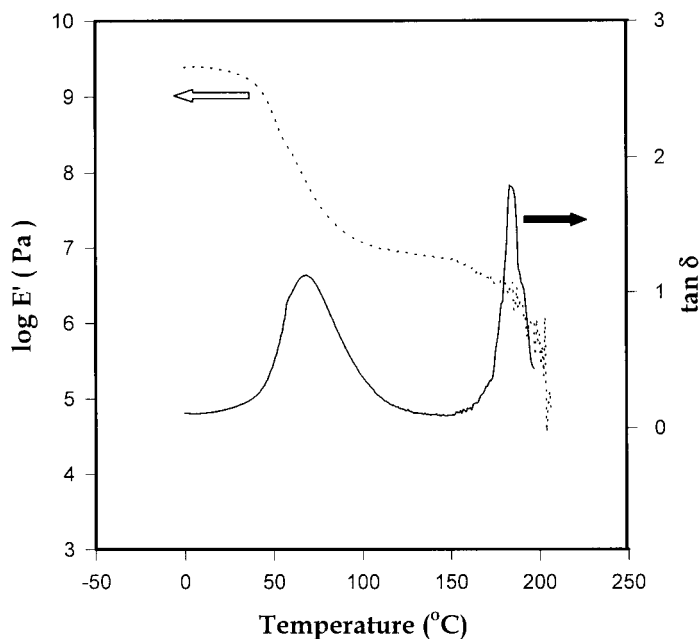
Figure 4 Dynamic mechanical properties of the 10 wt % SAA-blended small-sized PBMA latex film as a function of temperature storage modulus (E'); damping curve (tan δ).

ticles occur below the surface layer of SAA during annealing. As the annealing temperature is raised, more SAA molecules migrate to the surface of the latex film and concomitantly accumulate in the valleys between the particles.

The immiscibility between PBMA and SAA is the driving force for the migration of SAA during film formation. Figure 3(b) is AFM surface image showing that the interparticle spaces between PBMA particles are filled with migrated SAA resin.[17]

The particle size of PBMA latex (ca. 473.9 nm) is much bigger than that of SAA-fortified latex (ca. 73 nm). Discrepancy in particle sizes may bring about controversy on comparison between the two. To make sure that two damping peaks in the blend arise from the immiscibility not from the size effect, we carried out DMA experiment for the blend of small-sized PBMA latex and SAA solution under the same condition as that of the 10 wt % of SAA-blended PBMA latex. Small-sized PBMA latex was prepared using the conventional surfactant, sodium dodecylbenzenesulfonic acid, SDBS, and purified by serum replacement. The two damping peaks in Figure 4 definitely confirm the immiscibility between PBMA and SAA.

Tensile strength buildup of polymer films, prepared from aqueous dispersion of latex particles, is dependent not only on the nature of bulk ma-

terial property, but also the degree of particle coalescence, i.e., the extent of interdiffusion of chain segments across the particle boundary.[18] Table III compares the tensile properties of polymer films before and after blending. The postadded SAA could hinder interdiffusion between PBMA core particles during film formation. The decrease in tensile strength in the blend is generally observed in incompatible blends, and has been attributed to the retarded interdiffusion of core polymer particles.[19] As mentioned above, the SAA-adsorbed surface of the PBMA particles acts as a barrier to prevent or retard polymer diffusion between particles in films. The tensile property of blending sample was found to be inferior to that of

Table III Tensile Strengths of Prepared Latex Samples

Sample I.D.	Tensile Strength (MPa)
PBMA	22
Blend-S10 (10 wt % of SAA-blended PBMA latex film)	19
Blend-S20 (20 wt % of SAA-blended PBMA latex film)	17

PBMA latex film. Initially, the stress is proportional to the strain and Hooke's law is obeyed. The Young's moduli can be obtained from the slope. As the strain is increased, the curve decreased in slope until it reaches a maximum. This is conventionally known as the yield point.[20]

SAA-Fortified System

For the conventional emulsion polymerization of styrene examined by Harkins, the most important sites of particle generation are monomer-swollen emulsifier micelle. The amphiphilic polymers, which consist of both hydrophobic and hydrophilic groups, can stabilize polymer particles and can form aggregates like micelles as the result of the intermolecular and/or intramolecular hydrophobic interactions.[3] The particle formation in emulsion polymerization using SAA would be affected by the properties of SAA aggregates. In emulsion polymerization using SAA, SAAs are concentrated on the surface of the final latex polymer. The latex was stabilized by SAA, which results in formation of very small and stable particles without any surfactant. The particle size of PBMA latex prepared in the presence of SAA is illustrated in Figure 6. Generally speaking, it shows smaller particles than that prepared in the conventional emulsion polymerization. At SAA
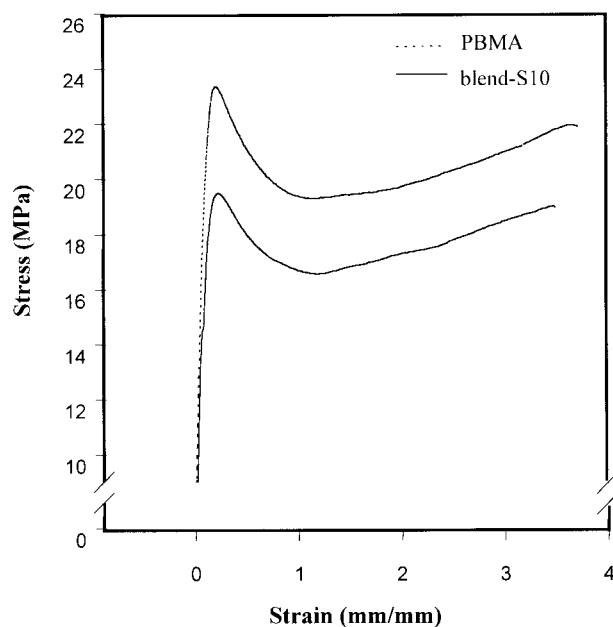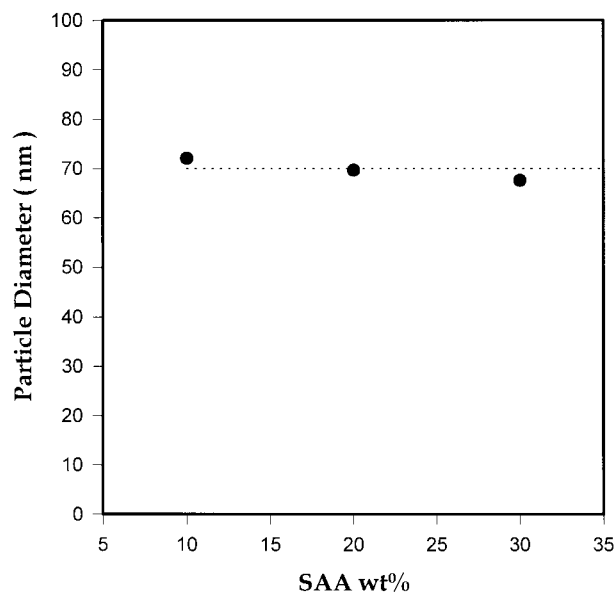

Figure 6 Particle size analysis of latex particles.

concentration of 10 wt % and above, it is also interesting to note that the particle size is similar.

The miscibility between PBMA and SAA resin in SAA-fortified emulsion has been confirmed by the occurrence of single, sharp glass transition temperatures in Figure 7. Interestingly, two glass transitions, signifying incompatibility in the blend, turned into a single intermediate glass transition in SAA-fortified emulsion system. It can be also interpreted from the improved miscibility between two phases without using any additives. The phase separation sometimes occurs during film formation resulting in a decrease in film properties. The SAA used as polymeric emulsifier would be grafted to main chains and keeps sticking to them during film formation showing no phase separation. Due to the increased amount of the SAA resin having higher T_g, 20 wt % of SAA-fortified PBMA latex film shows the slightly higher glass transition temperature than 10 wt % of SAA-fortified PBMA latex film.

Thin-layer chromatography was used to identify the grafted SAA in the PBMA latex. A DDI water–ammonium hydroxide mixture, a suitable developing solvent for ungrafted SAA and toluene in which SAA was insoluble, was a developing solvent for ungrafted PBMA.

The TLC separation was made in a two-stage development: the initial developer, a water–ammonium hydroxide mixture, was chosen so that
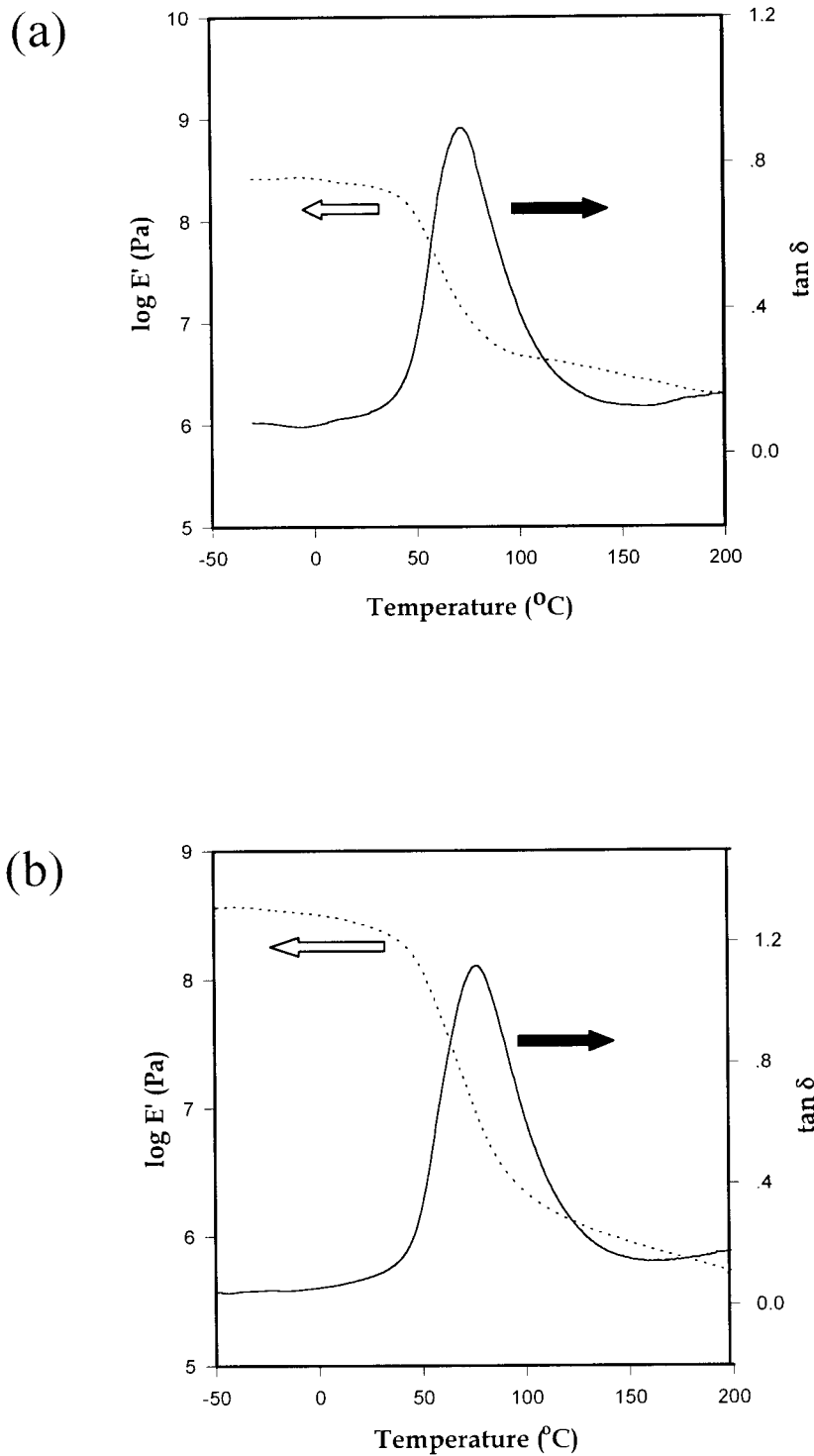

Figure 5 Stress vs. strain plots for PBMA and the 10 wt % SAA-blended films.

Figure 7 Dynamic mechanical properties of SAA-fortified PBMA latex films as a function of temperature; storage modulus (E'); damping curve (tan δ); (a) 10 wt % SAA, (b) 20 wt % SAA.

the ungrafted SAA migrated up to the solvent front, whereas the ungrafted PBMA and SAA-grafted PBMA remained immobile on the starting level. In practice, the solvent front was set at 15 cm above the starting level. This development was repeated more than three times.

After the initial development had been completed, the plate was subjected to secondary development with toluene as the developing solvent. With this procedure, only the ungrafted PBMA migrated up to a solvent front set at 7 cm. The sample was separated into three components. Figure 8 shows the TLC-FID chromatographic scanning curves of the blend and RFE-S10. The TLC-FID analysis confirmed that the PBMA latex contained a SAA-grafted portion, which indicated that the grafting of PBMA to SAA occurred during emulsion polymerization. This grafting in the PBMA latex probably resulted from chain transfer of monomer to SAA.[3]

Not all of incorporated SAA was grafted to PBMA in RFE-S10, and Figure 8 exhibited it very well. Ungrafted SAA might be dispersed in matrix PBMA without phase separation, which can be predicted from DMA spectrum. In the case of RFE-S20, more than 60% of incorporated SAA appeared to combine with PBMA polymer chains. Figure 9 shows the increased grafting efficiency of SAA. The more SAA is incorporated, the more SAA would be grafted to PBMA polymer chains. This result would be due to the larger surface area resulted from the smaller particle size.

In the blend, the SAA-grafted PBMA peak did not appear, indicating postadded SAA did not react with PBMA polymer chains. It is clear that
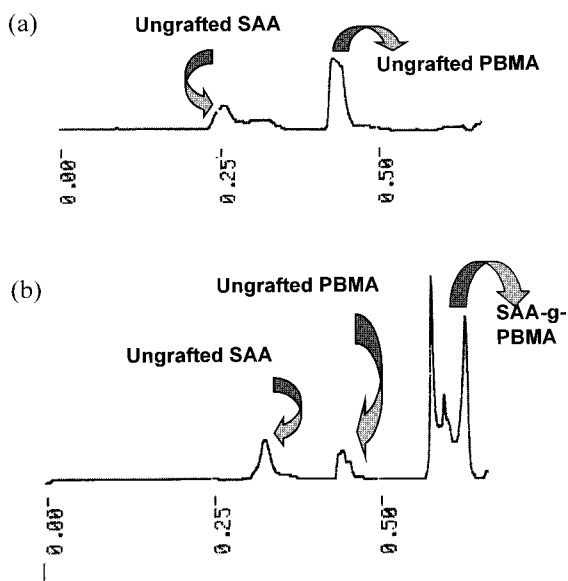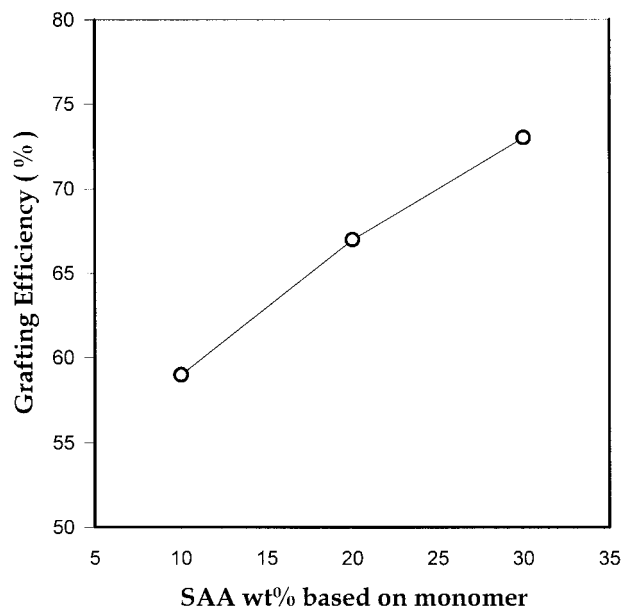
Figure 9 Grafting efficiency of SAA as a function of SAA concentration.

SAA has its own domains in the blend to show two relaxations in DMA spectra.

To investigate the miscibility between SAA-fortified latex and ASR, the DMA experiment for the blend of SAA-fortified PBMA and SAA was carried out. SAA-fortified PBMA (10% wt %) and 10 wt % SAA solution were blended. The DMA spectra for the blend film shows one single relaxation. As a result, SAA-fortified PBMA was found to be compatible with SAA. The core/shell structured SAA-fortified latex particles became miscible with SAA. The SAA would be well dispersed in the matrix phase without phase separation when it was blended with SAA-fortified PBMA. One damping peak in Figure 10 shows that SAA is compatible with SAA-fortified PBMA.

The behavior and contribution of SAA in emulsion polymerization were found to be different from those of conventional polymeric surfactants. The SAAs not only act like surfactants but also provide their own characteristics to latex for the SAA-fortified emulsion system.

CONCLUSIONS

This article addresses the issue of polymer interdiffusion in films prepared from latex particles on whose surface acid groups are adsorbed or
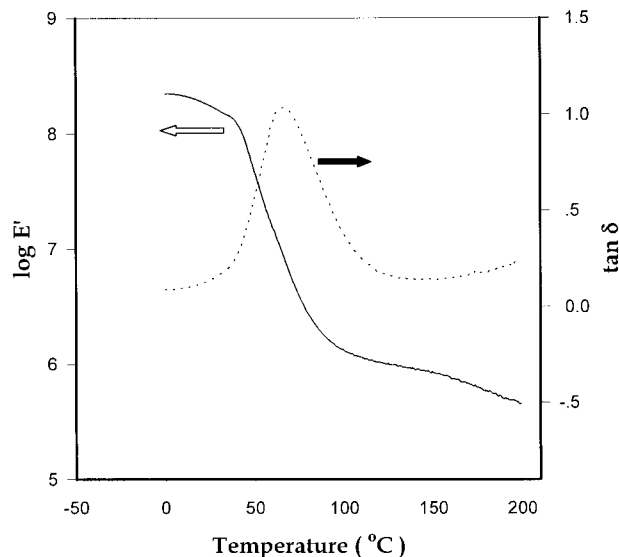
Figure 8 TLC-FID chromatographic scanning curves of PBMA latex prepared with SAA; (a) 10 wt % SAA-blended PBMA, (b) 10 wt % SAA-fortified PBMA.

Figure 10 Dynamic mechanical properties of the blend of 10 wt % SAA-fortified PBMA latex and SAA as a function of temperature; storage modulus (E'); damping curve (tan δ).

grafted. This SAA layer on the PBMA particle surface could act as a barrier to prevent or retard polymer diffusion between cells in these films. This retardation reduces the mechanical properties of films. SAA-fortified latex particles would be core/shell structured, and the miscibility between PBMA and SAA is clearly improved due to the grafting reaction.

The following conclusions have been drawn from this present investigation:

1. Dynamic mechanical analysis shows two glass transition temperatures for the blends, implying that the blends are immiscible.
2. SAA resin adsorbed onto PBMA particles tends to migrate to interparticle spaces during film formation, and holds its own domain in the phase-separated state.
3. The driving force for the migration of SAA resin is immiscibility of PBMA and SAA.
4. We successfully prepared PBMA latex using SAA as a polymeric emulsifier.
5. SAA-fortified PBMA latex film shows one single damping peak in DMA spectra indicating improved miscibility due to grafting reaction between PBMA and SAA.

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