

Surface and Interfacial FT-IR Spectroscopic Studies of Latexes. VIII. The Effect of Particle and Copolymer Composition on Surfactant Exudation in Styrene-*n*-Butyl Acrylate Copolymer Latex Films

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SYNOPSIS

Polarized attenuated total reflection Fourier transform infrared (ATR FT-IR) spectroscopy was used to identify the mobility and surfactant exudation of sodium dioctyl sulfosuccinate (SDOSS) surfactant molecules to the film-air (F-A) and film-substrate (F-S) interfaces in styrene/*n*-butyl acrylate (Sty/*n*-BA) latex films. It was found that, depending upon the latex particle composition, the surfactant molecules could be driven to the F-A or F-S interfaces. The primary factors that governed the direction of exudation were the compatibility of the latex components, interfacial film-substrate surface tension, and the chemical composition of the latex particles. Concentration, as well as orientation, of the hydrophilic $\text{SO}_3^- \text{Na}^+$ surfactant ends changed as a function of depth and the latex particle composition. © 1993 John Wiley & Sons, Inc.

INTRODUCTION

Synthesis of polymers, using emulsion polymerization, appears to be an attractive methodology because it results in a colloidal dispersion of polymer or copolymer latex particles in an aqueous medium. Such dispersions upon coalescence form continuous polymer films and many useful synthetic routes for latex preparation are now well documented. Along with the practical aspects of latex synthesis, several debatable theories, concerning the mechanisms of coalescence and the processes associated with it, were proposed. Recently, we have addressed the issue of physicochemical interactions between individual latex components and the film interfacial properties that resulted from these interactions.^{1,2} These issues are particularly pertinent in a context of the polymer or copolymer composition, particle composition, surfactant compatibility, and appear to be significant both from conceptual and practical points of view,

and in an effort to understand the principles governing latex film formation, during which surfactant mobility plays an important role.

In response to these concerns, our recent efforts focused on monitoring molecular processes and interactions that are responsible for the dynamics, mobility, orientation, and the effect of external sources on surfactant molecules in ethyl acrylate/methacrylic acid (EA/MAA) coalesced latex films.³⁻⁷ During the course of these studies, it became apparent that the surfactant-copolymer interactions in the presence of water may have a significant effect on many macroscopic properties, such as adhesion and durability. Ideally, one would expect that after the latex aqueous dispersion is deposited on a substrate, and proper coalescence conditions are met, a transparent, void-free film should form. This ideal scenario, however, can be disturbed by other processes that occur simultaneously. One of these processes is the diffusion of relatively mobile surfactant molecules through the film, during and after latex coalescence. This issue is particularly pertinent because, after a sufficient amount of water evaporates, the latex particles begin to come into contact with one another. Although one may argue about specific

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processes and propose many theories, the net effect is such that the latex particles undergo deformations to form a continuous polymer phase. We indirectly addressed some of these issues in ethyl acrylate/methacrylic acid (EA/MAA) latex studies, and many potential applications of the hard, soft, or hard-soft latex particles stimulated our interest in styrene/*n*-butyl acrylate (Sty/*n*-BA) latex copolymers. In this work, we will examine how the presence of soft, hard, and a combination of both latex particles may affect the mobility of the sodium dioctyl sulfosuccinate (SDOSS) surfactant molecules to the film-air (F-A) and film-substrate (F-S) interfaces. An ultimate goal of this study is to identify those factors that may mandate latex suspension and latex film stability, and influence dynamics of the small molecules in polymer networks formed from latexes.

EXPERIMENTAL

Latex Preparation

Styrene (Sty), *n*-butyl acrylate (*n*-BA) and methacrylic acid monomers (Aldrich Chem. Co.) were copolymerized by a semicontinuous emulsion copolymerization process, using the experimental apparatus reported in the previous studies.³ In a typical synthesis, sodium dioctyl sulfosuccinate (SDOSS, Aerol OT-100, American Cyanamid) surfactant was dissolved in 90 w/w % of double deionized (DDI) water (Millipore Filtration System). While stirring, the reaction vessel was purged with nitrogen and the temperature of the solution was raised to 75°C. After 30 min at 75°C, the remaining 10 w/w % DDI water, containing dissolved potassium persulfate initiator (Aldrich Chem. Co.), was introduced into the system and was stirred for 5 min. At this point, 20 w/w % of the thoroughly mixed monomers (or single monomer), was slowly added over a period of 5 min to form the seeds for latex particles. After 10 min, when a white milky emulsion was observed, the remainder of the monomer was fed for over 5 h at a rate of 20 drops/min. The temperature was maintained at 75°C and the latex emulsion was continuously stirred at this temperature for 30 min after the last drops of the starting materials were added. While stirring, the reaction vessel was gradually cooled to 40°C for a period of about 2 h.

Five separate monomer mixtures were prepared: 100% Sty, 75/25% Sty/*n*-BA, 50/50% Sty/*n*-BA, 25/75% Sty/*n*-BA, and 100% *n*-BA. The latexes, synthesized using these monomer ratios, were formulated to achieve 40 w/w % solids. Each mixture

also contained 5 mol % (based on a total number of moles of monomer) of methacrylic acid, 0.01 mol % of SDOSS, and 0.0025 mol % potassium persulfate. The preparation of pure Sty and *n*-BA homopolymers and copolymers was accomplished by repeated dissolution in chloroform and precipitation in ethanol.

Latex Film Preparation

Approximately 100–150 μm latex films were cast on a polytetrafluoroethylene (PTFE) substrate and were allowed to coalesce for 72 h at ambient temperature and humidity.

Spectroscopic Analysis and Latex Characterization

All spectroscopic procedures, using polarized attenuated total reflectance Fourier transform infrared (ATR FT-IR) spectroscopy, have been described previously.⁸ Latex particle size was analyzed using a Coulter N4S light scattering instrument (Coulter Electronics). The following mean particle diameters were detected: 128 nm–100% Sty; 107 nm–75/25% Sty/*n*-BA; 74 nm–50/50% Sty/*n*-BA; 73 nm–25/75% Sty/*n*-BA; 72 nm–100% *n*-BA.

RESULTS AND DISCUSSION

In the previous studies, we³⁻⁶ established that the presence of the 1046 and 1056 cm⁻¹ bands at the F-A and F-S interfaces was attributed to the surfactant -SO₃⁻Na⁺ hydrophilic entities that formed hydrogen bonding with water molecules in the presence of copolymer EA/MAA latex carboxylic acid groups. In essence, such environment causes the S-O electronic structure to be so perturbed that the S-O stretching band, originally detected for SDOSS at 1050 cm⁻¹, splits into two bands: the 1046 cm⁻¹, due to associations with H₂O, and the 1056 cm⁻¹, due to hydrogen bonding with the COOH groups. In this study, we will use a similar approach and analyze the SDOSS surfactant exudation, but the focus will shift to Sty/*n*-BA copolymer latexes. Specifically, we will examine the effect of Sty/*n*-BA latex and the latex particle composition on the surfactant mobility and subsequent concentration changes at the F-A and F-S interfaces.

To set the stage for further analysis, surfactant exudation in the latex, prepared from a 50/50 Sty/*n*-BA monomer mixture, will be compared to the latex films, obtained by mixing equal amounts of latex particles containing 100% polystyrene and

100% poly (*n*-BA). Such an approach will allow us to maintain the same overall Sty/*n*-BA composition of the suspension, while varying the latex particle composition. Traces A, B, and C of Figure 1 illustrate perpendicular (TE) polarized ATR FT-IR spectra, recorded from the F-A interface of the 50/50 Sty/*n*-BA at 60, 50, and 40° angle of incidence. For reference purposes, the FT-IR spectrum of SDOSS of surfactant is shown in trace D, with the S—O stretching band at 1050 cm⁻¹. While polarized ATR FT-IR spectra were recorded to establish the orientation of surfactant hydrophilic groups, a spacial distribution of the SDOSS molecules at the F-A and F-S interfaces can be monitored by varying the angle of incidence⁹; the greater the angle of incidence, the shallower the depth of penetration, which, for this experimental setup, is approximately of 3–5 μm. It should be noted that all ATR spectra, in order to be corrected for spectral distortions resulting from optical effects,¹⁰ were corrected using the recently developed algorithm based on the Kramers–Kronig transform.¹¹ Analysis of the spectral region, presented in Figure 1, indicates that there is a relatively small amount of surfactant molecules present at this interface. This is demonstrated by the weak intensity of the 1046 cm⁻¹ band, and the practically undetectable intensity at 1056 cm⁻¹, the latter a result of the overlap with the significantly stronger neighboring 1066 cm⁻¹ latex copolymer band. Because the spectra recorded at various surface depths show

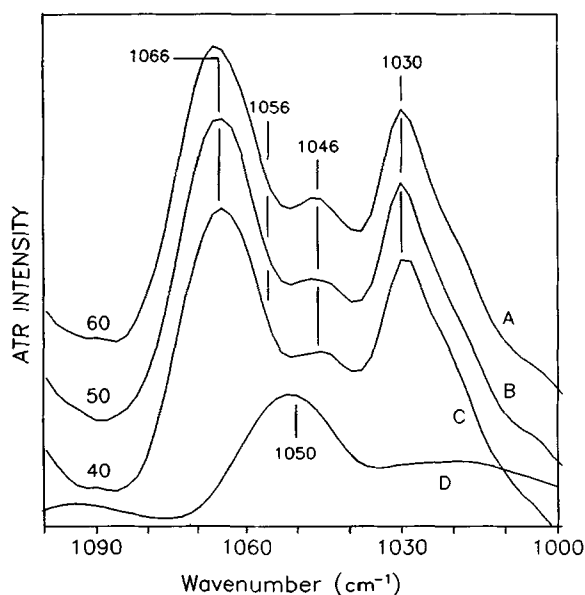


Figure 1 Perpendicular (TE) polarized ATR FT-IR spectra, recorded at various angles of incidence from the F-A interface of the 50/50 Sty/*n*-BA latex cast on PTFE: (trace A) 60°, (trace B) 50°, (trace C) 40°, and (D) transmission spectrum of SDOSS surfactant.

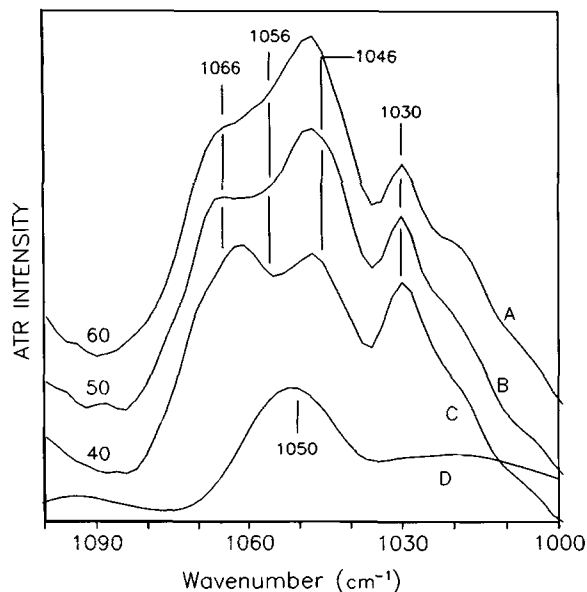


Figure 2 Perpendicular (TE) polarized ATR FT-IR spectra, recorded at various angles of incidence from the F-S interface of the 50/50 Sty/*n*-BA latex cast on PTFE: (trace A) 60°, (trace B) 50°, (trace C) 40°, and (D) transmission spectrum of SDOSS surfactant.

similar features, the concentration levels of the SDOSS surfactant at the F-A interface do not change across the examined film thickness. Although we indicated earlier that we shall use perpendicular and parallel polarized spectra to assess possible surfactant orientation changes at the F-A and F-S interfaces, parallel polarized spectra, recorded from the F-A interface for the 50/50 Sty/*n*-BA latex, show the same features and, therefore, are not presented.

The situation changes drastically when the same 50/50 Sty/*n*-BA latex is analyzed at the F-S interface. Perpendicular (TE) polarized ATR FT-IR spectra are shown in Figure 2. The spectral analysis, reported in Figure 2, as well as in the remaining figures of this article, has the same format as does the F-A interfaces in Figure 1, with traces A, B, and C corresponding to the increasing penetration depths, and trace D being the spectrum of SDOSS surfactant. Similar to the F-A interface spectra, the 1056 cm⁻¹ is not resolved, due to a strong overlap with the latex copolymer bands, but the 1046 cm⁻¹ band increases drastically as the depth of penetration decreases. This behavior is attributed to the increased concentration of the surfactant molecules at the F-S interface. As we recall the origin of the 1046 cm⁻¹ band, the existence of this band is attributed to the presence of water molecules near the SO₃⁻Na⁺ hydrophilic ends of the surfactant molecule. As the penetration depth increases (from spec-

trum A to C), the 1046 cm^{-1} band decreases, because the lower concentration levels of surfactant result in a fewer interactions in the bulk. Similar to Figure 1, when parallel polarized light is used for F-S interface analysis, no spectral differences are detected, indicating that the $\text{SO}_3^- \text{Na}^+$ groups have no preferential orientation at the F-A and F-S interfaces. Therefore, the spectra are not shown. However, as demonstrated by the spectral intensity changes recorded at various angles of incidence, there is a significant excess of surfactant molecules at or near the F-S interface.

Considering these observations, let us go back to the synthetic procedures employed in this study and realize that when the monomers are mixed before they are polymerized, the reactivity ratios, as well as other factors, will affect the latex copolymer particle composition. In the case of the 50/50 Sty/*n*-BA monomer mixture, discussed in Figures 1 and 2, an evaluation of the reactivities ratios indicates that, under continuous feed conditions, the latex particle composition will change across the particle. The reactivity ratios r_1 and r_2 ($r_1 = k_{11}/k_{12}$ and $r_2 = k_{22}/k_{21}$) for styrene/*n*-butyl acrylate vary, depending upon the source, ranging from 1.23 to 0.44 for r_1 , and from 0.29 to -0.106 for r_2 , respectively.¹² Without detailing the accuracy of the reactivity ratio determinations, the reactivity ratios favor styrene monomer reactions, to produce polystyrene over the styrene reactivity with *n*-butyl acrylate polymer. Based on these assessments, nonuniform type of the latex particle would be expected. However, because equal amounts of each monomer were introduced simultaneously, it is unlikely that the core of the latex particle predominantly contains polystyrene. Under such experimental conditions, it is possible that there is a diminishing gradient of polystyrene distribution (or increasing content of *n*-butyl acrylate segments) when proceeding toward the latex particle surface. Such a scenario is possible because styrene has higher hydrophobicity toward water, thus it will quickly migrate inside the surfactant micelles to polymerize. Although *n*-BA is less hydrophobic and exhibits less affinity towards reacting with styrene, it will still react. As a result, the latex particles, synthesized from a 50/50 mixture of Sty/*n*-BA, can be envisioned as having higher concentrations of *n*-BA near the surface and significantly fewer styrene blocks than are mixed in equal amounts of polySty and poly(*n*-BA) polymers. For such a latex copolymer, the data presented in Figures 1 and 2 indicate surfactant deficiency at the F-A interface and its excess at the F-S interface. Whereas the former can be attributed to the fact that SDOSS surfactant is more compatible with *n*-

butyl acrylate surface segments, the excess of surfactant at the F-S interface is a result of the ability of the surfactant to lower the interfacial surface tension between the copolymer and the PTFE. Similar observations were observed for ethyl acrylate/methacrylic acid/SDOSS latex.¹³ It should be noted that, when the interfacial surface tension attracts surfactant molecules, allowing them to assemble at the F-S interface, the effect of the water flux will be reduced, relative to the driving force induced by interfacial surface tension. The additional influence on the mobility and subsequent diffusion of the surfactant molecules will be derived from the mutual compatibility of SDOSS and the copolymer. In this context, compatibility should be understood not in a thermodynamic sense, but rather as a sufficient magnitude of favorable interactions, so that water flux will not carry surfactant molecules to the F-A interface.

In view of the above considerations, let us maintain the overall monomer ratio in the latex composition using the approach outlined in the experimental section, but vary the individual particle composition. This can be accomplished by mixing pure polystyrene and poly(*n*-butyl acrylate) latex particles in equal molar ratios (0.15 mol), and letting the mixture coalesce. Figure 3 illustrates the ATR FT-IR spectra, recorded from the F-A interface, obtained at various surface depths using parallel polarized light. It appears at the 1046 cm^{-1} band increases at shallower depths and, at same time, the 1056 cm^{-1} band become more pronounced. This observation indicates that there is not only a substantial amount of water and hydrogen-bonded acid groups, associated with the surfactant molecules near this interface, but also that the 1046 cm^{-1} band parallels the 1056 cm^{-1} counterpart, suggesting that the acid-surfactant interactions appear to be more pronounced.

At this point, it is appropriate to reconsider the results presented in Figure 1 and 2, and to remind ourselves that the 1056 cm^{-1} band was almost undetectable for the latex prepared from a 50/50 monomer mixture. Although heavy band overlap with the copolymer latex bands was identified as one issue, the 1056 cm^{-1} band, attributed to the $\text{SO}_3^- \text{Na}^+ \cdots \text{HOOC}$ -associations, is perhaps much weaker due to fewer acrylate groups available for these interactions. This, combined with the spectral overlap, is believed to be the reason for the inability to resolve the 1056 cm^{-1} band.

The 1046 and 1056 cm^{-1} bands are even stronger when the polarization of the IR incident beam changes to perpendicular. These results are shown in Figure 4. It is evident that the dominating bands

in the spectra are again at 1046 and 1056 cm^{-1} , indicating that, at the F-A interface, there are orientation changes of the $\text{SO}_3^- \text{Na}^+$ groups when the water-surfactant and acid-surfactant interactions occur. In contrast to the F-A interface showing an excess of surfactant, the F-S interface spectra exhibit only small amounts of surfactant at this interface. This is depicted in Figure 5 for the perpendicular polarized incident light. Again, because the spectra recorded using parallel polarization show essentially the same features, they are not presented.

As discussed earlier, mixing Sty and *n*-BA monomers prior to synthesis results in a composition gradient across the latex particle, whereas the mixture of polystyrene and poly(*n*-butyl acrylate) results in a uniform composition latex particles. Let us compare the results presented in Figures 1 and 4, and 2 and 5. For the randomly polymerized copolymer latex, no surfactant excess at either interface (Fig. 1) is detected. The situation changes drastically for the latex prepared from the mixture of polystyrene and poly(butyl acrylate) latex homopolymers: an excessive amount of the surfactant diffuses to the F-A interface (Fig. 4). At this point, the issue of compatibility and interfacial surface tension becomes essential. In the previous studies, we³⁻⁵ examined a few surfactants and established that when surfactant and copolymer latex are compatible, little or no exudation of the surfactant molecules is detected. In fact, among tested ionic and nonionic surfactants,

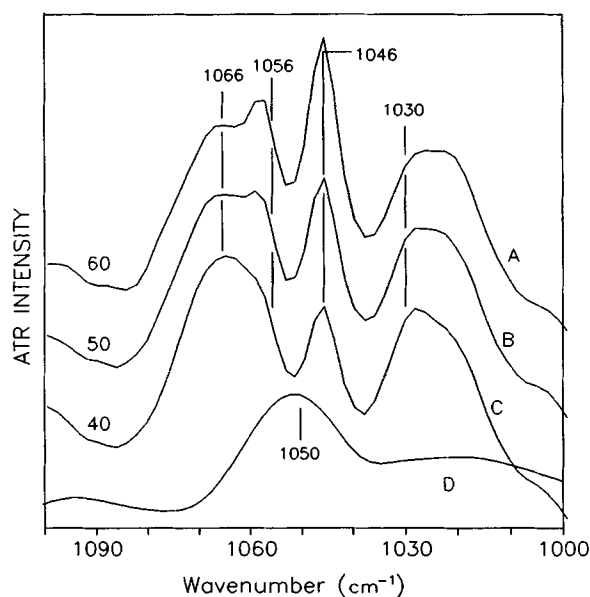


Figure 3 Parallel (TM) polarized ATR FT-IR spectra, recorded at various angles of incidence from the F-A interface of the mixture of polystyrene and poly(*n*-BA) latex cast on PTFE: (trace A) 60°, (trace B) 50°, (trace C) 40°, and (D) transmission spectrum of SDOSS surfactant.

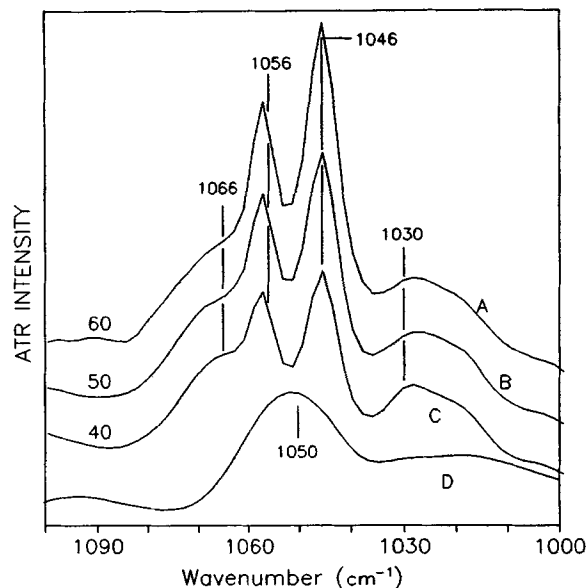


Figure 4 Perpendicular (TE) polarized ATR FT-IR spectra, recorded at various angles of incidence from the F-A interface of the mixture of polystyrene and poly(*n*-BA) latex cast on PTFE: (trace A) 60°, (trace B) 50°, (trace C) 40°, and (D) transmission spectrum of SDOSS surfactant.

the latter appeared to remain uniformly distributed throughout the coalesced film. We also identified that the surface tension of the substrate may have a significant effect, not only on the amount of sur-

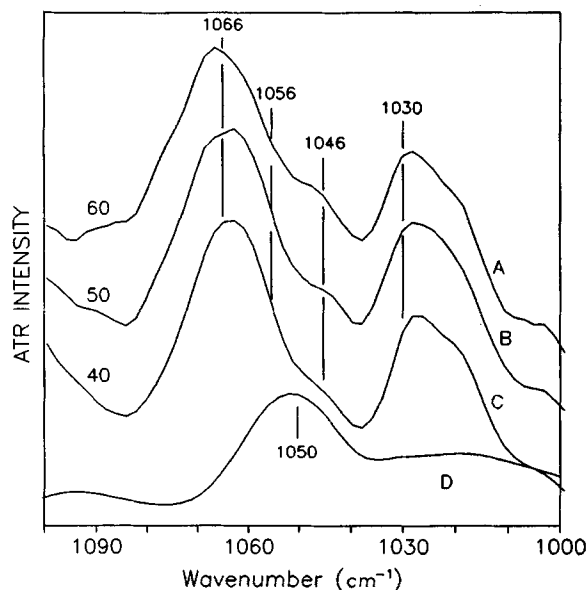


Figure 5 Perpendicular (TE) polarized ATR FT-IR spectra, recorded at various angles of incidence from the F-S interface of the mixture of polystyrene and poly(*n*-BA) latex cast on PTFE: (trace A) 60°, (trace B) 50°, (trace C) 40°, and (D) transmission spectrum of SDOSS surfactant.

factant exuded to a given interface, but may also significantly influence the direction of exudation. The latter is due to a magnitude of the interfacial surface tension. Since in this study PTFE was used as a substrate, its surface tension (approximately 18.5 mN/m) would have a similar effect on both latex films. However, when a 50/50 monomer mixture of Sty/*n*-BA is polymerized, more *n*-BA groups are at the latex particle surface, keeping the SDOSS surfactant at this surface, because the SDOSS surfactant is more compatible with *n*-BA. In contrast, when a mixture of pure polystyrene and pure poly(*n*-butyl acrylate) latex particles forms an aqueous suspension, those SDOSS molecules which served to stabilize polystyrene particles, will be expelled from the polystyrene surface, since SDOSS is relatively incompatible with polystyrene. This excess will be discreetly dispersed in water, which, upon water evaporation, will move to the F-A interface with water flux. In this case, water flux plays a significant role and the effect of interfacial surface tension is minimized because the presence of pure polystyrene and poly(*n*-butyl acrylate) particles at the PTFE surface altered the excess of surface energy.

If the above hypothesis is valid, let us disturb the latex mixture by modifying the latex composition by mixing equal amounts (0.1 mol) of 100% Sty,

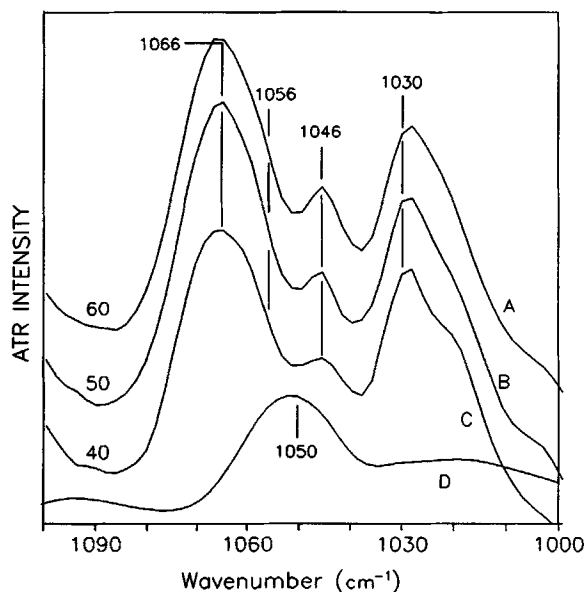


Figure 6 Parallel (TM) polarized ATR FT-IR spectra, recorded at various angles of incidence from the F-A interface of the Sty/*n*-BA latex, prepared by mixing equal amounts of 100% Sty, 75/25, 50/50, 25/75 Sty/*n*-BA, and 100% *n*-BA, and cast on PTFE: (trace A) 60°, (trace B) 50°, (trace C) 40°, and (D) transmission spectrum of SDOSS surfactant.

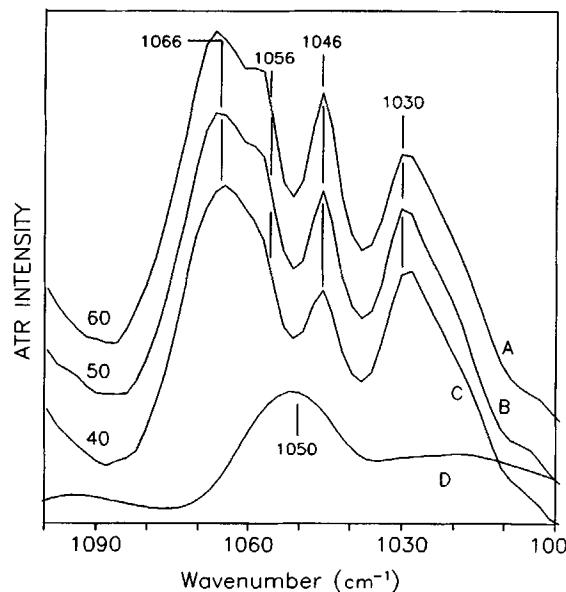


Figure 7 Perpendicular (TE) polarized ATR FT-IR spectra, recorded at various angles of incidence from the F-A interface of the Sty/*n*-BA latex, prepared by mixing equal amounts of 100% Sty, 75/25, 50/50, 25/75 Sty/*n*-BA, and 100% *n*-BA, and cast on PTFE: (trace A) 60°, (trace B) 50°, (trace C) 40°, and (D) transmission spectrum of SDOSS surfactant.

75/25, 50/50, 25/75% of Sty/*n*-BA, and 100% *n*-BA latex suspensions, prior to latex coalescence. From such a mixture, one would expect to have soft (pure poly(*n*-BA)), hard (pure polystyrene), and the latex particles, possibly containing an excess of Sty for a 75/25 composition, and an excess of poly(*n*-BA) for a 25/75 composition involved in the coalescence process. Figure 6 illustrates the spectra of such coalesced films, recorded from the F-A interface, using a perpendicular polarized light. Although, at first glance, the F-A spectra appear to have features similar to those recorded for the latex prepared from the 50/50 Sty/*n*-BA monomer mixture (Fig. 1), the 1046 cm⁻¹ band appears to increase as the penetration depth decreases. This is demonstrated by comparing trace C (40° angle of incidence) and trace A (60° angle of incidence). In contrast to the 50/50 Sty/*n*-BA monomer latex mixture, which did not indicate the F-A and F-S orientation changes, demonstrated by a lack of spectral changes between parallel and perpendicular polarized light measurements, Figure 7 shows a sequence of spectra obtained using perpendicular polarized incident light. It is apparent that the 1046 cm⁻¹ band significantly increases and, as the penetration depth decreases, the 1046 cm⁻¹ becomes stronger, and the 1056 cm⁻¹ band becomes more pronounced. It is apparent that for such a mixture

of latex particles, less surfactant exudation is observed. These results appear to fit between the previously discussed 50/50 monomer mixture (Figs. 1 and 2) and mixed polystyrene and poly(*n*-BA) latexes (Figs. 3 and 4). Similar to the previous findings, Figure 8 illustrates perpendicular polarized ATR FT-IR spectra, recorded at the F-S interface; almost no surfactant bands are detected at this interface, indicating that the effect of interfacial surface tension can be diminished by the latex particle composition. This, combined with the compatibility of latex components, will allow one to control surfactant as well as other small molecules' mobility in polymer networks.

CONCLUSIONS

Polarized ATR FT-IR spectroscopy was used to examine the effect of Sty/*n*-BA latex particle composition on the mobility and concentration of SDOSS surfactant molecules in Sty/*n*-BA latex films. This study shows that the latex particle composition may significantly influence the direction of surfactant exudation to the film-air or film-substrate interfaces. It appears that the magnitude and

the direction of exudation are affected by factors such as interfacial film-substrate surface tension, water flux during coalescence, and a chemical compatibility of the latex components. When a mixture of polystyrene and poly(*n*-BA) latex particles is coalesced, surfactant molecules are displaced from the polystyrene particles and exude to the film-air interface. For the same latex composition, but prepared by a semicontinuous polymerization of the styrene and *n*-butyl acrylate monomer mixture, the surfactant molecules are being attracted to the film-substrate interface, resulting from the excess of film-substrate interfacial energy.

The authors are thankful to Hitachi Chemical Company for supporting this research program and to the Miles Incorporated Foundation for the Summer Undergraduate Research Grant to the Department of Polymers and Coatings (JPK).

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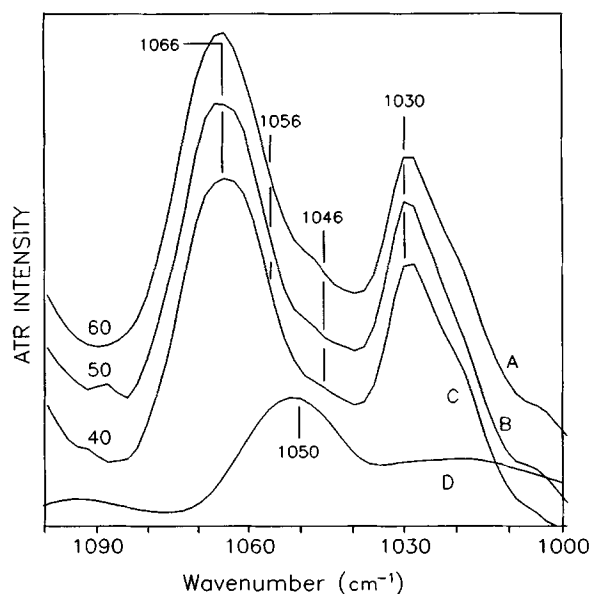


Figure 8 Perpendicular (TE) polarized ATR FT-IR spectra, recorded at various angles of incidence from the F-S interface of the Sty/*n*-BA latex, prepared by mixing equal amounts of 100% Sty, 75/25, 50/50, 25/75 Sty/*n*-BA, and 100% *n*-BA, and cast on PTFE: (trace A) 60°, (trace B) 50°, (trace C) 40°, and (D) transmission spectrum of SDOSS surfactant.

Received March 15, 1993

Accepted March 15, 1993