Polymer Vol. 37 No. 7, pp. 1117–1122, 1996
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0032-3861/96/$15.00+0.00

The effect of end-sulfonated polystyrene on the interfacial tension of nylon-6/polystyrene blends

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(Received 25 April 1995; revised 28 August 1995)

The effect of reactive interfacial agents on the interfacial tension of nylon-6/polystyrene (PS) blends was studied. Two types of Li end-sulfonated polystyrene (e-SPS), namely (ω-SPS and α,ω-SPS) were used as the reactive interfacial agents. The interfacial tension in the melt state was measured by the breaking-thread method. As the e-SPS content was increased, the interfacial tension of the nylon-6/PS blend decreased and reached a saturation value, as observed in the case of block copolymers. An effect of the structure of e-SPS on the interfacial tension was observed: for ω-SPS, the saturation value of the sulfonate group concentration was very low ($2 \times 10^{-5}\,\mathrm{mol\,l^{-1}}$) and no molecular-weight effect was observed; however, for α,ω-SPS, the saturated interfacial tension was attained at a relatively higher sulfonate group concentration than that of ω-SPS, while the lower molecular weight of α,ω-SPS reduced the saturated interfacial tension to a lower value.

(Keywords: interfacial tension; nylon-6/polystyrene blend; end-sulfonated polystyrene)

INTRODUCTION

The interfacial tension between two different polymers has been an important subject of study due to its effect on the blend morphology. In addition efforts at reducing the interfacial tension of the immiscible blends have been carried out by modification of the interface using interfacial agents which interact physically or chemically at the interface[1–3].

Block copolymers and graft copolymers have been used in efforts to compatibilize immiscible polymers where the chemical compositions of the latter are equal to or compatible with the copolymer blocks. Hence, their effect on the interfacial tension has been studied both theoretically and experimentally[4–8]. However, block copolymers possess several limitations as interfacial agents[9]. Therefore, much recent research has been concentrated on the use of reactive interfacial agents which react with the functional group in one component of the blend, thus resulting in the *in situ* formation of block or graft copolymers at the interface. It is also reported that the mechanical properties of the polymer blends is greatly improved by the use of such reactive interfacial agents[10–12].

It is known that interfacial agents where the interfacial interaction is by hydrogen bonding or ion–dipole interaction act as reactive compatibilizers[13–19]. Sullivan and Weiss reported that the compatibility of amorphous polyamide and sulfonated PS ionomer (SPS) is improved by hydrogen bonding between the amide and sulfonate groups, which is confirmed by studies of the morphology and Fourier transform infra-red (*FT* i.r.) spectroscopy of the blends[13]. Kramer and coworkers found that there are more block copolymers at the interface in a blend system which is hydrogen bonded with block copolymers at the interface than in the corresponding blend system which is not hydrogen bonded with the block copolymers[19]. Therefore, it is of interest to study the interfacial properties of immiscible polymer blends which have been compatibilized with reactive interfacial agents.

Our study has focused on the effect of reactive interfacial agents on the interfacial tension of immiscible blends. Nylon-6 and polystyrene (PS) are common commercially available polymers and their blends are immiscible. For the nylon-6/PS blends, ω-Li-sulfonated PS (ω-SPS) and α,ω-Li-sulfonated PS (α,ω-SPS) were used as the reactive interfacial agents. The breaking-thread method, which is known to be suitable for high-molecular-weight system[8,20], was used for interfacial tension measurements. The effect of structure and sulfonate group concentration of the e-SPS on the interfacial tension were also investigated.

The breaking-thread method

A cylindrical Newtonian fluid, in a Newtonian fluid

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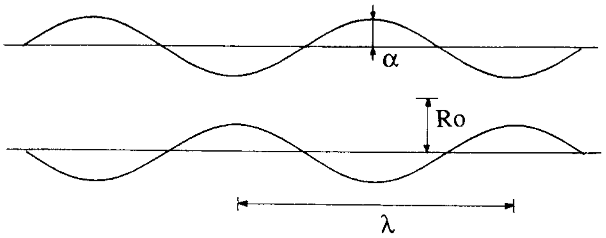

Figure 1 Sinusoidal distortions on a liquid cylinder

medium in which no flow field is present experiences sinusoidal distortions which result in breakup into spheres (*Figure 1*). This phenomenon occurs in order to equilibrate the viscous force and the interfacial tension between the cylindrical fluid and matrix fluid. Only those distortions with a wavelength (λ) longer than the initial thread circumference ($2\pi R_0$) continue to grow, due to the decrease of interfacial area.

According to Tomotika's original analysis[21], the distortions of the thread grow exponentially with time (equation (1)) and the interfacial tension can be expressed as shown in equation (2), as follows:

$$\alpha = \alpha_0(\exp(qt)) \tag{1}$$

$$\gamma = \frac{2q\eta_c R_0}{\Omega(\lambda, p)} \tag{2}$$

where α_0 and α are the original amplitude and the amplitude at time t, respectively, of the thread q is the growth rate of the thread distortion, γ is the interfacial tension between the thread and matrix, R_0 is the original radius of the thread, η_c is the zero shear viscosity of the matrix phase, and $\Omega(\lambda, p)$ is the function of the viscosity ratio of thread and matrix ($p = \eta_d/\eta_c$, where η_d is the zero shear viscosity of the thread phase) and the wavelength of thread distortion.

In contrast to other methods for interfacial tension measurement, the breaking-thread method does not require a knowledge of the polymer density in the melt and equilibrium is attained in a relatively short time. Thus this method is found to be suitable for polymer systems with small density differences or with high molecular weights[8,20].

EXPERIMENTAL

Materials

Polystyrene (PS, Dow Styron 685) and nylon-6 (Tong Yang Nylon 1011BRT) were used as the matrix material and the thread material for the breaking-thread method, respectively. Nylon-6 was dried under vacuum at 80°C for 2 days before use. Anionically polymerized Li end-sulfonated PS (ω-SPS, α,ω-SPS) with narrow polydispersities[22] were supplied by KIST Polymer Research Division. Tetrahydrofuran and dimethylformamide were used without further purification. The number-average molecular weights of polymers used in this study are summarized in *Table 1*.

Table 1 Characteristics of the materials used in this study

Material	$\overline{M}_n$	η^a (P)	Loss angle, δ (degree)
Nylon-6^b	16 500	3 331	90
PS^c	324 000	36 670	84.4
ω-sulfonated PS			
ω-SPS20	20 000	–	–
ω-SPS100	100 000	–	–
α,ω-disulfonated PS			
α,ω-SPS16	16 200	–	–
α,ω-SPS100	100 000	–	–

[a] Measured at 240°C; P = 0.1 N s m^{-2}
[b] Tong Yang Nylon 1011BRT
[c] Dow Styron 685

Zero shear viscosity measurements

The zero shear viscosities of PS (η_c) and nylon-6 (η_d) were measured by using a Rheometrics dynamic spectrometer (RDS-II, Rheometrics Inc.) over the shear rate range from 10^{-1} to 10^3 rad s^{-1} under a nitrogen atmosphere at 240°C. Since the content of e-SPS in the e-SPS/PS blends was very low, the zero shear viscosities of the blends were assumed to be the same as that of PS. The zero shear viscosity values which were obtained at 10^{-1} rad s^{-1} are given in *Table 1*.

Sample preparation

PS and e-SPS were blended in a THF/DMF (vol/vol = 9/1) solution, followed by precipitation in methanol. The precipitates were dried in a vacuum oven at 80°C for 2 days. The dried e-SPS/PS blends were compression moulded into sheets with a thickness of 0.5 mm using a Ferrotype plate under a hot press and then cut into slices with dimensions $5 \times 5 \times 0.5$ mm^3. Nylon-6 thread was prepared by drawing a small amount of resin melted on a hot plate; the thread diameter range was 20–50 μm. A short thread with a length of $\sim$ 3 mm was carefully placed between two slices of the e-SPS/PS blend and the sandwiched sample was then located between a glass slide and a cover glass.

Interfacial tension measurements

The breaking-thread method was performed under an optical microscope (Axioplan, Zeiss) with a heating stage. The sample was heated to 180°C at a heating rate of 3°C min^{-1} and then heated to 240°C at 1°C min^{-1} under a N_2 atmosphere. When the distortions of the thread started to increase, photographs were taken at regular time intervals (*Figure 2*). The distortion amplitude (α) was calculated by using equation (3) and the plot of the relative amplitude (log $(2\alpha/D_0)$) against time gave the growth rate(q) (equation (4)) from which the interfacial tension (γ) was calculated (equation (2)). The values of $\Omega(\lambda, p)$ in equation (2) were obtained from Figures 1 and 2 of Chappelear's paper[20]:

$$\alpha = \frac{b-a}{4} \tag{3}$$

$$\log\left(\frac{2\alpha}{D_0}\right) = \frac{q}{2.303}t + C \tag{4}$$

where a and b are the minimum and maximum diameters, respectively, D_0 is the original diameter of the thread, and $C = \log(2\alpha_0/D_0)$.

RESULTS AND DISCUSSION

The interfacial tension between nylon-6 and PS obtained

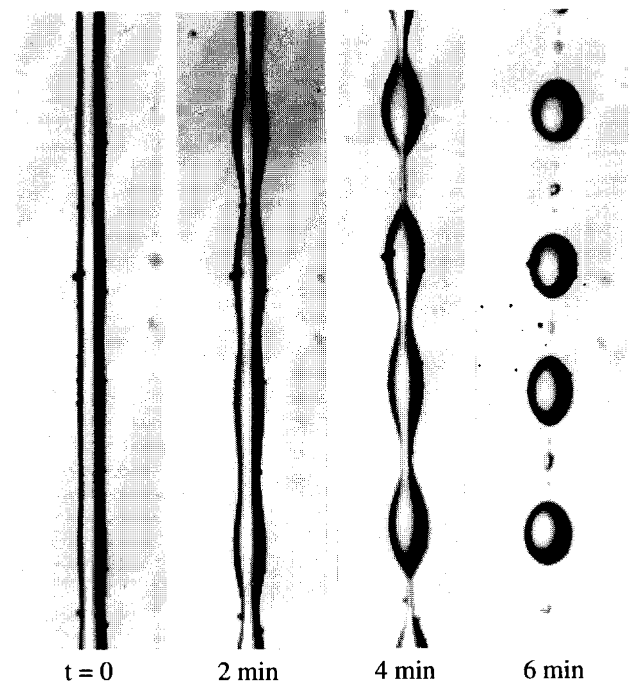

Figure 2 Typical examples of sinusoidal distortions of nylon-6 in e-SPS/PS blends (temperature = 240°C; $D_0 = 41\,\mu$m)

from this study was $7.3\,\mathrm{dyn\,cm^{-1}}$. This value is much lower than the previously reported value of $20\,\mathrm{dyn\,cm^{-1}}$, also measured via the breaking-thread method by Elemans *et al.*[8]. It is conjectured that this difference resulted from the viscosity ratio (p) difference. To obtain the best results from the breaking-thread method, the value of L/D_0 for the thread should be much larger than 60 and the viscosity ratio (η_d/η_c) should be much smaller than unity[8]. Since the viscosity ratio in our experiment was 0.09, which is smaller than that used by Elemans *et al.* (0.425), it is estimated that $7.3\,\mathrm{dyn\,cm^{-1}}$ is a more reliable value for the interfacial tension of the nylon-6/PS system.

The predicted interfacial tension values of nylon-6/PS obtained from various theories are shown in *Table 2*. According to the fractional polarity theory, the interfacial tension between immiscible polymer melts is well approximated by the harmonic mean equation[23]:

$$\gamma = \gamma_1 + \gamma_2 - 4\left(\frac{\gamma_1^{d}\gamma_2^{d}}{\gamma_1^{d} + \gamma_2^{d}} + \frac{\gamma_1^{p}\gamma_2^{p}}{\gamma_1^{p} + \gamma_2^{p}}\right) \tag{5}$$

where γ_1 and γ_2 are the surface tensions of the two polymers and the superscripts d and p refer to the dispersion and polar components of the surface tension, respectively. The tabulated values of γ^d and γ^p taken from the literature[26] were used in the calculation, and $2.8\,\mathrm{dyn\,cm^{-1}}$ was obtained for the interfacial tension of nylon-6/PS at 240°C. In addition, the theories of Helfand and Tagami[24] and Helfand and Sapse[25], which were expressed as equations (6) and (7), gave values of 3.24 and $3.43\,\mathrm{dyn\,cm^{-1}}$, respectively:

$$\gamma_\infty = \rho_0 bkT(\chi_{AB}/6)^{1/2} \tag{6}$$

$$\gamma = (2/3)kT\alpha^{1/2}\left(\frac{\beta_A^3 - \beta_B^3}{\beta_A^2 - \beta_B^2}\right) \tag{7}$$

Table 2 Prediction of values for the interfacial tension of nylon-6/PS using various theories

Theory	$\gamma_{\mathrm{PS,\,nylon\text{-}6}}$ (dyn cm^{-1})	Ref.
Fractional polarity theory	2.8	23
Helfand-Tagami	3.3	24
Helfand–Sapse	3.5	25
Present experiments	7.3	–

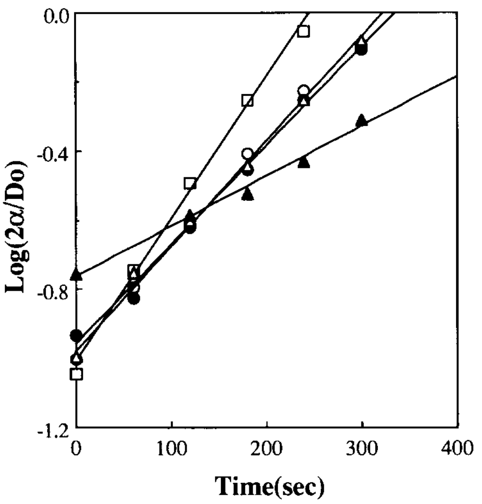

Figure 3 Relative amplitude vs. time for nylon-6/PS systems containing e-SPS (1 wt% e-SPS content in the PS matrix): (□) nylon-6/PS; (○) ω-SPS100; (●) ω-SPS20; (△) α,ω-SPS100; (▲) α,ω-SPS16

$$\alpha = \chi_{AB}/V_r \tag{8}$$

$$\beta_K = (1/6)\rho_{0K}b_K^2 \tag{9}$$

where γ_∞ is the interfacial tension at infinite molecular weight, ρ_0 and ρ_{0K} are the pure densities of a monomeric unit of the polymer and polymer K, respectively, b and b_K are the statistical segment (monomer) lengths of the polymer and polymer K, respectively, and V_r is the molar volume of the reference segment. The values for χ_{AB} in equations (6) and (8) were obtained from Hildebrand's solubility parameter relationship ($\delta_{PS} = 9.1$, $\delta_{N6} = 6.7$ (in $\mathrm{cal\,cm^{-3}})^{1/2}$)[27]. Although these theories involve certain errors in themselves, they can predict approximate values for the interfacial tension, and our result is closer to these predicted values. Therefore, it is inferred that the value for the interfacial tension between nylon-6 and PS obtained from our experiments is more reliable than that reported by Elemans *et al.*[8].

Figure 3 shows the relative amplitude *versus* time for nylon-6 thread which was embedded in a PS matrix containing 1 wt% e-SPS. A tendency for a reduction of the interfacial tension can be predicted from the slopes of the lines from equation (4). The slope of the nylon-6/pure PS curve is the steepest, with the slope decreasing as e-SPS is added. Therefore, the interfacial tension was reduced by the reactive interfacial agent, as expected. In addition, the interfacial tension of the α,ω-SPS16 system is lower than those of the ω-SPS100, α,ω-SPS20, and α,ω-SPS100 systems, where the interfacial tensions of the latter are similar to one another.

When the breaking-thread method is applied to systems which have reactive interfacial agents at the

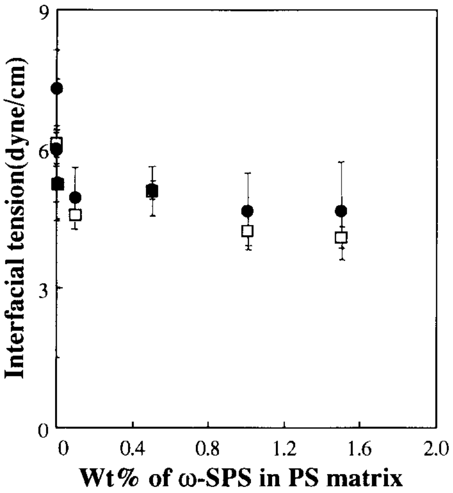

Figure 4 Interfacial tension as a function of ω-SPS content in the PS matrix: (●) ω-SPS100; (□) ω-SPS20

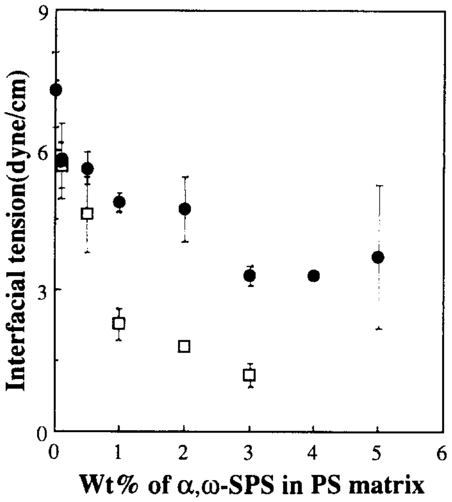

Figure 5 Interfacial tension as a function of α,ω-SPS content in the PS matrix: (●) α,ω-SPS100; (□) α,ω-SPS16

interface, one should be cautious about two particular points. The first of these is that the reaction rate may affect the growth rate of the thread distortion, i.e. if the interfacial reaction is completed below the thread-distortion temperature, then the interfacial reaction rate is meaningless. However, if the reaction temperature is similar to the distortion temperature, or if the reaction rate is very slow and the reaction is not completed at the thread-distortion temperature, then the growth rate of the thread distortions may decrease as the interfacial reaction proceeds. In this study, the specific interactions between nylon-6 and e-SPS, which are known to involve hydrogen bonding and ion–dipole interactions[13,14,16–18], require low activation energies for interaction, and therefore we have assumed that the interfacial interaction is in a state of equilibrium at 240°C; the plots showing a constant slope with time in *Figure 3* verify our assumption.

The other point to note is that it is difficult to obtain uniform distortions along the thread for a reactive interface, as was reported by Elemans *et al.* who studied the PS high-density polyethylene (HDPE) system with PE-*b*-PS block copolymers as interfacial agents[8]. They chose those measurements with the best agreement between X_{exp} and X_{m} where X is the dimensionless wave number, $2\pi R_0/\lambda$, X_{exp} is X from the experiments and X_{m} is that value of X which corresponds to the dominant wavelength (λ_{m}) at which the amplitude grows fastest. In this present study, there were difficulties in obtaining uniform distortions at high sulfonate concentrations and large scatterings in the data show that the breaking-thread method has a limit to measuring the interfacial tension of reactive interfaces at high concentrations of the reactive functional groups. For these reasons, four measurements with the best agreement between X_{exp} and X_{m}($X_{\mathrm{m}} = 0.6$ in this study) for one system were selected for calculating the interfacial tension.

The interfacial tension as a function of the e-SPS content for two different molecular-weight samples of e-SPS are given in *Figures 4* and *5*. From *Figure 4*, it can be seen that the interfacial tension decreases rapidly at very low ω-SPS contents (less than 0.1 wt%) and levels off to a saturation value of 4.5 dyn cm^{-1} as the ω-SPS content increases. The reduction in interfacial tension is ~40% for ω-SPS. Anastasiadis *et al.*[6] obtained an interfacial tension reduction of 50% when using PS-*b*-polybutadiene (PBD) block copolymer in the PS/PBD system. Their results showed that the interfacial tension reduction does not occur above the critical micelle concentrations (CMCs) of the block copolymers. Fleischer *et al.*[9] reported that interfacial modification with end-functionalized interfacial agents reduces the interfacial tension by up to 70%. They used carboxy-terminated polybutadiene (PBD–COOH) and amine-terminated polydimethyl siloxane (PDMS–NH_2) as the interfacial agents for reducing the interfacial tension of the PBD/PDMS system. Comparing our results for ω-SPS with the above-mentioned results of other groups, ω-SPS is the effective interfacial agent for reducing the interfacial tension of the nylon-6/PS system with the trend in interfacial tension reduction being similar to the case with block copolymers.

Figure 5 shows that the interfacial tension decreases to the saturation value at a content greater than 3 wt% of α,ω-SPS. Although the α,ω-SPS16 system shows no data above a content of 3 wt%, it is estimated that the value for the interfacial tension may be saturated at around this level. Furthermore, at this same content, α,ω-SPS16 shows a lower interfacial tension than α,ω-SPS100, due to its higher sulfonate group concentration. At the saturation value the interfacial tension reductions are 50% for α,ω-SPS100 and 84% for α,ω-SPS16. The effects of α,ω-SPS on the interfacial tension are different from those of ω-SPS. It is conjectured that these features are due to their structural differences and different molecular weights.

There may be three types of interaction in our nylon-6/PS/e-SPS blends, namely hydrogen bonding and ion–dipole interactions between the sulfonate groups and the amide groups at the interface, which directly affect the interfacial tension, plus ionic bonding between the sulfonate groups in the matrix. Although the exact interphase structure is not clear at this present time, near the interface the molar ratio of amide groups to sulfonate groups is much higher than unity before interactions begin to occur and relatively large quantities of the sulfonate groups near the interface may interact with the

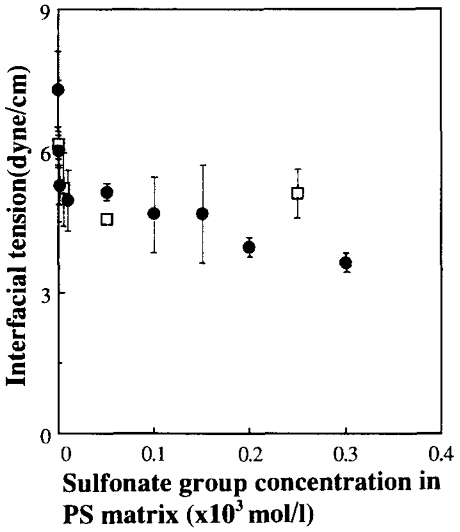

Figure 6 Interfacial tension as a function of the sulfonate group concentration of ω-SPS: (●) ω-SPS100; (□) ω-SPS20

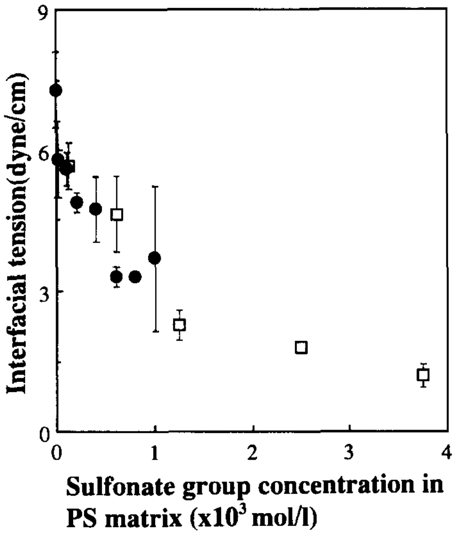

Figure 7 Interfacial tension as a function of the sulfonate group concentration of α, ω-SPS: (●) α, ω-SPS100; (□) α, ω-SPS16

amide groups, which results in a reduction in the interfacial tension. Ionic bonding between sulfonate groups is well known[28] and in this system it is estimated that the sulfonate group association is occurring above the critical concentration of e-SPS. In order to see this concentration effect more clearly, the interfacial tension as a function of sulfonate concentration is plotted in *Figures 6* and *7*.

Figure 6 shows that the interfacial tension is saturated at very low concentrations of sulfonate groups (2×10^{-5} mol l^{-1}) and the saturation value of the interfacial tension is determined by the sulfonate group concentration, i.e. the molecular weight of ω-SPS does not seem to affect the interfacial tension. At concentrations below the saturation concentration, it would be difficult for ω-SPS units in the matrix to associate with each other due to their extremely low concentration. Hence, up to the critical concentration the interfacial tension decreases as the sulfonate concentration increases. However, if association of sulfonates in the matrix is possible, no further interfacial tension reduction can occur. In addition, associated ω-SPS cannot act as a reactive interfacial agent since the functionality is destroyed by association in the form of a doublet. Therefore, for ω-SPS the interfacial tension is saturated at the low concentration of sulfonate groups at which the association of these groups occurs, and thus only the sulfonate concentration affects the interfacial tension.

On the other hand, α, ω-SPS showed a somewhat different behavior to ω-SPS (*Figure 7*). The interfacial tension reaches its saturation value at around 6×10^{-4} mol l^{-1} for α, ω-SPS100 and at a concentration of greater than 3.8×10^{-3} mol l^{-1} for α, ω-SPS16. These saturation concentrations are much higher than that found for ω-SPS. Moreover, unlike ω-SPS, the molecular weight of α, ω-SPS affects the saturation value of the interfacial tension. It is speculated that this difference results from the presence of 'living' sulfonates in the α, ω-SPS associated units. The α, ω-SPS species may also associate in the form of doublets at low sulfonate concentrations, at a similar level to the association concentration found for ω-SPS. Although α, ω-SPS association occurs, two sulfonate groups per doublet remain and these can continue to act as reactive interfacial agents. Thus, the interfacial tension decreases continuously with increasing sulfonate group concentration, due to these 'living' sulfonates. As the concentration of α, ω-SPS increases, the association of doublets or the formation of multiplets reduces the concentration of active sulfonate groups near the interface, which results in no further reduction in the interfacial tension. Therefore, α, ω-SPS induces the saturated interfacial tension condition at higher sulfonate group concentrations than ω-SPS.

CONCLUSIONS

The effect of e-SPS on the reduction in interfacial tension of nylon-6/PS has been investigated by using the breaking-thread method. The interfacial tension between PS and nylon-6 obtained from this study was 7.3 dyn cm^{-1}. Although e-SPS has a low sulfonate group concentration, it was found to be effective in reducing the interfacial tension of the nylon-6/PS blend. The interfacial tension behaviour was similar to that observed in the case of block copolymers in which the interfacial tension decreases and reaches the saturation value. The effect of ω-SPS on the interfacial tension was found to be different from that of α, ω-SPS. For ω-SPS, the saturation sulfonate group concentration was very low (2×10^{-5} mol l^{-1}) and a molecular-weight effect was not observed. However, for α, ω-SPS the saturated interfacial tension was attained at relatively higher sulfonate group concentrations than ω-SPS, with the lower-molecular-weight α, ω-SPS sample giving a lower value for the saturated interfacial tension. It is proposed that these differences resulted from the structural differences between ω-SPS and α, ω-SPS.

ACKNOWLEDGEMENT

This work was supported by a research grant from the Korea Science and Engineering Foundation (Grant No. 93-03-00-02).

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