Oil Absorbents Based on Styrene–Butadiene Rubber

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ABSTRACT: Four oil absorbents based on styrene–butadiene (SBR)—pure SBR (PS), 4-tert-butylstyrene–SBR (PBS), EPDM–SBR network (PES), and 4-tert-butylstyrene-EPDM-SBR (PBES)—were produced from crosslinking polymerization of uncured styrene–butadiene rubber (SBR), 4-tert-butylstyrene (tBS), and ethylene–propylene–diene terpolymer (EPDM). The reaction took place in toluene using benzoyl peroxide (BPO) as an initiator. Uncured SBR was used as both a prepolymer and a crosslink agent in this work, and the crosslinked polymer was identified by IR spectroscopy. The oil absorbency of the crosslinked polymer was evaluated with ASTM method F726-81. The order of maximum oil absorbency was PBES > PBS > PES > PS. The maximum values of oil absorbency of PBES and PBS were 74.0 and 69.5 g/g, respectively. Gel fractions and swelling kinetic constants, however, had opposite sequences. The swelling kinetic constant of PS evaluated by an experimental equation was $49.97 \times 10^{-2} h^{-1}$. The gel strength parameter, *S*, the relaxation exponent, *n*, and the fractal dimension, *d_f*, of the crosslinked polymer at the pseudo-critical gel state were determined from oscillatory shear measurements by a dynamic rheometer. The morphologies and light resistance properties of the crosslinked polymers were observed, respectively, with a scanning electron microscope (SEM) and a color difference meter. © 2003 Wiley Periodicals, Inc. J Appl Polym Sci 89: 1818–1824, 2003

Key words: oil absorbent; styrene–butadiene rubber; swelling kinetic constant; gel strength

INTRODUCTION

A variety of oil absorbents have been widely used to absorb oil-like liquids,^{1,2} especially for oil and some environmental hormones spilled on water. These are crosslinked polymeric networks with hydrophobicity. Among them, alkylacrylate and aromatic polymers with nonpolar side chain have been attracting more attention by scientific and applied research groups.^{3–6} It is known that there are a lot of double bonds on the main chains of uncured styrene-butadiene rubber (SBR) and that vulcanized SBR has excellent mechanical properties. Ethylene-propylene-diene terpolymer (EPDM) has high elasticity and outstanding resistance to degradation by heat, light, oxygen, and ozone. In this work uncured SBR was used not only as a polymeric monomer but also as a crosslink agent. Elastic polymer networks were prepared with graft crosslinking polymerization of SBR, 4-tert-butylstyrene (tBS), and EPDM. The reaction took place in a suitable solvent using benzoyl peroxide (BPO) as an initiator. The crosslinked polymer was identified by IR spectroscopy. The oil absorbencies of these oil absorbents were measured with ASTM method F726-81, and the swelling kinetic constants were evaluated by an experimental equation. The gel strength parameter, *S*, the relaxation exponent, *n*, and the fractal dimension, d_{f} , at the pseudo-critical state, light resistance, and morphologies of the crosslinked polymers were investigated.

EXPERIMENTAL

Materials

4-*tert*-Butylstyrene (tBS; Aldrich Chem., Milwaukee, WI) was purified by standard procedures. Benzoyl peroxide (BPO; Aldrich Chem.) was recrystallized from a methanol solution. EPDM, which has ethylidene norbornene as a termonomer (Aldrich, 50:50 ethylene:propylene, $M_n = 50,500$, $M_w = 102,000$), and SBR (Kosyn Co., bound styrene 23.5%, $M_n = 95,000$, $M_w = 520,000$), was used as received. All other chemicals were distilled prior to use.

Instrumentation

Infrared (IR) spectra were obtained with a Perkin– Elmer 1330 spectrometer. The morphologies of polymers were observed with a Hitachi S-4200 (Japan) scanning electron microscope. Light resistance was determined using a Fade-o-Meter (Atlas) (at 60°C and 65% relative humidity) and a color difference meter (NP-101DP). Oscillatory shear measurements were conducted in a Rheometrics RDA-II dynamic spec-

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trometer with parallel-plate geometry (25 mm in diameter) at 25°C.

Procedures

Preparation of crosslinked SBR (PS)

A given amount of uncured SBR was dissolved in a certain volume of toluene in a flask equipped with a modified Hopkins cooler, thermometer, and nitrogen gas inlet. The amounts of solvent used were 5, 10, 15, 20, 25, 30, and 35 mL/g of polymer. BPO (2.5 wt %), based on the weight of SBR, was introduced into the solution. The reaction lasted 48 h at 70°C. The product of the reaction was precipitated in excess methanol with stirring. The precipitate was collected by filtration and then dried in a vacuum oven until it was a constant weight. It was further extracted with tetrahydrofuran (THF) for 1 day to obtain sol-free crosslinked polymer SBR.

Preparation of crosslinked EPDM-SBR (PES)

EPDM and uncured SBR with a given weight ratio were dissolved in a determined volume of toluene in a flask equipped with a modified Hopkins cooler, thermometer, and nitrogen gas inlet. BPO (2.5 wt %), based on the total weights of EPDM and SBR, was added to the solution. The reactions were kept at 70°C for 48 h. The rest procedure was the same as the above.

Preparation of crosslinked tBS-SBR (PBS)

tBS and uncured SBR, with a given weight ratio, were dissolved in a determined volume of toluene in a flask equipped with a modified Hopkins cooler, thermometer, and nitrogen gas inlet. BPO (2.5 wt %), based on the total weights of tBS and SBR, was added to the solution. The reactions were kept at 70°C for 48 h. The rest of the procedure was the same as the above.

Preparation of crosslinked tBS-EPDM-SBR (PBES)

Except that tBS was added as an additional monomer into the reaction system, the procedure used was the same as that used for the preparation of crosslinked EPDM–SBR.

The gel fraction of polymer was estimated using the following equation:

Gel fraction (%) =

$$\frac{\text{Weight of dried gel after extraction}}{\text{Weight of polymer before extraction}} \times 100 \quad (1)$$

Oil-absorption test

The oil absorbencies of the crosslinked polymers obtained from graft crosslinking polymerization were determined using ASTM F726-81. First, 0.1 g of polymer was put into a stainless-steel mesh ($4 \times 4 \times 2$ cm) that had been immersed in oil solution (10 wt % crude oil diluted with toluene) and weighed beforehand. The sample and the mesh were picked up from the oil together, drained for 20 s, tapped with filter paper to remove excess oil from the bottom of the mesh, and then weighed on a balance. The oil absorbency was calculated by following formula:

Oil absorbency
$$(g/g) =$$

$$\frac{\text{Weight of absorbed oil in sample}}{\text{Weight of sample before oil-absorption}} (2)$$

To study the kinetics of oil absorption, the above measurements were repeated from time to time. To determine maximum oil absorbency, the tests were allowed to stand for more than 7 days. The maximum oil absorbency is the value that has reached the point of absorptive equilibrium of the polymer.

Swelling kinetics

According to Yao and Zhou,⁷ the swelling rate of the crosslinked polymers can be described by the following experimental equation:

$$dQ/dt = K(Q_{max} - Q)$$
(3)

where Q_{max} is the maximum oil absorbency, Q is the characteristic oil absorbency, and K is the swelling kinetic constant.

Integration from eq. (3) gives

$$-\ln(Q_{\max} - Q) = Kt + C \tag{4}$$

where *t* is the characteristic swelling time and *C* is the integration constant. By considering that

$$\ln(Q_{\max}/(Q_{\max}-Q)) = Kt \tag{5}$$

The characteristic time is defined as that at Q = 0.632 Q_{max} .⁸

Oscillatory shear experiments

The material at gel point, termed the critical gel (CG), is in an intermediate state between liquid and solid. In dynamic mechanical experiments^{9–24} the CG has been shown to have a power law behavior in frequency. The storage modulus, G', and the loss modulus, G'', at gel point are

$$G'(\omega) = G''(\omega) / \tan(n\pi/2) = \Gamma(1-n)\cos(n\pi/2)S\omega^n$$
(6)

where the gel strength, *S*, and relaxation exponent, *n*, are the only material parameters; $\Gamma(1 - n)$ is the gamma function, and ω is the frequency. The relaxation exponent is physically restricted to 0 < n < 1. The gel strength is known as gel stiffness and has rather unusual dimensions (Pa sⁿ). Power law mechanical behavior is an expression of mechanical selfsimilarity and is a consequence of structural self-similarity, which evolves during crosslinking.¹⁰ Theories of gelation describe the cluster growth process. Muthukumar²⁵ developed a theoretical model in which it is assumed the variations in the strand length between crosslinking points of the CG network give rise to changes in the excluded volume interactions. The conjecture is that increasing strand length will enhance the excluded volume effect. If the excluded volume interaction is fully screened, the relaxation exponent for a polydisperse system may be written as

$$n = d(d + 2 - 2d_f) / 2(d + 2 - d_f)$$
(7)

where d (d = 3) is the spatial dimension and d_f is the fractal dimension that relates the mass of a molecular cluster to its radius of gyration by $R^{d_f} \sim M$.

In this work the gel strength, S, the relaxation exponent, n, and the fractal dimension, d_{fr} of the polymer and some composites in the pseudo-critical gel state were determined from dynamic mechanical experiments. Here, the pseudo-critical gel means the polymer network from which all sol materials were removed and that was then immersed in a good solvent until there was swelling equilibrium. The oscillatory shear measurements were performed within the linear viscoelastic regime, in which the storage (G') and loss (G'') moduli were independent of the strain amplitude, that is, the G' and G'' curves were parallel to each other, and power laws in frequency were observed over the considered frequency domain.

Oscillatory shear measurements were conducted in a Rheometrics RDA-II dynamic spectrometer with parallel-plate geometry (25 mm in diameter) at 25°C. All samples were immersed in toluene over 7 days until equilibrium swelling was reached and then were cut to a size 2 mm thick and 25 mm in diameter. The values of the strain amplitude were checked to ensure that all measurements were carried out within the linear viscoelastic regime, in which the dynamic storage modulus (*G'*) and loss modulus (*G''*) are independent of the strain amplitude. The oscillating sweep measurements were carried out in the approximate frequency (ω) domain of 0.1–100 Hz and small strains (0.1%–1%).



Figure 1 IR spectra of (a) uncrosslinked PBS and (b) crosslinked PBS.

RESULTS AND DISCUSSION

Characterization

The structures of the uncrosslinked and crosslinked PBS are indicated by IR spectra, as shown in Figure 1(a,b). The characteristic absorption bands of PBS appear at 3020 cm⁻¹ (stretching vibration of aromatic C—H bond), 2900 cm⁻¹ (stretching vibration of aliphatic C—H bond), 1465 cm⁻¹ (CH₂; scissoring), 1380 cm⁻¹ (CH₃; bending), and 900–790 cm⁻¹ (aromatic = C—H bond; out-of-plan vibration). The vinyl stretching vibration at 1640 cm⁻¹ still remains in the uncrosslinked PBS, but it disappears in the crosslinked one. Similar phenomena were seen for PS, PES, and PBES, though they are not shown here.

Oil absorbency

Effect of solvent amount on maximum oil absorbency

Of all the reaction conditions, such as solvent amount, weight ratio of SBR to EPDM or weight ratio of tBS to EPDM and SBR, initiator concentration, reaction temperature, and reaction time, solvent amount was found to be one of the most important factors, affecting the crosslinking and the equilibrium value of oil absorbency. The greater the amount of solvent, the higher was the equilibrium value (or the maximum value) of oil absorbency. This is because the crosslinked polymer networks wrap a large quantity of solvent in it during the reaction. These cavities shrink after drying; however, they recover their volume or further swell when immersed in a suitable



Figure 2 Effect of solvent amount on maximum oil absorbency and gel fraction of PS networks.

solution. For instance, curve (1) in Figure 2 shows the influence of solvent amount on the maximum oil absorbency of PS networks yielded from polymerization with 2.5 wt % BPO as an initiator in toluene at 70 °C for 48 h. The gel fractions are shown with curve (2) in Figure 2. Maximum oil absorbency increased with an increase in the amount of solvent, whereas the gel fraction decreased until the polymeric concentration arrived at the threshold of crosslinking.²⁶ For PS the threshold of crosslinking was 30 mL/g, over which the gel fraction was equal to zero. The maximum value of oil absorbency for PS at a solvent amount of 30 mL/g was 63.18 g/g. To ensure crosslinking polymerization, the solvent amount for the four polymer networks was fixed at 10 mL/g unless otherwise specified.

Effect of SBR content on maximum oil absorbency

The effect of the weight ratio of SBR to EPDM on oil absorbency is shown in Figure 3. The polymers are



Figure 3 Time dependence of oil absorbency for PBES as a function of SBR content. SBR–EPDM (g/g): (1) 4.00, (2) 1.86, (3) 1.00, (4) 0.54; gel fraction (%): (1) 90.5, (2) 78.9, (3) 69.8, (4) 49.1.



Figure 4 Time dependence of oil absorbency for PBES as a function of tBS content. tBS/(SBR + EPDM) (g/g): (1) 0.00, (2) 0.2, (3) 0.5, (4) 0.8, (5) 1.0; gel fraction (%): (1) 96.7, (2) 89.8, (3) 69.8, (4) 55.9, (5) 39.2.

PBES networks. These were produced from crosslinking polymerization at 70°C for 48 h with a weight ratio of tBS to (EPDM + SBR) of 0.5 g/g, and a BPO content of 2.5 wt %. The maximum value of oil absorbency for PBES increased with a decreasing weight ratio of SBR to EPDM down to 1.00 g/g, but thereafter it decreased with any further decreasing in SBR content. SBR had two roles in the crosslinking polymerization, that of polymeric monomer as well as crosslink agent. The increase in the maximum value of oil absorbency at lower SBR contents was expected because the more the crosslink agent, the higher the crosslink degree of the network, resulting in a lower maximum value of oil absorbency.²⁷ On the other hand, the gel fractions went up with increasing SBR content (see caption of Fig. 3 for data). The exact reason why the maximum value of oil absorbency decreased with further decrease in SBR content is not clear, however. A similar result can be seen in PES networks.

Effect of tBS content on maximum oil absorbency

Figure 4 gives the effect on oil absorbency of the weight ratio of tBS to EPDM and SBR in PBES networks. The PBES polymers were obtained from crosslinking polymerization at 70°C for 48 h with a weight ratio of SBR to EPDM of 1.00 g/g and a BPO content of 2.5 wt %. The oil absorbency maximum value of PBES increased with an increasing weight ratio of tBS to (EPDM + SBR) up to 0.5 g/g, and then decreased with any further increase in tBS concentration. The gel fraction, however, decreased monotonously with increasing tBS. These results can be understood as follows: tBS is a monomer and has a structure of 4-tert-butyl on the styrene, which will give some space effect in the network. So more tBS content will support large cavities in the network, and the network will absorb much oil. On the other hand,



Figure 5 Time dependence of oil absorbency for four polymer networks: (1) PS, (2) PES, (3) PBS, (4) PBES; gel fraction (%): (1) 97.8, (2) 96.7, (3) 80.1, (4) 69.8.

many more monomers will give rise to a shortening of the strand length between crosslinking points of the network, and the gel turns out to be stiff. As a result, the pores in the gel cannot swell very well, and the maximum value of oil absorbency will decline. In addition, there are more chances to form a homopolymer, poly(tBS), when tBS content increases. At the same time, the concentration of the crosslinking agent (SBR) decreases, so that the gel fraction always decreases with increasing tBS content.

Comparison of the four polymeric networks

Figure 5 shows the oil absorbency of the four polymeric networks, PS, PES, PBS and PBES, as a function of time. The networks were obtained from crosslinking polymerization in toluene at 70 °C for 48 h, with solvent in a concentration of 10 mL/g and 2.5 wt % BPO. The weight ratios were: SBR to EPDM 1.00 g/g and tBS to (EPDM + SBR) 0.5 g/g in the PBES reaction; SBR–EPDM = 1.00 g/g in PES; and tBS–SBR = 1.00 g/g in PBS. Their gel fractions were in the order



Figure 6 Scanning electron micrographs of (a) PS, (b) PES, (c) PBS, (d) PBES (× 1000).

of PS > PES > PBS > PBES. The oil absorbency maximum values, however, appear opposite the sequences. Among them, PBES had the highest value, 74.0 g/g, with PBS next at 69.5 g/g. This result may be explained by the space effect of 4-tert-butylstyrene.

Morphologies of networks

The morphologies of the polymeric networks are shown in Figure 6. The polymers were yielded from the same reaction conditions as shown in Figure 5. There were many small pores in the crosslinked PS. These pores will support large surfaces in the polymeric network. PES is an interpenetrated elastomer network (IEN) and its morphology seems a rough set. Large pores are seen in the PBES network. The morphology of a crosslinked polymer will influence the rate of oil absorption.

Swelling kinetics

The swelling rates of PBES were given in Figures 3 and 4. According to eq. (5), the swelling kinetic constant (K) was determined and shown in Table I. Values of K increased with decreasing SBR content but decreased as tBS concentration increased. The reason for this result is the same as that mentioned above.

The swelling rates of the four polymeric networks are given in Figure 5. Their oil absorption characteristics are indicated in Table II. The order of the swelling kinetic constant, K, was PS > PES > PBS > PBES. This order is opposite to that for maximum oil absor-

TABLE I					
Absorption Characteristics of PBES in Oil Solution					

	1				
Sample	$Q_{\rm max}({\rm g}/{\rm g})$	$t_{\rm max}$ (h)	Q (g/g)	<i>t</i> (h)	$K \times 10^2 ({ m h}^{-1})$
SBR/EPDM (g/g)					
4.00	52.61	168	33.25	34.9	2.86
1.86	63.69	120	40.25	20.0	4.99
1.00	74.00	120	46.77	18.3	5.46
0.54	46.78	120	29.56	6.0	16.66
tBS/(SBR + EPDM) (g/g)					
0.00	42.65	120	26.95	8.8	11.34
0.20	55.45	144	35.04	17.3	5.78
0.50	74.00	120	46.77	18.3	5.46
0.80	67.19	120	42.46	19.1	5.24
1.00	63.58	144	40.18	19.7	5.08

The other reaction conditions were SBR/EPDM = 1.00 (g/g) (tBS)/(SBR + EPDM) = 0.5 (g/g), solvent amount 10 mL/g, initiator concentration 2.5 wt %, reaction temperature 70°C, and reaction time 48 h in addition to changed condition.

$K \times 10^2$ (h ⁻	h^{-1})
49.97	,
11.34	:
11.11	
5.46	
	11.34 11.11 5.46

TABLE II Absorption Characteristics of Four Polymeric Networks in Oil Solution

tBS/SBR = 1.00 (g/g) in PBS polymeric network; SBR/EPDM = 1.00 (g/g) in PES; tBS/(SBR + EPDM) = 0.50 (g/g) and SBR/EPDM = 1.00 (g/g) in PBES. The other reaction conditions were solvent amount 10 mL/g, BPO 2.5 wt %, 70°C for 48 h.

bency. Pure SBR network, PS, had the highest value of K, 49.97 × 10⁻² h⁻¹. The rate of oil absorption may depend mainly on the surfaces of the polymer or the number of pores. Maximum oil absorbency of a polymer relies not only on the volume of the cavities but on the number of pores as well. Many small pores in PS, as shown in Figure 6, will be consistent with the results of the highest *K* and the lowest maximum value of oil absorbency.

Light resistance

Light resistance was determined using color difference (ΔE). The ΔE versus irradiation time for the polymers is given in Figure 7. As Figure 7 shows, a polymer that includes 4-tert-butylstyrene will be sensitive to light. It may be that 4-tert-butyl- has chemical activity that is sensitive to light. EPDM, however, has good resistance to light, oxygen, and ozone. Adding EPDM will improve the light resistance property of the polymer, as can be seen in Figure 7 for PBES and PBS. PBES has better light resistance than PBS.

Parameters of pseudo-critical gels

In the pseudo-critical gel state, G' and G'' curves become parallel to each other, and the power law behavior ($G' \sim G'' \sim \omega^n$) in frequency is observed (see Fig. 8).



Figure 7 Plot of color difference versus irradiation time for samples (1) EPDM, (2) PES, (c) PS, (d) PBES (5) PBS.

From Figure 8 it can be seen that G' > G'' during the whole period of measurements. The gel strength, *S*, and relaxation exponent, *n*, were calculated from eq. (6) and the fractal dimension, d_f , from eq. (7). They are listed in Table III. The gel strength of PES is twice that of PBES. It is explicit that 4-tert-butylstyrene will weaken the gel strength of crosslinked PES. The relaxation exponent, *n*, and the fractal dimension, d_f , have only a little difference among them. This may suggest that the strand lengths between crosslinking points are similar and mainly depend on PES. Similar results were observed for PBS and PS.

CONCLUSIONS

Uncured SBR was used as both a polymeric monomer and a crosslink agent in this work. Four oil absorbents based on SBR were obtained from crosslinking polymerization in toluene at 70°C for 48 h using BPO as an initiator (2.5 wt % dependent on total weight of monomers including polymeric monomer). Of all reaction conditions investigated, solvent amount was found to be one of the most important factors affecting the degree of crosslinking and the maximum value of oil absorbency of the polymeric network. 4-tert-Butylstyrene grafted into a polymer makes it sensitive to light



Figure 8 Plot of *G*' and *G*" versus frequency for $(\blacktriangle, \triangledown)$ gel PES and $(\blacksquare, \textcircled)$ gel PBES, where \blacktriangle and \blacksquare are *G*' and \blacktriangledown and \blacksquare are *G*".

TABLE III						
Characteristic	Data	for	Pseudo-Critical	Gels		

Sample	(Oil absorbency) _{max} (g/g)	п	S (Pa · s ⁿ)	d_f
PES	42.65	0.26	1230	2.26
PBES	74.00	0.25	509	2.27

The reaction conditions were SBR/EPDM = 1.00 (g/g), tBS/SBR + EPDM = 0.50 (g/g), solvent amount 10 mL/g, BPO 2.5 wt %, 70°C, 48 hours.

and weakens the gel strength of the polymeric network, but increases the value of maximum oil absorbency. There are many small pores in pure SBR polymeric network PS. As a result, among the four polymeric networks it had the highest value for the swelling kinetic constant, K, 49.97 × 10⁻² h⁻¹, and the lowest maximum value of oil absorbency, 32.71 g/g. The polymer tBS–EPDM–SBR (PBES), however, had the highest value, 74.00 g/g, but its swelling rate, K, was the lowest, 5.46 × 10⁻² h⁻¹. In contrast with the four networks, the order was PBES > PBS > PES > PS for equilibrium oil absorbencies and the opposite sequence for the gel fractions and swelling kinetic constants.

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