Compatibilization of Polymer Blends with High-Molecular-Weight Peroxides

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ABSTRACT: A novel approach for the compatibilization of heterogeneous polymer blends with interface-active high-molecular-weight peroxides is presented. This three-step approach includes the synthesis of an anchor peroxide copolymer, the preparation of an interface-active, peroxide-containing graft copolymer (precompatibilizer) on its basis, and the localization of the precompatibilizer at the interfaces of the polymer blends during reactive blending with the *in situ* formation of compatibilizer macromolecules. We found that the precompatibilizer incorporating polypropylene fragments compatibilized blends of polypropylene with polystyrene, polyethylene, and unsaturated polyester resin. This verified a certain universality of the approach proposed for the compatibilization of polymer blends. © 2005 Wiley Periodicals, Inc. J Appl Polym Sci 96: 232–242, 2005

Key words: graft copolymers; blends; compatibilization; reactive processing

INTRODUCTION

More and more frequently, the contemporary chemistry of macromolecular compounds demands an elaboration of new "smart" materials, which should neatly perform their roles under different physicochemical conditions. In the field of polymer blends, this concerns the creation of new compatibilizers. The destination of these compounds is an assurance of an affinity to immiscible polymer materials, that is, (1) a decrease in the interfacial tension and (2) an increase in the interfacial adhesion between the general components of the polymer blends. It is well known[1,2] that the majority of carbon-chain polymer materials are immiscible with each other. During physical blending, their melts are phase-separated, tend to coalescence, and display low adhesion between phases. The latter problems result in the worsening of blend characteristics in comparison with the virgin polymers. Many techniques have been used to increase the compatibility of blended polymers,[1,3] and a common method for the majority of them is the introduction of copolymers of various architectures to the interfaces.[4,5] These copolymers, known as compatibilizers, consist of fragments partially miscible with the general components of the polymer blends. Compatibilization methods are generally divided into nonreactive ones (when preliminary prepared compatibilizers are introduced into the blend)[6–8] and reactive ones (when compatibilizer macromolecules are formed *in situ* during blending).[9] Nonreactive methods demand the creation of compatibilizers for every pair of blended polymers, although Vilgis and Noolandi[10] proposed an approach to search for a universal compatibilizer, of which fragments could differ completely from the general components of the polymer blend. However, the reactive compounding of polymers is considered as a much more promising method. In the latter case, the compatibilizer is formed directly during blending from macromolecules of the blended polymers. Thus, its blocks, tails, or fragments are partially miscible with the major blend components.

Conventional compatibilization reactions basically involve a condensation mechanism, such as, epoxide plus anhydride, oxazoline plus carboxylic acid, isocyanate plus carboxylic acid, acyl lactam plus amine, carbodiimide plus carboxylic acid, and anhydride plus amine.[11–14] Many of them are discussed in detail by Xanthos and Dagli,[3] Orr et al.,[15] and Koning et al.[16] These processes can be conducted from solution[17] during the *in situ* polymerization of one blend component in the presence of a second polymer, which could possibly result in the interpenetration of structures,[18] or during extruder blending[19] and can include the formation of block and/or graft copolymers[20] and crosslinking.[21] Compatibilizers formed *in situ* possess fragments chemically

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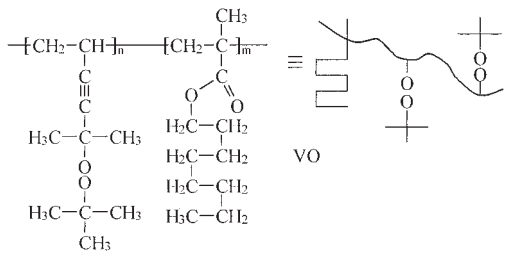
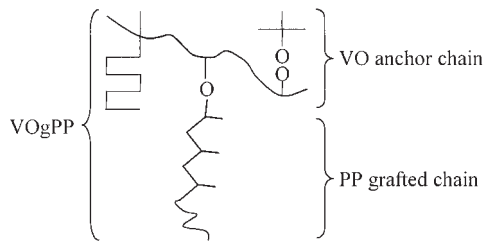


Scheme 1 Structure of the random anchor peroxide-containing polymer VO.



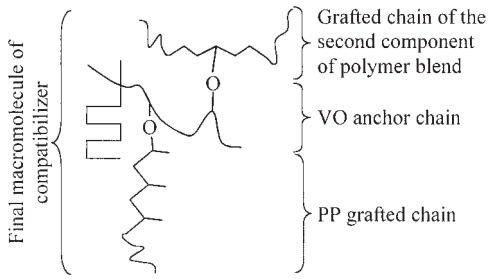
Scheme 2 Structure of the precompatibilizer VOgPP.

identical to the segments of corresponding blend components, and it has been suggested that they are mainly placed across the interfaces.[22,23] Such a variety of compatibilization methods have been developed because of the limitations of each method to certain sets of polymer materials or the need for additional preliminary modification of the virgin components.

However, practically all of the polymers blended today are carbon chains by nature or incorporate carbon-chain fragments. For the latter, this means that they possess a universal property to participate in free-radical reactions, which are widely used for polymer modification. Mostly, the radical processes are initiated by low-molecular-weight peroxides[24–33] or by the application of high-energy irradiation.[34–39] Teh and Rudin[40,41] used low-molecular-weight peroxides and chain-transfer agents and showed the utility of radical processes for blend compatibilization. Although this approach has been successfully applied so far,[25,42–44] one can imagine that low-molecular-weight substances may be not suitable and efficient enough under the blending conditions because of their volatility and the initiation of radical processes not only across the interfaces but also in the bulk of the materials, where they can be harmful. To avoid this significant drawback, it is purposeful, in our opinion, to incorporate the sites of free-radical generation into the structure of high-molecular-weight compounds. To a certain extent, this was realized by Boutevin et al.[45] on the modification of polyolefins and poly(vinyl chloride) via ozonation followed by the graft polymerization of the second blend component fragments initiated by all of the incorporated peroxide groups. Authors have shown the possibility of using prepared modified polymers as emulsifiers for polymer blends. However, there has been no attempt to use such graft copolymers with a part of the peroxide groups kept for the final *in situ* formation of the compatibilizing system during blend preparation. The latter route should allow one to incorporate the fragments of the components just used in the blend and, with respect to the common polymer property mentioned to participate free-radical reactions, should display a universality for the compatibilization of a number of blends simultaneously.

We propose a new approach for the compatibilization of polymer blends with interfacial-active peroxides that consists of three general stages.

1. Synthesis of an anchor peroxide-containing copolymer of 2-tert-butylperoxy-2-methyl-5-hexene-3-yne and octyl methacrylate (VO). Scheme 1 represents the structure of an anchor peroxide random copolymer VO, which is proposed for the preparation of compatibilizers. Units of the peroxide monomer of this copolymer provide the initiation of the radical processes of chain transfer and grafting at the temperatures of polymer blend preparation. Octyl methacrylate (OMA) units provide this copolymer (and its derivatives) partial miscibility with polyolefins necessary for the further preparation of precompatibilizer.
2. Precompatibilizer-VO-graft-polypropylene (VOgPP)-was prepared via grafting of copolymer VO with polypropylene chain (PP) (see Experimental). Its structure is shown in Scheme 2. It incorporates the grafted fragments of the first component of the polymer blend (in this study, PP), which provides interfacial activity, and peroxide fragments suitable for the initiation of radical processes.
3. *In situ* formation of compatibilizer macromolecules during the reactive blending of polymers. When the graft copolymer (Scheme 2) is added to the polymer blend it is localized across the interfaces and initiates radical processes that result in the grafting of macromolecules of the second



Scheme 3 General structure of the *in situ* formed compatibilizer macromolecules.

component of polymer blend, thereby, forming compatibilizer macromolecules (Scheme 3).

Thus, in this article, we present a new approach for polymer blend compatibilization that, for the first time, uses interface-active, peroxide-containing copolymers, which we named *precompatibilizers* (Scheme 2). Each of these precompatibilizers uses the universal ability of carbon-chain polymers to participate the free-radical reactions. Because of the structural features, one precompatibilizer is able to compatibilize a large variety of polymer blends, in each case forming compatibilizer macromolecules (Scheme 3) that fit just to the polymer pair being blended. As a basis for precompatibilizer creation, we applied a random copolymer of 2-*tert*-butylperoxy-2-methyl-5-hexene-3-yne (VEP) and OMA. The grafting of PP to this copolymer led to the formation of the VOgPP precompatibilizer used in this study.

EXPERIMENTAL

The synthesis of an interface-active, peroxide-containing precompatibilizer (VOgPP, Scheme 2) for the compatibilization of PP blends with other carbon-chain polymers was presented earlier.[46,47] It consists of two stages, the first of which is the synthesis of the random copolymer of VEP and OMA (VO, Scheme 1), and the second one is the preparation of VOgPP, performed in a DACA Micro-Compounder (volume = 4.5 cm^3; DACA Instruments, Goleta, CA) in accordance to parameters optimized by means of full-factorial, second-order orthogonal design: temperature = 175°C, VO concentration in PP [Aldrich 428183 (Munich, Germany); number-average molecular weight (M_n) $\approx$ 5400 g/mol, weight-average molecular weight (M_w)/M_n = 3.63] = 18 wt %, and reaction time = 12 min. The VO had a M_n of about 6500 g/mol with a M_w/M_n of 1.1 and contained 14 mol % VEP units. After the removal of nongrafted VO and the products of VO and PP decomposition and destruction by Soxhlet extraction, VOgPP was used as an approximately 35 wt % blend with PP; the quantity of grafted VO in accordance with quantitative IR analysis (with an adsorption band of carbonyl groups at 1732 cm^{-1}) was about 22 wt %. The active oxygen content in VOgPP calculated with the activation parameters obtained from model experiments[46] was 0.7 mmol/g.

Ellipsometric mapping

Specimens for ellipsometric mapping were prepared as follows. Highly polished silicon wafers (obtained from Wacker-Chemitronics, Burghausen, Germany) were first cleaned in an ultrasonic bath with dichloromethane (three times for 5 min each), then placed in a cleaning solution [$NH_4OH/H_2O_2/H_2O$ = 1/1/1 (by volume)] at 60°C for 1 h, and then washed several times with Millipore water (18 MΩ cm). After they were dried with a nitrogen stream, the silicon substrates were placed into an argon-filled glove box and immersed into a 3-glycidoxypropyl trimethoxysilane (GPS; Aldrich) solution (1 wt %) in dry toluene for 16 h. The modified substrates were rinsed several times with dry toluene and ethanol in an ultrasonic bath to remove unattached GPS. Maleic anhydride terminated polypropylene (PP-MA; $M_n \approx$ 9100 g/mol, M_w/M_n = 2.64) was spin-coated from a 1 wt % toluene solution onto the surface of the GPS-modified silicon substrates. The substrates were then placed into a vacuum oven at 170°C for 24 h to graft the PP chains covalently. Unattached PP-MA was removed by Soxhlet extraction with toluene for 5 h. After the substrates were dried, the grafted PP layer was mapped with a high-speed *in situ* 44-wavelength ellipsometer (J.A. Woollam Co., Lincoln, NE). VO was spincoated from a 1 wt % toluene solution onto the surface of the PP-MA modified silicon substrates. The thickness of these VO films before grafting was 35 ± 4 nm. Then, the substrates were placed into a vacuum oven at 130°C for 24 h, and unattached VO was removed by Soxhlet extraction with toluene for 5 h. An ellipsometric map was built for the VO layer grafted to the PP layer.

Blends of PP and polystyrene [PS; PP/PS = 30/70; PP amorphous, Aldrich 428183; PS 143 E (BASF AG, Ludwigshafen, Germany)] and PP and polyethylene [PE; PP/PE = 10/90; PP Novolen 1106 H (Targor); PE Hostalen GC 7260 (Elenac GmbH)] were prepared as follows. The precompatibilizer was premixed at ambient conditions with granules of PP and the second polymer component (PS or PE) used for blend preparation. Reactive blending was performed by means of a DACA Micro-Compounder at 190°C for 10 min. The prepared blends were extruded as strands into a container with water.

Blends of PP with unsaturated polyester resin (UPR) dissolved in styrene [uncured and cured compositions; PP/UPR–styrene compositions of 3/97–20/80 w/w; PP amorphous, Aldrich 428183; UPR PN-15 (Ukraine); styrene, Aldrich] were prepared as follows. PP without precompatibilizer or with VOgPP was added to UPR melt-heated to 190°C in a steel mixing chamber [Universal Laboratory AID type MPW-309 (Mechanika Precyzyjna) with a steel turbine blade and an internal chamber diameter/blade diameter of 3/1]. The use of this device was grounded on the preliminary experiments, during which the maximum fineness and stabilization (see Table I) of the PP dispersion phase were achieved. Thus, mixing was performed at 2000 rpm for 15 min at 190°C. After that, styrene heated to 50°C was added to the mixing chamber (with the heat turned off) to achieve a UPR/styrene ratio of 60/40 (w/w). At this time, the PP particles

TABLE I
Initial Formulas and Properties of the PP/UPR–Styrene Compositions Compatibilized with VOgPP

	Component content in the composition (wt %)				Composition properties	
No.	PP	UPR	Styrene	VOgPP	Stability (days)	Charpy unnotched impact strength (kJ/m^2)
1	0	60.0	40.0	0	—[a]	5.8 ± 0.7
2	3.0	58.2	38.8	0	1	2.9 ± 0.3
3	20.0	48.0	32.0	0	—[b]	—[c]
4	4.8	57.0	38.0	0.2	52	6.3 ± 0.8
5	9.6	54.0	36.0	0.4	35	6.6 ± 0.7
6	3.9	57.1	38.0	1.0	>180	8.3 ± 0.8
7	6.2	55.3	36.8	1.6	>180	7.3 ± 0.8
8	3.0	57.0	38.0	2.0	>180	5.2 ± 0.6
9	9.4	52.9	35.3	2.4	>180	7.5 ± 0.6

[a] Composition did not contain a polyolefin dispersed phase (homogeneous).
[b] Stratification occurred at discharge from the mixing chamber; at lower PP contents, the stratification onset time increased steadily to about 1 day.
[c] Cured samples could not be prepared because of composition stratification.

solidified, and the UPR phase dissolved gradually in styrene with simultaneous cooling to 20°C for 20 min. The liquid composition obtained could be stored as long as necessary at ambient conditions (see Results and Discussion). For composition curing, benzoyl peroxide (40 wt % blend in dibutyl phthalate, Aldrich) in a quantity of 5 wt % and 2,4-dimethylaniline (Aldrich) in a quantity of 0.15 wt % (both based on the total quantity of UPR and styrene) were added to the composition before it was cast to the mold. After casting, the composition curing proceeded at room temperature for 24 h to produce solid specimens 0.5 × 1.0 × 8.0 cm^3 in size.

Scanning electron micrographs were obtained from the cryofractured surfaces by means of either a scanning electron microscope (DSM982 Gemini; Figs. 3 and 4, shown later) or a scanning electron microscope LEO VP435 (Fig. 6, shown later, both Carl Zeiss AG, Oberkochen, Germany) at 10 kV.

RESULTS AND DISCUSSION

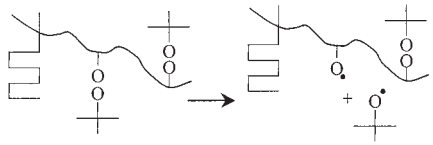
During the specific polymer blending with the precompatibilizer, grafting reactions occurred that formed an *in situ* compatibilizer, which should have been highly efficient just for the system where it was formed. As shown in the compatibilization scheme (Fig. 1), the precompatibilizer macromolecules had to fit the following demands: they had to be localized across the interfaces, and the reactivity of their peroxide groups had to be high enough to provide sufficient grafting of the second polymer. The presence of ditertiary peroxide fragments at the interface caused the generation of highly reactive *tert*-butoxyl and methyl radicals,[48] which were efficient for hydrogen atom detachment[25] and macroradical formation. The combination of the macroradicals of the second polymer blend component with precompatibilizer macroradicals led to the formation of the desired compatibilizer (Fig. 1).

Interaction of high-molecular-weight peroxide with carbon-chain polymers

To form efficient compatibilizing structures, the polyperoxide VO had to be grafted with both major blend components. To study the reactivity, we conducted model investigations of polymeric peroxides with PP and PS surfaces.

Figure 2 represents the ellipsometric mapping of a VO layer grafted to PP and PS layers, which in turn, were grafted to GPS-modified silicon wafers. Ungrafted VO molecules were removed from the surface after the grafting process. The results shown confirmed the ability of the VO molecules to interact efficiently with low-reactive polymer surfaces. A VO macromolecule grafting with PP as an example can be schematically represented as the following set of elementary reactions:

1. VO peroxide group decomposition:



2. Hydrogen atom detachment from the PP macromolecule:

3. β-Scission:[49]

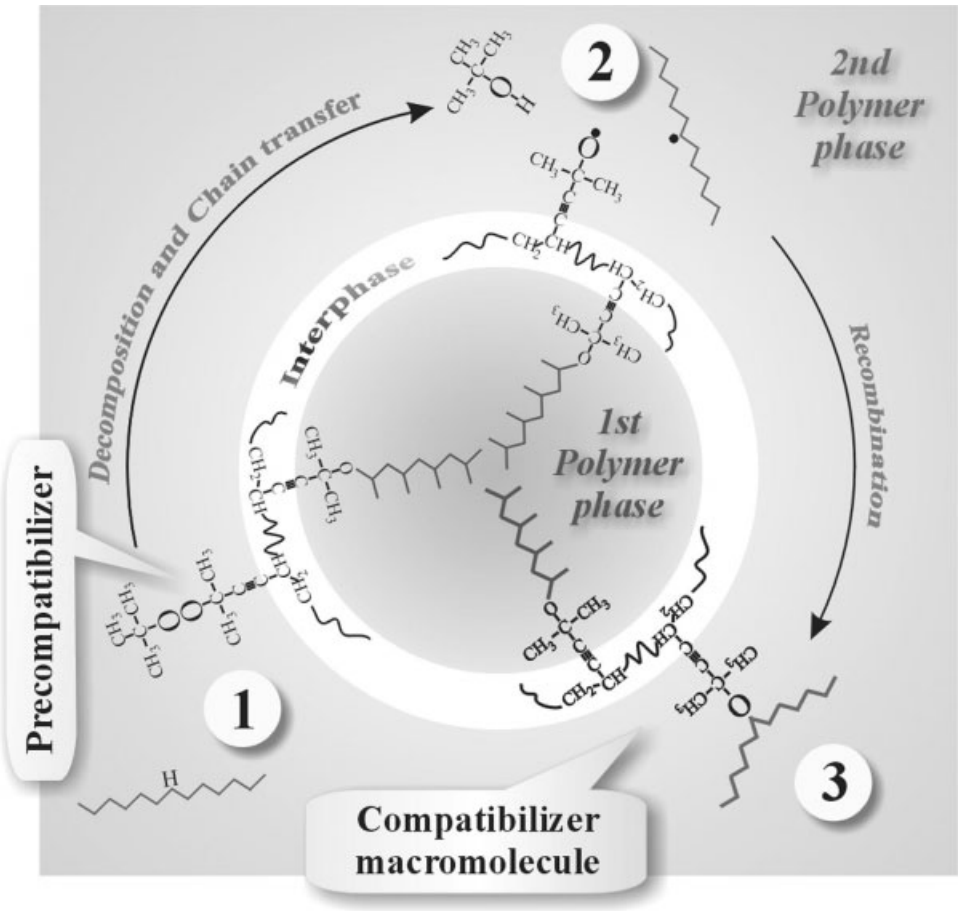
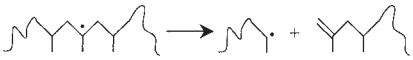
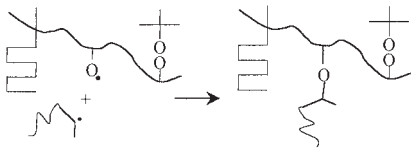


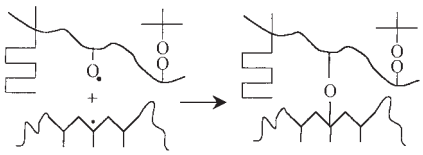
Figure 1 Schematic representation of compatibilizer macromolecule formation during the process of reactive blending of the two polymers with the participation of a peroxide-containing precompatibilizer.



4. Recombination with a scissed PP macroradical:



5. Recombination with a nonscissed PP macroradical:



The conditions for the formation of the VOgPP precompatibilizer were chosen in such a way that about half of the peroxide groups in the virgin VO were consumed during grafting with PP. VOgPP was supposed to display an interface activity similarly to the amphiphilic behavior of surfactants for aqueous systems where one part of molecule is hydrophilic and the other part is hydrophobic. In our case, this meant that the PP chain was compatible with the PP phase and that the anchor VO chain was not compatible, which forced the precompatibilizer to locate at the surface of the PP phase in the blend.

PP/PS polymer blend preparation

To verify the possibility of the VOgPP application for the compatibilization of polymer blends, we used it for the reactive blending of PP and PE and PP and PS.

Figure 3 presents scanning electron micrographs of the cryofractured surfaces of PP/PS (30/70) blends with varying contents of graft copolymer. The decrease in the dispersed particle size (PP) and their more regular form, which was observed with precompatibilizer content increases, proved the blend compatibilization by VOgPP. In our opinion, compatibilization was caused by VOgPP anchor chain migration to the particle surface, whereas the grafted PP chain of VOgPP remained in the bulk (or, more correctly, in the

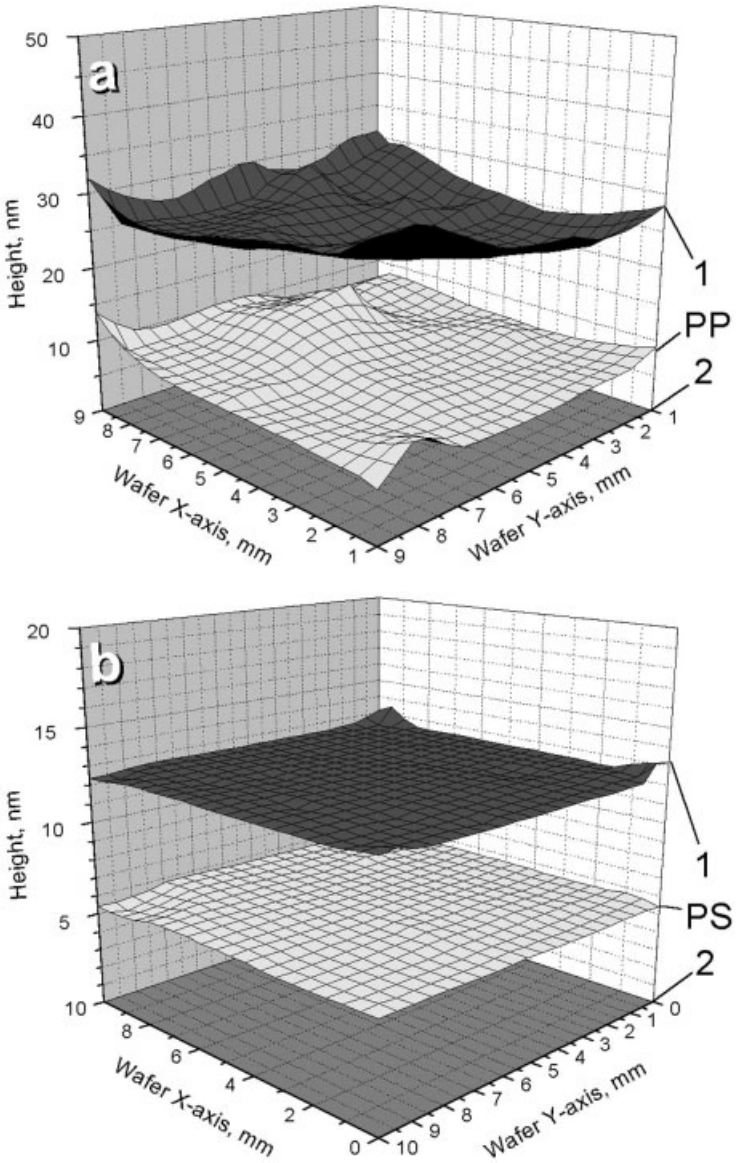
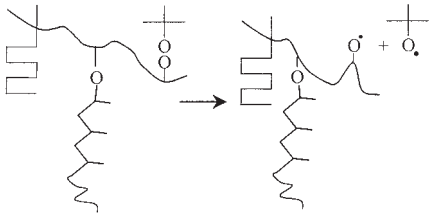


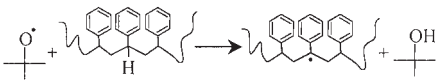
Figure 2 Ellipsometric mapping of the (1) VO layer grafted to (a) PP and (b) PS layers, which in turn, were grafted to a (2) GPS-modified silicon wafer.

surface layer) of the PP phase. This process itself may not have led to the decrease in interfacial tension, but it was a necessary condition for the initiation of radical processes across the interfaces in accordance with the following scheme:

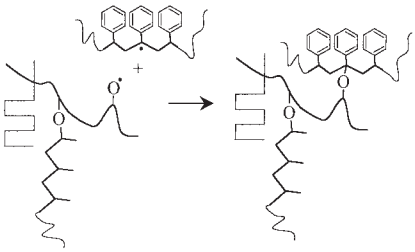
1. VOgPP peroxide group decomposition:



2. Proton detachment from the PS macromolecule:



3. Recombination with the PS macroradical and the formation of the final compatibilizer macromolecule:



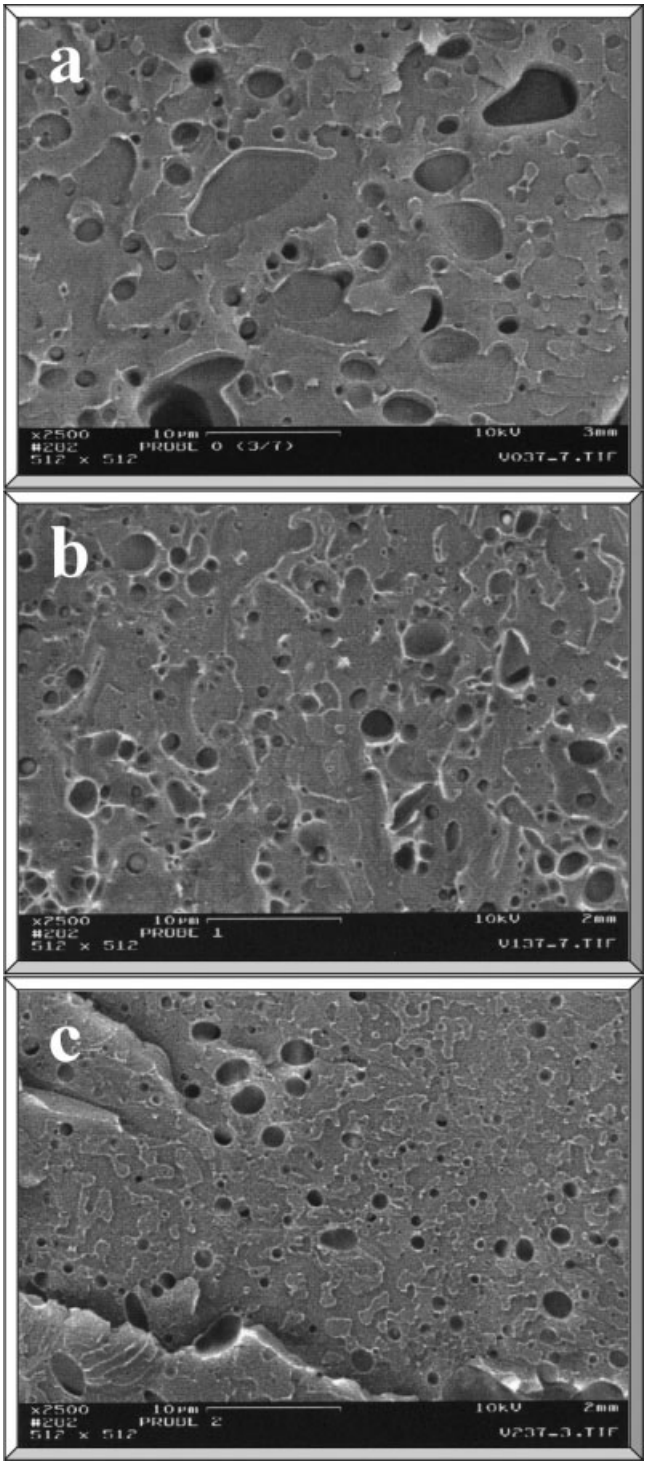


Figure 3 Scanning electron micrographs of the cryofractured surfaces of PP/PS (30/70) blends (a) without VOgPP and with (b) 0.3 and (c) 0.9 wt % VOgPP.

In this way, the localization of radical processes at the interfaces led to the formation of a highly efficient compatibilizer, of which the side chains were chemically identical in nature to both blend components. An analogous explanation for the compatibilizing effect of low-molecular-weight peroxide is reported elsewhere.[40,41,50] Also, the results obtained confirmed the high compatibilizing efficiency of the system proposed. For example, in the case of VOgPP use in polyolefin/PS blends, the concentration of active oxygen for the achievement of the compatibilization effect was 1.5–3.5 $\times 10^{-7}$ mol/g of the blend, whereas when low-molecular-weight dicumene peroxide[41] was used, this concentration needed to be on the order of 1.8–9.2 $\times 10^{-5}$ mol/g of the blend for similar systems, which is about 50 times higher.

An attempt to create inverse polymer blends (PP/PS = 70/30) with 1.5 wt % of precompatibilizer was not very successful because of the substantial divergence between the viscosities of the continuous and the dispersed phases. The PP melt had a much lower viscosity than the dispersed PS phase. Shear forces during blending were not high enough to reach the necessary fragmentation of the dispersed phase, and only large particles were observed. However, essential changes at the interfaces in the blend were caused by the addition of a relatively high quantity of precompatibilizer. For a clear demonstration of the effect of the precompatibilizer utilization, micrographs are shown in Figure 4 of a specimen that did not contain VOgPP and one containing a sufficiently large amount of it. The arrows point to the zones of interlinked phases induced by the formation of compatibilizer macromolecules because of the addition of 1.5 wt % VOgPP, whereas in the nonreactive blend [Fig. 4(a)], the phase adhesion was poor. Only at the bottom of the shown particle were signs of adhesion visible.

Furthermore, a viscosity decrease was found for the PS/PP blends [Fig. 5(a)] that was characteristic for the conduction of compatibilization with the use of radical processes.[40] The peroxide activity led to changes in the conformational and molecular weight parameters of the blend components, especially in the layers close to the phase boundaries. Even if the process led to the formation of highly efficient compatibilizers, undesired side reactions could occur. In PP, radical processes cause degradation and, therefore, a reduction in its melt viscosity. The complex melt viscosity of blends is dominated by the matrix, which was in our case the PS phase. Therefore, the observed decrease in the complex viscosity of the blend and the changes in the ratio between the storage modulus and the loss modulus [Fig. 5(b)] due to the addition of precompatibilizer may have been caused by the interfacial effects described by Teh and Rudin,[40] especially by the reduction of the average matrix polymer molecular weight due to the favorable grafting of high-molecular-weight PS chains to the dispersed phase. However, with a high amount of active peroxides, the melt viscosity of the blend was higher than with low precompatibilizer addition. This finding showed the effects of

reduced particle sizes and, therefore, the increased interfacial area and enhanced interfacial adhesion.

PP/PE polymer blend preparation

Also, the precompatibilizer VOgPP could be applied not only for the compatibilization of the PP/PS blend but also for PP blends with the majority of polymers capable for participation in free-radical chain transfer and recombination reactions.

With VOgPP, we compatibilized PP/PE 10/90 blends.[46] Micrographs of the cryofractured surfaces made with different precompatibilizer quantities are presented in Figure 6. Minor differences between the chemical and physical properties of PE and PP led to a not so clear visualization of their blend micrographs, as in the case of the PP/PS blends. Despite this, a comparison of the blends without precompatibilizer and blends with 1 wt % VOgPP allowed us to unambiguously determine that a significant refinement in blend morphology had occurred. It is very likely that the chemistry of the compatibilizer formation was similar to the case of PP/PS blend compatibilization. The difference was that the PE chains were grafted to the precompatibilizer instead of the PS one:

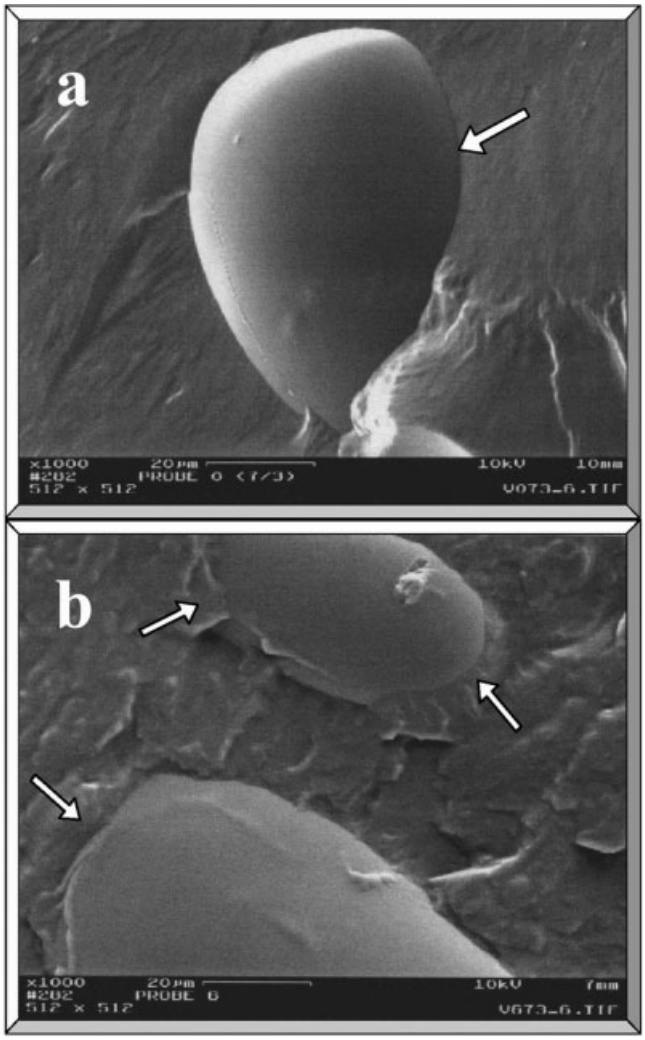


Figure 4 Scanning electron micrographs of the cryofractured surfaces of PP/PS (70/30) blend samples prepared (a) without VOgPP and (b) with 1.5 wt % VOgPP.

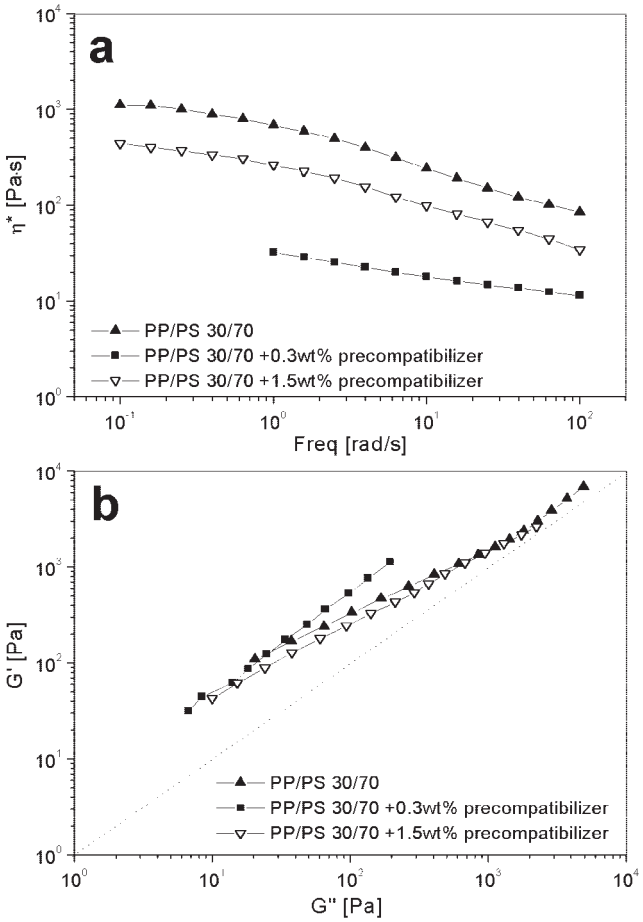
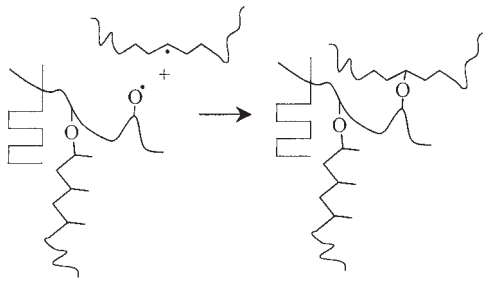


Figure 5 (a) Complex melt viscosity (η^*) and (b) storage modulus (G') versus loss modulus (G'') at 190°C for PP/PS(30/70) samples without VOgPP and with different amounts of VOgPP (Freq = frequency).



P polymer blend preparation

The creation of thermoplastic/thermoset polymer blends is of great interest.[51–55] To check the possibility of compatibilization of thermoplastic/thermoset poly-

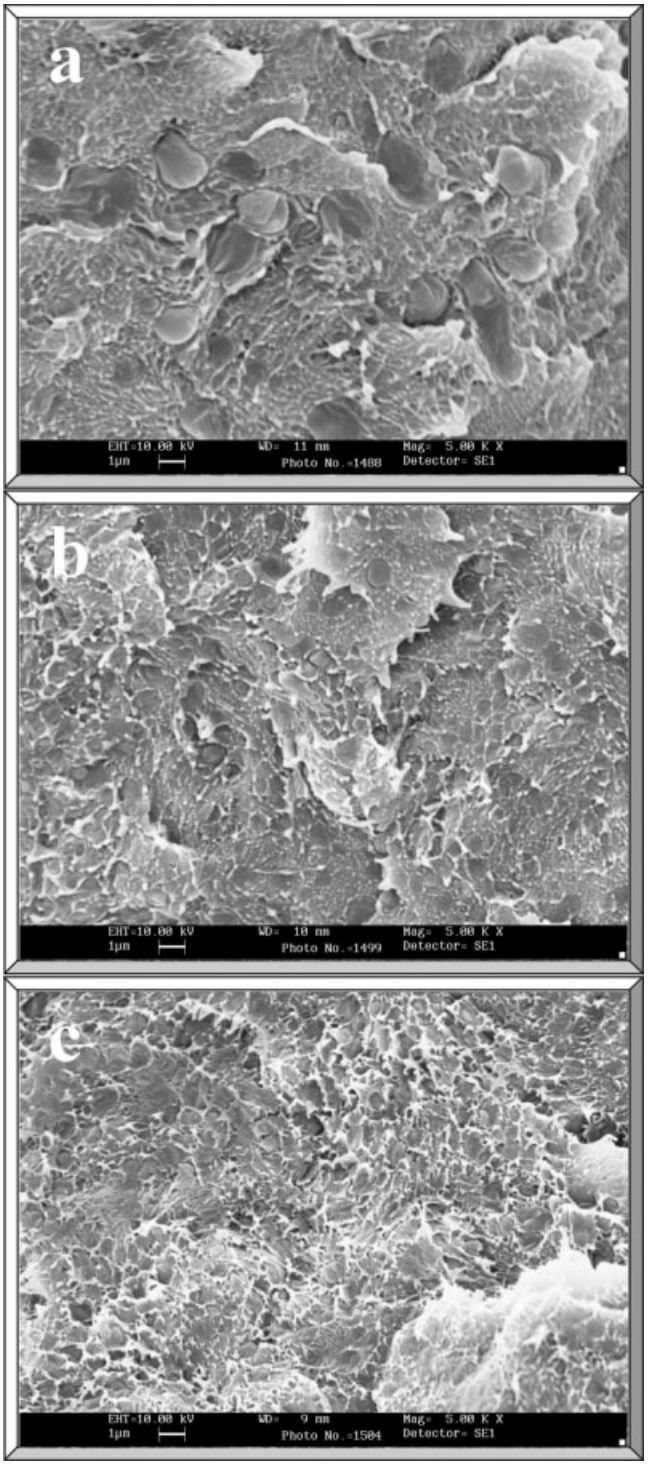


Figure 6 Scanning electron micrographs of the cryofractured surfaces of PP/PE (10/90) blend samples prepared (a) without VOgPP and with (b) 0.2 and (c) 1.0 wt % VOgPP.

mer blends, we studied a blend of PP with UPR. The task was solved in two stages. In the first stage, a dispersion of polyolefin in the prepolymer, which was a noncured solution of UPR in styrene (UPR–styrene), was obtained. The UPR–styrene phase was cured in the second stage, which resulted in a homogeneous UPR/styrene matrix resin with a dispersed PP phase.

In the first stage, conclusions about the compatibilizing efficiency of the precompatibilizer could be made through the evaluation of the stability of the polyolefin dispersion in the UPR/styrene solution. One can see from the data in Table I that the dispersions obtained without precompatibilizer were instable: at a high PP content, stratification began at once after discharge from the mixing chamber, and at a PP content near 3 wt %, stratification began after approximately 24 h. The incorporation of a small quantity of VOgPP in the blend during PP dispersal in the UPR melt increased the dispersion stability drastically: at 0.2–0.4 wt % VOgPP content, the stability increased the dependence on the dispersed phase content to 35–52 days. Stratification did not take place at all during the observation period of 6 months on the addition of 1–4 wt % precompatibilizer.

After composition curing, the compatibilization effect was confirmed by positive changes in the mechanical properties because it is well known[56] that with the improvement of the interfacial interaction dispersion phase–matrix, a transfer of stresses from the matrix to the dispersion phase also improves. If no precompatibilizer was present, even small amounts of added PP reduced the impact strength drastically. However, the addition of 0.2–2.4 wt % (on total blend weight; Table I) VOgPP led to an increase in the impact strength of up to 45% (this increase was also dependent on the weight part of the PP phase). When we recalculated the VOgPP quantity with respect to the PP dispersion phase from the data given in Table I, it was revealed that the maximum impact strength was achieved at 5–10 wt % VOgPP [on PP phase (Fig. 7); these data accounted for the purity of VOgPP (~35 wt %) with the remaining quantity (~65 wt %) of PP from additives combined with the quantity of the PP phase]. Further increases in the precompatibilizer quantity lowered the impact strength of the samples.

Thus, the proposed approach allowed the compatibilization of polymer blends, both of the thermoplastic/thermoplastic and thermoplastic/thermoset types, with the same peroxide precompatibilizer.

CONCLUSIONS

The use of interface-active high molecular peroxides is a universal and effective approach for the compatibilization of immiscible polymer blends, both of the thermoplastic/thermoplastic and thermoplastic/thermoset blend types. The radical processes localized at the interface led to the formation of compatibilizer molecules with fragments of both polymers to be compatibilized. The resulting partial compatibility to both

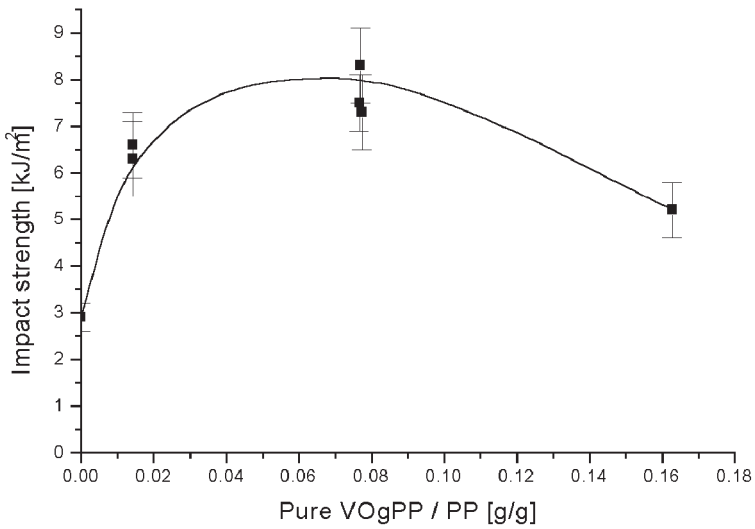


Figure 7 Dependence of the impact strength of the cured PP/UPR–styrene blends on the weight ratio of VOgPP/PP accounting for the purity of VOgPP (~35 wt %).

blend phases caused the high efficiency of the *in situ* formed compatibilizer. Because the majority of carbon-chain polymers were able to participate in the free-radical processes, additional treatments of the virgin materials were not necessary. The method described will allow researchers to create flexible schemes of universal compatibilization of different polymer blend systems.

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