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Compatibility of the Polystyrene/Polyolefin Blends Containing Block Copolymers I. Processing Behaviour and Characterization

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The processing behaviour and mechanical and thermal properties of the polystyrene/ polyethylene or polystyrene/polypropylene blends containing block copolymers of KRATON type as compatibilizing agents have been studied. It has been established that the compounding on a twin screw extruder followed by γ -irradiation assures satisfactory homogeneity of the blends and also good mechanical properties.

A thermomechanical method was developed to characterize the blends. The variation of the α -relaxation or glass transition temperatures with blend composition as well as the variation of the other thermal characteristics such as melt flow rate, melting and crystallization temperatures, Vicat temperatures, *etc.*, exhibit the important differences in respect to the average values indicating a good compatibilization of the components.

Keywords: Polystyrene/polyolefin blends; Compatibility; Thermomechanical method; Processing

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1. INTRODUCTION

Polystyrene (PS)/polyolefin (PO) blends are intensively studied due to both theoretical and practical interest to combine the properties of an amorphous and a crystalline polymer. The behavior of these blends depends on many interrelated variables such as polymer properties, blending and processing conditions, morphology, *etc.* [1,2]. Due to the structural and morphological differences they are typical incompatible blends, but the components offer the possibility to combine very different properties so a great number of studies were devoted to improve the compatibility and to study the change in their morphology and characteristics. With this aim the following solutions have been applied: reactive processing [3, 4], utilization of the compatibilizing agents [5-9], superficial treatment by irradiation [10] or combined procedures of compatibilizing agents use and cold plasma irradiation [11-13].

Block copolymers give a much stabilized dispersion state of blends with the drastic particle size reduction to submicron sizes even for only $\sim 2 \text{ wt\%}$ level of compatibilizing agent by their emulsifying and dispersing activity [14, 15].

This paper reports on a new way of compounding of polystyrene with polyethylene (HDPE) or polypropylene (PP) by combining a conventional method of the incorporation of block copolymers as SEBS and SEP of KRATON type compatibilizing agents with a γ -irradiation technique and by selecting the most suitable processing conditions in an effort to control the nature and properties of the interface and the interphase so that a stable morphology of the blends to be obtained.

2. EXPERIMENTAL

2.1. Materials

Polystyrene (PS), high density polyethylene (HDPE), isotactic polypropylene (PP), styrene-b-ethylene-co-butylene-b-styrene triblock copolymer (SEBS) and styrene-b-ethylene-propylene diblock copolymer (SEP) used were commercial polymers. Their characteristics are given in the Tables I and II.

No.	Characteristic	HDPE	PP	PS
0	Supplier	ARPECHIIM, Pitesti, Romania	PETROTEL, Ploiesti, Romania	CAROM, Borzesti, Romania
1	Density $(g cm^{-3})$	0.9631	0.9052	1.0482
2	Melt flow index (g/10 min.)	1.26*	2.69**	2.50***
3	Melting interval (°C)	136-145	168-175	115-175
4	Crystallization temperature (°C)	130	138	

TABLE I Characteristics of the homopolymers

*190°C/2.16 kg; ** 230°C/2.16 kg; *** 200°C/5 kg.

TABLE II Physico-chemical properties of the compatibilizing agents supplied by SHELL company

No.	Characteristic	SEBS (KRATON G 1652)	SEP (KRATON G 1702)
1	Density (g cm ⁻³)	0.91	0.91
2	Architecture and S/ER or S/PR ratio	Triblock, 29/71	Diblock, 28/72
3	Relative molecular weight	Low	High
4	Melt flow index $(g/10 \text{ min} 190^{\circ}\text{C}, m = 21.6 \text{ kg})$	10	_
5	Viscosity, 25% toluene solution, (cP)	1350	> 50.000
6	Brookfield viscosity, melt at 177°C (cP)	1500	
7	Glass temperature, Tg (°C)	- 42	- 42
9	Physical form	Powder	Powder

S/ER -ethylene rubber and S/PR propylene rubber.

2.2. Blend Preparation

PS/PO blends with weight composition given in the Table III were prepared. The SEBS was used as compatibilizing agent in the PS/PE blends and SEP in PS/PP blends.

The mixing was performed in three ways. The mixing conditions for each series were the following:

(a) First dry blending and then fusing and mixing for 15 to 20 min in a Brabender Plasticorder-Torque Rheometer-Dusseldorf batch mixer at 200°C, the rotor speed of 60 rpm, and the shear ratio 50/100. The time of mixing was defined by the attainment of constant torque values and this occurred generally in about 3-4 min. After batch mixing the extrudates as fibers with $\Phi = 3$ mm were obtained on the Brabender extruder from all blends.

Code of the blend	HDPE (% wt.)	PP (% wt.)	PS (% wt.)	SEBS (KRATON G 1652) (% wt.)	SEP (KRATON G 1702) (% wt.)
5 PS/5 PE(a)	49		50	1	
6 PS/4 PE(a)	35		60	5	-
7 PS/3 PE(a)	25	-	70	5	-
5 PS/5 PP(a)	-	49	50	_	1
6 PS/4 PP(a)	-	35	60	-	5
7 PS/3 PP(a)		25	70	_	5
5 PS/5 PE(b)	50		48.5	1.5	-
6 PS/4 PE(b)	40		58.5	1.5	-
7 PS/3 PE(b)	30	-	68	2.0	-
5 PS/5 PP(b)	-	50	48	-	2
6 PS/4 PP(b)	-	35	60	-	5
7 PS/3 PP(b)		25	70	-	5
5 PS/5 PE(c)	45	-	50	5	-
6 PS/4 PE(c)	35	_	60	5	-
7 PS/3 PE(c)	25	_	70	5	-
7 PS/3 PP(c)	_	30	68	-	2

TABLE III PS/PO blend composition

- (b) The compounding was performed by a reactive extrusion by means of a co-rotating twin-screw extruder, from BI-VIS-CLEXTRAL-Torino, Italy, computer assisted, operating with the following parameters: the temperatures of the ten zones varied between 120-233-188°C for PS/PE blend and 130-240-196°C for PS/PP blend for feeding zone, middle zones and output zone, respectively, screw speed of 200 and 300 rpm, pressure of 600 MPa and 46 MPa, energy consumption of 1177 and 3003 W for PS/PE and PS/PP blends, respectively, 20 modular elements computer-assisted assure an extensive mixing and a good temperature control. At least three combinations of processing parameters were tested selecting the most suitable according to quality and properties of materials. The optima processing parameters for PS/PO blends on a reactive co-rotating twin-screw extrusion-injection compounding installation provided with 20 modular elements and L/D = 4have been established such as: for PS/PE blends: temperature of 230°C, pressure of 600 MPa and screw speed of 200 rpm while for PS/PP blends the processing parameters were: temperature of 240°C, pressure of 4600 MPa and screw speed of 300 rpm.
- (c) Intensive mixing and granulating on a HENCKEL mixer provided with a counter-rotating twin-screw extruder, KO-BUSS MDK/140

type using the following parameters: temperature of the feeding zone of 190°C, temperature of the extrusion zone of 220°C, die temperature of 230°C and rotation speed of 200 rpm.

After mixing and processing, the extrudated or injected specimens have been irradiated with an ILU source at an intensity of 8 A that produces 0.05 MGy/10 min. Irradiation time was 10 min.

The mixing ratios around middle composition range were selected, because the final purpose of this project is to obtain asymmetric membranes [16, 17] based on these blends.

2.3. Methods of Investigation

2.3.1. Processing Behavior

Processing behavior has been appreciated from two kinds of experiments: (1) Brabender experiments in batch mixer and extrusiometer in the conditions described above. (2) Injection procedure on a KUASSY 100/25 machine, working parameters varying in the following limits: temperature: $180-230^{\circ}$ C, injection pressure: 800-1300 MPa, injection speed: 10-30 mm s⁻¹. The length and mass of the injected coil in a special mold have been determined.

2.3.2. Physical Properties

Physical properties determined according to well-known standard methods were: density, melt flow index (CEAST-Torino, instrument), melting interval and crystallization temperature (Boetius VEB ANALYTIK PMMK 78 Dresden micromass), Vicat temperature (CEAST-Torino, instrument).

Physico-mechanical characteristics were determined on at least 10 specimens $(120 \times 10 \times 4 \text{ mm})$ according to the standard methods as follows: tensile strength (STAS-6642/1973) on a FP 10 type HECHERT VEB-THURINGER dynamometer; flexural strength (ISO-178/1998) by means of a KARL-FRANK BAVARIA, IZOD impact strength (STAS-7310/1987) and CHARPY impact strength (STAS-5801/1986) on a CEAST-TORINO Italy.

2.3.3. Thermomechanical Method

Thermomechanical method is based on the change in the flexural (bending) characteristics *versus* temperature at a very slow speed of action of the applied force. The thermomechanical analysis has been performed on a CEAST Torino modified instrument to measure the flexural deflection under a stress of 1.8 Nm^{-2} with a constant heating rate of $2^{\circ}\text{Cmin}^{-1}$ controlled by a SAMSUNG regulator. The precision of the temperature determination was $\pm 0.2^{\circ}\text{C}$ and precision of the deflection measurement was $\pm 0.01 \text{ mm}$.

The determination of the glass transition temperature of the polystyrene by DSC measurements for PS/PE blends is very difficult due to the overlapping of this transition with the melting of the high density polyethylene. In order to overcame this situation, an adapted DSC method was also utilized, using the HDPE homopolymer as reference material. The DSC curves were recorded on a METTLER DSC 12 E under a 50 cm³min⁻¹ nitrogen flow, at 10°C min⁻¹ heating rate.

3. RESULTS AND DISCUSSION

3.1. Processing Behavior

Torque versus time curves recorded on Brabender plastograph are presented in Figures 1 and 2 for the PS/PE and PS/PP blends, respectively. In the first case, the curves of the blends are placed between those of components, although at the beginning, all mixtures exhibit higher torques than of PS or HDPE. This is a normal situation taking in the view the energy consumption for compounding of the blends. After 4-6 min. of mixing a constant torque value, close to that of HDPE, was obtained for all blends – Figure 1. In the second case, the curves of the PS/PP blends – Figure 2 – lay above the curves of the both components, the torque increases with PS increasing content.

Among the blends, the 5 PS/5 PP one has the lowest torque values, therefore a good processability and low energy consumption – Table IV – and therefore a best processing behavior in respect to that of PS. In the case of PS/PE blends it can be observed that the energy consumption decreases increasing PS content of the blend, the minimum value exhibiting the 7 PS/3 PE blend.



FIGURE 1 Torque – time curves of the PS (------), HDPE (.....), 5 PS/5 PE (-----), 6 PS/4 PE (-----) and 7 PS/3 PE (-----).



FIGURE 2 Torque – time curves of the PS (-----), PP (-----), 5 PS/5 PP (-----), 6 PS/4PP (------) and 7 PS/3 PP (------).

The torques of the previously compounded blends on the twinscrew equipments were, as expected, lower than those of the blends prepared directly on Brabender plastograph, because in this case a

Sample	Energy consumption on Brabender processing (J/15 min.)
HDPE	224.60
5 PS/5 PE(c)	219.46
6 PS/4 PE(c)	224.77
7 PS/3 PE(c)	215.87
5 PS/5 PP (c)	180.75
6 PS/4 PP(c)	195.74
7 PS/3 PP(c)	210.77
PP	135.60

TABLE IV The energy consumption for the mixing of the PS with PO on Brabender plastograph

supplementary energy was already consumed for mixing on compounding installation.

The variation of the length (L) and mass (m) of the injected coil have been used to determine the behavior during the extrusion-injection processing. These characteristics were optima (maximum values) in the following conditions: temperature of 210° C, pressure of 1300 MPa, injection speed of 20 min s^{-1} . The dependence of the length and mass of the injected coil on the blend composition is given in Figures 3 and 4 for the PS/PE and PS/PP blends, respectively. It can be easily



FIGURE 3 Variation of the length and the mass of the injected coil with composition of the PS/PE.



FIGURE 4 Variation of the length and the mass of the injected coil with composition of the PS/PP blends.

remarked that for the PS/PE blends, the both processing characteristics increased with PS content (Fig. 3), the highest values for the length and mass of the injected coil exhibits the 7 PS/3 PE blend. The PS/PP blends exhibit maximum values of these characteristics for a 50/50 mixing ratio, than increasing PS content, the values decreased, although the mass of the injected coil showed higher value than that of PS for the other two studied mixing ratios.

Therefore on both processing equipments the 7 PS/3 PE and 5 PS/5 PP blends exhibit the best processing behavior.

3.2. Physico-mechanical Characterization of the PS/PO Blends

The specimen shape obtained on the three compounding equipments is different, so only the percentage variation of the tensile strength can be used to compare the quality of the obtained materials.

In the three cases, a significant increase of the physico-mechanical properties such as ultimate tensile strength, flexural strength, impact strength of the PS/PO blends in respect to the homopolymer components of the blends, especially in respect to PS – Figures 5 and 6 Tables V and VI-was found. The increase of the tensile strength is of maximum 45% for the blends obtained on Brabender plastograph – Figure 5a – and higher than 52% for the blends prepared



FIGURE 5 Average percentage increase of the ultimate tensile strength (TS) and elongation at break (Elong) in respect with the value of the PE or PS versus composition of the PS/PE blend prepared on Brabender plastograph (a) and on a twin-screw extruder-injection installation (b).

on extrusion-injection compounding device – Figure 5b. The tensile strength of the PS/PP blends has lower values compared with the value of the PP, but a little higher compared with the value corresponding to PS – Figures 6a and 6b.

Unfortunately the elastic components was decreased both for PS/PE and PS/PP blends.

The best values were obtained for the bending and impact resistance, the both being very important for practical applications – Tables V and VI.

The values of the physico-mechanical properties of the blends increase with PS content. The increase of the flexural strength (bending



FIGURE 6 Average percentage increase of the ultimate tensile strength (TS) and elongation at break (Elong) in respect with the value of the PP or PS *versus* composition of the PS/PP blend prepared on Brabender plastograph (a) and on a twin-screw extruder-injection installation (b).

resistance) of the 7 PS/3 PE blend in respect to that of HDPE is about 2 times while, the IZOD impact strength increased of 3-4 times in respect to the value of HDPE and 11-12 times in respect to the value of PS.

The physico-mechanical properties of the PS/PP blends – Table VI – exhibit similar variation in respect to the values of the components as that presented for PS/PE blend. The flexural and the impact strengths significantly increased. The PS/PP blends exhibit the

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No.	Characteristic	5 PS/5 PE (b)	5 PS/5 PE (c)	6 PS/4 PE (b)	6 PS/4 PE (c)	7 PS/3 PE (b)	7 PS/3 PE (c)	Sd	HDPE
_	Tomilo structh $M^{-2} \sim 10^{-3}$	366	242	VLC	367	101	362	000	750
T	I CHISHE SHERE IN INTEL X IO)	<i></i>		1/0	700	100	COC	070	007
0	Elongation at break (%)	17	20	9	×	0	0	0	450
ŝ	Bending resistance $(Nm^{-2} \times 10^{-3})$	143	138	182	184	202	206	120	<u> 06</u>
4	IZOD impact strength (kJ m $^{-2}$)	8.5	8.4	10.5	9.8	12.8	11.2	1.0	3.0
S	CHARPY impact strength (kJ m ⁻²)	5.42	5.04	7.26	6.21	8.64	8.16	0.0	1.2

TABEL V Physico-mechanical properties of HDPE, PS and their blends of series (b) and series (c)

		indrid imaiinina	TAD AL 4 0, 1 1 0 01		Saurae mue (a) eau	5	
No.	Characteristic	5 PS/5 PP (b)	6 PS/4 PP (b)	7 PS/3 PP (b)	7 PS/3 PP (c)	PS	ΡP
	Tensile strength $(Nm^{-2} \times 10^{-3})$	340	346	363	348	320	390
7	Elongation at break (%)	17	20	9	œ	0	0
3	Bending resistance $(Nm^{-2} \times 10^{-3})$	157	160	175	163	120	96
4	IZOD Impact strength (kJ m ⁻²)	6.6	7.5	9.2	8.2	1.0	1.4
5	CHARPY impact Strength (kJ m ⁻²)	5.2	5.4	6.5	6.1	0.0	1.2

TABEL VI Physico-mechanical properties of PS, PP and their blends of series (b) and series (c)

improvement of the flexural and impact strength in respect to that of PP of 2 times and 4.5-6.5 times respectively, while the increase of the flexural strength in respect to PS reached even an increase of 9 times.

The improvement of the physico-mechanical properties is a proof for a good compatibilization of the components. However the systems remain in a state of microphases' separation so explaining the increase of the impact resistance.

The examination of the values of the blends compounded by the ways b and c indicate that the way b is the best method of compounding, because all physico-mechanical indices have higher values than those of the specimens obtained applying the way c of compounding, although the differences are not so important, both ways being suitable for preparation of the blends with satisfactory properties.

Comparing the properties of the blends of series b containing $\sim 2 \text{ wt\%}$ SEBS or SEP compatibilizing agent with the properties of the blends of series c containing 5 wt% SEBS or SEP clearly results that the increase of the compatibilizing agent over 2 wt% does not lead to the further increase of the mechanical properties. This conclusion is in accordance with the literature results [14, 15].

3.3. Thermal Characterization of the Blends

The data of Table VII give the dependence of the global thermal characteristics on the blend composition. The melt flow rate has higher values than that of HDPE. The 5PS/5 PE blend exhibits the highest values of melt flow rate (MFI). The density of the blends increases with PS content.

The melting and crystallization temperatures of the blends are intermediary between those of the components. The incorporation of the PS amorphous component in HDPE or PP crystalline component leads to the considerable enlargement of the melting interval and to the decrease of the crystallization temperature. The Vicat temperature of the blends exhibits higher values than that of PS.

All global thermal properties are particular for each blend and different from the values calculated as simple average of the properties of the components. It can be concluded that the systems exhibit a good compatibility degree.

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seir blends	7 PS/3 PP	1.0132	1.73	115-160	127	103
S, PP and th	6 PS/4 PP	1.0004	2.42	120-157	128	105
he HDPE, P	5 PS/5 PP	0.9882	2.52	125-150	130	107
eratures of t	PP	0.936	2.69	168-172	138	150
verage temp	ΡS	1.0482	2.50	115-175	I	96
and Vicat a	7 PS/3 PE	1.0008	1.95	115-165	125	102
ystallization	6 PS/4 PE	0.9901	2.47	120155	127	106
g interval, cr	5 PS/5 PE	0.9802	2.95	125-150	128	108
nsity meltin	HDPE	0.963	1.26	136–145	130	ł
TABEL VII Melt flow rate, de	Characteristic	Density $(g \text{ cm}^{-3})$	Melt flow rate (g/10 min)	Melting or softening interval (°C)	Average crystallization temperature (°C)	Average Vicat temperature (°C)

The relaxation phenomena determined from flexural characteristics at low heating rate are close related on the morphology of the blends.

The bending deflection versus temperature curves are presented in Figures 7 and 8 for PS/PE and PS/PP blends, respectively. The temperature of the beginning of the deflection was assigned to a α -relaxation temperature according to the literature data [18]. The strain of HDPE becomes evident below 20°C and is developing on a large temperature interval from $< 20^{\circ}$ C to 58° C as a rubbery behaviour. PS is a rigid polymer resistant to flexural deformation up to 88.5°C when the deflection starts and is total inside 8°C temperature interval. The slope of the curve is very high so a viscous flow can be supposed. In the case of the PS/PE blends, the flexural deflection increases from 60 to 63 and to 67.5°C with increasing PS content for 50, 60 and 70 wt.%, respectively and the shape of the curve is particular for each blend, this situation being much better evidenced for PS/PP blend. The flexural deflection for blends increases from 46.5 to 58 and 65°C, respectively, with PS increasing content from 50, 60 and 70 wt% - Figure 8.



FIGURE 7 Thermomechanical curves of PS, HDPE and their blends.



FIGURE 8 Thermomechanical curves of PS, PP and their blends.

The curve of the 5 PS/5 PP blend is similar with that of PP between -20 and 40° C temperature interval, then the curve is parallel with that of PS. In the curve of the 7 PS/3 PP blend a transition is recorded in a narrower temperature interval of 21°C (from 67.5 to 88.5°C).

The slope of the thermomechanical curve of 7 PS/3 PP blend is very high, it exhibiting a viscous flow behavior inside 15° C (from 65 to 80°C). The end of deflection of all blends is 88.5°C very close to glass transition temperature of PS.

The α -relaxation temperatures of the both kinds of blends are higher than the additive average values – Figure 9. A similar variation was found also in Vicat temperatures and also in Tg of polystyrene determined by DSC – Table VIII and Figures 10 and 11.

By using HDPE as reference material, the glass transition temperature of PS appears very clear – Figure 10 and their dependence on the composition can be established – Table VIII. The small increase of the Tg values of the blends in respect with that of PS, especially for PS/PE blends could be due to of a possible crosslinking reaction that takes place during γ -irradiation that is much probable in the blends containing HDPE. The melting temperature and the melting heat of the PO in the mixtures are lower than those



FIGURE 9 α -relaxation and glass transition temperatures versus composition of the PS/PE (a) and PS/PP (b) blends.

of unmixed PO and crystallinity degree decreased very much – Table VIII. This is in good accordance with the increase of the impact strength of the blends.

To explain the dependence of the α -relaxation and glass transition temperatures on the blend composition must taken into account both the possibility of an interphasic layer formation [19] and also the changes occurring during γ -irradiation [20, 21].

No.	Sample	$Tg(^{\circ}C)$	Tm (°C)	$\Delta Hm~(Jg^{-1})$	Crystallinity degree* (%)
1	PS	102.5	_		_
2	HDPE	-	137	217.9	77
3	PP		175	88.12	35.2
4	5 PS/5 PE	105	135	53.48	18.9
5	6 PS/4 PE	108	134.5	45.56	16.1
6	7 PS/3 PE	107	134	35.70	12.6
7	5 PS/5 PP	105.5	164	39.02	15.6
8	6 PS/4 PP	105	163.5	25.98	10.4
9	7 PS/3 PP	105.5	162	13.34	5.3

TABLE VIII DSC results for the PS, PO and their blends

* Crystallinity degree is the ratio $\Delta H_m / \Delta H_m^0$, where ΔH_m is melting heat of the sample and ΔH_m^0 is melting heat for 100% crystalline PO, taken from literature, they are 283 J/g for PE and 250 J/g for PP [22].



FIGURE 10 DSC curves of the PS (_____), 5 PS/5 PE (-----), 6 PS/4 PE (------) and 7 PS/3 PE (_____).

An interphasic layer with particular properties which assures an increase of the physico-mechanical properties and of the relaxation/ transition temperatures should appear during intensive mixing of the three components PS/PO and KRATON-type copolymers as compatibilizing agents. The γ -irradiation leads to the formation of the cross-links. Both modifications lead to observed changes in mechanical and thermal properties.



FIGURE 11 DSC curves of the PS (----), PP (----), 5 PS/5 PP (----), 6 PS/4 PP (----), 7 PS/3 PP (----).

4. CONCLUSIONS

The compatibility and properties of PS/PO blends are strongly dependent on the processing conditions. The optima processing parameters for PS/PO blends on a reactive co-rotating twin-screw extrusion-injection compounding installation provided with 20 modular elements and L/D = 4 have been established such as: for PS/PE blends: temperature of 230°C, pressure of 600 MPa and screw speed of 200 rpm and for PS/PP blends at temperature 240°C, pressure 4600 MPa and screw speed 300 rpm.

7 PS/3 PE and 5 PS/5 PP blends exhibit a good processing behaviour.

On the basis of the processing behaviour and mechanical properties has been established that an amount of 2 wt% compatibilizing agent is enough to achieve a satisfactory compatibilization of the blends.

A thermomechanical method based on flexural characteristics was proposed for rapid thermal characterization of the thermoplastic and rigid polymers and polymer blends.

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