

Study on Compatibility of PP–STC Blends Functionalized by Ultraviolet Irradiation

RONG GUAN

Polymer Research Institute, Chengdu University of Science and Technology, Chengdu, 610065, China

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ABSTRACT: To enhance the compatibility of polypropylene (PP) with sericite–tridymite–cristobalite (STC), polypropylene was irradiated by low-energy ultraviolet (UV) irradiation within 2.5 h to introduce the oxygen-containing groups onto the PP chains. From blending the UV-irradiated polypropylene with STC, the effects of the compatibility on the structure and properties of PP–STC blends could be investigated by differential scanning calorimetry, thermogravimetry, thermomechanical analysis, wide-angle X-ray diffraction, scanning electron microscopy, mechanical properties, and melt index measurements. The experimental results show that for the UV-irradiated PP–STC blends, the degree of crystallinity is smaller than those with an unirradiated PP–STC blend, the thermal degradation temperatures are increased and the thermal stability is improved. Furthermore, with the UV time varying from 0 to 2.5 h, the melt index increases from 2.73 to 3.39 g/10 min, and the mechanical properties improve, exhibiting stiffening and toughening effects. Also a toughened morphology can be observed because of the enhanced interaction between UV-irradiated polypropylene and STC. © 2000 John Wiley & Sons, Inc. *J Appl Polym Sci* 77: 96–103, 2000

Key words: polypropylene; ultraviolet irradiation; compatibility; blend; structure and property

INTRODUCTION

Polypropylene (PP) is a widely used and valuable plastic since its properties put it between the categories of engineering and general plastics. Through blending, filling, and reinforcing, it is possible to prepare polypropylene-based engineering plastics,¹ that is, to prepare high-performance PP materials. In polypropylene blends, the compatibility, which relates to the phase interaction of the components in the blends, is a very important factor when determining the properties of the blends.^{2,3} However, because polypropylene is a nonpolar polymer, it is usually incom-

patible with inorganic fillers or engineering plastics. The usual way to enhance the compatibility of polypropylene blends is by adding compatibilizers, such as poly(styrene-*block*-ethylene/butylene) (SEBS),⁴ glycidylmethacrylate-grafted polypropylene (PP-*g*-GMA),⁵ and maleic anhydride-grafted polypropylene (PP-*g*-MAH),⁶ or by pretreating inorganic fillers with silane-containing additives. However, preparing the compatibilizers by the melting, solution and solid techniques is usually complex, even toxic under some conditions. The residual monomer in the compatibilizers may damage the electrical, thermal, and hygienic properties of polypropylene blends. When grafting the MAH onto polypropylene chains, the possibility for continued MAH homopolymerization into block side chains has been suggested to be unlikely based on ceiling temperature considerations⁷ (i.e., at typical melt temper-

Correspondence to: R. Guan (rongguan@public.wh.hb.cn).
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ature of ca. 190°C); however, a portion of the grafted MAH groups may form crosslinks between polypropylene chains.⁸ Furthermore, pre-treating inorganic fillers with silane-containing additives will decrease the tensile strength and Young's modulus of polypropylene blends.

Since 1990 Xi Xu⁹ and his research group have developed a new way to functionalize polyethylene (PE) by using γ -ray, electron beam, ultraviolet (UV), and microwave irradiation techniques to irradiate PE in an air atmosphere without adding any other additives and without chemical pollution, successfully introducing the polar groups onto PE chains. They then blended such functionalized PE with inorganic fillers and engineering plastics, and they obtained stiffened and toughened PE materials. But polypropylene is quite different from PE upon irradiation—it is quite vulnerable among alkenic polymers because of its tertiary hydrogen atom.¹⁰ Upon irradiation, especially in air, with polypropylene there occurs mainly a chain scission,^{11–13} its amount depending on the irradiation energy time, temperature, and so forth. The higher and longer the energy and time, the more chain scissioned will be the polypropylene. As the chain scission of polypropylene is unavoidable during the processing, grafting, and even crosslinking¹⁴ and because a small amount of chain scission will not affect the main properties of polypropylene, it seems acceptable to allow polypropylene degrade lightly.

This article discusses the exploration using low-energy UV to irradiate polypropylene in order to introduce the oxygen-containing groups onto the polypropylene chains. Through the investigation using FTIR, ESCA, ultraviolet spectrum, and elementary analysis methods, the $-\text{C}(=\text{O})\text{OC}-$, $-\text{C}(=\text{O})\text{CH}_3$ and $-\text{CH}_2\text{C}(=\text{O})\text{CH}_2-$ groups have been introduced onto the polypropylene chains.¹⁵ Since polypropylene is not expected to absorb light at wavelengths of more than 200 nm, such oxygen-containing groups could be introduced onto its chains because small quantities of external impurities such as conjugated double bonds and/or carbonyl groups may be responsible for the absorption of radiation of more than 200 nm.¹¹ This article reflects a different condition—a difference in UV irradiation conditions—than that represented in all the other literature. Here, UV irradiation was done using long wavelengths and a low-energy UV source and a short irradiation time, because we tried to keep the chain scission of polypropylene to a minimum. According to the literature,^{16–18} a short wavelength (254

nm), (for example, more than 50 h) a higher-energy UV source, and a longer UV irradiation time have been used, which caused the severe chain scission that leads to the deterioration of polypropylene properties.¹¹ There is a lot of literature dealing with polypropylene photodegradation mechanism and photolysis products,^{19–23} in which it has been shown that ketone is the major product of the photodegraded polypropylene. But fewer articles tell about the interesting use of the oxygen-containing groups at the polypropylene chains to enhance the compatibility of polypropylene blends.

The aim of the investigation detailed in this article was to use polypropylene functionalized by UV irradiation to enhance the compatibility of polypropylene with sericite–tridymite–crystalalite (STC) blends. We examine the structure and properties of ultraviolet-irradiated PP–STC (80 : 20 wt %) blends and reveal the effects of enhanced compatibility on the structural and properties of PP–STC blends. Differential scanning calorimetry (DSC), thermogravimetry (TG), and thermo-mechanical analysis (TMA) were used to contrast the thermal properties of UV-irradiated PP–STC blends with the thermal properties of UV-irradiated PP. Wide-angle X-ray diffraction further showed the crystallinity variation, and mechanical and melt index measurements were carried out to show the property changes. Scanning electron microscopy (SEM) depicted the toughened morphology for the UV-irradiated PP–STC blends.

EXPERIMENTAL

Materials

A commercial grade of isotactic PP (PP2401, Yanshan Petrochemical Company, China) was used, with weight-average molecular weight (M_w) of 240,000 and a melt index of 2.5 g/10 min. The filler sericite-tridymite-cristobalite (STC), a product of Sichuan Powder Engineering Center of Research and Development (China), has a density of 2.7–3.0 g/cm³, an average diameter of 1.6 μm , and a specific surface area of 6.91 m²/g. The major compositions of STC are listed in Table I.

Ultraviolet Irradiation

A 500-W Ga-I lamp manufactured by Chengdu Lamp Factory (China) was used. The Ga-I lamp

Table I Chemical Compositions of Sericite-tridymite-cristobalite

Component	Content by Weight	Component	Content by Weight
SiO ₂	70.69%	Al ₂ O ₃	16.24%
Fe ₂ O ₃	3.11%	ZnO	2.37%
K ₂ O	6.07%	MgO	0.16%
CaO	1.22%	TiO ₂	0.14%

has a tubular shape, with a wavelength in the range of 340–370 nm, and the UV intensity is 3.2×10^{-2} W/m²; the UV lamp is initiated by a trigger. The UV irradiation process was carried out at room temperature in air. The PP pellets (which contained the antioxidants when manufactured in the factory) were placed in a disk container and irradiated for different time intervals varying from 0.5 to 2.5 h, with the distance between the lamp and the substrate being about 28 cm.

Preparation of PP–STC Bends

To prohibit polypropylene degradation, 0.25% tetrakis[methylene-3-(3,5-di-*tert*-butyl-4'-hydroxyphenyl) propionate]methane and 0.25% dilauryl thiodipropionate (which has a synergistic effect when used with the former antioxidant) have been added into PP–STC blends. The UV-irradiated PP pellets, STC filler, and antioxidants were blended on a two-roll mill at $168 \pm 2^\circ\text{C}$ for 12 min in an aerobic atmosphere. The blending temperature is a few centigrade degrees above the melt point of polypropylene in order to allow polypropylene to have a certain melt strength to process. The milled PP–STC blends were then melted and pressed at $192 \pm 2^\circ\text{C}$, 4 MPa for a few minutes in a hydraulic press, then cold-pressed in another hydraulic press under about 8 MPa pressure for 15 min at room temperature to prepare the testing plates. These PP–STC plates were then cut into bar specimens according to the test standards.

Differential Scanning Calorimetry

Calorimetry measurements were carried out on a DuPont 2910 thermal analysis system with software V4.10. The sample weighing was carried out on a DuPont TG2950 with specimens of 3–4 mg each, and the thermograms were recorded under nitrogen flow (1×10^{-5} m³/min), using a heating rate of 10°C/min from room temperature to

190°C. To erase any thermal history, a reheating scan (the second heating run) was conducted with the same heating rate after the specimen was heated to 190°C and cooled down in air. The melting temperature (T_m) and heat of fusion or enthalpy (ΔH_f) were measured from thermograms. The degree of crystallinity was also determined from $X = \Delta H_f / \Delta H_f^{\circ} \%_o$ (where for PP, the equilibrium heat of fusion ΔH_f° is 209 J/g^{24,25}).

Thermogravimetry

Thermogravimetry (TG) measurements were carried out on a DuPont 2950 thermal analysis system at a heating rate of 10°C/min and with a nitrogen flow 1×10^{-5} m³/min. Temperature was set from room temperature to 600°C.

Thermomechanical Analysis

Thermomechanical analysis (TMA) was performed using a DuPont 2000 thermomechanical analyzer at a heating rate of 5°C/min from room temperature to 160°C. The dimensions of the specimen were 4 mm long, 4 mm wide, and 4 mm thick.

Wide-Angle X-ray Diffraction

WAXD analysis was performed using a MaxIII A (Rigaku) wide-angle X-ray diffractometer (Ni-filtered CuK α radiation). The high voltage was 35 kV, tube current 20 mA, and the wavelength 1.54 nm in the diffraction angle range $2\theta = 10\text{--}49^\circ\text{C}$.

Scanning Electron Microscope

SEM photographs were taken on a HITACHI-8520 electron microscope. The morphology of the cross section of tensile samples was observed on a carbon replica and golden-coated sample.

Melt Index

The melt index of PP–STC blends was measured using a CS-127 capillary viscometer of the extrusion type under a 230°C, 2.16 kg load.

Mechanical Property Measurement

Mechanical property measurements were carried out on an Instron 4302 all-purpose tester. The specimens were cut into dumbbell shapes, with the size of the specimen and the test conditions following the ASTM D268 conditions. The impact strength measurement was carried out according

Table II First Melting DSC Data of PP-STC (80 : 20) Blends

Sample	UV Time (h)	T_p (°C)	Enthalpy (J/g)	Crystallinity (%)
B1	0.0	166	68.0	32.5
B2	0.5	165	62.4	29.9
B3	1.0	165	64.9	31.1
B4	1.5	165	66.6	31.9
B5	2.0	163	67.0	32.1
B6	2.5	165	71.7	34.3

to ISO180-1993E. The results reported here are the average of five tests.

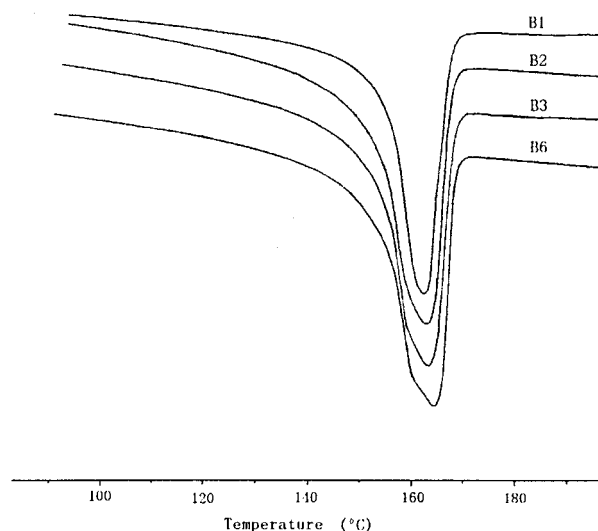
RESULTS AND DISCUSSION

Thermal Characterization

Thermal characterization gives a fairly good idea of changes taking place between the UV irradiated polypropylene and the filler STC. The author's work¹⁵ has shown that within 2.5 h of the onset of UV time, the UV-irradiated polypropylene displayed two melting peaks, around 150°C and 165°C, which corresponding to the β -phase and α -phase, respectively, of polypropylene. And the degree of crystallinity of UV-irradiated polypropylene (when UV time is more than 1 h) is larger than those of the unirradiated polypropylene. Blending the UV-irradiated polypropylene with STC, produced thermal properties that are summarized in Tables II and III. From Tables II and III and from Figure 1, it is clearly evident that there is only one melting peak for the UV-irradiated PP-STC blends, and the temperature of the melting peak is around 165°C, corresponding to the typical melting point

Table III Second Melting DSC Data of PP-STC (80 : 20) Blends

Sample	UV Time (h)	T_p (°C)	Enthalpy (J/g)	Crystallinity (%)
B1	0.0	166	77.0	36.8
B2	0.5	164	63.6	30.4
B3	1.0	163	66.7	31.9
B4	1.5	164	72.7	34.8
B5	2.0	163	73.6	35.2
B6	2.5	163	73.9	35.4

**Figure 1** Melting thermograms of PP-STC (80 : 20) blends.

of polypropylene's α -phase spherulite. After UV irradiation, the melting peak temperature of PP-STC blends reduced by about 1 degree to 3°C. This small reduction can be attributed to the light chain scission of polypropylene and the defects caused by UV irradiation, as can be seen from the small increase in the melt index for the UV-irradiated polypropylene.¹⁵

An interesting observation during the heating cycles relates to changes in the heat of fusion values (ΔH_f) and the degree of crystallinity. Under the experimental conditions examined, the heat of fusion and the degree of crystallinity of UV-irradiated PP-STC blends are smaller than those of the unirradiated PP-STC blend (except for the UV 2.5-h PP-STC blend in the first heating run). These results are contrary to the results with the UV-irradiated polypropylene.¹⁵ For example, at 2.5 h UV time, the crystallinity of the PP-STC blend is 35.4% (in the second run) compared with 36.8% for the unirradiated PP-STC blend, while polypropylene is 69.8%¹⁵ compared with 34.8% for the unirradiated polypropylene. This suggests there should be a phase interaction between UV-irradiated polypropylene and STC: If there is no the interaction between the UV-irradiated polypropylene and STC, the amount of crystallinity is expected to increase higher than the values in Table II or Table III after polypropylene is UV irradiated since more polypropylene is available for recrystallization and as this polypropylene is no longer interacting with STC and in the absence of miscibility, STC has less of

Table IV T_G Analysis of PP-STC (80 : 20) Blends

Sample	UV Time (h)	T_1 (°C)	T_p (°C)	T_f (°C)	T_{50} (°C)
B1	0.0	393	428	462	434
B2	0.5	416	442	475	458
B3	1.0	429	450	481	467
B4	1.5	431	455	482	468
B5	2.0	395	429, 459	473	440
B6	2.5	440	467	484	471

an influence on depressing crystallinity. Moore and Kaur²⁶ have reported the same phenomenon in the case of PEO blends.

After showing a small decrease, the heat of fusion and the degree of crystallinity of the UV-irradiated PP-STC blends increases with increasing UV time, and the heat of fusion is higher and the degree of crystallinity is larger in the second heating run than those in the first heating run. This accounts for the chain scission resulting in small molecules with higher freedom of movement, which may allow them to rearrange and order themselves in the solid phase,²⁷ leading to crystallinity increasing. The more there's chain scission of polypropylene, the larger the crystallinity. In addition, during the first heating run some defective structure areas have been adjusted, and some defective segmental crystals rearranged into the crystal lattice to recrystallize, in doing so making the crystallinity higher in the second heating run.

The thermal degradation behavior of PP-STC (80 : 20) blends was investigated by thermogravimetric analysis. To show the detailed thermal degradation data, the initial degradation temperature (T_i), the final degradation temperature (T_f), the degradation temperature at half weight loss (T_{50}), and the peak degradation temperature of the derivative thermogravimogram (T_p) were obtained from the thermogravimograms and are listed in Table IV. It is apparent that the UV-irradiated PP-STC blends degrade at a higher temperature while the unirradiated PP-STC blend degrades at a lower temperature. That is, the degradation temperatures of the UV-irradiated PP-STC blends are higher than those of the unirradiated PP-STC blend. Therefore, the UV-irradiated PP-STC blends are more thermal stable than the unirradiated PP-STC blend. For instance, at 2.5 h UV time, the UV-irradiated PP-STC blend shows definite improvements in the

initial, final, half weight loss, and peak degradation temperatures by as much as 47°C, 22°C, 37°C, and 39°C, respectively, relative to the unirradiated PP-STC blend, while the corresponding UV-irradiated polypropylene is -5°C, -10°C, 1°C, and -5°C, respectively.¹⁵ Clearly, the thermal stability of the UV-irradiated PP-STC blends is improved. The necessity of an interaction between the UV-irradiated PP and STC, which enhances the compatibility of PP-STC blends, is possibly the reason for the enhancement of the thermal stability of the UV-irradiated PP-STC blends.

The thermal deformation of PP-STC blends by temperature was investigated by the thermomechanical analysis, shown in Figure 2. The UV-irradiated PP-STC blends show a smaller thermal deformation than those of the unirradiated PP-STC blend at the same temperature; therefore, the thermal stability related to the thermal mechanical deformation of the UV-irradiated PP-STC blends is also improved. This should be a result of the enhanced interaction between UV-

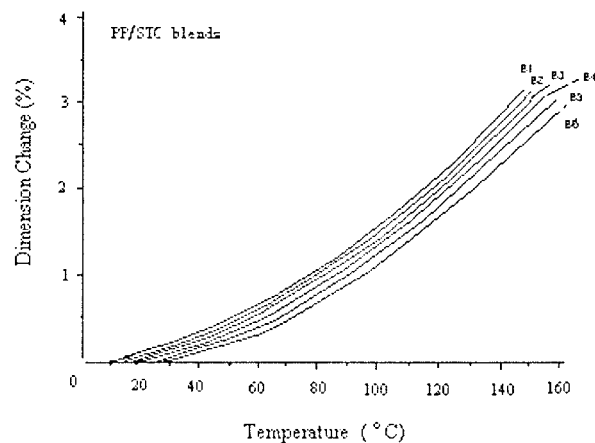


Figure 2 Dimension change versus temperature curves of PP-STC (80 : 20) blends.

Table V WAXD Analysis of PP-STC (80 : 20) Blends

Sample	UV Time (h)	Crystallinity (%)	d-Spacing of PP-STC Crystalline Plane (Å)			
			(110)	(040)	(111)	(131)
B1	0.0	42.3	6.157	5.157	4.180	4.006
B2	0.5	37.5	6.133	5.142	4.153	4.008
B3	1.0	39.5	6.224	5.206	4.201	4.024
B4	1.5	40.4	6.178	5.176	4.196	4.037
B5	2.0	41.9	6.166	5.169	4.185	4.005
B6	2.5	42.1	6.202	5.197	4.194	4.039

irradiated PP and STC, the polar groups introduced onto the polypropylene chains, and the increased crystallinity.

Wide-Angle X-ray Diffraction Characterization

Table V lists the WAXD crystallinity and the d-spacing of the crystalline plane of the typical polypropylene's α -phase spherulite. The degree of crystallinity of the UV-irradiated PP-STC blends is lower than those of the unirradiated PP-STC blend, while the degree of crystallinity of the UV-irradiated PP-STC blends increases with increasing UV time. This tendency toward variation in crystallinity is similar to DSC analysis (in the second heating run of DSC analysis). Still the WAXD crystallinity of the UV-irradiated PP-STC blends is smaller than that of the UV-irradiated polypropylene.¹⁵ As in the DSC analysis, this proves that there is some phase interaction between the UV-irradiated PP and STC. This phase interaction depresses the crystallization of PP-STC blends, thus making the UV-irradiated PP-STC blends show a lower crystallinity. In addition, in the UV-irradiated blends, there are not any identifiable crystallographic transitions, but

there are some crystal structure changes, characterized by the variation of crystalline planes (110), (040), (111), and (131).

Properties of PP-STC Blends

The tensile strength (σ), elongation at break (ϵ_b), and Young's modulus (E) of PP-STC blends were obtained from the stress-strain curves and are presented in Table VI. The notched impact strength is also listed in Table VI. It can be seen that the tensile strength, elongation at break (after UV time is more than 30 min), Young's modulus, and notched impact strength of the UV-irradiated PP-STC blends are increased, although some increase only a small amount. Young's modulus had the maximum increase, where a improvement by as much as 404 MPa can be observed at 2 h UV time, while the increase is only 124 MPa for the UV-irradiated polypropylene under the same UV irradiation conditions.¹⁵ So it some phase interaction between the UV-irradiated polypropylene and STC enhancing the compatibility, stiffness, and toughness of the UV-irradiated PP-STC blend may be inferred. It

Table VI Mechanical Properties of PP-STC (80 : 20) Blends

Sample	UV Time (h)	σ (MPa)	ϵ_b (%)	E (MPa)	Notched Impact Strength (J/m)
B1	0.0	29.6	8.7	1791	39.9
B2	0.5	31.1	8.7	1868	46.7
B3	1.0	31.1	9.3	1890	47.2
B4	1.5	31.8	13.3	1871	48.5
B5	2.0	32.9	10.9	2195	48.5
B6	2.5	30.3	9.1	2048	49.8

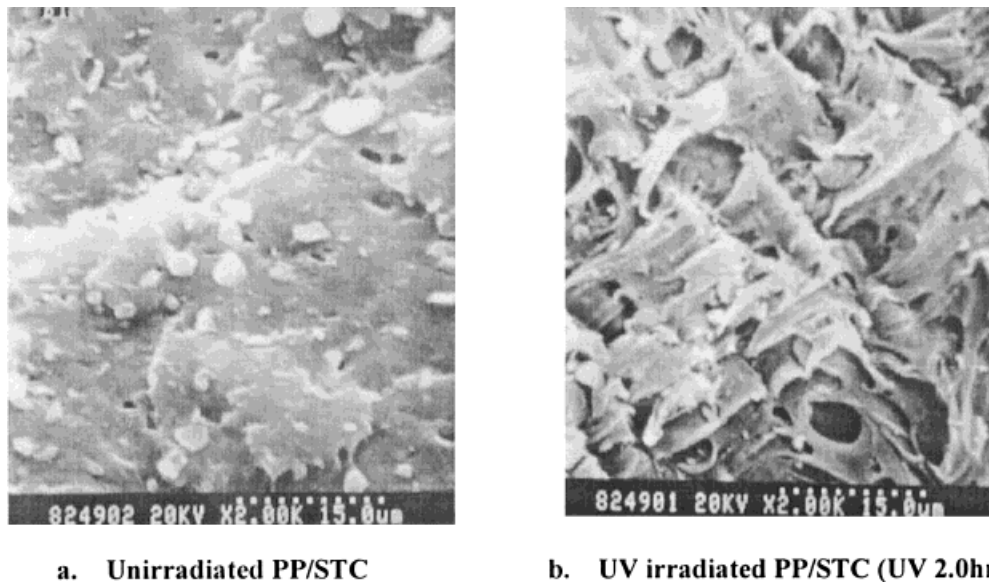


Figure 3 SEM photograph of the tensile fracture surface of PP-STC (80 : 20) blends.

is therefore appropriate to seek a morphological explanation to explain this effect.

Figure 3 shows the SEM photographs of the cross section of PP-STC blends. The STC particle is clearly observed in Figure 3(a) of the unirradiated PP-STC blend and is poorly dispersed in the polypropylene matrix, while in the Figure 3(b) of the UV-irradiated PP-STC blend, the STC particle is closely connected with the polypropylene matrix and is well dispersed. Moreover, the polypropylene matrix displays a fibrous morphology. Hence, from the above facts, a phase interaction between the UV-irradiated polypropylene and STC is clearly demonstrated.

The melt index of PP-STC blends, which reflects the rheological behavior at low shear rate, is shown in Figure 4. As the UV irradiation time increases to 2.5 h, the melt index of PP-STC blends increases from 2.7 g/10 min to 3.4 g/10 min, the result of the chain scission of the polypropylene being UV irradiated. But under the same UV irradiation conditions, the melt index of the polypropylene increases from 2.5 g/10 min to 3.3 g/10 min.¹⁵ Apparently, the rate of increase in the melt index of the PP-STC blends is lower. This indirectly reflects an interaction between the UV-irradiated PP and STC that depresses the melt index increase for the UV-irradiated PP-STC blends.

CONCLUSIONS

A novel approach, using the polypropylene functionalized by short-time UV irradiation to enhance the compatibility of PP-STC blends, has been carried out in this study. Through studies of DSC, T_G , TMA, WAXD, SEM, the mechanical properties and melt index measurements, it has been demonstrated that UV-irradiated polypropylene does enhance the compatibility of the PP-

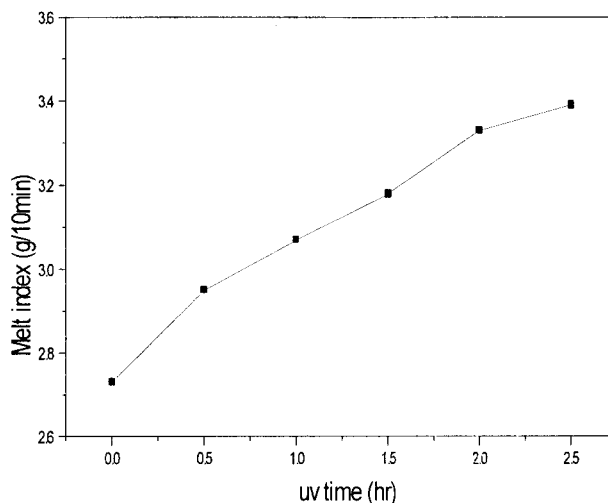


Figure 4 Melt index versus UV time of PP-STC (80 : 20) blends.

STC blend, although in some cases this compatibility effect is not remarkable due to polypropylene chain scission that reduces tensile strength and elongation at break.

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