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Reactive compatibilization of LLDPE/PS blends with a new type of Lewis acid as catalyst

Yanlong Liu · Qiang Shi · Zhuo Ke · Ligang Yin · Jinghua Yin

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Abstract The reactive compatibilization of LLDPE/PS (50/50 wt%) was achieved by Friedel–Crafts alkylation reaction with a combined Lewis acids (Me_3SiCl and $InCl_3{\cdot}4H_2O$) as catalyst. The graft copolymer at the interface was characterized by Fourier transform infrared spectroscopy and the morphology of the blends was analysized by scanning electron microscopy. It was found that the combined Lewis acids had catalytic effect on Friedel–Crafts alkylation reaction between LLDPE and PS, and the catalytic effect was maximal when the molar ratio of $InCl_3{\cdot}4H_2O$ to Me_3SiCl was 1:5. The graft copolymer LLDPE–g–PS was formed via the F–C reaction and worked as a tailor-made compatibilizer to reduce the interfacial tension. The mechanical properties of reactive blend with combined Lewis acids as catalyst was notably improved compared to that of physical LLDPE/PS blend and serious degradation had been decreased compared to the reactive blend system with $AlCl_3$ as catalyst; we interpreted the above results in term of acidity of combined Lewis acids.

Keywords Reactive compatibilization · PE/PS blends · Friedel–Crafts alkylation reaction · Combined Lewis acids

Introduction

Compabilization of polystyrene (PS) and polyethylene (PE) blends has been the subject of considerable research and development in recent decades [1–3]. A convenient route to reactive compatibilize PE/PS blends is the use of the Friedel–

Y. Liu · Q. Shi · Z. Ke · L. Yin · J. Yin (✉)
Changchun Institute of Applied Chemistry, CIAC-CAS Changchun,
130022 Jilin, People's Republic of China
e-mail: yinjh@ciac.jl.cn

Crafts alkylation reaction (F–C reaction) in the melt. The graft copolymer PE–g–PS is formed at interface via F–C reaction, which acts as a “tailor-made compatibilizer” and can strongly modify the morphology, interfacial adhesion and final mechanical properties of blend [4, 5].

$AlCl_3$ is a strong Lewis acid catalyst that was most used to initiate the F–C alkylation reaction on the melt [1–7]. Baker et al. [1] had investigated the catalytic effect of different Lewis acid on the PE/PS blends, they found $SnCl_4$, $FeCl_3$ and $AlCl_3 \cdot 6H_2O$ had no catalytic effect and only $AlCl_3$ can catalyst the reaction effectively. Gao et al. [4] adopted $AlCl_3$ to increase the compatibilization of the LLDPE/PS and LLDPE/HIPS blend, they examined the structure of graft copolymer and confirmed that LLDPE segments were grafted to the para position of the benzene rings of PS. Diaz et al. [3, 5] applied $AlCl_3$ to compatibilize the PE/PS blends, they studied the relation between the initial PE MW and the structure of the graft copolymer, the influence of the graft copolymer architecture and content on the efficiency of blend compatibilization was studied as well [6]. Although $AlCl_3$ successfully initiates the F–C alkylation reaction on the melt state, it has the inherent shortcomings: (1) it is sensitive to humidity and (2) it initiates the degradation of PS at high $AlCl_3$ loading [2, 4, 6, 7].

In recent years, the utilization of indium salts has been dramatically expanded due to their characteristic properties: (1) they readily provide carbon nucleophiles by transmetallation, and (2) can be used in water [8, 9]. However, the low Lewis acidity of the indium salts considerably limits the scope of applicable substrates [10]. To increase the Lewis acidity, Onishi et al. used combined catalyst system, $InCl_3$ with chlorosilane (R_3SiCl), and they reported that the catalytic effect of combined system was comparable to that of $AlCl_3$ in the solution [9, 10]. In this context, the combined Lewis acids are promised to substitute $AlCl_3$ to compatibilize the PE/PS blends in the melt. Here, a combined Lewis acid ($InCl_3 \cdot 4H_2O$ and Me_3SiCl) was chosen to in situ compatibilize LLDPE/PS blend. The effect of the combined catalyst on the formation of graft copolymer, morphology and mechanical properties of blends and degradation of PS are detailed investigated. The possible mechanism is tentatively proposed as well.

Experimental

Materials

PS (GPPS 200D) was purchased from Daqing Inc. Its weight–average molecular weight (M_w) of 2.64×10^5 g mol^{-1} and polydispersity index (M_w/M_n) of 2.49 was determined by gel permeation chromatography (Waters Scientific model 150-CV) with calibration curves for standard PS. LLDPE (DFDA 7042) was purchased from Jilin Petrochemical Co., its M_w is 9.7×10^4, and polydispersity 3.75. The Lewis acids, trimethylchlorosilane (Me_3SiCl, chemical pure) and indium chloride ($InCl_3$, analytical grade), were purchased from Sinopharm Chemical Reagent Co. Ltd. All samples were used without further purification.

Preparation of LLDPE/PS blends

The reactive blend LLDPE/PS (50/50 wt%) was carried out in a mixer (Thermo-Haake, Germany), fitted at 180 °C, 50 rpm and 40 g charges. Before adding the LLDPE/PS blends to the mixer, the combined Lewis acids were mixed for 5 min. After the complete melting and mixing of LLDPE/PS blends was reached, a quantity of combined acids was added to the melt mixture. The reactive blend was performed for 5 min.

Extraction of LLDPE/PS blends

To isolate the PS free polymer, the LLDPE/PS blends obtained were first dissolved in boiling xylene and then precipitated in cold methanol. The precipitates were filtrated and dried in vacuum for 48 h. The purified blends were then extracted by THF to remove unreacted PS homopolymer. The grafting yield was calculated based on conventional graft degree concept [11] by the ratio of weight of reacted PS to the initial loading weight of LLDPE, as shown in Eq. 1. The “Weight $\mathrm{PS}_{\mathrm{graft}}$” is the weight of PS in graft copolymers, the “Weight $\mathrm{LLDPE}_{\mathrm{initial}}$” is the weight of LLDPE in blends and the results were shown in Table 1. The free PS homopolymer in THF was precipitated by methanol, and the precipitates were filtrated, then dried in vacuum for 48 h. For comparison, the physical blends were extracted by THF to dissolve the PS and the free PS was obtained.

$$\text{Grafting yield (\%)} = \frac{\text{Weight PS}_{\text{graft}}}{\text{Weight LLDPE}_{\text{initial}}} \times 100 \tag{1}$$

FTIR spectroscopy analysis

The samples of physical and reactive blends extracted by THF were tested with a Bruker Vertex 70 Fourier transform infrared spectroscopy (FTIR) instrument. Its resolution is 4 cm^{-1} and the scan number is 32. Samples were prepared by pressing at 180 °C for 20 s between two plates applying a pressure of 2.5 MPa.

Table 1 The grafting yield with different loading of combined Lewis acids

LLDPE/PS (wt%)	Catalyst (mmol)		Grafting yield (%)
	$\mathrm{InCl_3{\cdot}4H_2O}$	$\mathrm{Me_3SiCl}$	
50/50	0	0	0
50/50	0.75	3.75	3.3
50/50	1.5	7.5	8.6
50/50	2.25	11.25	13.2
50/50	3	15	12.7
50/50	3.75	18.75	15.3

Emitting scanning electron microscope (ESEM) analysis

The morphologies were examined by an emitting scanning electron microscope (Xl 30 ESEM FEG scanning electron microscope, FEI Company) at accelerating voltage 15 kV. The cryogenically fractured surfaces of specimens were coated with a thin film of gold to prevent charging.

GPC analysis

The free PS homopolymers extracted from LLDPE/PS blends were used for Gel permeation chromatography (GPC) analysis, all the GPC data were obtained with a Waters 410 GPC apparatus. All the samples were dissolved in THF at the same concentration. Then, the sample was injected at 35 °C.

Measurements of mechanic properties

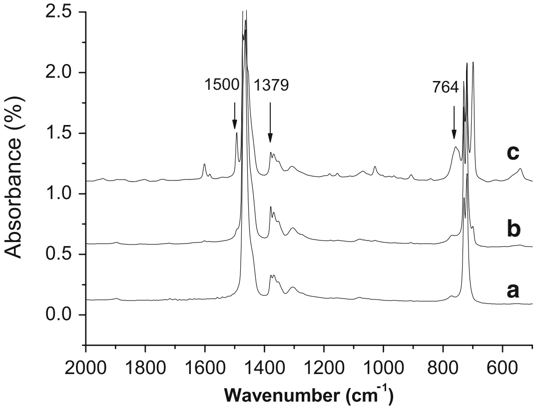
The specimens used for the tensile and impact tests were press molded at 180 °C for 5 min (10 MPa) and then kept at room temperature for 3 days before testing. The tensile test was performed using the dog-bone test specimen with a GB/T 1,040–1,992 standard at room temperature, with a crosshead speed of 50 mm/min. The dimension of the gauge section of the tensile test specimen was 20 × 4 × 1 mm. The Izod impact test was carried out with a XJU-22 impact tester (Chengde test machine company) at room temperature. The diameter and thickness of the specimens were 12 and 3 mm, respectively. The impact strength was taken to be the area under the force–displacement graph in Joules.

Results and discussion

FTIR spectra

Figure 1 shows representative IR spectra of LLDPE (a), sample of physical blends extracted by THF (b) and reactive blend extracted by THF (c). The spectrum of LLDPE shows the peak at 1,379 cm^{-1}, which has been attributed to absorbance of methylene group [11, 12]. The spectrum of the sample of physical blends extracted by THF shows the similar absorbance peak as that of neat LLDPE, indicating that PS has been extracted completely from the physical blends. The spectrum of reactive blend extracted by THF shows the characteristic absorbance of PS at 1,500 and 764 cm^{-1}, the absorption bands at 1,500 cm^{-1} is due to the C=C in-plane stretch vibration of the benzene ring, while the absorption bands at 764 cm^{-1} is due to the aromatic C–H deformation of the substituted benzene ring [1, 13]. The above results confirm that the combined Lewis acids catalyze the F–C alkylation between LLDPE and PS [6, 7].

Fig. 1 FTIR spectra of samples. **a** LLDPE; **b** physical blend extracted by THF; and **c** reactive blend extracted by THF



Morphology of blend

Figure 2 shows the SEM pictures of cryogenically fractured surfaces of LLDPE/PS (50/50 wt%) blends with different amount combined Lewis acids. The corresponding grafting yields of PS are shown in Table 1. There is no grafting yield in physical blend. With the increase of combined Lewis acids addition, the grafting yield increases, indicating increased graft copolymer content in blends. For physical blend (Fig. 2a), particles of PS and the holes left by them during the fracture process can be observed. The fracture is clearly inter-particle and the border is detached. In contrast, the reactive blends catalyzed by combined Lewis acids exhibit a trans-particle fracture (Fig. 2b–f). This change in the fracture mode, resulting from the compatibilization reaction, is consistent with an improvement in interfacial adhesion [3, 6]. In Fig. 2b, when 0.75 mmol $InCl_3{\cdot}4H_2O$ and 3.75 mmol Me_3SiCl is added, the border of LLDPE and PS is still clear, owing to the low grafting yield of PS part in blend (Table 1). When 1.5 mmol $InCl_3{\cdot}4H_2O$ and 7.5 mmol Me_3SiCl is added, it becomes indistinctive compared to that of physical blend, indicating the formation of graft copolymer at interface (Fig. 2c) [4]. With increase of catalysts, a higher PS grafting yield in blends can be found (Table 1), the effect of some elastic chain shrinking after fracture, leading to ring shaped edges around the particles can be observed. These edges are more prominent in Fig. 2d and 2e, providing the evidence of critical micelle concentration [6]. So, no obvious difference of morphology can be observed with further increase of catalyst (Fig. 2f).

Mechanical properties of blends

The effect of combined Lewis acid ($InCl_3{\cdot}4H_2O$ and Me_3SiCl) on the mechanical properties is studied. The results of tensile and impact testing are tabulated in Table 2 in terms of tensile strength, elongation at break and Izod impact strength. It is known mechanical properties, such as tensile strength and impact strength, are important criteria for assessing the compatibilization effect [6, 14], and there is close relation between tensile and impact properties of the blend and the interfacial

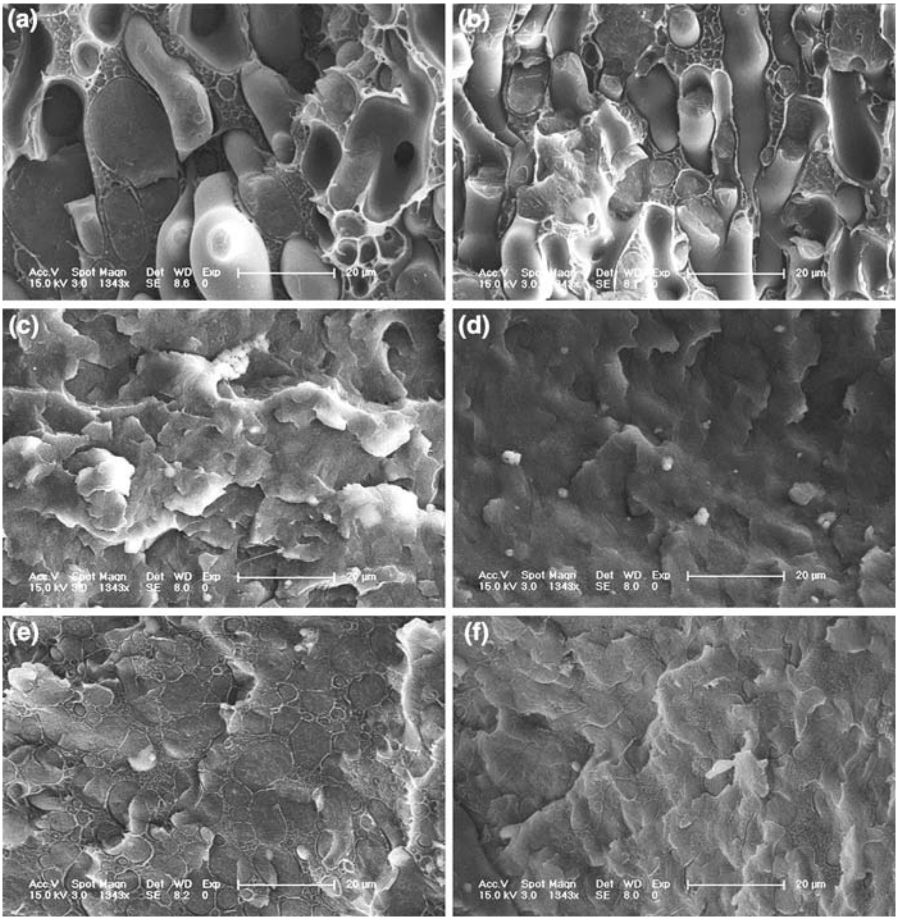


Fig. 2 SEM micrographs of cryogenically fractured surfaces for LLDPE/PS with different amount of combined Lewis acids: **a** 0; **b** 0.75 mmol $InCl_3 \cdot 4H_2O$; **c** 1.5 mmol $InCl_3 \cdot 4H_2O$; **d** 2.25 mmol $InCl_3 \cdot 4H_2O$; **e** 3 mmol $InCl_3 \cdot 4H_2O$; **f** 3.75 mmol $InCl_3 \cdot 4H_2O$. The mole ratio of $InCl_3 \cdot 4H_2O$ to Me_3SiCl is fixed at 1:5

adhesion [15]. In Table 2, the physical blend of LLDPE/PS (50/50 wt%) shows poor tensile property as expected, owing to the weak interaction between LLDPE and PS. The addition of 1.5 mmol $InCl_3 \cdot 4H_2O$ or 7.5 mmol Me_3SiCl alone to LLDPE/PS blend does not enhance the tensile strength and Izod impact strength, indicating that Me_3SiCl or $InCl_3 \cdot 4H_2O$ alone has no catalytic effect on this blend system. A substantial increase of tensile and impact strengths can be observed when the combined Lewis acids of $InCl_3 \cdot 4H_2O$ and Me_3SiCl are used, showing that it may be the combined system of $InCl_3 \cdot 4H_2O$ and Me_3SiCl that catalyzes the F–C alkylation [10]. The tensile and impact properties vary with the composition of combined Lewis acids (ratio of $InCl_3 \cdot 4H_2O$ to Me_3SiCl). The elongation at break is also important criteria for estimating the compatibilization [6], but within the scope of Lewis acids that have been used (Table 2), the elongation at break of blends has

Table 2 Mechanical properties of reactive LLDPE/PS blends with combined Lewis acids as catalysts

LLDPE/PS (wt%)	Catalysts loading (mmol)		Tensile strength (MPa)	Elongation at break (%)	Izod impact strength (kJ/m^2)
	$InCl_3 \cdot 4H_2O$	Me_3SiCl			
50/50	0	0	7.9 ± 1.1	5.6 ± 1.3	4.0 ± 1.3
50/50	0	7.5	6.8 ± 0.9	6.0 ± 0.2	3.9 ± 0.3
50/50	1.5	0	7.4 ± 0.8	5.1 ± 0.8	4.2 ± 0.8
50/50	1.5	1.5	11.9 ± 0.5	5.6 ± 0.3	5.8 ± 0.7
50/50	1.5	4.5	13.8 ± 0.6	5.9 ± 1.0	6.3 ± 0.6
50/50	1.5	7.5	15.9 ± 0.7	6.4 ± 0.9	8.6 ± 1.1
50/50	1.5	10.5	12.6 ± 0. 5	5.1 ± 1.0	5.3 ± 0.6

no obvious change at different Lewis acids ratio. This is caused by co-continuous phase structure of the 50/50 weight ratio of LLDPE/PS. According to Leibler [16], when the blends forming co-continuous structure, the physical connection within rigid phase increases the ability to deformation and flow. Thus, during the tensile process, the elongation at break is low even if the blends are reactive compatibilized. Some authors have observed the same results in PPE/Surlyn [17] and PA/PP systems [18]. When rubbery compatibilizer is added in PE/PS system, this property increases a little [19]. In this study, the maximum of mechanical properties is obtained when the mole ratio of $InCl_3 \cdot 4H_2O$ to Me_3SiCl is set at 1:5. Thus, the molar ratio of $InCl_3 \cdot 4H_2O$ to Me_3SiCl is fixed at 1:5 in the subsequent studies.

The effect of content of combined Lewis acids on the mechanical properties of blends is studied, and traditional Lewis acid catalyst $AlCl_3$ is also investigated for comparison. These results are listed in Table 3. Table 3 shows that enhancement of tensile and impact properties is nearly linear with the adding amount of $InCl_3 \cdot 4H_2O$ and Me_3SiCl, and the maximum of mechanical properties are obtained at 0.6 wt% (about 2.25 mmol $InCl_3 \cdot 4H_2O$). With the increase of combined Lewis acids, the elongation at break increases to some extent. The mechanical properties vary slightly even at high loading of combined Lewis acids (1.0 wt%). As a contrast, an obvious enhancement of tensile properties of blends is obtained when the addition of $AlCl_3$ is 0.2 wt% (about 0.6 mmol $AlCl_3$). However, further increase loading of $AlCl_3$ causes dramatic decrease of tensile strength and Izod impact strength, which has been proved to be resulted from degradation of PS [4, 6]. Thus, the advantage of combined Lewis acids are obvious: (a) the mechanical properties are substantially improved; (b) the degradation of PS are efficiently decreased.

GPC analysis

The GPC results of PS extracted from LLDPE/PS blends are listed in Table 4 in terms of number-averaged molecular weight (M_n), weight-averaged molecular weight (M_w) and polymer dispersion index (M_w/M_n). Table 4 shows that for the reactive LLDPE/PS blends catalyzed by combined Lewis acids. The degradation of PS does not occurred even at the high catalyst loading (1.0 wt%), while for the

Table 3 Mechanical properties of reactive LLDPE/PS (50/50 wt%) blends with different Lewis acids

Catalyst	Loading (wt%)	Tensile strength (MPa)	Elongation at break (%)	Izod impact strength (kJ/m^2)
$InCl_3 \cdot 4H_2O/Me_3SiCl$ (1:5 mol%)	0.2	11.9 ± 0.5	3.6 ± 0.3	5.8 ± 0.7
	0.4	15.9 ± 0.7	6.4 ± 0.9	8.6 ± 1.1
	0.6	17.5 ± 1.0	11.2 ± 3.4	7.7 ± 0.8
	1.0	18.6 ± 0.3	9.3 ± 2.4	5.4 ± 0.4
$AlCl_3$	0.1	14.1 ± 0.9	4.7 ± 0.5	4.17 ± 0.3
	0.2	17.1 ± 0.6	6.0 ± 0.5	6.64 ± 1.28
	0.3	12.2 ± 0.4	3.2 ± 0.7	2.61 ± 0.05

Table 4 GPC results of PS extracted from LLDPE/PS blends

Catalyst	Loading (wt%)	M_n ($\times 10^4$)	M_w ($\times 10^4$)	M_w/M_n
None[a]	None	11.8	29.1	2.46
$InCl_3 \cdot 4H_2O/Me_3SiCl$ (1:5 mol%)	0.2	12.9	29.2	2.27
	0.4	12.2	28.6	2.34
	0.6	14.3	32.9	2.29
	1.0	13.3	30.8	2.31
$AlCl_3$	0.1	13.3	29.6	2.22
	0.2	2.6	12	4.63
	0.3	2.1	12.5	5.99

[a] The PS homopolymer was extracted from physical blend

reactive LLDPE/PS blends catalyzed by $AlCl_3$, the substantial degradation of PS is observed even with 0.2 wt% loading. Interestingly, the molecular weights of PS extracted from blends catalyzed by the combined Lewis acid are slightly higher than that extracted from physical blends, indicating that the PS chains with low molecular weight are readily grafted with LLDPE [3]. The GPC results provide the direct evidence that the combined Lewis acids do not cause the degradation of PS.

Catalytic process

Based on experimental results and previous works [1, 2, 20], the mechanism of Friedel–Crafts benzene ring alkylation catalyzed by combined Lewis acids is tentatively proposed. As shown in Fig. 3, the reaction mechanism can be divided into four parts: combination, initiation, chain scission and grafting. The first step is appropriate combination between Me_3SiCl and $InCl_3 \cdot 4H_2O$ (Eq. 2). The Lewis acidity of the silicon center is enhanced by coordination from chloride to indium trichloride. The fact that combined Lewis acids is the true catalyst is supported by the results of Table 2, and Saito et al. have demonstrated that the combination between indium and silicone species is essential for the alkylation, and low

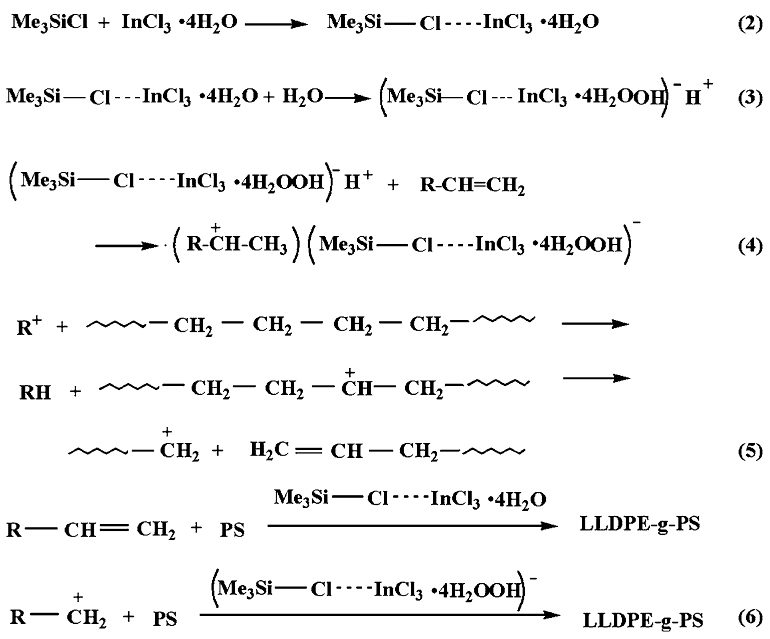


Fig. 3 The possible mechanism of Friedel–Crafts benzene ring alkylation catalyzed by combined Lewis acids

oxophilicity and high halophilicity of indium are the reasons of the reactivity [8–10]. The second step is initiation (Eqs. 3, 4). Combined Lewis acids react with impurities (for example, water) to form a complex, which then reacts further with unsaturated compounds to form the initial carbocation [1, 2, 20]. The third step is chain scission of LLDPE (Eq. 5). The initial carbocation attacks LLDPE, generating a macrocarbocation, then macrocarbocation undergoes a chain scission through electron rearrangement near the site of the carbocation [1]. The last step is grafting (Eq. 6). In the presence of combined Lewis acids, fragments of the LLDPE chain substitute for a proton from the benzene ring of PS, forming a LLDPE–g–PS copolymer [2, 20].

The degradation mechanism of PS on the addition of Lewis acids have been proposed by Pulanskzy et al. [21]. With $AlCl_3$ as catalyst, they suggested that PS chain cleavage occurred in the cationic process. The degradation mechanism is shown in Fig. 4. Impurity (for example, water) in conjunction with $AlCl_3$ formed a complex (Eq. 7), which affected hydrogen-abstraction (Eq. 8), and the transitory benzylcation induced β cleavage (Eq. 9).

Comparison of Eq. 4 (in Fig. 3) and Eq. 8 (in Fig. 4) reveals that the addition of the acid to unsaturated carbon bond and hydrogen abstraction by the acid is essential for the F–C alkylation and degradation of PS. As the addition of acid to carbon–carbon double bond is easier than hydrogen abstraction by the same acid [22, 23], three possible consequences will occur depending on the acidity of Lewis acid [10]. If the acidity of Lewis acid is too weak to add to unsaturated carbon–carbon double

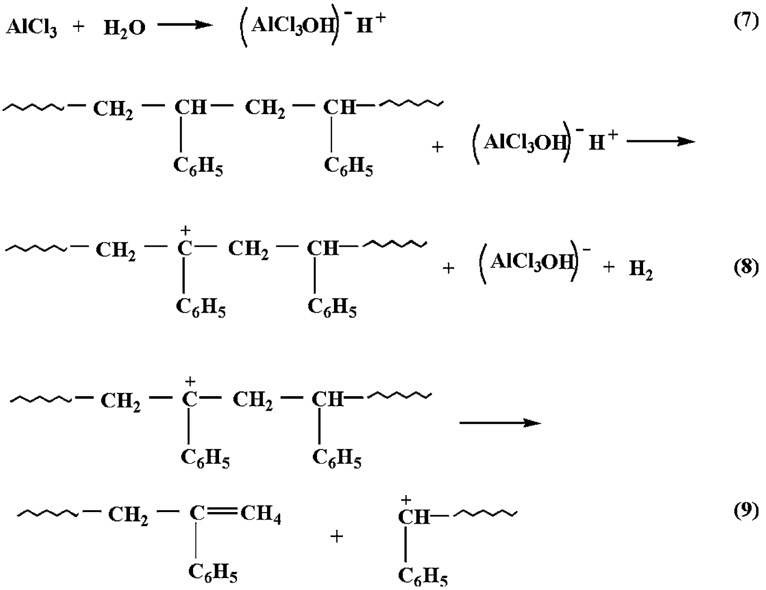


Fig. 4 The degradation mechanism of PS catalyzed by Lewis acids ($AlCl_3$)

bond, it can not initiate F–C alkylation. Since the acidity is very low [9], $InCl_3 \cdot 4H_2O$ and Me_3SiCl alone have no catalytic effect on LLDPE/PS blends. On the contrary, if the acidity of Lewis acid is strong enough, it will initiate both reactions. For this reason, the extensive degradation of PS occurred when $AlCl_3$ was added as catalyst [10, 21]. if the acidity of Lewis acid is appropriate, it will initiate F–C alkylation, while decrease degradation of PS. Based on the experimental results and above analysis, we can make a conclusion that it is because the appropriate acidity of combined Lewis acids that they have catalytic effect on F–C alkylation and do not cause substantial degradation of PS.

Conclusion

A new type of combined Lewis acids, $InCl_3 \cdot 4H_2O$ and Me_3SiCl in the molar ratio of 1:5, was investigated for in situ compatibilization LLDPE/PS blends. The experiments revealed that the combined Lewis acids had catalytic effect on Friedel–Crafts alkylation reaction between LLDPE and PS, and the graft copolymer LLDPE–g–PS was formed via the F–C reaction and worked as a tailor-made compatibilizer to reduce the interfacial tension; the mechanical properties of reactive blend with combined Lewis acids as catalyst was notably improved compared to that of physical LLDPE/PS blend and serious degradation had been decreased compared to the reactive blend system with $AlCl_3$ as catalyst; the appropriate acidity of combined Lewis acids was believed to be the main reason for this type of Lewis acids possessing above advantages.

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