

# Effect of Low Molar Mass Liquid Crystal and Lubricant on Miscibility and Thermal Properties of Syndiotactic Polystyrene Blends

Pichet Pahupongsab,<sup>1</sup> Supakanok Thongyai,<sup>1</sup> Sirirat Wacharawichanant,<sup>2</sup> Piyasan Praserttham<sup>1</sup>

<sup>1</sup>Department of Chemical Engineering, Faculty of Engineering, Center of Excellence on Catalysis and Catalytic Reaction Engineering, Chulalongkorn University, Bangkok 10330, Thailand

<sup>2</sup>Department of Chemical Engineering, Faculty of Engineering and Industrial Technology, Silpakorn University, Nakorn Pathom 73000, Thailand

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**ABSTRACT:** This research concerned with the miscibility and thermal properties of syndiotactic polystyrene (SPS) blended with several polymers such as poly( $\alpha$ -methyl styrene)—PaMS, poly(ethyl methacrylate)—PEMA, poly(*n*-butyl methacrylate)—PBMA, poly(cyclohexyl acrylate)—PCHA, and poly(*cis*-isoprene)—PIP. The SPS synthesized by using metallocene catalyst and modified-methylaluminoxane (MMAO) as cocatalyst. From the experimental results, it was found that the SPS can be miscible with PaMS, PEMA, PBMA, PCHA, and PIP. Furthermore, the effects of the addition of low molar mass liquid crystal (cyclohexyl-biphenyl-cyclohexane, CBC33) and lubricant (glycerol monostearate, GMS) on thermal properties of the blended polymers with SPS are con-

cerned. Both CBC33 and GMS can lower the crystallization temperature ( $T_c$ ) of the blend and can also slightly lower the melting temperature ( $T_m$ ) of the blends. This might be because of the higher mobility of SPS molecules induced the separation from the crystal both in the case of the depression of  $T_c$  or  $T_m$ . The higher mobility of the blends happened together with the further reduction of the melt viscosity of the blends according to CBC33 that can be proven by Motong et al. in 2008 (Motong et al., *J Appl Polym Sci* 2008, 107, 1108). © 2009 Wiley Periodicals, Inc. *J Appl Polym Sci* 114: 2053–2059, 2009

**Key words:** miscibility; thermal properties; syndiotactic polystyrene; liquid crystal; lubricant

## INTRODUCTION

Generally, polystyrene (PS) is one of the most important commodity polymers in the industry. Its applications range from high modulus, transparent grade to rubber modified, tough resins and blends with outstanding impact resistance and mechanical properties. Recently, coordination polymerization techniques were introduced for preparation of new polystyrene, which has an entirely new range of possibilities and the feasibility for preparation of a highly stereoregular, syndiotactic polystyrene (SPS) was demonstrated.<sup>1</sup> SPS prepared by coordination polymerization is a new semicrystalline thermoplastic material with high crystalline melting temperature (270°C) and excellent chemical resistance. However, because SPS has some economic disadvantages such as low strength at low temperature,<sup>2</sup>

higher processing temperature,<sup>3</sup> and low efficiency of polymerization catalyst, it has been restricted to a few applications. So, many researchers are still interested in blending SPS with secondary polymer materials and in the miscibility of its polymer blends.

The previous researches have studied the miscibility of polystyrene with several polymers, viz. polyphenyleneether (PPE), polyvinylmethylether (PVME), poly-2-chlorostyrene (PCS), polymethylstyrene (PMS), polycarbonate of tetramethyl bisphenol-A (TMPC), polycyclohexyl acrylate (PCHA), polyethylmethacrylate (PEMA), polycyclohexyl methacrylate (PCHMA) and etc.<sup>4</sup> Widmaier and Mignard<sup>5</sup> investigated the blends of PS of molecular weights from 4000 to 80,000 g/mol to poly( $\alpha$ -methylstyrene) of molecular weights from 55,000 to 300,000 g/mol by freeze-drying from benzene solutions. Glass transition temperature ( $T_g$ ) measurements by differential scanning calorimetry (DSC) indicate that the miscibility behavior of the polymers is very sensitive to change of molecular weights. A decrease in PS chain length changes a two-phase system into a miscible or partially miscible blend. Cimmino et al.<sup>6,7</sup> investigated the dependence of miscibility on composition and temperature in SPS/PVME blends by means of

Correspondence to: S. Wacharawichanant (iamsirirat@yahoo.com).

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solid-state NMR and DSC. The DSC experiments showed two  $T_g$  values corresponding to an SPS-rich phase (83 : 17 wt %) and a PVME-rich phase (13 : 87 wt %). For SPS/PPE blends, DSC and DMTA measurements give an intermediate single  $T_g$  value<sup>8-11</sup> between those of the pure components and obviously depended on the compositions. The  $T_g$  values of SPS and PPE are much different from each other (98 and 220°C, respectively), and this result constitutes an unambiguous proof of the blend miscibility within the whole composition range.

However, the excellent properties of polymer can become a cause of limitation in manufacturing processes especially the viscosity of melted polymers. The polymer blending procedure requires many complicated operations involving high temperature and high shear rate; therefore, their processing and manufacturing of polymer blends usually consume high energy. The processing properties of polymer blends can be modified by adding various additives, such as antioxidants, plasticizers, and others. Many additives can reduce melt viscosity of polymers to improve their processability, but most additives may cause many negative effects to important properties of polymers, especially the mechanical properties of the final products. Lubricant,<sup>12</sup> a small quantity polymers' additive, can provide a considerable decrease in resistance to the movement of chains or segments of amorphous polymer or at least partly amorphous structure. Low molar mass liquid crystals (LMMLCs) were also found to improve processability of polymers.<sup>13,14</sup> Patwardhan and Belfiore reported that addition of LMMLC to amorphous polymers could improve both processability and mechanical properties of the blends.<sup>15</sup> The addition of low molar mass liquid crystal (CBC33) can dramatically reduce the melt viscosity of polycarbonate as observed by the rheometer.<sup>13</sup>

This work investigates the miscibility and thermal properties of SPS, which synthesized by homogeneous half-metallocene catalyst system, blended with various polymers, and of these SPS blended properties after addition of low molar mass liquid crystal (CBC33) and lubricant (GMS).

## EXPERIMENTAL

### Materials

Styrene monomer purchased from Fluka Chemie A.G. was distilled from sodium under vacuum just before use. Trichloro(pentamethyl cyclopentadienyl)

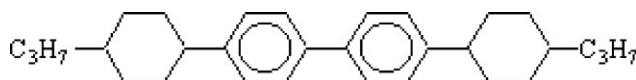


Figure 1 Structure of CBC33.

TABLE I  
Properties of Low Molar Mass Thermotropic  
Liquid crystals

Property	CBC33
Melting point (°C)	158
Smectic-nematic temperature (°C)	223
Clearing temperature (°C)	327
Molecular weight (g/mol)	403

titanium (IV) ( $Cp^*TiCl_3$ , 97.0%) was purchased from Aldrich. Modified methyaluminoxane (MMAO) 1.831 M in toluene was donated from Tosoh Akso, Japan. The low molar mass liquid crystal (LMMLC), CBC33, was purchased from Merck Co., Ltd. in the form of a white powder. The CBC33 structures that contain a cyclohexyl-biphenyl-cyclohexane backbone can be shown as Figure 1. Its molecular weight characteristics, transition temperatures, and other physical properties can be shown in Table I. The lubricant, glycerol monostearate (GMS), was kindly provided by Rikevita Ltd (Malasia) with the melting point of 65°C and the molecular weight of 358 g/mol. The chemical structure of GMS can be shown in Figure 2.

Poly(*n*-butyl methacrylate)—PBMA, poly( $\alpha$ -methyl styrene)—PaMS, poly(cyclohexyl acrylate)—PCHA, poly(cyclohexyl methacrylate)—PCHMA, poly(*cis*-isoprene)—PIP, poly(ethyl methacrylate)—PEMA were purchased from Scientific Polymer Products, Inc and used as received.

### Polymerization procedure

$Cp^*TiCl_3$  (~ 0.014 g) was stirred in 35 mL of toluene under argon atmosphere until dissolved. Styrene monomer (chemical reagent grade) was washed with 5% aqueous sodium hydroxide (NaOH) solution and distilled water, then distilled under reduced pressure.

Polymerization of styrene was carried out in a 250 cm<sup>3</sup> glass reactor equipped with a magnetic stirrer by introducing 46 mL of toluene, 32 mL of  $Cp^*TiCl_3$  dissolved in toluene, 13.6 mL of MMAO and 28.4 mL of styrene at the desired temperature of 25°C under argon atmosphere. The total volume of the polymerization mixture was 120 mL. The addition of styrene was regarded as the starting point of the polymerization reaction. After complete the

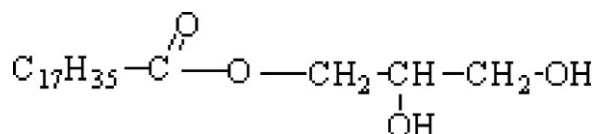


Figure 2 Structure of glycerol monostearate (GMS).

**TABLE II**  
**Polymerization of Styrene Using Cp\*TiCl<sub>3</sub> with MMAO<sup>a</sup>**

% Yield <sup>b</sup>	72.67 wt %
Catalytic activity	5084.67 g PS/mmol Ti-hr
% Syndiotactic index	93.38 %
$M_w^c$	1,943,500 g/mol
$M_n^c$	592,300 g/mol
Molecular weight distribution (MWD) <sup>c</sup>	3.3
$T_g^d$	97.90°C
$T_m^d$	271.41°C

<sup>a</sup> Conditions: [Cp\*TiCl<sub>3</sub>] =  $3.68 \times 10^{-4}$  M, [MMAO] = 1.83 M, [Styrene] = 2.06, Al/Ti = 563, 25°C.

<sup>b</sup> Calculated from (weight of synthesized polymer/weight of monomer)  $\times$  100.

<sup>c</sup> Obtained from GPC and MWD was calculated from  $M_w/M_n$ .

<sup>d</sup> Obtained from DSC.

desired reaction time, the reactions were terminated by the addition methanol followed by 10% HCl in methanol. The precipitated polymer was washed several times with methanol and dried at room temperature. The polymer obtained was extracted with refluxing methyl ethyl ketone (MEK) for 12 h to isolate the SPS portion of the polymer obtained from other isomers.

Soxhlet extractor was used for syndiotactic content determination. The obtain polystyrene was extracted with boiling methyl ethyl ketone (MEK) or 2-butanone to give syndiotactic (insoluble) and atactic/isotactic (soluble) polystyrene. A % syndiotactic index (% S.I.) is computed from

$$\% \text{ S.I.} = \frac{\text{Insoluble Weight of PS}}{\text{Total Weight of PS}} \times 100 \quad (1)$$

The melting temperature ( $T_m$ ) and  $T_g$  values of the polymers were determined by a Perkin-Elmer DSC-Diamond. The analyses were performed at the heating rate of 20°C/min in the temperature range 50–300°C. The molecular weight ( $M_w$ ) and molecular weight distribution (MWD) were investigated by gel permeation chromatography (GPC). Samples were prepared accurately at a concentration of approximately 0.5–1.0 mg/mL in the mobile phase and dissolved by using the PL-SP 260 at a temperature of 150°C for, approximately, an hour. The dissolved sampled were transferred into PL-GPC 220. The GPC measurement was performed at Thai Petrochemical Industry Public Co., Ltd.

#### Polymer blend preparation and characterization

The blends of SPS/polymers and their blends with, CBC33, the liquid crystal or, GMS, the lubricant were prepared by mechanical mixing using a digital hot plate at various compositions. The SPS/polymers

were mixed together with CBC33 or GMS at 310°C, and all the blended samples were kept at 300°C for 5 minutes before immediately quenched to 200°C and held for 20 minutes, before further cooled down to room temperature. The melting temperature ( $T_m$ ), the crystallization temperature ( $T_c$ ) and the glass transition temperature ( $T_g$ ) of the blends were determined by a Perkin-Elmer DSC-Diamond. The analyses were performed at the heating rate of 20°C/min in the temperature range 50–300°C.

## RESULTS AND DISCUSSION

### Polymerization of styrene

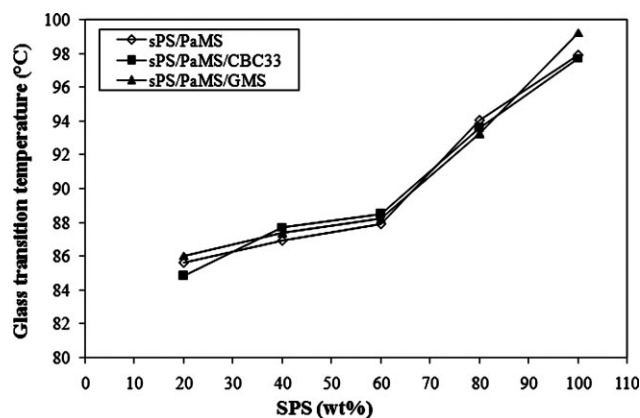
The results of the polymerization of styrene, using Pentamethylcyclopentadienyl titanium trichloride (Cp\*TiCl<sub>3</sub>) with modified-methylaluminoxane (MMAO) as cocatalyst, can be summarized as in Table II.

### Effect of LMLLC and lubricant on thermal properties of polymer blends

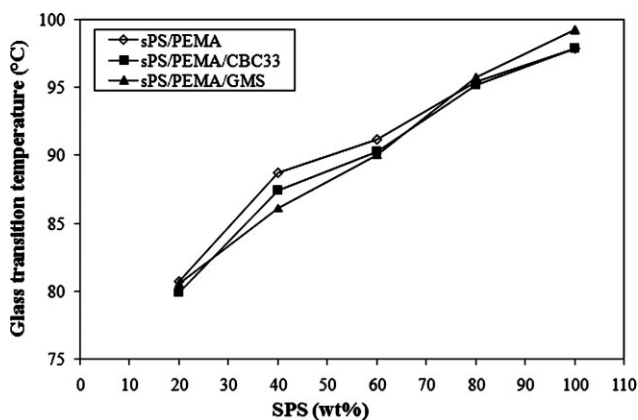
#### Glass transition temperature

The glass transition temperature is the characteristic of the amorphous part of the polymers. At  $T_g$ , a dramatic change occurs in the local movement of molecule level of polymer chain from glassy state to rubbery state, which this changes almost all of the physical and mechanical properties of polymer.<sup>16</sup>

The miscibility of binary blends is frequently ascertained by measurements of their  $T_g$ . Figure 3 shows  $T_g$  of each composition of SPS/PaMS blend. It is observed that the  $T_g$  of pure SPS and PaMS is 97.90 and 87.33°C, respectively. All the blends with different compositions exhibit single  $T_g$  which shifted to a higher temperatures in the same trend as the SPS content in the samples. This result may imply the miscibility of the two components in the blends under the DSC condition. The thermal



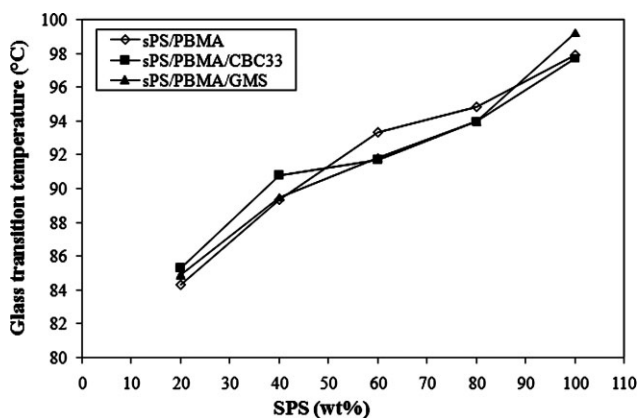
**Figure 3** Glass transition temperature of SPS/PaMS blends before and after adding CBC33 and GMS.



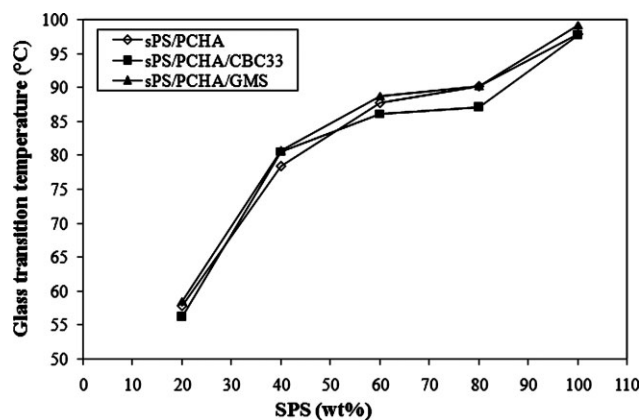
**Figure 4** Glass transition temperature SPS/PEMA blends before and after adding CBC33 and GMS.

characteristics of SPS/PaMS/CBC33 blends and SPS/PaMS/GMS blends are shown that  $T_g$  of binary blends are in the same vicinity of their blends with CBC33 and GMS. The difference between  $T_g$  of binary blends and their blends with additives are less than  $1^\circ\text{C}$ , and cannot be distinguished from each other. These phenomena may be resulted from the too small (1.0% w/w) amount of additives in the matrix phase of the binary blends that are not enough to plasticize the blends to such an extent that the significant reducing in  $T_g$  of the blends can be observed.

Figures 4–7 show  $T_g$  of each composition of SPS/PEMA, SPS/PBMA, SPS/PCHA, SPS/PIP blend. It is observed that all the blends with different compositions exhibit single  $T_g$ , which shifts to a higher temperature with the SPS content. This result may imply that the miscibility of the two components in the amorphous state of the blends. The values of  $T_g$  of binary blends are in the same vicinity to their blends with CBC33 and GMS. Thus, the additions of CBC33 and GMS have not significantly affected  $T_g$  of binary blends as same as SPS/PaMS blends.



**Figure 5** Glass transition temperature SPS/PBMA blends before and after adding CBC33 and GMS.

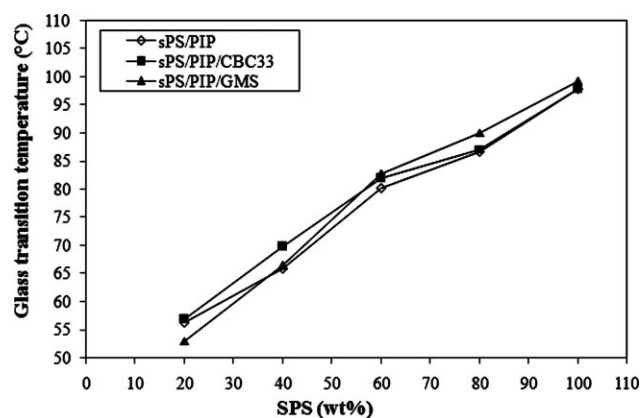


**Figure 6** Glass transition temperature SPS/PCHA blends before and after adding CBC33 and GMS.

From these results, it was found that the SPS have tendency to be miscible with PaMS, PEMA, PBMA, PCHA, and PIP by melt mixing method. The glass transition temperatures of all the blends with additives do not significantly change from additive less binary blends. This phenomenon proves that additives do not have significant direct plasticizing effects on glass transition temperature of pure binary blends when added in the small quantity.

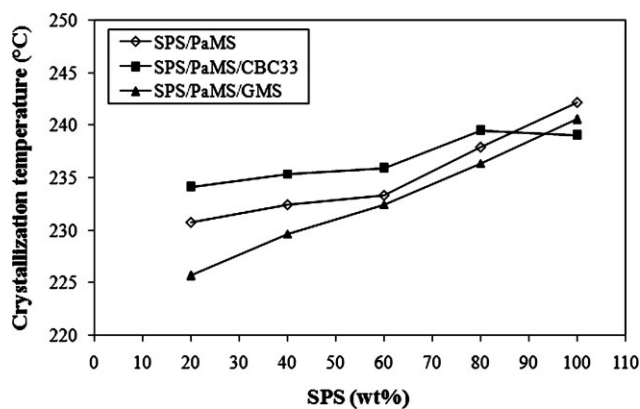
### Crystallization temperature

The crystallization temperature is the temperature that the sPS in the blends started to crystallize due to lower temperature from the cooling ramp rate in DSC. From Figure 8, when the crystallization temperature at cooling rate of  $20^\circ\text{C}/\text{min}$  ( $T_c$ ) of SPS/PaMS blends and their blends with additives were compared, they can be showed that addition of CBC33 have affected in the slightly increasing  $T_c$  of their blends in the amount of less than  $3^\circ\text{C}$  apart from the pure blend. These differences were located

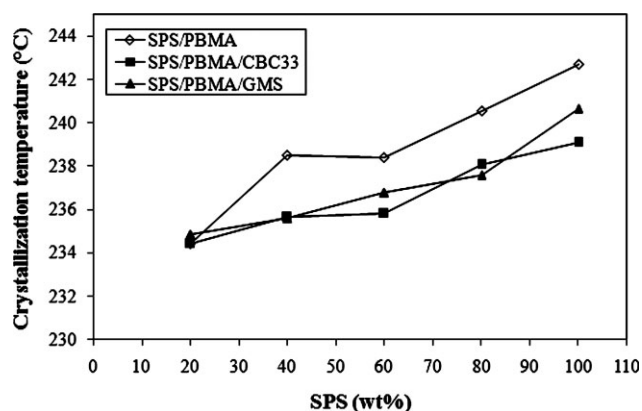


**Figure 7** Glass transition temperature SPS/PIP blends before and after adding CBC33 and GMS.





**Figure 8** Crystallization temperature of SPS/PaMS blends before and after adding CBC33 and GMS.



**Figure 10** Crystallization temperature of SPS/PBMA blends before and after adding CBC33 and GMS.

in error limit of the DSC that is less than  $\pm 5^\circ\text{C}$  apart from each other. But the addition of GMS has the adverse affected by slightly decreasing the  $T_c$  of their blends in the vicinity of less than  $3^\circ\text{C}$  apart from the pure blend. These differences still were in the error limit of the DSC, and the increases/decreases were not significant while the concentration of the blend changed. So, in the system of SPS/PaMS, the additives (CBC33, GMS) of the small amount in the SPS/PaMS blend were not changed significantly the crystallization temperatures of the blend. This might be because of too similar molecular structures between the SPS and PaMS.

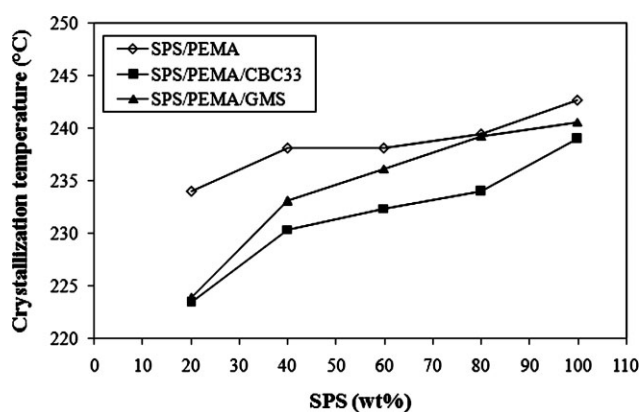
From Figure 9, it can be seen that the addition of CBC33 in SPS/PEMA blends affected the decreasing of  $T_c$  in their blends about  $4\text{--}11^\circ\text{C}$  from pure blend without CBC33. The addition of GMS also affected the decreasing  $T_c$  of their blends about  $2\text{--}10^\circ\text{C}$ . These temperature differences were significant and we can draw the conclusion that the additives tend to significantly decrease the  $T_c$  of SPS/PEMA blends, regarded the small amount of the additive.

For SPS/PBMA blends, the additions of CBC33 or GMS have affected in decreasing of  $T_c$  in their

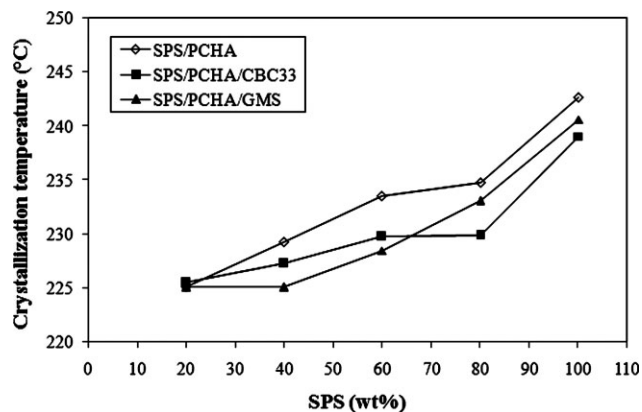
blends about  $3^\circ\text{C}$  apart from the pure blend  $T_c$  as shown in Figure 10. Although the decreases of  $T_c$  were in the error limit of the DSC, but the unanimous decreases in  $T_c$  implied the significant variations. Thus, the additions of CBC33 or GMS have significant trended in decreasing the  $T_c$  of SPS/PBMA blends, regarded the small amount of the additive.

Figure 11 shows the values of  $T_c$  of SPS/PCHA blends before and after addition of CBC33 or GMS. It can be concluded that the additions of CBC33 or GMS have affected in decreasing of  $T_c$  of the blends about  $2\text{--}5^\circ\text{C}$  from the pure blend. Thus, both additives have significant effects in decreasing the  $T_c$  of SPS/PCHA blends, regarded the small amount of the additive.

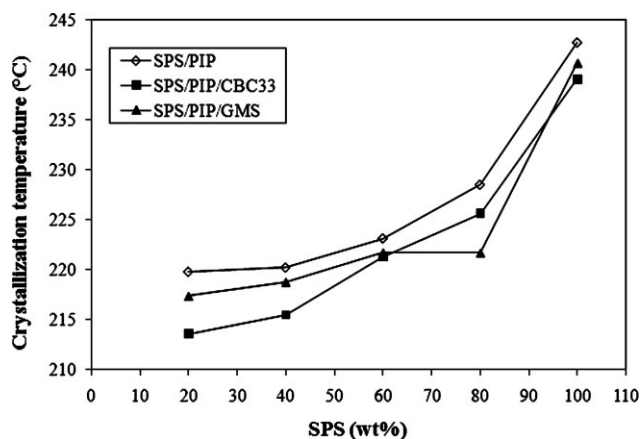
Similarly, for SPS/PIP blends, the addition of CBC33 or GMS have affected significantly in decreasing of  $T_c$  of the blends about  $2\text{--}7^\circ\text{C}$  from the pure blend as shown in Figure 12. Thus, both additives also have significant effects in decreasing the  $T_c$  of SPS/PIP blends from the pure blend, regarded the small amount of the additive.



**Figure 9** Crystallization temperature of SPS/PEMA blends before and after adding CBC33 and GMS.



**Figure 11** Crystallization temperature of SPS/PCHA blends before and after adding CBC33 and GMS.



**Figure 12** Crystallization temperature of SPS/PIP blends before and after adding CBC33 and GMS.

To summarize from these results, the effects of adding CBC33 or GMS resulted in the significantly slightly decreasing  $T_c$  of the polymer blends concerned except the blend with PaMS. These might be because CBC33 and GMS can reduce melt viscosity of the blends,<sup>13,17</sup> so the molecules of the polymers in the blend could move or separate easily. Therefore, the mobile SPS molecules have more difficulty to form the crystal from the induced of cooling temperature, because more mobile SPS molecules may tend to move apart from the order (crystal). The crystalline temperature will decrease according to the mobility of the molecules when added the CBC33 or the GMS due to the less order of the easily mobile chain molecules.

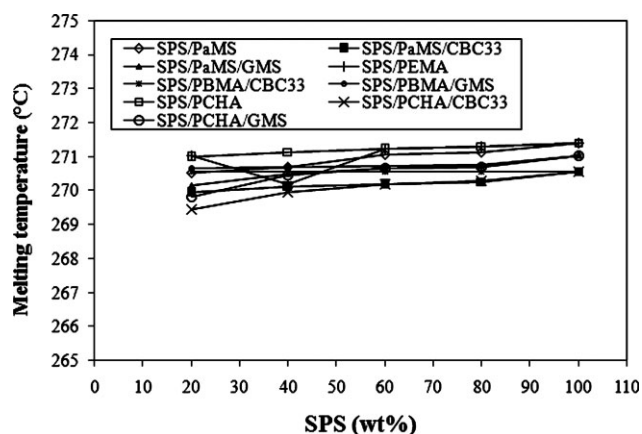
### Melting temperature

For polymer blends, the depression phenomenon of the crystalline melting point temperature ( $T_m$ ) usually happened from the lower of the blends' Gibbs free energy. From Figure 13, the crystalline melting temperature  $T_m$  of the pure binary blends and their blends with additives have unanimous lowering tendency than the  $T_m$  of the pure component of SPS, although the decreases were in the error limit of the DSC. The decreases were more pronounced with the additions of CBC33 or GMS which the  $T_m$  of the addition blends were unanimously and slightly lower than the  $T_m$  of the pure blends. In every systems concerned in this research, the crystalline melting points when added the CBC33 or GMS were lowered than the pure polymer blend. From melting point depression phenomenon, the crystalline melting point will be lower if the additives were added to the pure blends. However, because of the small amount of the CBC33 or GMS added, the quantities of the crystalline melting point depression will not only come from the melting point depression

phenomenon alone but also contribute from the addition of the CBC33. The decreasing in the crystalline melting point due to the addition of small molecule can be calculated from the equation below,<sup>18</sup>

$$\frac{1}{T_m} - \frac{1}{T_m^0} = -\frac{R}{\Delta H_f} \ln X_A \quad (2)$$

where,  $\Delta H_f$  represents the molar enthalpy of the fusion from the SPS crystal. To a first approximation, the melting point depression depends on the mole fraction of impurity ( $X_B$ ) and the mole fraction of crystallizable polymer (being  $X_A$ ). For polymer blends, the crystalline melting temperature depression phenomenon that results from the lower of the blend Gibbs free energy needed to have the large amount of  $X_B$  to overcome the effect from the molar enthalpy of the fusion. Thus, by this equation, the crystalline melting temperature of the binary blends and their blends with very small amount of additives will have very slightly tendency to lower the temperature from the pure component of the polymers. However, in this research, the effects in lowering the crystalline melting point ( $T_m$ ) might also come from the contribution from the addition of the CBC33 more than the melting point depression by normal additive. These might be because of the molecular mobility enhancement from both CBC33 and GMS,<sup>13,17</sup> the crystal of the blends will melt easier. The more mobile SPS molecules can slide and depart from each other easier than the blend's SPS molecules without additive; therefore, the crystal will melt easier than the polymer blend without the additive. Moreover, as we have concluded before, the ease of the moving of the SPS molecules may enhance the depression in the crystalline temperature ( $T_c$ ). By the same assumption, the systems will also have the tendency to be apart from the shape of the crystal (melting of the crystal) faster than the



**Figure 13** Melting temperature depression of their blends.

normal pure SPS molecules in the normal blend, so the  $T_m$  will be lower than usual.

### CONCLUSIONS

In this research, the miscibility and thermal properties of SPS blended with several polymers such as PaMS, PEMA, PBMA, PCHA, and PIP were investigated. The SPS was synthesized by using metallocene catalyst. From DSC, it was found that the SPS have tendency to miscible with PaMS, PEMA, PBMA, PCHA and PIP by melt mixing method. The glass transition temperatures of all the blends with additives do not significantly change from non additive binary blends. This phenomenon proves that additives do not have direct plasticizing effects on glass transition temperature of pure binary blends, regarded the small amount of additives.

Both CBC33 and GMS significantly decrease the crystallization temperature ( $T_c$ ) of polymer blends. Because CBC33 and GMS reduce melt viscosity of the blends,<sup>13,17</sup> the polymers' molecules could move or separate easily. The SPS in the blends might subject to more difficulty to ally their molecules in the shape of the crystal from the fast mobility melt of the polymer blends when added CBC33 or GMS. Therefore, when mobile melt molecule tends to form the crystal, it will hardly form the crystal in case of GMS and CBC33 addition in the blend. Thus, the ease of the moving of the molecule will enhance the depression in the crystalline temperature.

The CBC33 and GMS slightly decrease crystalline melting temperature ( $T_m$ ) of their blends. By the same assumption, because CBC33 and GMS reduced melt viscosity of the blends, crystal of the blends will melt easier. In other words, the  $T_m$  tends to decrease when added with GMS or CBC33 because of the effects from the mobility of the molecules. It

can be concluded that the effects of lowering the crystalline melting point largely come from the contribution from the addition of the small amount of CBC33 more than the normal melting point depression by normal additive.

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