# Polymer Blends of Polypropylene with Polydialkylsiloxane

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**ABSTRACT:** Polypropylene was blended with polydialkylsiloxane (alkyl : propyl and octyl). Some properties of the resultant polymer blends were investigated: the melt flow index at 230°C, bending modulus, and Izod impact strength. Furthermore, their morphology was observed using scanning and transmission electron microscopes. It was elucidated that polydipropylsiloxane and polydioctylsiloxane, which are finely dispersed as ciliated islands in the sea of polypropylene, are effective to increase the melt flow index and Izod impact strength of the polymer blends. © 1998 John Wiley & Sons, Inc. J Appl Polym Sci **67:** 1853–1861, 1998

**Key words:** polymer blend; polypropylene; polydimethylsiloxane; polydioctylsiloxane; mechanical properties; morphology

## **INTRODUCTION**

Many investigators have studied polymer blends<sup>1-3</sup>; for example, rubber-toughened plastics are well known. However, polydimethylsiloxane is rarely applicable for this purpose because of its poor compatibility with polyolefin, so polydimethylsiloxane, containing some amount of unsaturated groups, is blended with polyolefin by using a peroxide and/or mechanical shearing to induce free-radical formation.<sup>4-6</sup>

In this work, we prepared polydipropylsiloxane (PPSO) and polydioctylsiloxane (POSO) from dichlorosilane and propylene or octene, and they are blendable with polypropylene (PP). Since PPSO and POSO are elastic, owing to their flexible main chain, it was expected that they would be effective in making PP tougher by blending. Therefore, PPSO or POSO was blended with PP and some properties of the PP blends were investigated, especially regarding their moldability and toughness: melt flow index (MFI) at 230°C, bending modulus (BM), and Izod impact strength (IIS). Furthermore, the morphology was observed by using scanning and transmission electron microscopes.

## **EXPERIMENTAL**

#### Materials

The PPs used were a homopolymer (homo-PP, ME-140) and the block copolymer containing a poly(ethylene-*co*-propylene) component (*block*-PP, PN-630, 640, 650, and 670) from Tokuyama Corp. Their properties are shown in Table I.

PPSO and POSO were prepared according to the following scheme: (1) hydrosilylation of olefin to prepare dialkyldichlorosilane; (2) hydrolysis of dialkyldichlorosilane to prepare cyclotridialkylsilane; (3) polymerization of cyclotridialkylsiloxane. All reagents were of chemical grade and used without further purification.

## Hydrosilylation of Propylene

n-Hexane (150 mL) was put into a flask (300 mL) equipped with a stirrer, a thermometer, a reflux

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Table I Properties of homo-PP and *block*-PP

PP	MFI (g/10 min)	$\frac{BM}{(kg/cm^2)}$	IIS (kg cm/cm <sup>2</sup> )
homo-PP			
ME-140	10	18,000	2
$block ext{-}\operatorname{PP}^{\operatorname{a}}$			
PN-620	0.7	13,500	12
PN-630	2.5	14,500	11
PN-640	6.5	15,500	6
PN-650	10	15,500	5
PN-670	23	15,500	3

<sup>a</sup> Contents of the ethylene unit were 4.1-4.2%.

condenser using methanol-dry ice, connected to a N<sub>2</sub> gas bubbler, and a H<sub>2</sub>SiCl<sub>2</sub> gas inlet. Next, H<sub>2</sub>SiCl<sub>2</sub> gas (0.50 mol) was introduced over 1 h, and then a small amount of a propylene and isopropanol solution of H<sub>2</sub>PtCl<sub>6</sub> (catalyst,  $1 \times 10^{-5}$ mol) was successively added at 0°C. Subsequently, propylene (0.50 mol) was introduced at the rate of 0.2 mol/h, keeping the reaction temperature below 10°C. All H<sub>2</sub>SiCl<sub>2</sub> changed to the monopropyl substitute at this stage. Then, the reaction temperature was elevated to 25°C and propylene (0.52 mol) was similarily introduced to prepare (C<sub>3</sub>H<sub>7</sub>)<sub>2</sub>SiCl<sub>2</sub>, which was purified by distillation under reduced pressure (bp, 89°C/50 mmHg; yield, 87 g, 0.47 mol, about 100% by GC).

Colorless oil,  $MS_{\it m}/z$  184 and 186  $(M^+)$  and 141 and 143  $(M^+$  –43).

## Hydrosilylation of Octene

Hexane (200 g) and the isopropanol solution of  $H_2PtCl_6$  (1 × 10<sup>-4</sup> mol) were charged into a flask (3 L) equipped with a stirrer, a thermometer, a reflux condenser connected to a methanol-dry ice trap, and inlets of H<sub>2</sub>SiCl<sub>2</sub> and 1-octene, which were, respectively, subsequently introduced at the rates of 0.4 mol/h over 10 min at 20°C. After keeping the reaction mixture at 20°C for 10 min, it was confirmed that the monooctyl substitute of H<sub>2</sub>SiCl<sub>2</sub> was produced. Then, H<sub>2</sub>SiCl<sub>2</sub> and 1-octene were again introduced at the rates of 0.88 and 0.96 mol/h, respectively, keeping the reaction temperature below  $30^{\circ}$ C. The total amounts of H<sub>2</sub>SiCl<sub>2</sub> and 1-octene were 5.0 and 10.2 mol, respectively. The resultant  $(C_8H_{17})_2$ SiCl<sub>2</sub> was purified by distillation under reduced pressure (bp 140-145°C/0.3 mmHg; yield 1500 g, 4.6 mol, 95% by GC).

Colorless oil,  $MS_{\it mz}$  324 and 326 (M<sup>+</sup>) and 211 and 213 (M<sup>+</sup> –113).

## Hydrolysis of Dipropyldichlorosilane

A methyl isobutyl ketone (1 L) and 3N HCl agueous solution (500 mL) was charged into a flask (2 L) equipped with a stirrer, a thermometer, a dropping funnel, and a reflux condenser. Then,  $(C_3H_7)_3SiCl_2$  (1 mol) was added dropwise with stirring over 10 min and the reaction mixture was vigorously stirred for 1 h at room temperature. Next, the methyl isobutyl ketone layer of the reaction mixture was separated by using a separating funnel (3 L). The methyl isobutyl ketone solution was rinsed four times with water until no acid was detected and then dehydrated by using anhydrous  $MgSO_4$ . After removing  $MgSO_4$ , methyl isobutyl ketone was evaporated with the aid of an evaporator, and the residue was heated in methanol (200 mL) with stirring. Subsequently, the resultant methanol solution was removed to exclude the oligomer with hydroxy groups at the terminals. The residue consisted of cyclotri- and cyclotetradipropylsiloxane (trimer, 75% estimated by GC) and was distilled under reduced pressure to recover the trimer (bp 107-110°C/0.3 mmHg). Thus, cyclotridipropylsiloxane was prepared.

Colorless oil, yield: 78 g, 0.6 mol;  $MS_{mz}$ . 347 (M<sup>+</sup>) for the trimer; <sup>29</sup>Si-NMR, -11.1 ppm for the trimer and -22.2 ppm for the tetramer.

## Hydrolysis of Dioctyldichlorosilane

A methyl isobutyl ketone (1.5 L) and 3N HCl aqueous solution (1.5 L) was put into a flask (5 L)equipped with a stirrer, a thermometer, a reflux condenser, and a dropping funnel. (C<sub>8</sub>H<sub>17</sub>)<sub>2</sub>SiCl<sub>2</sub> (4.6 mol) was added dropwise at 60°C over 5 min with vigorous stirring and the reaction was carried out for 30 min. After cooling, the methyl isobutyl ketone layer was recovered with the aid of a separating funnel, then rinsed eight times with water until no acid could be detected and dehydrated by using anhydrous MgSO<sub>4</sub>. After filtration of  $MgSO_4$ , methyl isobutyl ketone was removed using an evaporator. The residual solid was dissolved in an acetone (1.5 L)-methanol (0.5 L) mixture at 50°C and then the solution was cooled to 15-18°C. Cyclic tri- and tetradioctylsiloxsane were precipitated and filtered. This procedure was repeated four times. Then, the recovered

solid was dried at  $100^{\circ}$ C under a vacuum. The product (810 g) consisted of cyclotri- and cyclote-tradioctylsiloxane (the trimer was 80% estimated by GPC).

Solid: IR (Si—O, neat) 1005 cm<sup>-1</sup> for the trimer and 1080 cm<sup>-1</sup> for the tetramer; <sup>29</sup>Si-NMR: -10.7 ppm for the trimer and -21.9 ppm for the tetramer; <sup>1</sup>H-NMR (Si—CH<sub>2</sub><sup>a</sup>—(CH<sub>2</sub><sup>b</sup>)<sub>6</sub>—CH<sub>3</sub><sup>c</sup>): a, 0.53; b, 1.30; c, 0.91 (ppm). GPC:  $M_n$ , 800 for the trimer and 1000 for the tetramer.

#### Polymerization of Cyclotridipropylsiloxane

An example is shown as follows: Cyclotridipropylsiloxane (3.0 mol) and  $(CH_3)_3SiOSi(CH_3)_3$  (adjuster of polymerization degree, 12 mmol) were put into a flask (1 L) equipped with a stirrer, a thermometer, and a N<sub>2</sub> gas bubbler and, subsequently,  $CF_3SO_3H$  (catalyst, 30 mmol) was added. The polymerization was carried out at room temperature for 16 h. The product was dissolved in hexane (3 L) and this solution was rinsed four times with water, using a separating funnel, until no acid could be detected.

The hexane solution was dehydrated by adding  $MgSO_4$ , which was then removed by filtration. Hexane was removed using an evaporator. The residual product was treated twice in hot acetone and successively twice in methanol with stirring in order to purify the polymer. Polydipropylsiloxane was thus prepared after drying at room temperature under a vacuum.

Solid: 310 g, yield 80%. GPC: polymerization degree, 750.

#### Polymerization of Cyclotridioctylsiloxane

An example is shown as follows: The mixture of cyclotri- and cyclotetradioctylsiloxanes (680 g; cyclotridioctylsiloxane, 667 mmol) and  $(CH_3)_3Si-OSi(CH_3)_3$  (3.3 mmol) were put into a separable flask (2 L) equipped with a stirrer, a thermometer, and a N<sub>2</sub> gas bubbler and heated to 40°C. Then, CF<sub>3</sub>SO<sub>3</sub>H (6.6 mmol) was added and the polymerization was carried out at 40°C for 16 h. The product was dissolved in hexane (3 L). The hexane solution was rinsed six times with water using a separating funnel (5 L) until no acid could be detected and then dehydrated using anhydrous MgSO<sub>4</sub>. After separating MgSO<sub>4</sub>, hexane was removed using an evaporator. The residue was

treated with a hot mixture of methyl isobutyl ketone (800 mL)-acetone (1.5 L) with stirring in order to exclude the cyclic compounds. This procedure was repeated three times. After extracting the residual solvents with methanol, the resultant polydioctylsiloxane was dried at room temperature under a vacuum.

 $\begin{array}{l} \label{eq:solid} Solid, 430 \mbox{ g GPC: polymerization degree, 480. $^1$H-NMRi} \ [Si-CH_2{}^a-(CH_2{}^b)_6-CH_3{}^c]: \mbox{ a, 0.53; b, 1.30; c, 0.91} \ (ppm); $$^{13}C-NMR$ (Si-C{}^a-C{}^b-C{}^c-C{}^d-C{}^e-C{}^e-C{}^d-C{}^e-C{}^e-C{}^d-C{}^e-C{$ 

Table II shows some examples of the resultant polymers, of which the polymerization degree is controllable by adjusting the [cyclotridialkylsilox-ane/( $CH_3$ )\_3SiOSi( $CH_3$ )\_3] ratio.

#### Measurement

PP and PPSO or POSO were blended at 190–200°C for 5 min using a pair of mixing rolls. Then, test pieces were molded by hot-pressing at 230°C for 5 min. MFI, IIS, and BM were measured according to JIS-K6758, 7110, and 7203, respectively, and used to provide the following ratios:

$$R(MFI) = \frac{MFI \text{ of } PP \text{ blend}}{MFI \text{ of } PP}$$
$$R(IIS) = \frac{IIS \text{ of } PP \text{ blend}}{IIS \text{ of } PP}$$
$$R(BM) = \frac{BM \text{ of } PP \text{ blend}}{BM \text{ of } PP}$$

NMR spectra were measured in  $CDCl_3$  using  $(CH_3)_4Si$  as an internal standard employing a GSX-270 from JEOL Ltd. (<sup>1</sup>H, 270 MHz; <sup>13</sup>C, 67.8 MHz; and <sup>29</sup>Si, 53.5 MHz). GC-MS spectra were measured using an M-2000 from Hitachi Ltd. The IR spectrometer used was a JIR-100 from JEOL Ltd. The polymerization degree was estimated by GPC (standard, polystyrene; solvent, HCCl<sub>3</sub>).

### Scanning Electron Microscopy

The test pieces were broken by bending at the liquid nitrogen temperature and the resultant fractures were treated with chloroform to exclude the poly(ethylene-*co*-propylene) component, PPSO or POSO, in advance, and then each sample was

		TMSO <sup>b</sup> /CTDS Molar Ratio	Polymerization				
Alkyl	CF <sub>3</sub> SO <sub>3</sub> H/CTDS <sup>a</sup> Molar Ratio		Temp (°C)	Time (h)	Conversion	Polymerization Degree (n)	
$C_3$	1/50	1/10	20	3	90	30	
	1/50	1/50	20	3	90	150	
	1/50	1/200	20	3	90	600	
	1/100	1/250	20	16	90	750	
$C_8$	1/100	1/20	40	16	80-90	55	
	1/100	1/50	40	16	80-90	150	
	1/100	1/100	40	16	80-90	290	
	1/100	1/200	40	16	80-90	480	
	1/100	1/300	40	16	8-90	630	

<sup>a</sup> Cyclotridialkylsiloxane.

<sup>b</sup> (CH<sub>3</sub>)<sub>3</sub>SiOSi(CH<sub>3</sub>)<sub>3</sub>.

pretreated by gold-ion sputtering for 3 min at 1.2 kV and 8-10 mA. The fracture was observed using a scanning electron microscope, JSM-220, from JEOL Ltd. The accelerated voltage and the probe current were 15 kV and 5 mA, respectively.

## **Transmission Electron Microscopy**

The test piece was embedded in an epoxy resin and then treated with  $RuO_4$  for 48 h after trimming. The ultrathin sample was prepared using an ultramicrotome: a Reichert-Nissei ULTRACUT-N from Leica AG. The transmission electron microscope was a JEM 200CX from JEOL Ltd., attached to an energy dispersion X-ray analyzer, TN-502, from Tracol Northern Co.

# **RESULTS AND DISCUSSION**

First, we examined the compatibility of polydimethylsiloxane and polydialkylsiloxane (PASO; alkyl:propyl and octyl) with PP by kneading both polymers on a hot plate (180°C) for 5 min using a spatula when the weight ratio of polydimethylsiloxane or PASO/PP was 1/10. Polydimethylsiloxane (polymerization degree, 230) is hardly compatible and the resultant polymer became white. On the other hand, PASO is quite compatible and the resultant polymers show quite good transparency. This comes from the affinity of their relatively larger alkyl groups to PP.

Also, PASO has the characteristics that its main chain is flexible and the relatively larger alkyl group has an affinity for PP. Accordingly, it

was expected that the blending of PASO with PP could make the resultant PP blend tougher and easier for molding. In fact, preliminary studies elucidated that the blending of PPSO is effective to increase the MFI of the PP blend and the blending of POSO makes the IIS of the *block*-PP blend greater, as shown in Table III. PPSO is more effective in making the R(MFI) larger in the case of the *block*-PP blend than in the case of the homo-PP blend, and the smaller the polymerization degree, the more effective the PPSO. Also, PPSO makes the R(IIS) of the *block*-PP blend and the R(BM) of the homo- and *block*-PP blends decrease slightly, and POSO makes the R(MFI) and R(IIS) of the *block*-PP blend greater and the R(BM) of the *block*-PP blend smaller. Figure 1 shows the relationship between the PPSO/PP ratio and R(MFI). R(MFI) increases with increasing PPSO/PP ratio significantly in the case of the block-PP blend and less in the case of the homo-PP blend. It is not clear as to why PPSO is more effective in the case of the *block*-PP blend.

Figure 2 shows the relationships between the PASO/PP ratio and the R(IIS) and R(BM) of the *block*-PP blends. In both cases of PPSO and POSO, R(BM) similarly decrease with increasing PASO/PP ratio. On the other hand, with an increasing PPSO/PP ratio, R(IIS) decreases a little in the case of PPSO, but sharply increases in the case of POSO to the POSO/PP ratio =  $6 \times 10^{-2}$  and then decreases. Evidently, POSO is effective as an additive to make *block*-PP tougher.

Next, Figure 3 shows the dependencies of the R(IIS) and R(BM) of *block*-PP blends on the polymerization degree of POSO. The polymerization

		R(MFI)		R(IIS)		R(BM)	
PASO	Polymerization Degree	homo-PP	block-PP	homo-PP	block-PP	homo-PP	block-PP
PPSO	30	1.2	1.8	1.0	0.9	0.9	0.9
	750	1.1	1.4	1.0	0.9	1.0	1.0
POSO	480	1.1	1.3	1.0	4.0	1.0	0.9

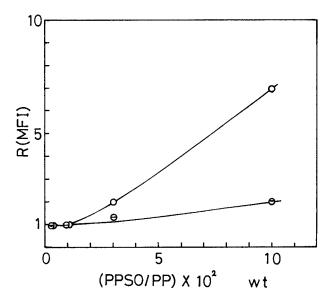
Table III Blending Effect of PASO to Some Properties of PP Blends

PASO/PP = 0.03 by weight.

degree has no effect on R(BM) but has an effect on R(IIS). With increasing the polymerization degree of POSO, R(IIS) increases at first and then levels off in the range of the polymerization degree of 150–630.

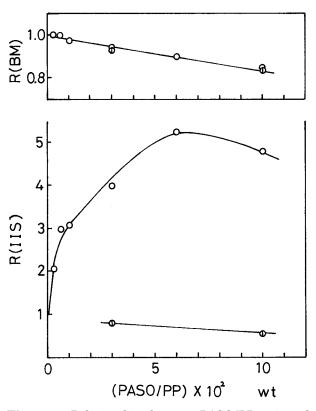
Figure 4 shows the relationships between the MFI of *block*-PP and the R(IIS) and R(BM) of *block*-PP blends. The smaller the MFI of the *block*-PP, the larger is the R(IIS) of the *block*-PP blends. On the other hand, R(BM) is unaffected by the increase of MFI of the *block*-PP. It is interesting that the use of *block*-PP, with a relatively smaller MFI and larger polymerization degree, is preferable. Since *block*-PP with a larger polymerization degree is relatively tougher, the meaning of the results shown in Figure 4 is important from the viewpoint of practical application.

As described above, it is elucidated that PPSO is effective in increasing the R(MFI) of a PP blend

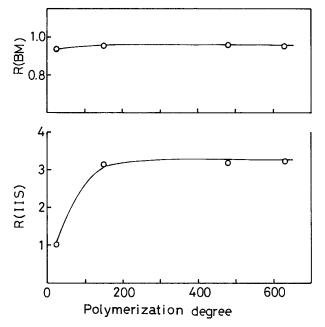


**Figure 1** Relationships between PPSO/PP ratio and R(MFI): ( $\ominus$ ) homo-PP (ME-140); ( $\bigcirc$ ) block-PP (PN-620); PPSO, n = 30.

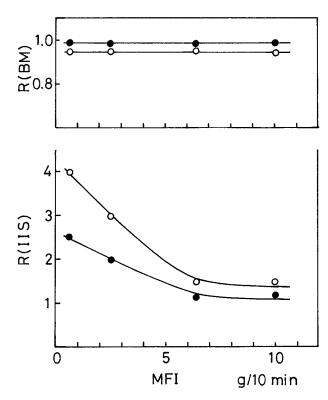
in particular, and POSO is effective in increasing the R(IIS) with no significant decrease of R(BM), when the POSO/PP ratio is relatively smaller and the MFI of the *block*-PP used is relatively smaller. Probably the former effect is ascribed to the more flexible main chain of PPSO owing to its less bulky propyl group than octyl group. On the other hand, the effect of the latter is inexplicable only by the elasticity of POSO, of which the relatively longer octyl group could be incorporated into the texture of the *block*-PP. The details should be studied further.



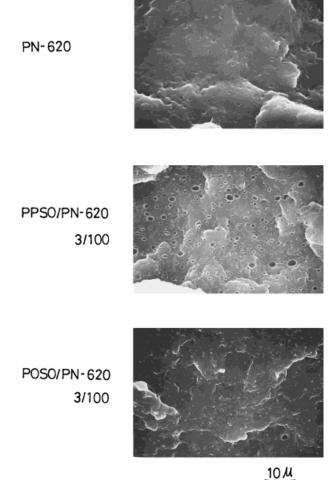
**Figure 2** Relationships between PASO/PP ratio and R(IIS) and R(BM) of *block*-PP (PN-620) blends: ( $\bigcirc$ ) PPSO, n = 750; ( $\bigcirc$ ) POSO, n = 480.



**Figure 3** Dependencies of R(IIS) and R(BM) of POSO-*block*-PP (PN-630) blends on polymerization degree of POSO. POSO/*block*-PP = 0.03 by weight.



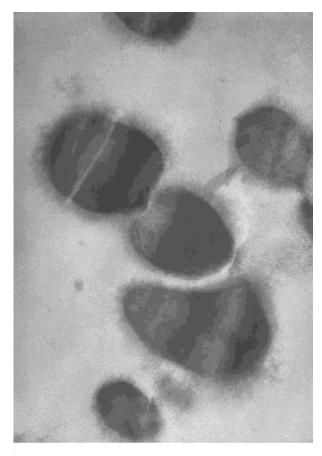
**Figure 4** Relationships between MFI of *block*-PP and R(IIS) and R(BM) of POSO-*block*-PP (PN-620) blends. POSO/PN-620: ( $\bullet$ ) 0.01 and ( $\bigcirc$ ) 0.03 by wt; POSO, n = 480.



**Figure 5** Scanning electron micrographs of fracture of *block*-PP, PPSO-*block*-PP blend, and POSO-*block*-PP blend. PASO/*block*-PP (PN-620) = 0.03 by weight.; PPSO, n = 750; POSO, n = 480.

# Scanning Electron Microscopy

As is well known, the dispersion state of a rubber component in rubber-toughened plastics is critically important in relation to their mechanical properties. Accordingly, we investigated the dispersion state of PASO in *block*-PP blends using scanning electron microscopy. Figure 5 shows scanning electron micrographs of the fractures of *block*-PP, a PPSO-*block*-PP blend, and a POSO*block*-PP blend. There are many minute cavities in the case of *block*-PP, which are formed by the exclusion of the poly(ethylene-*co*-propylene) component soluble in chloroform. However, the numbers of the cavities in the cases of PPSO- and POSO-*block*-PP blends are larger than in the case of the *block*-PP. Furthermore, the cavities in



0.5µm

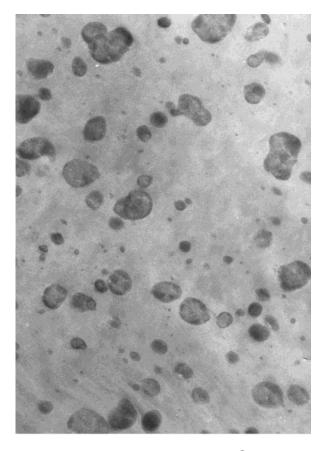
Figure 6 Transmission electron micrograph of POSO-homo-PP (ME-140) blend. PP/homo-PP (ME-140) = 10/90 by weight.

the case of POSO are smaller than those in the case of PPSO. This probably means that POSO is more easily compatible with *block*-PP than PPSO, due to its larger alkyl group. However, there is no way to recognize which cavity is caused by exclusion of the poly(ethylene-*co*-propylene) component or the PASO components.

## **Transmission Electron Microscopy**

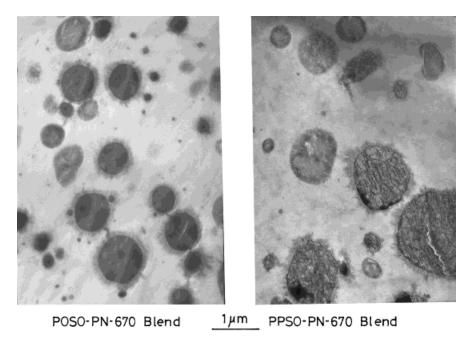
Figure 6 shows a transmission electron micrograph of the POSO-homo-PP blend. POSO is finely dispersed in the homo-PP phase. It is notable that the POSO particles have cilia on their boundaries, probably showing good affinity of POSO for homo-PP. Figure 7 shows a transmission electron micrograph of *block*-PP. The poly-(ethylene-*co*-propylene) component is dispersed as small particles with no cilia on their boundaries. Figure 8 shows transmission electron micrographs of PPSO- and POSO-*block*-PP blends. Both PPSO and POSO are dispersed as fine particles with cilia on their boundaries, and the poly-(ethylene-*co*-propylene) component is observed as fine particles with no cilia as shown in Figure 7. Furthermore, the POSO particles are smaller than the PPSO particles and this is consistent with the results shown in Figure 5.

Next, in order to clarify the interrelation between POSO and the poly(ethylene-*co*-propylene) component, some details were investigated. Figure 9 shows a transmission electron micrograph of the POSO-homo-PP blend. Here, an energydispersion X-ray microanalyzer was used to detect elemental Si. No Si was detected at point 1 in the homo-PP phase, but Si can be detected at point 2 in the ciliated particles of the POSO component. Figure 10 shows transmission electron micro-

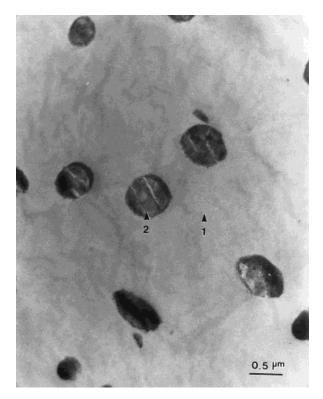


2µm

**Figure 7** Transmission electron micrograph of *block*-PP (PN-640).



**Figure 8** Transmission electron micrographs of PPSO- and POSO-*block*-PP (PN-670) blends. PPSO/*block*-PP = 5/95 and POSO/*block*-PP = 10/90 by weight. PPSO, n = 750; POSO, n = 480.



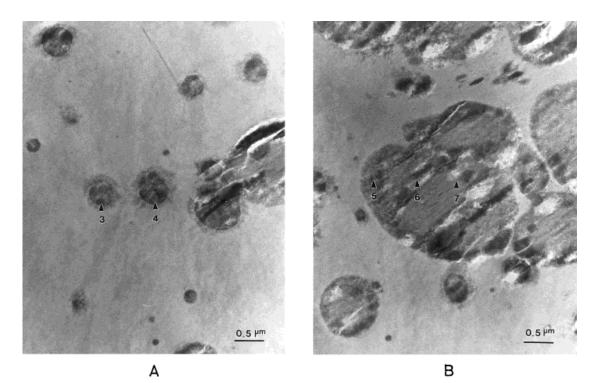
**Figure 9** Transmission electron micrograph of POSO-homo-PP (ME-140) blend. POSO/homo-PP = 10/90; POSO: n = 480.

graphs of the POSO-*block*-PP blend. Si is detectable at points 3 and 4 in the ciliated particle of POSO, as shown in Figure 8. On the other hand, no Si was detected at points 5-7 in the particle with no cilia, which is of the poly(ethylene-*co*-propylene) component. Therefore, it is concluded that POSO is not blended into the poly(ethylene-*co*-propylene) component. However, it is obscure now as to why the cilia are formed. Presumably, the formation of the ciliated particles could be a key point regarding the excellent effectiveness of POSO to give the POSO-*block* PP blend toughness, but further study is needed.

# CONCLUSION

homo-PP or *block*-PP was blended with PPSO or POSO. The resultant polymer blends were studied regarding their MFI, BM, IIS, and morphology:

- 1. PPSO is effective in increasing the MFI of both the homo- and *block*-PP blends, and this effect is much more in the case of the *block*-PP blend.
- 2. POSO is effective in increasing the MFI and IIS of the block PP blend, and the effect regarding IIS is outstanding.



**Figure 10** Transmission electron micrographs of POSO-*block*-PP (PN-670) blend. POSO/*block*-PP = 5/95; POSO: n = 480.

- 3. Blending of PPSO or POSO somewhat decreases the BM of homo- and *block*-PP blends.
- 4. PPSO or POSO is dispersed as fine particles with cilia in the case of homo- or *block*-PP.

PPSO or POSO is finely dispersed as minute ciliated particles in the PP phase, and it is probably the reason why PPSO and POSO are effective modifiers of PP. However, it is obscure as to how and why the cilia are formed on the boundary of the particles and this needs further investigation in the future.

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