

# Study on Morphology of Ternary Polymer Blends. II. Effect of Composition

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**ABSTRACT:** The composition effect on morphology of polypropylene/ethylene-propylene–diene terpolymer/polyethylene (PP/EPDM/PE) and polypropylene/ethylene-propylene–diene terpolymer/polystyrene (PP/EPDM/PS) ternary blends has been investigated. In all of the blends, polypropylene as the major phase was blended with two minor phases, that is, EPDM and PE or PS. From morphological studies using the SEM technique a core–shell morphology for PP/EPDM/PE and separated dispersed morphology for PP/EPDM/PS were observed. These results were found to be in agreement with the theoretical predictions. The composition of components affected only the size of dispersed phases and had no appreciable effect on the type of morphology. The size of each dispersed phase, whether it forms core or shell or disperses separately in matrix, can be related directly to its composition in the blend. © 2001 John Wiley & Sons, Inc. *J Appl Polym Sci* 82: 1138–1146, 2001

**Key words:** ternary polymer blends; composition effect; morphology; relative interfacial energy; melt viscosity ratio

## INTRODUCTION

In recent years increasing interest has been devoted to polymeric alloys because blending is an important route for preparation of materials with synergistic and tailored properties.<sup>1–4</sup> So far, developments of polymer blends have been mainly focused on two-component systems. However, such systems typically have a matrix-dispersed morphology with relatively large dispersed phases and weak interfacial adhesion, which in many cases results in poor mechanical properties. A common approach to alleviate this problem involves the addition (or the *in situ* formation) of an

interfacially active agent, or so-called compatibilizer, to the blend.<sup>5</sup>

Multicomponent polymer blends, which consist of at least three immiscible polymers, are a new emerging area in the field of polymeric materials. A large range of phase morphology then becomes available and directly influences the whole set of properties.

For systems containing two minor phases dispersed in a continuous matrix, three distinct types of phase morphology have to be considered. The first situation corresponds to the independent dispersion of the two minor components. The second extreme situation is where one minor component forms a shell around small domains of the second one. The third situation is the intermediate case, where mixed phases of the two minor components are formed without any ordered organization.<sup>5–7</sup>

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**Table I Properties and Producers of Polymers Used**

Polymer	Density (g/cm <sup>3</sup> )	$T_m$ (°C)	MFI (g/10 min)	Mooney Viscosity ML(1 + 4), 125°C	C <sub>2</sub> Content (%)	ENB Content (%)	Producer
HDPE	0.964	130	0.35 <sup>a</sup>	—	—	—	5200 B, Iran Petrochemical
PP	0.9	165	0.27 <sup>b</sup>	—	—	—	MOPLEN 60R, Iran Petrochemical
EPDM1	0.86	—	—	30	50	5	Buna AP241, Bayer Co.
EPDM2	0.87	—	—	61	70	5	Buna AP447, Bayer Co.
PS	1.05	185	1.6 <sup>c</sup>	—	—	—	1070, Iran Petrochemical

<sup>a</sup> 190°C/2.160 kg.<sup>b</sup> 230°C/2.160 kg.<sup>c</sup> 200°C/5 kg.

The type of morphology and the size of dispersed phases in binary or ternary systems are important factors that determine mechanical properties and rheological behavior of polymeric blends.<sup>6,8-10</sup> The type of morphology and the size of dispersed phases can be affected by composition, melt viscosity of the components, interfacial interaction, and processing parameters.<sup>5,6</sup>

The main objective of the present work was to study the effect of composition on morphology of ternary PP/EPDM/PE and PP/EPDM/PS blends.

**Table II Nomenclature, Components, and Composition of Binary Blends**

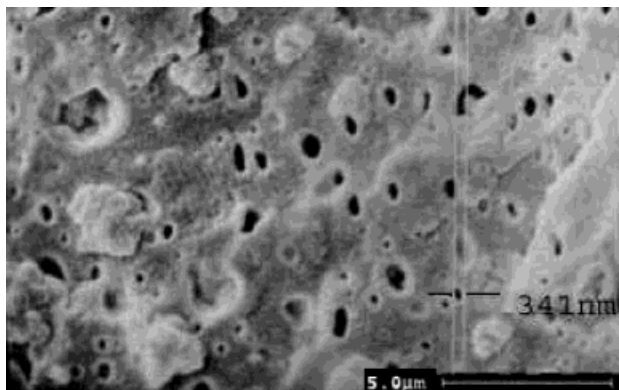
Sample Code	Composition (wt %)	
	Major Phase	Minor Phase
B <sub>1</sub>	PP (95)	EPDM1 (5)
B <sub>2</sub>	PP (90)	EDPM1 (10)
B <sub>3</sub>	PP (85)	EPDM1 (15)
B <sub>4</sub>	PP (80)	EDPM1 (20)
B <sub>5</sub>	PP (95)	HDPE (5)
B <sub>6</sub>	PP (90)	HDPE (10)
B <sub>7</sub>	PP (85)	HDPE (15)
B <sub>8</sub>	PP (80)	HDPE (20)
B <sub>9</sub>	PP (95)	EDPM2 (5)
B <sub>10</sub>	PP (90)	EDPM2 (10)
B <sub>11</sub>	PP (85)	EDPM2 (15)
B <sub>12</sub>	PP (80)	EDPM2 (20)
B <sub>13</sub>	PP (95)	PS (5)
B <sub>14</sub>	PP (90)	PS (10)
B <sub>15</sub>	PP (85)	PS (15)

In another investigation carried out by the same authors the morphology of these ternary systems with regard to interfacial interaction and melt viscosity of components has been reported.<sup>11</sup> The

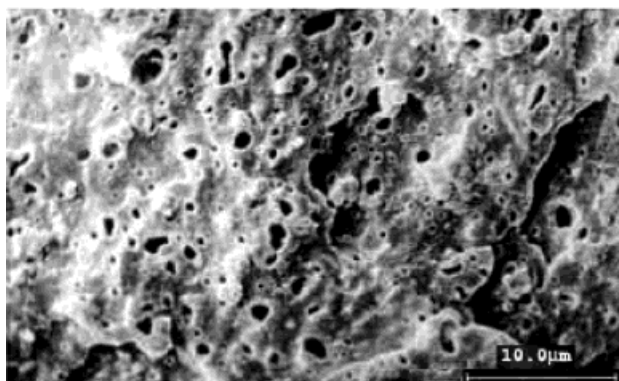
**Table III Nomenclature, Components, and Composition of Ternary Blends**

Sample Code	Comparison (wt %)		
	Major Phase	Minor Phase (1)	Minor Phase (2)
T <sub>1</sub> <sup>a</sup>	PP (85.5)	EPDM1 (9.5)	HDPE (5)
T <sub>2</sub>	PP (81)	EPDM1 (9)	HDPE (10)
T <sub>3</sub>	PP (72)	EPDM1 (8)	HDPE (20)
T <sub>4</sub>	PP (63)	EPDM1 (7)	HDPE (30)
T <sub>5</sub> <sup>b</sup>	PP (76)	EPDM1 (19)	HDPE (5)
T <sub>6</sub>	PP (72)	EPDM1 (18)	HDPE (10)
T <sub>7</sub>	PP (64)	EPDM1 (16)	HDPE (20)
T <sub>8</sub>	PP (56)	EPDM1 (14)	HDPE (30)
T <sub>9</sub> <sup>c</sup>	PP (85.5)	EPDM2 (9.5)	PS (5)
T <sub>10</sub>	PP (81)	EPDM2 (9)	PS (10)
T <sub>11</sub>	PP (72)	EPDM2 (8)	PS (20)
T <sub>12</sub>	PP (63)	EPDM2 (7)	PS (30)
T <sub>13</sub> <sup>d</sup>	PP (76)	EPDM2 (19)	PS (5)
T <sub>14</sub>	PP (72)	EPDM2 (18)	PS (10)
T <sub>15</sub>	PP (64)	EPDM2 (16)	PS (20)
T <sub>16</sub>	PP (56)	EPDM2 (14)	PS (30)

<sup>a</sup> In blends of T<sub>1</sub> to T<sub>4</sub> EPDM1/(PP + EPDM1) is 10 wt %.<sup>b</sup> In blends of T<sub>5</sub> to T<sub>8</sub> EPDM1/(PP + EPDM1) is fixed at 20 wt %.<sup>c</sup> In blends of T<sub>9</sub> to T<sub>12</sub> EPDM2/(PP + EPDM2) is fixed at 10 wt %.<sup>d</sup> In blends of T<sub>13</sub> to T<sub>16</sub> EPDM2/(PP + EPDM2) is fixed at 20 wt %.



(a)



(b)

**Figure 1** Scanning electron micrographs of PP/EPDM1 blends at different EPDM1 content: (a) 5 wt %; (b) 20 wt %.

spreading coefficient and relative interfacial energy concepts have been used to explain the effect of interfacial interaction on morphology of ternary polymer blends.<sup>5,6,12</sup> We have introduced three simplified equations for relative interfacial energy for three different phase structures.<sup>5,11</sup>

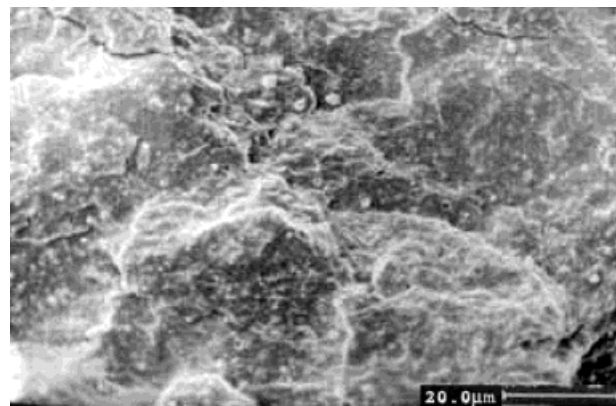
$$(\text{RIE})_{B+C} = \left( \sum A_i \gamma_{ij} \right)_{B+C} / K = x^{2/3} \gamma_{AB} + \gamma_{AC} \quad (1)$$

$$(\text{RIE})_{B/C} = \left( \sum A_i \gamma_{ij} \right)_{B/C} / K = (1+x)^{2/3} \gamma_{AB} + \gamma_{BC} \quad (2)$$

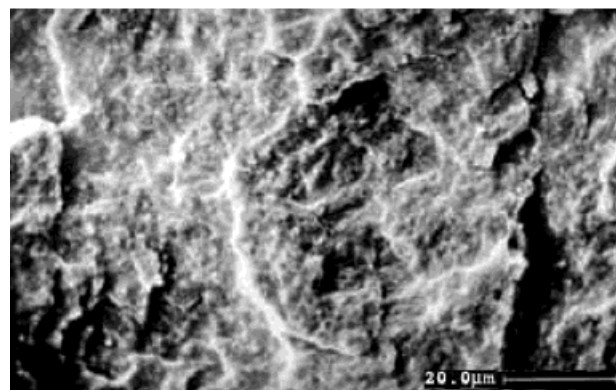
$$(\text{RIE})_{C/B} = \left( \sum A_i \gamma_{ij} \right)_{C/B} / K = x^{2/3} \gamma_{BC} + (1+x)^{2/3} \gamma_{AC} \quad (3)$$

where  $(\text{RIE})_{B+C}$  denotes the relative interfacial energy for the separately dispersed morphology of

two minor components,  $(\text{RIE})_{B/C}$  denotes the morphology in which the B phase encapsulates C, and  $(\text{RIE})_{C/B}$  denotes the morphology in which the C phase encapsulates B.  $\gamma_{ij}$  is the interfacial tension and  $\chi$  is a composition-dependent parameter, as the volume ratio of minor phases. The dominant phase morphology will be the morphology with the lowest relative interfacial energy. The steady-state torque (from Brabender torque versus time plots) has been used as a measure of viscosity, to study the effect of viscosity ratio on morphology and particle size of the dispersed phase.<sup>6–11</sup> For core-shell morphology, we have used the ratio of average steady-state torque of two minor phases to the matrix, to predict the dispersed phase size as a whole,<sup>11</sup> and torque ratio of the core to shell for prediction of core size.<sup>6</sup> As reported earlier,<sup>11</sup> the PP/EPDM/PE (70/15/15 wt %) ternary system shows a core-shell morphology where EPDM encapsulates the PE core, whereas in the PP/

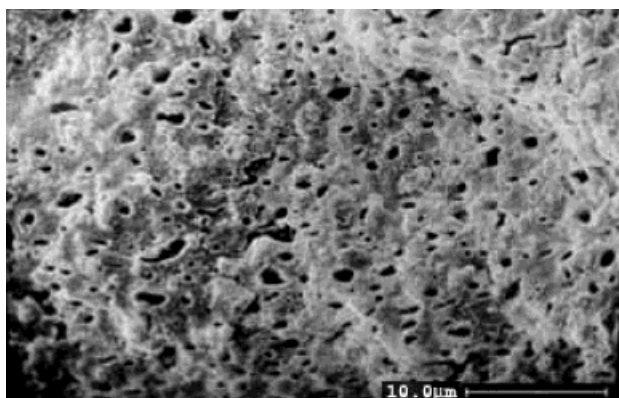


(a)

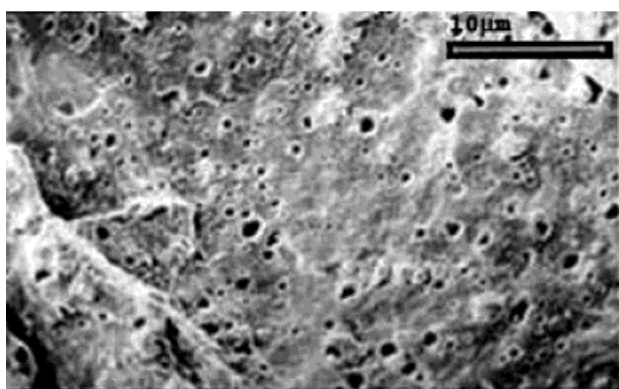


(b)

**Figure 2** Scanning electron micrographs of PP/HDPE blends at different HDPE content: (a) 5 wt %; (b) 20 wt %.



(a)



(b)

**Figure 3** Scanning electron micrographs of PP/EPDM2 blends at different EPDM2 content: (a) 5 wt %; (b) 20 wt %.

EPDM/PS (70/15/15 wt %) ternary system, EPDM and PS disperse separately in the PP matrix.

## EXPERIMENTAL

### Materials

One type of high-density polyethylene (HDPE), one type of isotactic polypropylene (PP), two types of ethylene-propylene-diene terpolymer (EPDM), and one type of polystyrene (PS) were used for blending. The main properties of these polymers and their producers are listed in Table I.

### Blend Preparation

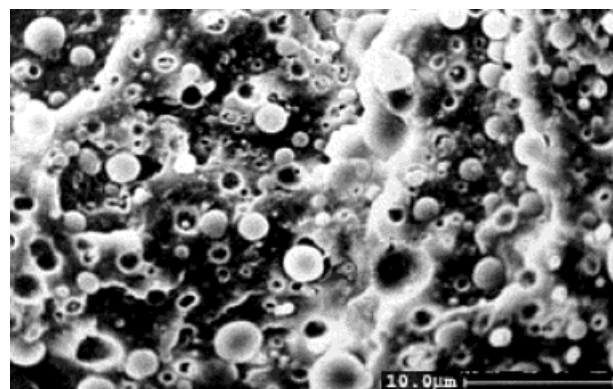
Binary and ternary blends varying in constituents and composition, listed in Tables II and III, were studied. These blends were prepared by melt-mixing in a Brabender internal mixer with roller-type rotors at 190°C and 60 rpm. As shown

in Table II, for binary blends, the minor phase content (EPDM, HDPE or PS) was varied between 5 to 20 wt %. For ternary blends, the EPDM/(PP + EPDM) ratios were kept constant at 10 and 20 wt % and the other minor phase content (HDPE and PS) was varied between 5 to 30 wt %.

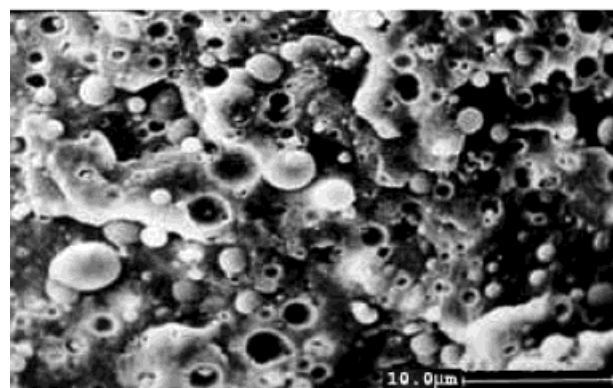
For preparation of binary blends, the minor phase (EPDM or HDPE) was charged into molten PP in the mixer and mixing was carried out for 3 min. For preparing ternary blends, EPDM was added to molten PP in the mixer and after 3 min of mixing, the second minor phase (PS or PE) was charged into the mixer and mixing was continued for a further 3 min.

### Morphology Observations

Morphologies of the blends were studied using scanning electron microscopy (SEM S360; Cambridge Instruments, Worcester, MA). SEM micrographs were taken from cryogenically fractured surfaces of blend specimens. The fractured sur-

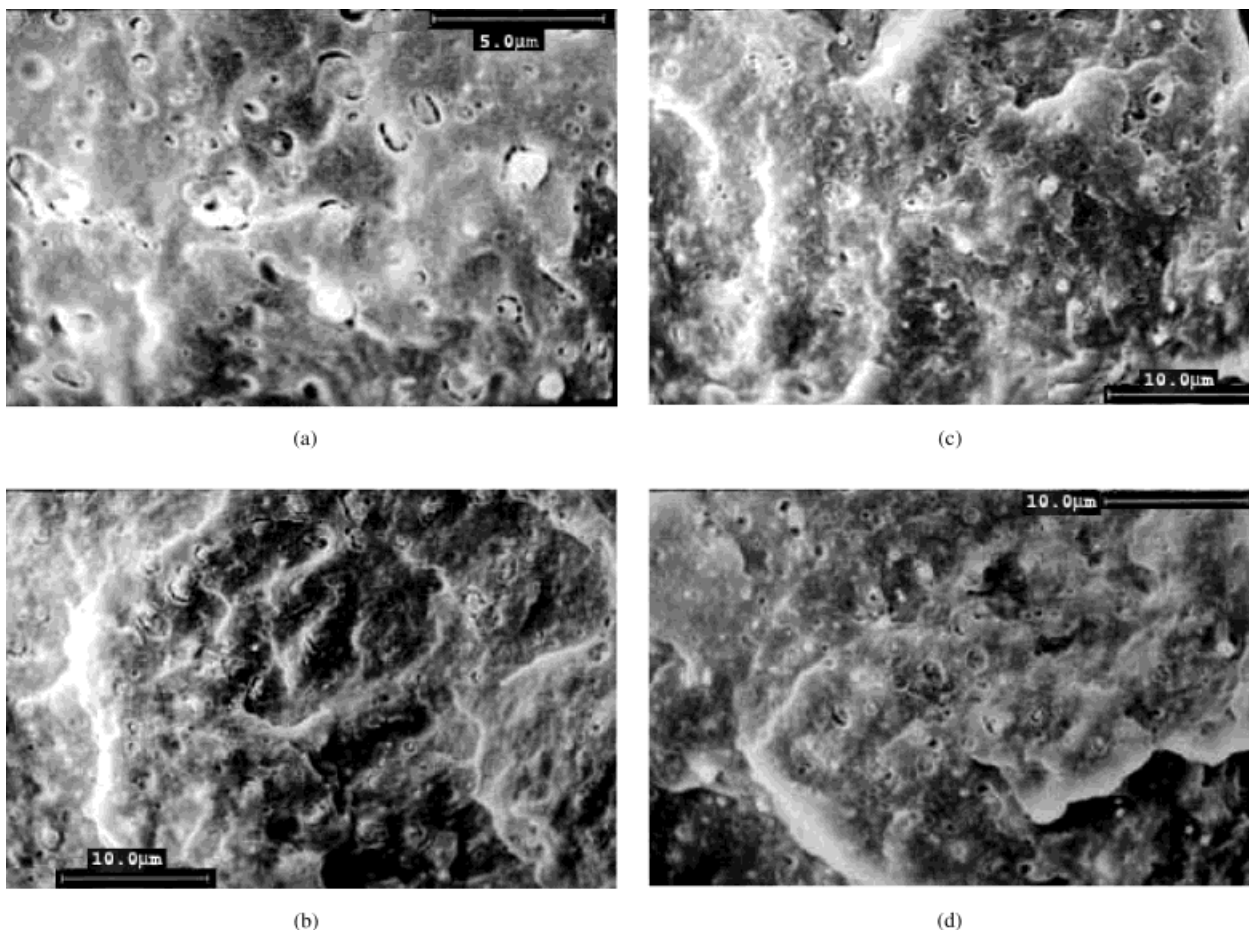


(a)



(b)

**Figure 4** Scanning electron micrographs of PP/PS blends at different PS content: (a) 5 wt %; (b) 20 wt %.



**Figure 5** Scanning electron micrographs of different samples of PP/EPDM1/PE blends: (a) T<sub>1</sub>; (b) T<sub>3</sub>; (c) T<sub>5</sub>; (d) T<sub>7</sub>.

faces of PP/rubber/PE blends were etched by cyclohexane for 24 h at room temperature, to remove the rubber phase, and coated with gold before viewing.

## RESULTS AND DISCUSSION

### Binary Blends

Four binary blends each of PP/EPDM1, PP/EPDM2, PP/HDPE, and PP/PS were studied, as

**Table IV** Interfacial Tensions at 190°C

Interface	Interfacial Tension at 190°C (dyn cm <sup>-1</sup> )
PP/PE	1.23
PP/EPDM1	0.32
PP/EPDM2	0.63
PE/EPDM1	0.39
PP/PS	4.06
PS/EPDM2	4.41

shown in Table II. It is generally known that in binary blends the size of the dispersed phase gradually increases with an increase in phase composition until phase inversion occurs.<sup>13–15</sup>

**Table V** Relative Interfacial Energies for (PP/EPDM/HDPE) Ternary Blends at Different Compositions

Sample Code <sup>a</sup>	$\chi = V_B/V_C$ <sup>b</sup>	RIE <sub>B+C</sub> <sup>b</sup>	RIE <sub>B/C</sub> <sup>b</sup>	RIE <sub>C/B</sub> <sup>b</sup>
T <sub>1</sub>	1.9	1.72	1.04	3.1
T <sub>2</sub>	0.9	1.53	0.88	2.24
T <sub>3</sub>	0.4	1.4	0.79	1.75
T <sub>4</sub>	0.23	1.35	0.758	1.56
T <sub>5</sub>	3.8	2.01	1.3	4.44
T <sub>6</sub>	1.8	1.7	1.03	3.02
T <sub>7</sub>	0.8	1.5	0.86	2.15
T <sub>8</sub>	0.47	1.42	0.8	1.82

<sup>a</sup> See Table III.

<sup>b</sup> B phase is EPDM and C is HDPE.

**Table VI Steady-State Torque of Components at 60 rpm and 190°C**

Polymer	Steady-State Torque at 190°C and 60 rpm (Nm)
PP	17
HDPE	19.2
EPDM1	16.5
EPDM2	41.2
PS	32.8

Figure 1(a) and (b) represent the SEM micrographs of PP/EPDM1 blends containing 5 and 20 wt % of rubbery phase (samples B<sub>1</sub> and B<sub>4</sub>). As expected, in these blends the rubber phase (appearing as dark holes for etched samples) forms a dispersed phase. Average particle sizes of samples B<sub>1</sub> and B<sub>4</sub> measured by image analysis were 0.94 and 1.52  $\mu\text{m}$ , respectively, increasing with increasing minor component content, in accordance with the theoretical predictions.<sup>13–15</sup>

Figure 2(a) and (b) represent SEM micrographs of PP/HDPE blends containing 5 and 20 wt % of HDPE (samples B<sub>5</sub> and B<sub>8</sub>), respectively. It appears from the micrographs that contrast of the phases is not good enough to distinguish the existing two phases.

Figures 3 and 4 show SEM micrographs of PP/EPDM2 (samples B<sub>9</sub> and B<sub>12</sub>) and PP/PS (samples B<sub>13</sub> and B<sub>16</sub>). The results of the image analysis made on these blends showed that for PP/EPDM2 blends the average particle size of the minor phase increases from 1.26  $\mu\text{m}$  for 5 wt % to

1.76  $\mu\text{m}$  for 20 wt %. The increase in the EPDM particle size observed in these blends was found to follow almost the same trend as that of the PP/EPDM1 blends. However, these results demonstrate that the EPDM particle size in PP/EPDM2 blends was higher than that in PP/EPDM1 blends. This is mainly attributed to the higher viscosity of the minor phase in PP/EPDM2 blends compared to that of PP/EPDM1 blends.

In PP/PS blends the average particle size of PS particles increased from 1.8  $\mu\text{m}$  for the blend containing 5 wt % of PS to 2.31  $\mu\text{m}$  for 20 wt % of PS, again in agreement with the theoretical predictions.<sup>13–15</sup>

## Ternary Blends

### PP/EPDM/HDPE Ternary Systems

SEM micrographs of these blends are shown in Figure 5. As can be seen these blends show a core-shell-type morphology in which the PE core is encapsulated by the EPDM phase.

Equations (1) to (3) were used to predict the type of morphology. Relative interfacial energy (RIE) values for each morphology calculated from interfacial energy data at 190°C (Table IV)<sup>11</sup> are listed in Table V. From calculated results it was found that the morphology in which EPDM encapsulates HDPE, irrespective of their composition, has the lowest RIE. Comparing these results with SEM micrographs of the blends (shown in Fig. 5) shows that the experimental results are in good agreement with the above-mentioned theoretical prediction.

The steady-state torque ratio of components was used to predict the size variation of the dis-

**Table VII  $T_{av}$  and Torque Ratios at 190°C and 60 rpm**

Sample Code <sup>a</sup>	$T_{av}$	$T_{av}/T_{pp}$	$T_{HDPE}/T_{EPDM}$	Volume Fraction of Dispersed Phase <sup>b</sup>	Volume Fraction of HDPE in Dispersed Phase <sup>c</sup>
T <sub>1</sub>	17.42	1.024	1.16	0.145	0.34
T <sub>2</sub>	17.93	1.05	1.16	0.19	0.53
T <sub>3</sub>	18.44	1.09	1.16	0.28	0.71
T <sub>4</sub>	18.69	1.1	1.16	0.37	0.81
T <sub>5</sub>	17.04	1.0	1.16	0.24	0.2
T <sub>6</sub>	17.47	1.03	1.16	0.28	0.36
T <sub>7</sub>	18.01	1.06	1.16	0.36	0.56
T <sub>8</sub>	18.33	1.08	1.16	0.44	0.68

<sup>a</sup> See Table III.

<sup>b</sup> (HDPE + EPDM)/(PP + HDPE + EPDM).

<sup>c</sup> HDPE/(HDPE + EPDM).

**Table VIII** Number-Average Diameter of Dispersed Phase and Core Particle of PP/EPDM/PE Ternary Blends

Sample Code <sup>a</sup>	Number-Average Diameter ( $\mu\text{m}$ )	
	Core Particles	Dispersed Phase
T <sub>1</sub>	0.91	1.27
T <sub>2</sub>	1.2	1.32
T <sub>3</sub>	1.51	1.7
T <sub>4</sub>	1.63	1.75
T <sub>5</sub>	0.86	1.51
T <sub>6</sub>	1.16	1.55
T <sub>7</sub>	1.48	1.83
T <sub>8</sub>	1.56	1.88

<sup>a</sup> See Table III.

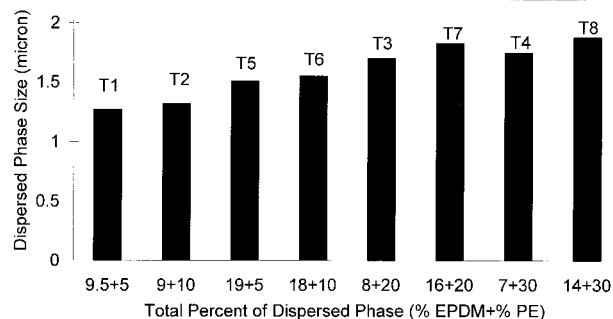
persed phase. The results of steady-state torque of each component at 60 rpm and 190°C, and corresponding average torque and torque ratios are given in Tables VI and VII.

The results of average diameter of HDPE core and dispersed phase as a whole for PP/EPDM/PE ternary blends (samples T<sub>1</sub>–T<sub>8</sub>), measured by image analysis, are given in Table VIII. These results showed that for PP/EPDM/PE ternary blends, with different HDPE content but constant EPDM/(PP + EPDM) ratios (10 and 20 wt %), the dispersed phase size as a whole and core size increase with increasing HDPE content (samples T<sub>1</sub>–T<sub>4</sub> and T<sub>5</sub>–T<sub>8</sub>). The dispersed phase size was also found to increase as a result of increasing EPDM content. The effect of increasing EPDM content on either core or shell, at constant HDPE content, can be evaluated by comparing number-average particle size data of blends T<sub>2</sub> with T<sub>6</sub> or T<sub>3</sub> with T<sub>7</sub> or T<sub>4</sub> with T<sub>8</sub> (Table VIII). From these data it was found that increasing EPDM content at constant HDPE content decreases the PE core size, which can be attributed to the compatibilizing effect of EPDM, for PP and PE phases.

The average particle size of dispersed particles plotted against total dispersed phase content is shown in Figure 6. The average diameter of HDPE core against HDPE content is shown in Figure 7. These figures demonstrate that increasing EPDM content results in increasing the dispersed particle size as a whole, whereas increasing HDPE content increases PE core size only.

#### PP/EPDM/PS Ternary Systems

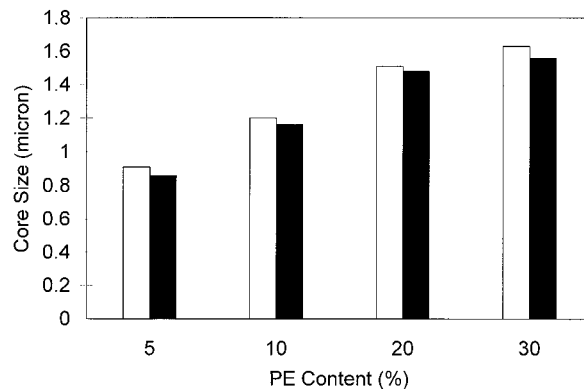
SEM micrographs of these blends are shown in Figure 8. From these micrographs, it was found

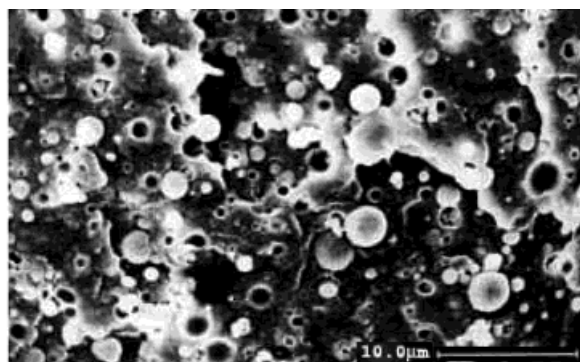
**Figure 6** Number-average diameter of dispersed phase (micron).

that in PP/EPDM/PS ternary blends, PS and EPDM phases disperse separately in the PP matrix. Equations (1) to (3) were used to predict the phase morphology of these blends.

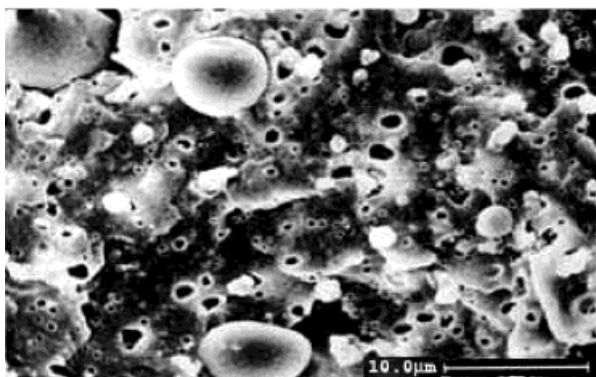
Results of relative interfacial energy (RIE) of these blends also suggest that a morphology with separately dispersed phases has the lowest RIE (Table IX), which is in good agreement with the above-described experimental results. The average particle sizes of each phase measured by image analysis are also listed in Table X. These results showed that increasing PS content at constant EPDM increases PS particle size for both groups of blends (T<sub>9</sub>–T<sub>12</sub> and T<sub>13</sub>–T<sub>16</sub>). A similar trend was found for the EPDM particle size.

The results given in Table X also show that the average rubber particle size in PP/EPDM/PS ternary blends is smaller than that of the PS particle. This can be explained in terms of interfacial tension between matrix and dispersed phase and melt viscosity ratio of two phases. The torque ratios of EPDM/PP and PS/PP are 2.4 and 1.93,

**Figure 7** Number-average diameter of PE cores (micron). Samples T<sub>1</sub>–T<sub>4</sub> ■ and samples T<sub>5</sub>–T<sub>8</sub> □ (micron).



(a)



(b)

**Figure 8** Scanning electron micrographs of different samples of PP/EPDM/PS blends: (a) T<sub>10</sub>; (b) T<sub>15</sub>.

respectively. Moreover,  $\gamma_{12}$  between PP and EPDM is  $0.63 \text{ dyn cm}^{-1}$ , whereas  $\gamma_{12}$  between PP and PS is  $4.06 \text{ dyn cm}^{-1}$ . From these data, together with the concept of the viscosity ratio and interfacial interaction effects, it is expected that

**Table IX** Relative Interfacial Energies for (PP/EPDM/PS) Ternary Blends at Different Compositions

Sample Code <sup>a</sup>	$\chi = V_B/V_C$ <sup>b</sup>	$\text{RIE}_{B+C}$ <sup>b</sup>	$\text{RIE}_{B/C}$ <sup>b</sup>	$\text{RIE}_{C/B}$ <sup>b</sup>
T <sub>9</sub>	1.9	5.02	5.69	15.01
T <sub>10</sub>	0.9	4.65	5.37	10.3
T <sub>11</sub>	0.4	4.4	5.197	7.45
T <sub>12</sub>	0.23	4.29	5.13	6.3
T <sub>13</sub>	3.8	5.59	6.23	22.21
T <sub>14</sub>	1.8	4.99	5.66	14.6
T <sub>15</sub>	0.8	4.6	5.34	9.8
T <sub>16</sub>	0.47	4.44	5.22	7.88

<sup>a</sup> See Table III.

<sup>b</sup> B phase in EPDM and C is PS.

**Table X** Number-Average Diameter of Dispersed Phases ( $\mu\text{m}$ )

Sample Code <sup>a</sup>	Number-Average Diameter ( $\mu\text{m}$ )	
	EPDM Particles	PS Particles
T <sub>9</sub>	1.38	1.7
T <sub>10</sub>	1.29	1.95
T <sub>11</sub>	1.3	2.18
T <sub>12</sub>	1.33	2.33
T <sub>13</sub>	1.68	1.86
T <sub>14</sub>	1.72	2.01
T <sub>15</sub>	1.75	2.12
T <sub>16</sub>	1.71	2.28

<sup>a</sup> See Table III.

the EPDM phase forms smaller particle size than does PS in the PP matrix.

## CONCLUSIONS

PP/EPDM/PE ternary blends showed a core-shell-type of morphology in which EPDM encapsulates PE in the PP matrix. The results also showed that in PP/EPDM/PE ternary blends, the size of the PE core and dispersed phase as a whole is directly related to PE content. Increasing EPDM content at constant amount of PE increases the dispersed phase size, while reducing PE core size. However, in PP/EPDM/PS ternary blends, in contrast to PP/EPDM/PE blends, minor phases (EPDM and PS) form separate phases dispersed in the PP matrix. The size of each dispersed phase in these blends can be related to its composition, interfacial interaction, and melt viscosity ratio between each minor phase and the matrix. The results also showed that EPDM particles are generally smaller than PS particles in the PP matrix because of their lower interfacial tension with PP. Finally, the morphology observed for both groups of ternary blends was found to be in good agreement with that predicted on the basis of relative interfacial energy (RIE) and spreading coefficient concepts.

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