

# Thermogravimetric Analysis of Blends of Low-Density Polyethylene and Poly(dimethyl siloxane) Rubber: The Effects of Compatibilizers

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**ABSTRACT:** The thermal stability of vulcanizates of low-density polyethylene (LDPE), poly(dimethyl siloxane) (PDMS) rubber, and their blends was studied by nonisothermal thermogravimetry. Four ethylene copolymers [ethylene methyl acrylate (EMA), ethylene vinyl acetate, ethylene acrylic acid, and a zinc-salt-based ionomer (Lotek 4200)] were used as compatibilizers for the blend systems. The thermograms and derivatograms of the blends showed that thermal degradation took place in two stages, whereas those for the base polymers showed single-stage degradation. Kinetic studies of the blends and pure components showed that the degradation followed first-order reaction kinetics. The activation energy at 10% degradation was determined

with the Freeman–Carroll method and was at a maximum (42.34 kcal/mol) for the 25:75 LDPE/PDMS rubber blend. The half-life at 200°C was evaluated by the Flynn–Wall method and was at a maximum (812.5 days) for the same blend. Out of four compatibilizers, EMA showed the maximum activation energy (34.25 kcal/mol) for degradation and a maximum half-life (695.3 days), indicating that EMA was the best compatibilizer for the blend system. © 2003 Wiley Periodicals, Inc. *J Appl Polym Sci* 90: 635–642, 2003

**Key words:** thermogravimetric analysis (TGA); activation energy; ionomers; crosslinking

## INTRODUCTION

The study of the thermal degradation of individual polymers and their blends is very important to predicting their suitability in the specific field of applications. The thermal stability and mode of decomposition of a polymer are largely dependent on the chemical structure of the chain segments or repeat units. Interestingly, the degradation characteristics of a polymer cannot be precisely predicted on the basis of those exhibited by low-molecular-weight model compounds. Even though the mode of decomposition is much the same, the polymers degrade or decompose at temperatures far lower than those of the corresponding model compounds. The degradation may primarily lead to chain scission or depolymerization reactions, resulting in a decrease in chain length or molecular weight due to the breaking of the main polymer chain backbone.

The thermal stability of polymer blends and alloys has been extensively studied by several authors.<sup>1–9</sup> Varughese<sup>10</sup> reported the thermal stability and flame-retardant characteristics of miscible blends of poly(vi-

nyl chloride) and epoxidized natural rubber (ENR). Paulmer et al.<sup>11</sup> reported the effect of crosslinking agents on the structure and thermal stability of millable polyurethane (PU) elastomer. They evaluated the effect of dicumyl peroxide (DCP) and toluene diisocyanate dimer (TDID) as crosslinking agents on the relative thermal stability of the PU composites. A mixture of DCP and TDID was also studied as the crosslinking agent. A dramatic improvement in the thermal stability of PU elastomer was observed because of synergism in the crosslinking mechanism achieved with a combination of DCP and TDID.

Santra et al.<sup>12</sup> studied the thermal stability of miscible blends of ethylene methyl acrylate (EMA) copolymer and poly(dimethyl siloxane) (PDMS) rubber. They reported that the degradation of the blends and the pure components followed first-order reaction kinetics. Moreover, the blends exhibited thermal stability intermediate between the two blend constituents. Mohanty et al.<sup>13</sup> studied the decomposition behavior of the blends of polyethylene-co-acrylic acid and ENR. Thermograms of ENR showed a clear two-stage degradation. On further investigation, it was observed that a multistage degradation occurred in the blends containing 70 and 50% ENR, whereas blends containing 30, 20, and 10% ENR exhibited two-stage degradations, indicating stronger chemical interactions between the blend constituents during melt processing.

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**TABLE I**  
**Materials**

Material	Trade name	Supplier	Specification
LDPE	Indothane 20XL020	IPCL (Vadodara, India)	SG = 0.919 MFI = 2.0 g/10 min, mp = 112°C
PDMS	Silastic WC-50	Dow Corning, Inc. (Midland, MI)	SG = 1.150 Brittle temperature = -39°C
EMA	Optema TC-120	Exxon Chemical, Inc. (Mumbai, India)	SG = 0.940, MFI = 6.0 dg/min, mp = 81°C, methyl acrylate content = 21%
EVA	—	Polyolefins Industries, Ltd. (Mumbai, India)	SG = 0.950, vinylacetate content = 28 wt %
EAA	Escor 5001	Exxon Chemical (Antwerp, Belgium)	SG = 0.930, Acrylic acid content = 6 wt %
Ionomer	Lotek 4200	Exxon Chemical (Antwerp, Belgium)	SG = 0.946, MFI = 3.0 g/10 min, acrylic acid content = 5 wt %, Zn salt of acrylic acid = 5 wt %
DCP	98% pure	Aldrich Chemical Co., Inc. (Los Angeles, CA)	mp = 39–41°C, decomposition temperature = 88°C

SG = specific gravity; MFI = melt flow index; mp = melting point.

Thermal stability of compatibilized blends of natural rubber (NR) and poly(methyl methacrylate) (PMMA) by addition of NR-g-PMMA was analyzed by thermogravimetry.<sup>14</sup> The compatibilized blends were more thermally stable than uncompatibilized blends. Effects of ethylene content and maleated EPDM content on the thermal stability and degradation kinetics of EPDM were also studied<sup>15,16</sup> with high-resolution thermogravimetric analysis (TGA) and modulated TGA. Modulated TGA showed that the rubber degradation was complex. Budrugaec<sup>17</sup> did rapid prediction of the thermal lifetime of polymeric materials by evaluating the activation energy ( $\Delta E$ ) with the isothermal degradation method and a rapid analyzing method. It was found that there was large difference between the  $\Delta E$  values evaluated by the conventional method and by the analyzing method, indicating a large error in the thermal lifetime prediction with the rapid method.

Silicone rubber has long been recognized as a specialty rubber for applications over a wide range of high and low temperatures. Low-density polyethylene (LDPE), being the most abundantly used commodity plastic, is used extensively as the base polymer for the insulation coating of wires and cables over a wide voltage range. Most synthetic rubbers [e.g., styrene-butadiene rubber, ethylene propylene diene rubber (EPDM), chloroprene rubber] have been replaced by LDPE for its low dielectric loss properties and for its superior mechanical properties at ambient temperatures and because of its lower cost. Santra et al.<sup>18</sup> attempted to compatibilize LDPE/PDMS blends with the help of EMA's physicommechanical properties. In this investigation, we tried to compatibilize LDPE/PDMS blends with the help of EMA and three ethylene copolymers [viz., ethylene vinyl acetate (EVA), ethylene acrylic acid (EAA), and Lotek] on the basis of thermal stability studies. This study corroborated the degradation temperatures,  $\Delta E$  for degradation, and

half-lives for base polymers, blends without compatibilizer, blends with EMA compatibilizer, and blends with different compatibilizers.

## EXPERIMENTAL

### Materials

The details for the materials we used are given in Table I.

### Preparation of the blends

PDMS rubber and ethylene copolymers (EMA, EVA, EAA, and Lotek ionomer) were melt-mixed in a Brabender plasticorder (model PLE-330) with cam-type rotors at different temperatures (Duisberg, Germany)<sup>19,20,13,21</sup> (Table II) at a rotor speed of 100 rpm for 6 min; then, LDPE was added and mixed for another 2

**TABLE II**  
**Blend Composition**

Blend code	LDPE (wt %)	PDMS (wt %)	Compatibilizer (wt %)	DCP (wt %)	Mixing temperature (°C)
A	100	0	0	1.5	180
B	75	25	0	1.5	180
C	50	50	0	1.5	180
D	25	75	0	1.5	180
E	0	100	0	1.5	180
F	75	25	1 (EMA)	1.5	180
G	75	25	2 (EMA)	1.5	180
H	75	25	6 (EMA)	1.5	180
I	75	25	2 (EVA)	1.5	160
J	75	25	2 (EAA)	1.5	150
K	75	25	2 (Lotek)	1.5	140
L	0	0	100 (EMA)	1.5	180
M	0	0	100 (EVA)	1.5	160
N	0	0	100 (EAA)	1.5	150
O	0	0	100 (Lotek)	1.5	140

TABLE III  
Degradation of the Base Polymers

Sample code	$T_i$ (°C)	$T_{1\max}$ (°C)	$T_{2\max}$ (°C)	$T_1$ (°C)	$T_{50}$ (°C)
A	305.26	425.18	—	554.82	407.50
E	363.00	491.50	—	560.00	491.50
L	331.90	476.20	—	516.25	396.82
M	396.15	428.18	—	507.85	484.67
N	385.18	465.35	—	518.54	458.65
O	345.15	485.16	—	520.17	450.63

min. The molten mass was then taken out and sheeted out on a two-roll 150 × 300-mm laboratory mill kept at room temperature. The sheet was again charged into the plasticorder at the aforesaid temperatures and re-mixed for another 2 min. The temperature was lowered down to 140°C, and a requisite quantity of DCP was added and mixed for 2 min before it was sheeted out on the two-roll mill. Subsequently, the sheets were compression-molded in a molding press (Moore, Birmingham, England) at the respective temperatures for 2 min at a pressure of 10 MPa, and then, we cooled the sheets under pressure by passing cold water (25°C) through the platens of the press. All other mixes according to Table II were prepared with the same procedure. For different compatibilizers, we chose different mixing temperatures to understand the extent of interaction with the blend components without causing any degradation.

### TGA

TGA and derivative TGA of the blends and pure components were carried out in a Stanton Redcroft simultaneous thermogravimetric analyzer (model STA 625) equipped with a computer data analyzer (version C 4.20, Dublin, Ireland) in a nitrogen atmosphere (flow rate = 50 mL/min) at a heating rate of 10°C/min. The weight of the sample was approximately 5 mg in all cases.

### Freeman–carroll method

$\Delta E$  for the 10% decomposition of the blend and the order of the degradation reaction were determined with the standard kinetic equation<sup>22</sup>

$$-dw/dt = Ae^{-\Delta E/RT}W^n \quad (1)$$

where  $dw/dt$  is the rate of the degradation reaction (mg/s),  $A$  is the preexponential factor ( $s^{-1}$ ),  $\Delta E$  is the activation energy (kcal/mol),  $R$  is the molar gas constant ( $1.987 \times 10^{-3}$  kcal/mol),  $T$  is the absolute temperature (K),  $W$  is the active weight at a particular time (mg), and  $n$  is the order of the reaction (dimensionless).

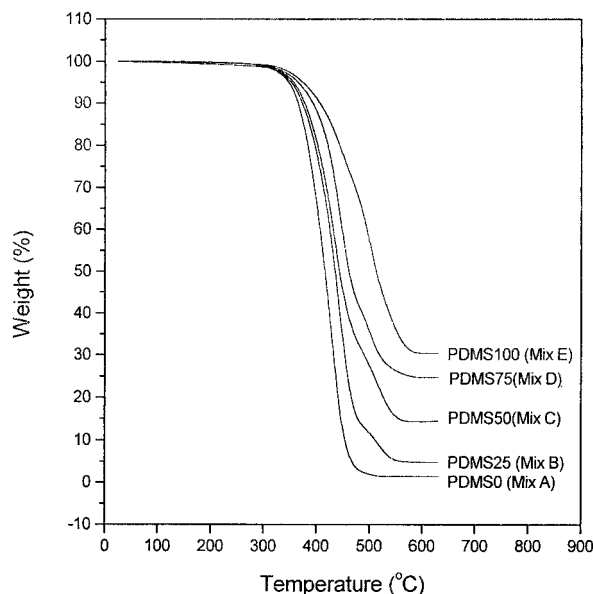


Figure 1 TGA of different LDPE/PDMS rubber blends.

The thermograms were analyzed graphically to determine the  $\Delta E$  value and half-life with the Freeman–Carroll and Flynn–Wall methods, respectively, with the help of the software package supplied by Stanton Redcroft (version C4.20).

### Phase morphology

The phase morphology of the cryogenically fractured surface of LDPE/PDMS blends was studied with scanning electron microscopy (SEM; Cam Scan Series-II). Strips (2 × 2 mm) were cut from the molded sheets and then fractured cryogenically. The samples were dried at 70°C in a vacuum drier for 4 h; afterward, they were kept in a desiccator until they cooled to room temperature. Subsequently, the fractured surfaces were sputter-coated with gold to facilitate scanning under SEM at a 0° tilt angle.

## RESULTS AND DISCUSSION

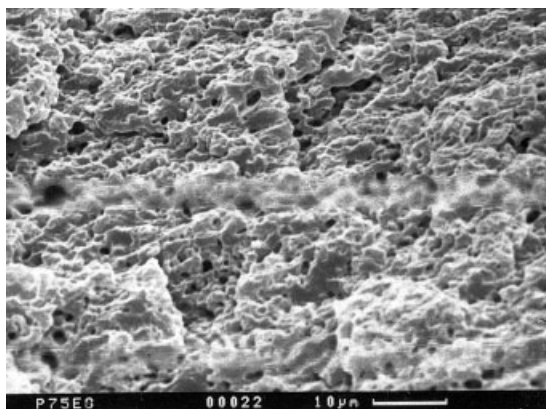
### Degradation temperature

#### Base polymers

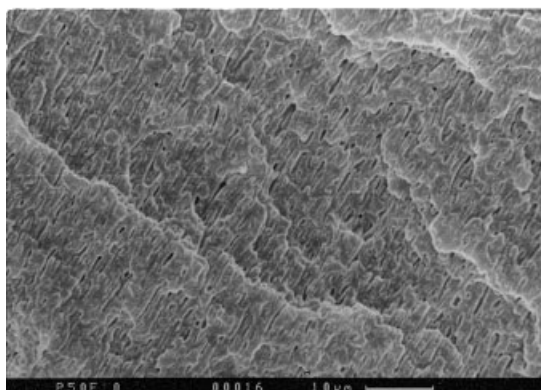
The characteristic degradation temperatures for the crosslinked base polymers obtained from the thermo-

TABLE IV  
Degradation Temperatures of LDPE, PDMS Rubber, and Their Blends

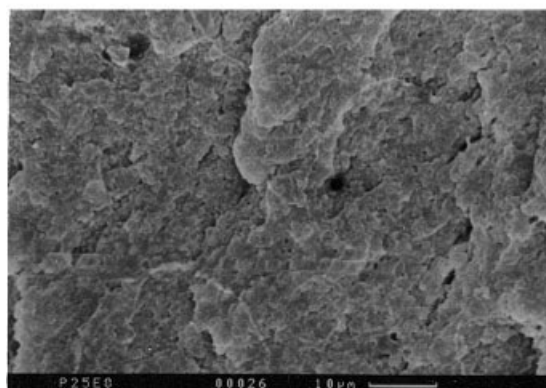
Sample code	$T_i$ (°C)	$T_{1\max}$ (°C)	$T_{2\max}$ (°C)	$T_f$ (°C)	$T_{50}$ (°C)
A	305.26	425.18	—	554.82	407.50
B	307.14	436.19	508.12	580.95	419.04
C	308.37	450.22	487.22	586.12	465.64
D	345.71	471.43	528.37	595.24	514.29
E	363.00	491.50	—	560.00	491.50



(a)



(b)



(c)

**Figure 2** SEM micrographs for LDPE/PDMS blends: (a) 75:25, (b) 50:50, and (c) 25:75.

grams are summarized in Table III. The initial degradation temperature ( $T_i$ ), corresponding to 1% decomposition for LDPE, PDMS rubber, EMA, EVA, EAA, and Lotek, were 305.26, 363, 331.90, 396.15, 385.18, and 345.15°C, respectively. It was clear from the data that

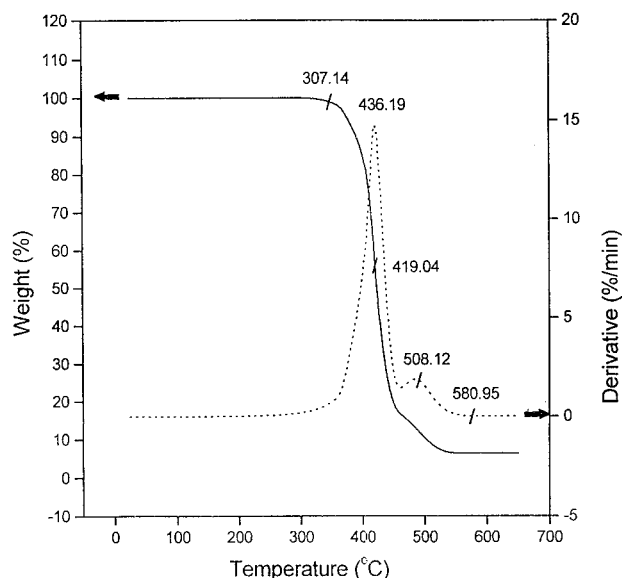
LDPE started an early degradation compared to the other base polymers.

### Blends without compatibilizer

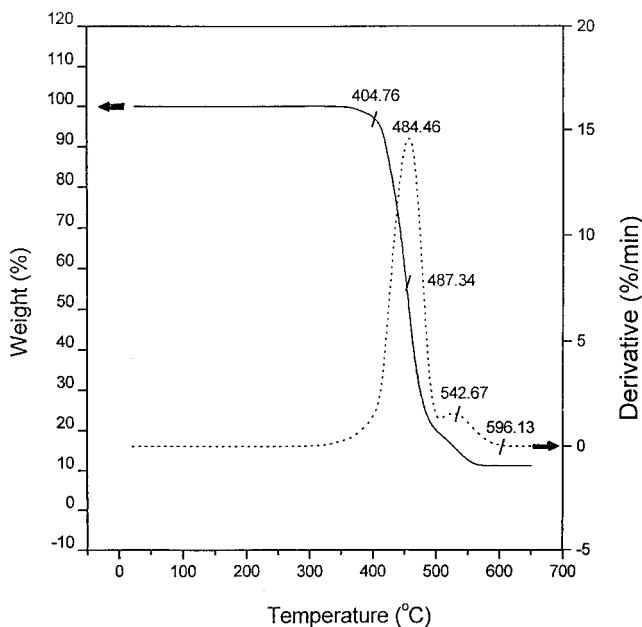
Thermograms of the crosslinked blends are shown in Figure 1, and the characteristic degradation temperatures are summarized in Table IV. It was clear from the data that with increasing proportion of PDMS rubber in the blends, the  $T_i$ , the maximum decomposition temperatures ( $T_{1\max}$  and  $T_{2\max}$ ), the final decomposition temperature ( $T_f$ ), and the temperature at which 50% weight loss occurred ( $T_{50}$ ) increased marginally. This may have been due to incompatibility between the blends, which led to two-phase morphology in the blends (Fig. 2). However, crosslinking by DCP brought out thermal stability to some extent by introducing crosslinks at the interface. Intermolecular and intramolecular crosslinking formation were also possible between LDPE and PDMS rubber. This was responsible for marginally enhancing the stability.

### Blends with EMA compatibilizer

With the incorporation of EMA copolymer in 75:25 LDPE/PDMS rubber blends from 0 to 6 wt % (Figs. 3–6.),  $T_i$  gradually increased and reached a maximum at 2 wt % EMA (i.e., 307.14°C for 0 wt %, 404.76°C for 1 wt %, 407.34 for 2 wt %, and 405.20°C for 6 wt % EMA). A similar trend was observed for  $T_{1\max}$ ,  $T_{2\max}$ ,  $T_f$  and  $T_{50}$  in the pure components and in the blends (Table V). The  $T_f$  corresponds to the temperature after which there is negligible weight loss.  $T_f$  for the blend containing 2 wt % EMA occurred at a temperature as high as 614.29°C, which indicates that this blend was

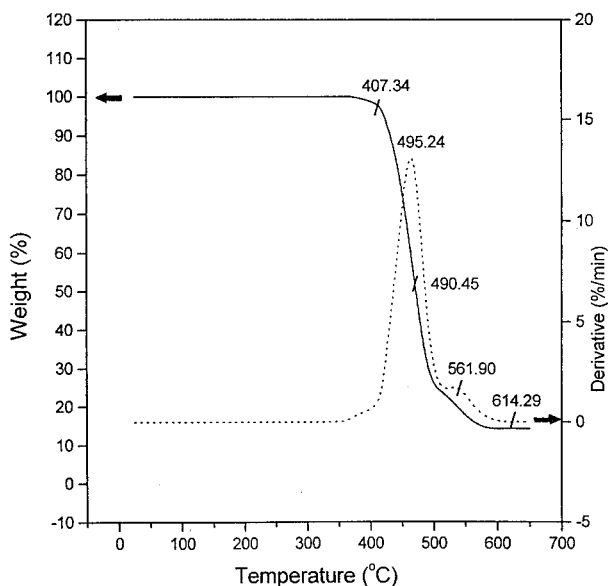


**Figure 3** TGA and derivative curves of 75:25 LDPE/PDMS rubber blends without EMA (mix B).

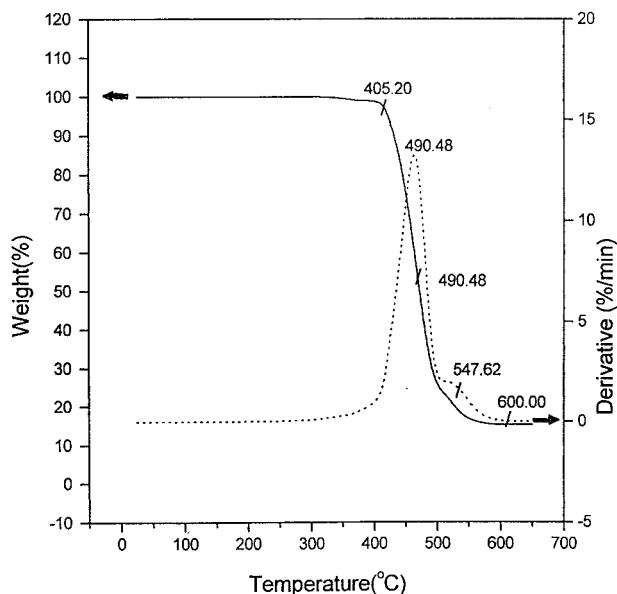


**Figure 4** TGA and derivative curves of 75:25 LDPE/PDMS rubber blends with 1 wt % EMA (mix F).

relatively more stable compared to all of the other blends. This was also reflected in the  $T_{50}$  values of the blends. LDPE/PDMS rubber blends are incompatible due to a high surface energy difference between the blend constituents. By introducing the EMA copolymer into the blend systems, we made the two blend constituents compatible through the formation of EMA-g-PDMS. The mechanism of compatibilization was already discussed in a previous article by Santra et al.<sup>23</sup> Thus EMA-g-PDMS acted as virtual bridge holding the two phases (LDPE continuous and PDMS



**Figure 5** TGA and derivative curves of 75:25 LDPE/PDMS rubber blends with 2 wt % EMA (mix G).



**Figure 6** TGA and derivative curves of 75:25 LDPE/PDMS rubber blends with 6 wt % EMA (mix H).

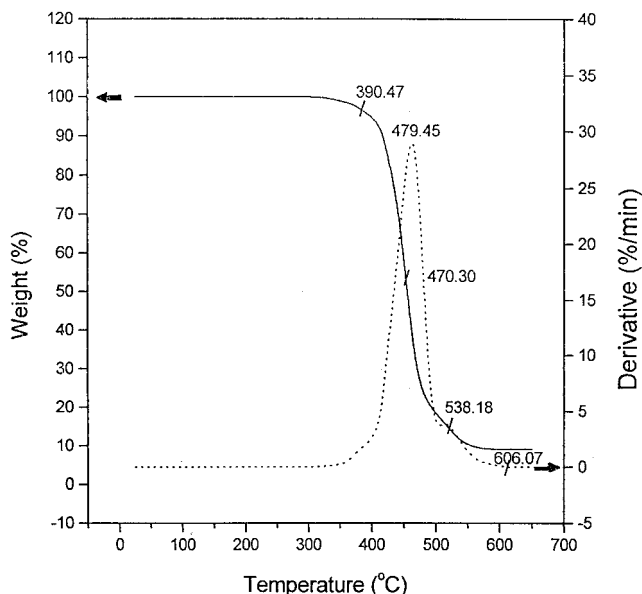
rubber dispersed) together. So the initiation temperature for degradation ( $T_i$ ) kept increasing as the EMA copolymer proportion increased in the system. Afterward, it reached a maximum at the optimum proportion of EMA copolymer, that is, 2 wt % in the 75:25 LDPE/PDMS rubber blend. At a higher proportion of the EMA copolymer (>2 wt %), EMA-g-PDMS rubber tended to form a separate phase, which thus lowered the thermal stability.

Blends with different compatibilizers

The other different ethylene copolymers (e.g., EVA, EAA, and Lotek 4200) were used as compatibilizers for the 75:25 LDPE/PDMS rubber blends. The degradation temperatures of the blends (Figs. 7–9.) are summarized in Table VI. The blend containing 2 wt % EAA showed the maximum  $T_i$ , whereas those for EMA, Lotek, and EVA followed a decreasing order. However,  $T_{1max}$  and  $T_{2max}$  were at maximum for EMA, whereas those EVA, Lotek, and EAA followed a decreasing trend. With the incorporation of compatibilizer, chemical interaction (e.g., grafting and crosslinking) and physical entanglement between the

**TABLE V**  
Effect of EMA on the Degradation Temperatures of 75:25 LDPE/PDMS Rubber Blends

Sample code	$T_i$ (°C)	$T_{1max}$ (°C)	$T_{2max}$ (°C)	$T_f$ (°C)	$T_{50}$ (°C)
B	307.14	436.19	508.12	580.95	419.04
F	404.76	484.46	542.67	596.13	487.34
G	407.34	495.24	561.90	614.29	490.45
H	405.20	490.48	547.62	600.00	490.48



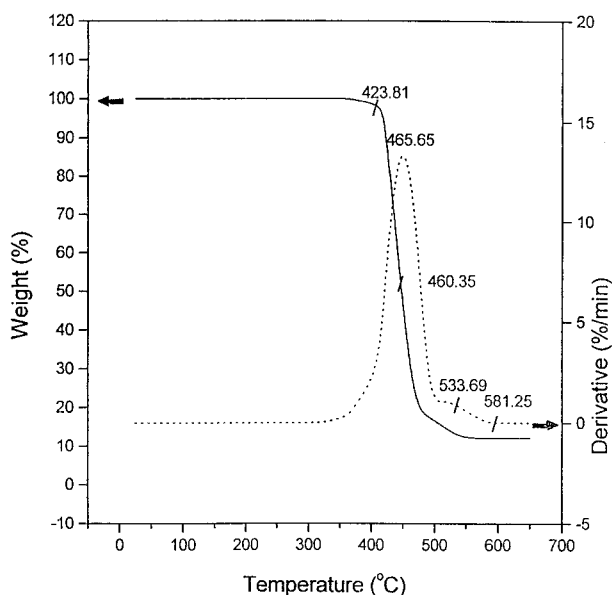
**Figure 7** TGA and derivative curves of 75:25 LDPE/PDMS rubber blends with 2 wt % EVA (mix I).

two constituents were possible at the interface only, but the bulk of the polymer remained uninfluenced. So individual components were likely to follow their own degradation route. This explains why the compatibilizer did not significantly improve the thermal stability.

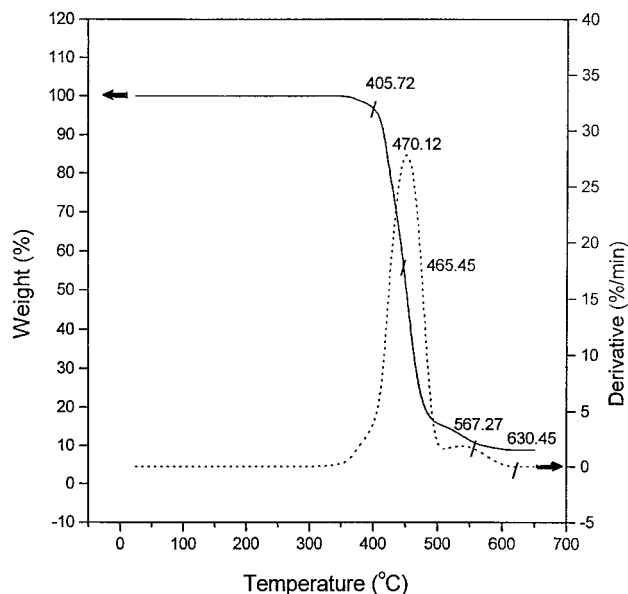
### $\Delta E$

#### Base polymers

$\Delta E$  for LDPE, PDMS, and EMA were 26.71, 22.67, and 16.41 kcal/mol, respectively (Table VII). At the initial



**Figure 8** TGA and derivative curves of 75:25 LDPE/PDMS rubber blends with 2 wt % EAA (mix J).



**Figure 9** TGA and derivative curves of 75:25 LDPE/PDMS rubber blends with 2 wt % Lotek 4200 (mix K).

stage of degradation for PDMS rubber, the elimination of oligomer occurred at a lower temperature from the chain ends, leading to a low  $\Delta E$  for degradation. However, PDMS actually decomposed over a wide range of temperatures, whereas LDPE and the other compatibilizers decomposed over a narrow range of temperatures.

#### Blends without compatibilizer

The theoretical and experimental  $\Delta E$  values for 75:25, 50:50, and 25:75 LDPE/PDMS rubber blends are shown in Table VII. With increasing content of PDMS rubber into the blends, the theoretical  $\Delta E$  decreased, as the  $\Delta E$  of PDMS rubber was lower than that of LDPE. However, the experimental  $\Delta E$  followed the reverse trend. This may have been due to the fact that in addition to the crosslinking of the blend constituents, PDMS rubber contained about 30 wt % inherent silica filler, as supplied by the manufacturer, which may have formed physical entanglements with the crystalline zone of LDPE and, thereby, increased LDPE/PDMS rubber interaction and the thermal stability of the blend systems.

**TABLE VI**  
Effect of Different Compatibilizers on the Degradation Temperatures of 75:25 LDPE/PDMS Rubber Blends

Sample code	$T_i$ (°C)	$T_{1max}$ (°C)	$T_{2max}$ (°C)	$T_f$ (°C)	$T_{50}$ (°C)
G	407.34	495.24	561.90	614.29	490.45
I	390.47	479.45	538.18	606.07	470.30
J	423.81	465.65	533.69	581.25	460.35
K	405.72	470.12	567.27	630.45	465.45

**TABLE VII**  
 **$\Delta E$  and Half-Life as Evaluated by the Flynn–Wall Method (and Verified by the Freeman–Carroll Method)**

Sample code	$\Delta E$ (kcal/mol)		Half-life at 200°C days)	
	Theoretical	Experimental	Theoretical	Experimental
A	—	26.71	—	5.7
B	25.70	31.54	7.65	624.5
C	24.69	36.46	9.60	726.7
D	23.68	42.34	11.55	812.5
E	—	22.67	—	13.5
F	25.86	33.16	7.70	673.1
G	26.03	34.25	7.75	695.3
H	26.68	32.27	7.94	680.2
I	25.94	29.25	7.72	653.7
J	26.01	30.75	7.75	665.1
K	26.02	32.19	7.78	679.6
L	—	16.41	—	4.9
M	—	12.16	—	3.5
N	—	15.25	—	5.1
O	—	16.10	—	6.5

#### Blends with EMA compatibilizer

As the EMA proportion in the blend increased, the  $\Delta E$  value increased and reached a maximum at 2 wt % EMA copolymer; then, it decreased. The increase in  $\Delta E$  may have been due to the chemical reaction (e.g., grafting and crosslinking) between EMA copolymer and PDMS rubber and the physical interaction between EMA copolymer and LDPE leading to cocrystallization.<sup>18</sup> The EMA-g-PDMS formed *in situ* during reactive processing<sup>18</sup> helped to bridge the LDPE matrix with PDMS rubber. At higher levels of EMA copolymer, it formed a separate phase in the compatibilized blend, which led to a lower thermal stability of the blends. The experimental values of  $\Delta E$  for all of the blends were higher than the theoretical values, as calculated with the additive rule. The theoretical activation energy of the thermal degradation of a polymer blend ( $\Delta E_b$ ) was calculated with the following relationship:<sup>24</sup>

$$\Delta E_b = W_1\Delta E_1 + W_2\Delta E_2 + \dots \pm \Delta E \quad (2)$$

where  $W$  is the weight fraction of each component and  $\Delta E_1$ ,  $\Delta E_2$ , and so on, are the activation energies for thermal degradation of each component.  $\pm\Delta E$  is the difference in the energy produced on blending as a result of the formation of polymer–polymer ter bonds. The difference between the experimental and theoretical  $\Delta E$  values was at a maximum for 2 wt % EMA (Fig. 10), which was in good agreement with the higher thermal stability of the crosslinked and compatibilized blend system.

#### Blends with different compatibilizers

As shown in Table VII, the experimental  $\Delta E$  values for 2 wt % EMA, EVA, EAA, and Lotek 4200 in 75:25

LDPE/PDMS rubber blends were 34.25, 29.25, 30.75, and 32.19 kcal/mol, respectively. So from this  $\Delta E$  data, we found that the blends containing 2 wt % EMA were the most stable blends.

#### Half-life

##### Base polymers

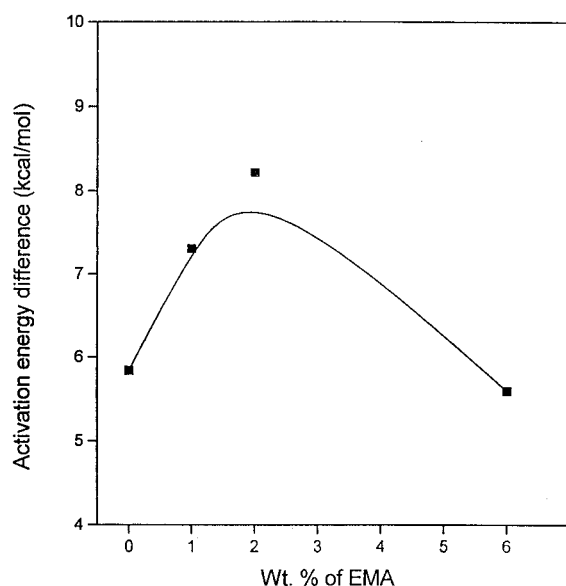
The half-lives for the base polymers were very small. They varied within 15 days approximately. They were characteristic features of the base polymers.

##### Blends without compatibilizer

The experimental half-life increased as the proportion of PDMS rubber in the blends increased (Table VII). This was because of a higher thermal stability of silicone rubber.

##### Blends with EMA compatibilizer

The half-life of the blends reflected their life span under higher temperature conditions. The half-lives of the blends were quite high compared to their individual components. With increasing EMA content up to 2 wt %, half-life increased from 624.5 days for 0 wt % EMA to 695.3 days for 2 wt % EMA copolymer. Beyond 2 wt % EMA copolymer in the blend, half-life decreased again. The half-life was at a maximum for the compatibilized LDPE/PDMS rubber blend containing 2 wt % EMA, which confirmed that it was the most thermally stable blend.



**Figure 10** Plot of the differences in experimental and theoretical  $\Delta E$  values with weight percentage of EMA in the blend.

### Blends with different compatibilizers

The half-life of 75:25 blends with different compatibilizers varied marginally (Table VII). The blends containing 2 wt % EMA, EVA, EAA, and Lotek 4200 showed half-lives of 695.3, 653.7, 665.1, and 679.6 days, respectively. Blends with EMA had the highest half-lives, whereas those with EVA had the shortest half-lives. As EMA reacted with the other blend constituents by chemical bond formation and physical interaction,<sup>18</sup> it gave the blend its maximum half-life. However, the other compatibilizers interacted with the blend constituents by physical interaction only, thereby producing less thermal stability compared to that of the EMA copolymer.

### CONCLUSIONS

1.  $T_g$  shifted toward higher temperatures for the blends containing 2 wt % copolymer.
2. The degradation reaction exhibited first-order kinetics for all of the blends.
3. The compatibilized blend vulcanizates exhibited a two-stage degradation.
4. The maximum half-life was at a maximum for the blend containing 2 wt % EMA.
5. The experimental  $\Delta E$  was higher than theoretically calculated value for all of the blends, and the difference was highest with EMA compatibilizer.

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