

# Mechanical Properties of Thermoplastic Elastomers Based on Silicone Rubber and an Ethylene–Octene Copolymer by Dynamic Vulcanization

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**ABSTRACT:** Thermoplastic vulcanizates (TPVs) are a special class of thermoplastic elastomers that are generally produced by the simultaneous mixing and crosslinking of a rubber with a thermoplastic polymer at an elevated temperature. Novel peroxide-cured TPVs based on blends of silicone rubber and the thermoplastic Engage (an ethylene–octene copolymer) have been developed. These TPVs exhibit very good overall mechanical and electrical properties. With an increasing concentration of dicumyl peroxide, the tensile strength, modulus, and hardness of the TPVs increase, whereas the elongation at break decreases. Signif-

icant correlations have been obtained from oscillating disc rheometer torque values with various physical properties, such as the modulus and tension set of the TPVs. The aging characteristics and recyclability of the silicone-based TPVs are also excellent. Scanning electron microscopy photomicrographs of the TPVs have confirmed a dispersed phase morphology. © 2008 Wiley Periodicals, Inc. *J Appl Polym Sci* 108: 1079–1085, 2008

**Key words:** blends; elastomers; thermoplastics; rubber; vulcanization

## INTRODUCTION

Nowadays, thermoplastic elastomers (TPEs) are among the rapidly growing materials in the polymer industry and have extremely high potential growth in the future. They combine the processing characteristics of thermoplastics with the elastic and mechanical properties of conventional vulcanized rubbers. TPEs have both advantages and disadvantages in comparison with conventional vulcanized rubbers, including the following:

### Advantages:

- No vulcanization and very little compounding are required.
- They are suitable for methods of thermoplastic processing, such as injection moldings, blow molding, thermoforming, and heat welding.
- Scrap can be recycled and reused without significant deterioration in performance.
- Properties can very easily be manipulated for different requirements by just a change in the blend ratio.

### Disadvantages:

- TPEs soften or melt at an elevated temperature, above which they lose their rubbery character.

- On extended use, TPEs show creep behavior.<sup>1–3</sup>

TPEs based on rubber/thermoplastic compositions are generally classified into two main categories: the first category consists of simple blends and is commonly designated as thermoplastic elastomeric olefins, and in the second category, the rubber phase is dynamically vulcanized in the presence of a suitable crosslinking agent, leading to a thermoplastic vulcanizate (TPV) or dynamic vulcanizate.

The first TPE was introduced to the market in 1972 by Fisher.<sup>4</sup> Significant improvements in the properties of these blends were achieved in 1978 by Coran and coworkers<sup>5,6</sup> by fully vulcanizing the rubber phase under dynamic shear while maintaining the thermoplasticity of the blends. These blends were further improved by Abdou-Sabet and Fath<sup>7</sup> in 1982 by the use of phenolic resins as curatives. A series of extensive studies on dynamically vulcanized TPEs were carried out by Coran and Patel in the 1980s.<sup>8–15</sup> Bhowmick and coworkers<sup>16–24</sup> also reported various TPEs and TPVs. Very recently, Naskar and coworkers<sup>25–30</sup> extensively studied the effects of various peroxides, including multifunctional peroxides, as crosslinking agents for polypropylene (PP)/ethylene–propylene–diene rubber (EPDM) TPVs. TPVs are typically characterized by finely dispersed (micrometer-sized) crosslinked rubber particles distributed in a continuous thermoplastic matrix. Generally, the rubber particle size varies in the range of 0.5–2  $\mu\text{m}$ .

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TABLE I  
Properties of Engage-8440 (Ethylene–Octene Grade)

Density (g/cm <sup>3</sup> )	Melt index (°/min) <sup>a</sup>	Mooney viscosity (ML <sub>1+4</sub> ) <sup>b</sup>	Total crystallinity (%)	Hardness (Shore A)	DSC melting peak (°C) <sup>c</sup>
0.897	1.6	16	27	92	94

<sup>a</sup> At 190°C and 2.16 kg.

<sup>b</sup> At 121°C.

<sup>c</sup> The rate was 10°C/min.

A literature survey has indicated that there is enough opportunity to generate new materials through the blending of existing polymers, especially to improve properties, to cover up the deficiency of one polymer by another and also to reduce cost. This survey also reveals that there has been growing interest in the use of TPVs or dynamic vulcanizates in the last couple of decades. Currently, TPVs are the fastest growing elastomer market with an annual growth rate of about 15%. Commercialized dynamic vulcanizates are commonly based on blends of EPDM and PP and to a lesser extent on combinations of natural rubber, butyl rubber, or nitrile rubber with PP or polyethylene.

Silicone rubbers are notable for their following general characteristics: very wide service temperature range (−100 to +250°C); excellent resistance to attack by oxygen, ozone, and sunlight; excellent nonstick and nonadhesive properties; low toxicity; optical transparency; good electrical insulation properties; and low chemical reactivity. However, silicone rubber has few limitations. Possible limitations include the low tensile strength of vulcanized rubbers, poor hydrocarbon oil and solvent resistance, high gas permeability, and somewhat high cost.

Only limited research has been pursued so far in the field of silicone-rubber-based TPVs. There is hardly any openly published literature available in this field because of commercial sensitivity. A few research works in a related field have been patented by Dow Corning Corp.<sup>31–33</sup> Potential areas of application of silicone-based TPVs could be the wire and cable industries and soft-touch appliances.

In 1996, Dow Chemical first commercialized a polymer (ethylene–octene copolymer) known as Engage. The mechanical behavior of Engage spans

from the medium hardness of an elastomer to the high hardness range of polyolefins, thereby bridging the gap between rubber and plastics. Engage can be used as a thermoset as well as a thermoplastic polymer. Several grades are available in the market. In this particular work, thermoplastic-grade Engage has been chosen as the thermoplastic component of TPVs because it has the following special characteristics: excellent physical properties including toughness and low-temperature ductility, excellent impact resistance, excellent processability, and good mechanical properties. Despite the different chemical natures of silicone rubber and Engage, the polymers have very similar solubility parameter values of approximately 7–8 (cal/cm<sup>3</sup>)<sup>1/2</sup>, and so using these two polymers as the blend constituents without the addition of any compatibilizer is expected to lead to very good TPVs.

The main objective of this work is an extensive study of the effects of peroxide as a crosslinking agent in silicone/Engage TPVs at a fixed blend ratio on the mechanical and electrical properties.

## EXPERIMENTAL

### Materials

Silicone rubber [polydimethylsiloxane (PDMS)], supplied by GE Silicones (India), had a specific gravity of 0.9 g/cc and a Mooney viscosity (ML<sub>1+4</sub>) at 100°C of 45. Engage-8440 (ethylene–octene grade) was supplied by DuPont Dow Elastomers (USA). Table I<sup>34</sup> shows various properties of Engage, which has a crystallinity of only 27%.

Dicumyl peroxide (DCP; 98%) was used as the crosslinking agent, and triallyl cyanurate (TAC; 50%) was used as the coagent (booster for peroxide).

TABLE II  
TPV Compositions (phr) at Different DCP Concentrations at a Fixed PDMS/Engage Blend Ratio

Component	E <sub>0</sub>	E <sub>1</sub>	E <sub>2</sub>	E <sub>3</sub>	E <sub>4</sub>	E <sub>5</sub>	E <sub>6</sub>	E <sub>7</sub>
PDMS	100	100	100	100	100	100	100	100
Engage-8440	50	50	50	50	50	50	50	50
DCP (98%)	0	0.5	1.0	1.5	2.0	2.5	3.0	4.0
TAC (50%)	0	2.0	2.0	2.0	2.0	2.0	2.0	2.0

**TABLE III**  
**Mechanical and Electrical Properties of the Different Blends**

Sample	Tensile strength (MPa)	Elongation at break (%)	Young's modulus (MPa)	Modulus (MPa)			Hardness		Tension set at 25°C (%)	Volume resistivity $\times 10^{-13}$ ( $\Omega$ cm)
				100%	200%	300%	Shore A	Shore D		
E <sub>0</sub>	3.0	533	8.0	1.4	1.7	2.1	40	9	19	1.2
E <sub>1</sub>	3.2	473	8.5	1.6	1.9	2.3	43	10	16	1.5
E <sub>2</sub>	4.0	560	8.7	1.7	2.0	2.5	50	11	14	1.8
E <sub>3</sub>	4.1	583	8.8	1.7	2.0	2.4	50	11	12	2.2
E <sub>4</sub>	4.2	530	9.1	1.8	2.0	2.5	50	11	11	2.5
E <sub>5</sub>	4.5	570	10.0	1.9	2.2	2.7	50	11	10	2.9
E <sub>6</sub>	4.2	512	10.0	2.2	2.7	3.3	53	12	8	5.8
E <sub>7</sub>	3.3	512	9.8	1.6	2.0	2.4	50	12	8	6.1

### Preparation of TPVs

The TPV compositions employed for this work are shown in Table II. The experimental variable was the level of the peroxide curing agent. All TPVs were prepared by a batch process in a Brabender PLE 330 plasticorder (Germany) with a mixing chamber volume of 70 cc. The mixer temperature was kept at 120–130°C with a constant rotor (cam-type) speed of 80 rpm. Engage and silicone rubber (PDMS) were first mechanically melt-mixed. After 6 min of mixing, the coagent (TAC) was added and was followed by DCP. The mixing was continued for another 4 min to complete the dynamic vulcanization process in the Brabender plasticorder. Immediately after mixing, the molten mass was passed through a cold two-roll mill to achieve a sheet about 2 mm thick. The sheet was cut and pressed (2 mm thick) for 4 min in a compression-molding machine (Moore Press) (United Kingdom) at 130°C. Teflon sheets were placed between the molded sheet and the press plates. The sheet was then cooled at room temperature under pressure. Test specimens were die-cut from the compression-molded sheet and used for testing after 24 h at room temperature.

### Testing procedures

Tensile tests were carried out according to ASTM D 412-98 on dumbbell-shaped specimens with a Zwick 1445 tensile testing machine (Germany) at a constant

crosshead speed of 500 mm/min. Young's modulus was determined from the initial slope of the stress-strain curve. The hardness of the samples was measured with a durometer (Shore A and Shore D per ASTM D 2240). A tension set test was carried out at room temperature after stretching of the samples for 10 min at 100% elongation according to ASTM D 412-98.

The curing characteristics of only silicone rubber (without Engage) containing different concentrations of DCP were determined with a Monsanto R100S rheometer (USA), which is an oscillating disc rheometer (ODR), at 130°C for 30 min.

An aging test was carried out with the samples kept in an aging oven at 70°C for 72 h. This aging condition was chosen because the TPVs contained Engage as one of the blend constituents, which has a melting temperature of only 94°C.

Recyclability tests were carried out by placement of the residual molded TPV samples again in the Brabender plasticorder at 120–130°C followed by compression molding of the samples at 130°C and subsequent testing.

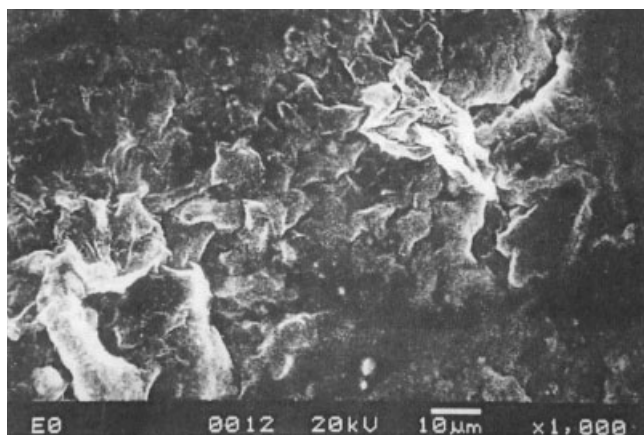
The direct-current volume resistivity was measured with an Agilent 4339B high-resistance meter and 16008B resistivity cell (USA).

The phase morphology of the various blends was investigated with a JEOL JSM 5800 digital scanning electron microscope (Japan). The sample surface was sputter-coated with gold and then examined under the scanning electron microscope. The images were

**TABLE IV**  
**Formulations of the Samples Used for the ODR Experiments (phr) at 130°C and Corresponding Data**

Sample	PDMS	DCP (98%)	TAC (50%)	Minimum ML (dN m)	Maximum MH (dN m)	Delta torque (dN m)	T <sub>90</sub> (min)
R-1	100	1.0	2.0	5.00	71.00	66.0	21
R-2	100	1.5	2.0	4.50	73.00	68.5	17
R-3	100	2.0	2.0	5.00	77.00	72.0	21
R-4	100	2.5	2.0	7.50	83.00	75.5	20
R-5	100	3.0	2.0	7.50	88.00	80.5	13
R-6	100	4.0	2.0	6.00	82.00	76.0	9

MH is Maximum torque and ML is Minimum torque.

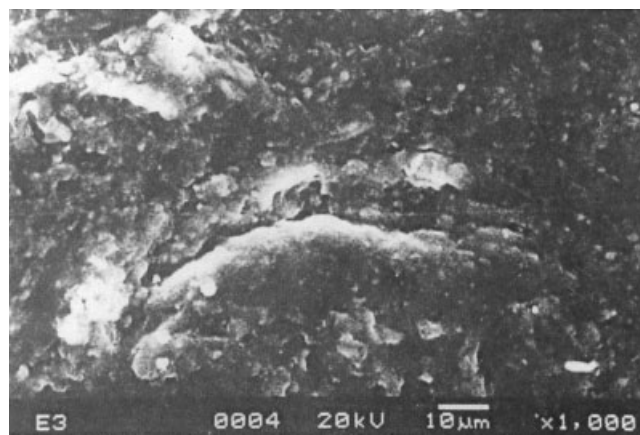


**Figure 1** SEM photomicrograph of the PDMS/Engage TPV in the absence of DCP.

obtained at a tilt angle of  $0^\circ$  with an operating voltage of 20 kV.

### RESULTS

Table III shows various mechanical properties of PDMS–Engage TPVs at different DCP concentrations. The table shows that the tensile strength is a function of the concentration of the crosslinking agent. With increasing concentrations of DCP, the tensile strength of TPVs increases first, then reaches a maximum at a particular concentration (2.5 phr), and then decreases. Moreover, with increasing DCP concentrations, Young's modulus and modulus values at 100, 200, and 300% also increase. Hardness values of TPVs are also found to be a function of the peroxide concentration. However, only a small increase in the hardness values can be observed. The average Shore A hardness values vary between 43 and 53. With the concentration of DCP increasing, the volume resistivity values of TPVs increase slightly because of the formation of a more compact network structure. The values also indicate good electrical properties of the silicone-based TPVs, and this is an important characteristic of cable insulating materials.



**Figure 2** SEM photomicrograph of the PDMS/Engage TPV in the presence of DCP (1.5 phr).

To get better insight into the data obtained, it is important to understand the vulcanization characteristics of the DCP/TAC combination in pure silicone gum compounds (without any Engage) from ODR. Table IV shows formulations of the samples for rheograms containing only PDMS, DCP, and TAC and corresponding results.

The delta torque (maximum torque – minimum torque) values, as obtained from the ODR, generally correlate with the crosslinking efficiency of the peroxide, which is defined as the number of moles of chemical crosslinks formed per mole of peroxide.  $T_{90}$  is the time elapsing until 90% of the maximum torque value is achieved; this parameter is important for defining the optimal vulcanization for a compound. It has been observed that delta torque values gradually increase with an increasing concentration of DCP. Optimum cure time ( $T_{90}$ ) values are also affected by the peroxide concentrations.

Figures 1 and 2 show the scanning electron microscopy (SEM) photomicrographs of the  $E_0$  and  $E_3$  samples in the absence and presence of 1.5 phr DCP, respectively. The morphology changes from a cocontinuous phase to a dispersed phase (where PDMS is dispersed in the continuous matrix of Engage). Simi-

**TABLE V**  
Changes in the Modulus, Tensile Strength, and Elongation at Break After Aging (72 h at  $70^\circ\text{C}$ )

Sample	Modulus (MPa)						Tensile strength (MPa)			Elongation at break (%)		
	100%			300%			Before aging	After aging	Change (%)	Before aging	After aging	Change (%)
	Before aging	After aging	Change (%)	Before aging	After aging	Change (%)						
$E_0$	1.4	1.6	+14.3	2.1	2.3	+9.5	3.0	2.6	-13.3	533	506	-5.1
$E_1$	1.6	1.6	0	2.3	2.2	-4.3	3.2	2.8	-12.5	473	475	+0.4
$E_2$	1.7	1.8	+5.9	2.5	2.5	0	4.0	3.7	-7.5	560	509	-9.1
$E_3$	1.7	1.8	+5.9	2.4	2.3	-4.2	4.1	3.1	-24.4	583	506	-13.2
$E_4$	1.8	1.8	0	2.5	2.4	-4.0	4.0	3.2	-20.0	530	509	-5.1
$E_5$	1.9	1.9	0	2.7	2.6	-3.0	4.5	4.0	-11.1	570	540	-5.3
$E_6$	2.2	2.3	+4.5	3.3	3.3	0	4.2	4.1	-2.4	512	476	-7.0
$E_7$	1.6	1.8	+12.5	2.4	2.5	+4.2	4.3	3.3	-23.2	512	500	-2.3

lar photomicrographs of TPVs have been obtained at various other DCP concentrations and are therefore not shown here.

Table V shows various mechanical properties of TPVs before and after aging. However, no linear behavior for the parameters has been observed in a comparison of the influence of DCP before and after aging. Most of the TPVs show very good retention of properties even after aging, and this indicates good resistance to heat. Peroxide curing provides strong C—C linkages as crosslinks, and this is reflected in the better aging behavior.

Table VI shows the data for the recycling test, which shows very good recyclability of the silicone-rubber-based TPV samples because of very good retention of all the mechanical properties after recycling.

DISCUSSION

There are many plausible explanations for the changes in the physical properties of the silicone-based TPVs related to the variations in the concentrations of DCP. In principle, the crosslinking process of high polymers by DCP can be divided into three successive steps.<sup>35–37</sup> The first step is the homolytic decomposition of DCP and generation of cumyloxy free radicals. This step is the rate-determining step of the overall reactions. These cumyloxy radicals can further undergo β scission to produce highly reactive methyl radicals and acetophenone. The second step is the abstraction of hydrogen atoms from the silicone polymer, resulting in stable peroxide decomposition products such as methane, acetophenone, and 2-phenyl propanol-2 and silicone polymeric radicals. The final step consists of the combination of two such silicone polymeric radicals to produce a stable C—C crosslink, which has very high bond strength. The general crosslinking mechanism of a peroxide is shown in Figure 3. In general, the higher the DCP concentration is, the higher the amount is of generated reactive methyl radicals and hence the higher the extent is of crosslinking in the silicone rubber phase. Increases in the tensile strength, modulus, and hardness of TPVs with increasing concentrations of peroxide can thus be explained by the higher extent of crosslinking in the PDMS phase. It should be noted, however, that there is a possibility that DCP could also take part in crosslinking the Engage phase to some extent because of its chemical structure. However, crosslinking in the PDMS phase is predominating here and is mainly controlling the final phase morphology and consequently the properties of the blends. In addition, during the process of dynamic vulcanization of silicone rubber and ethylene–octene copolymer in the presence of DCP/TAC, there is a possibil-

TABLE VI  
Mechanical Properties Before and After Recycling

Sample	Tensile strength (MPa)			Elongation at break (%)			Modulus (MPa)								
	Before recycling	After recycling	Change (%)	Before recycling	After recycling	Change (%)	100%		200%		300%				
							Before recycling	After recycling	Change (%)	Before recycling	After recycling	Change (%)	Before recycling	After recycling	Change (%)
E <sub>0</sub>	3.0	3.0	0	533	500	-6.2	1.4	1.5	+7.1	1.7	2.0	+17.6	2.1	2.2	+4.8
E <sub>1</sub>	3.2	3.2	0	473	498	+3.2	1.6	1.8	+12.5	1.9	2.1	+10.5	2.3	2.4	+4.3
E <sub>2</sub>	4.0	3.5	-12.5	560	465	+0.9	1.7	2.1	+23.5	2.0	2.4	+20.0	2.5	2.8	+12.0
E <sub>3</sub>	4.1	3.9	-4.9	583	550	-4.8	1.7	2.1	+23.5	2.0	2.3	+15.0	2.4	2.7	+12.5
E <sub>4</sub>	4.0	4.4	+10.0	530	570	+7.0	1.8	2.3	+27.7	2.0	2.6	+30.0	2.5	3.0	+20.0
E <sub>5</sub>	4.5	3.1	-31.1	570	500	-12.3	1.9	2.2	+15.8	2.2	2.3	+4.5	2.7	2.5	-7.4
E <sub>6</sub>	4.2	2.9	-33.3	512	440	-14.0	2.2	1.7	-22.7	2.7	2.1	-22.2	3.3	2.2	-33.3
E <sub>7</sub>	4.3	3.0	-30.2	512	505	-1.4	1.6	1.5	-6.3	2.0	1.9	-5.0	2.4	2.5	+4.2

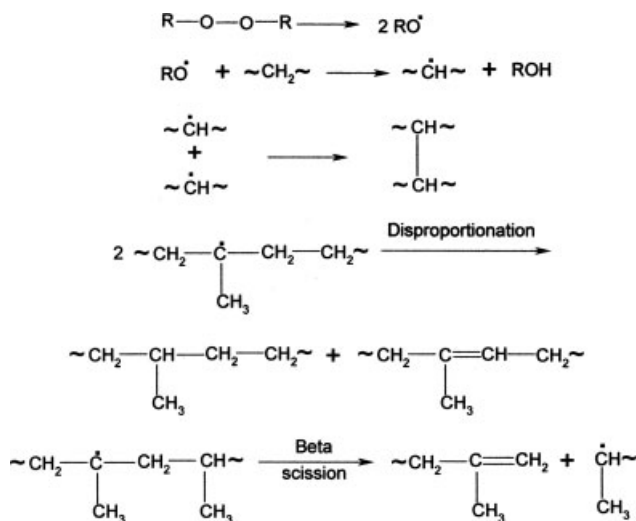


Figure 3 General crosslinking mechanism of a peroxide.

ity of generation of *in situ* graft links of silicone rubber and ethylene-octene copolymer at the interface (which is very difficult to prove by any analytical technique because of the extremely small amount of grafts being generated), which can in turn enhance the compatibility between the two phases and also can improve the final mechanical properties of the TPVs. Furthermore, in the presence of DCP, especially at a higher concentration level, the crystallinity of Engage might change (as observed in a few differential scanning calorimetry experiments: 27, 24, and 22% at DCP concentrations of 0, 2.5, and 3.0 phr, respectively), and this is reflected in the deterioration of physical properties of the TPVs beyond a concentration of 2.5 phr.

The main objective of the dynamic vulcanization process is to generate the TPV typical phase morphology. In binary systems, generally the major component forms the matrix phase, and the minor com-

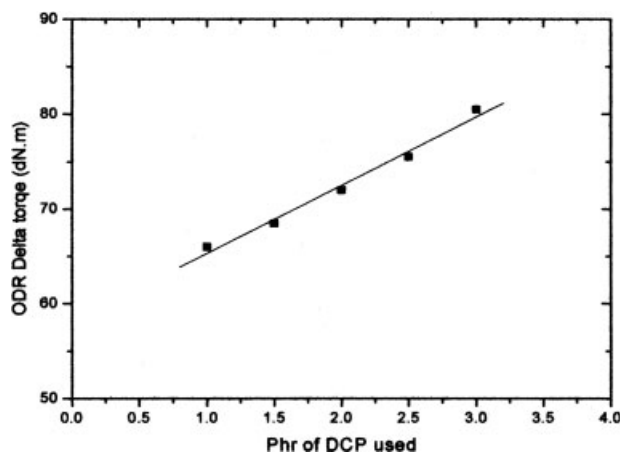


Figure 4 Relation between the ODR delta torque and DCP concentration.

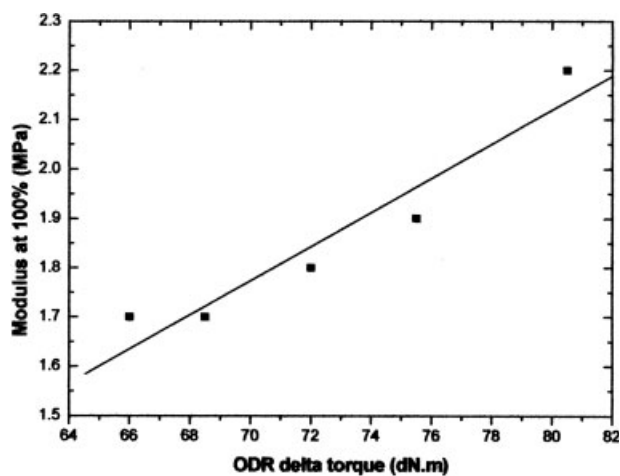


Figure 5 Modulus of TPV at 100% elongation as a function of the ODR delta torque.

ponent forms a dropletlike dispersed phase. However, in the case of dynamic vulcanization, it has been well established in various reports that a cocontinuous phase morphology may exist before the addition of a crosslinking agent because of a rubber to thermoplastic viscosity ratio of  $\approx 1$ .<sup>1-3</sup> Immediately after the start of the selective crosslinking process, the viscosity of the rubber becomes very high, and with this, the stresses in the material increase very strongly, leading to the breakup of the cocontinuous rubber phase into small dispersed particles, as observed from the SEM photomicrograph.

The delta torque (maximum torque – minimum torque) values obtained from ODR generally correlate with the crosslinking efficiency of the peroxide, which is defined as the number of moles of chemical crosslinks formed per mole of peroxide. It should be noted, however, that the latter could be measured by

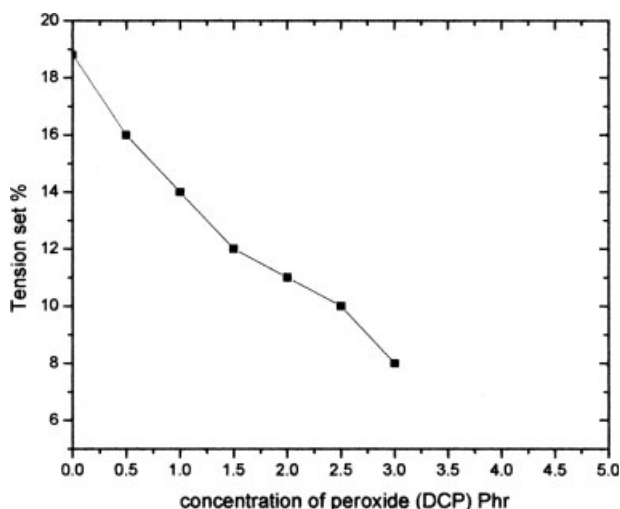


Figure 6 Tension set of TPV as a function of the DCP concentration.

static vulcanization only in the absence of Engage; which is not exactly a one-to-one comparison to dynamic vulcanization, because of the lack of a high shear rate and the longer timescales. Excellent correlations exist between ODR delta torque values and various physical properties, namely, the modulus at 100% and tension set of TPVs as a function of the concentration of DCP (as mentioned in Tables III and IV), which are shown in Figures 4–6.

It has also been observed that the modulus at 100% of TPVs increases linearly with the ODR delta torque obtained at different DCP concentrations. On the other hand, the tension set (%) decreases with increasing DCP concentrations, indicating better elastic recovery of the TPVs.

### CONCLUSIONS

Novel TPVs based on blends of silicone rubber and the thermoplastic Engage have been developed. These TPVs exhibit very good overall mechanical and electrical properties. With an increasing concentration of DCP, the tensile strength, modulus, and hardness of TPVs increase and then reach their maximum. However, optimum physical properties have been found at the DCP concentration of 2.5 phr. Important correlations have been obtained from the difference of the maximum and minimum torque values with various physical properties of TPVs, namely, the tensile strength, modulus, tension set, and crosslinking density. The aging characteristics and recyclability of silicone-based TPVs have also been found to be excellent. SEM photomicrographs of the TPVs confirm the dispersed phase morphology.

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