# Electron-Beam Irradiation of Poly(vinyl chloride)/Epoxidized Natural Rubber Blends in Presence of Trimethylolpropane Triacrylate

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**ABSTRACT:** Electron-beam initiated crosslinking of poly(vinyl chloride)/epoxidized natural rubber blends, which contained trimethylolpropane triacrylate (TMPTA), was carried out over a range of irradiation doses (20–200 kGy) and concentrations of TMPTA (1–5 phr). The gel content increased with the irradiation dose and the TMPTA level, although the increase was marginal at higher doses and higher TMPTA levels. Blends containing 3–4 phr TMPTA achieved optimum crosslinking, which in effect caused the maximum tensile strength (TS) at a dose of 70 kGy. A further addition of TMPTA caused a decline in the TS above 40 kGy that was due to embrittlement, which is a consequence of excessive crosslinking and the breakdown of the network structure. The possible formation of a more open network as a result of the breakdown of the network structure was further confirmed by the modulus results. Dynamic mechanical analysis (tan $\delta$ curve) and scanning electron microscopy studies on samples irradiated at 0 and 200 kGy were undertaken in order to gain further evidence on the irradiation-induced crosslinking. The plasticizing effect of TMPTA prior to irradiation and the formation of microgels upon irradiation were also discussed. © 2001 John Wiley & Sons, Inc. J Appl Polym Sci 81: 1926–1935, 2001

**Key words:** irradiation; dose; level; crosslinking; trimethylolpropane triacrylate

## INTRODUCTION

High energy irradiation ($\gamma$ and electron beam) is a well-known technique for the modification of polymers.[1–4] Several factors have contributed to the growth of the commercial irradiation processing industry over the past 40 years. These include the availability of powerful electron accelerators and large $\gamma$ irradiation facilities; an improved understanding of the irradiation chemistry underlying irradiation polymerization, scission, and crosslinking; and the ability to process materials at ambient temperatures and fast processing rates.[5–10] Electron-beam processing uses high energy electrons from an accelerator to initiate polymerization and crosslinking reactions in suitable matrices, thus enhancing their specific physical and chemical properties.[11] There have been extensive studies concerning the irradiation of pure polymers. We reported the irradiation processing of polymers and some of our findings on surgical rubber gloves,[12] poly(vinyl chloride) (PVC),[13] polypropylene,[14,15] and hydrogel coatings.[16,17]

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However, there is little fundamental work in the scientific literature on the effects of irradiation on polymer blends. Blends of PVC and epoxidized natural rubber (ENR), which were found to be miscible, attracted interest in the field of polymer blends.[18–22] Recent work by Ratnam and Zaman[23,24] showed the potential for modification of PVC/ENR blend properties by electron-beam irradiation. Detailed studies on the stabilization of PVC/ENR blends demonstrated that the addition of tribasic lead sulfate[25] improved the blend properties upon irradiation. The effects of several types of antioxidants on the irradiation crosslinking of PVC/ENR blends was also reported.[26] The influence of the functionality of acrylates, such as trimethylolpropane triacrylate (TMPTA), 1,6-hexanediol diacrylate, and 2-ethylhexyl acrylate, on the efficiency of irradiation-induced crosslinking of PVC/ENR blends indicated that TMPTA could be useful as an efficient crosslink enhancer.[27] However, there was no attempt to study the effect of TMPTA loading on the enhancement of PVC/ENR blend properties upon irradiation. In this article the electron-beam initiated modification of a PVC/ENR blend in the presence of TMPTA is investigated. The influence of the level of TMPTA and the dose of electron-beam irradiation on the crosslinking, tensile, thermal, and morphological properties of a 50/50 PVC/ENR blend is described by using the blend without TMPTA as the control.

## EXPERIMENTAL

### Materials

The ENR (Epoxyprene 50) with a 50% epoxidation level was supplied by Guthrie Polymer Ltd. as a free sample. The PVC ($K = 66$; $M_n = 66{,}000$; grade MH66, 6519) was purchased from Industrial Resin (M) Ltd.

The tribasic lead sulfate (TS-100M) used as the PVC stabilizer was purchased from Lonover Scientific Suppliers Ltd. (London). The TMPTA was a product of UCB Asia Pacific. These compounds were used as received.

### Formulations

The 50/50 PVC/ENR blends were prepared by mixing 50 parts of PVC with 50 parts of ENR50. The recipes are given in Table I.

**Table I Recipes of PVC/ENR Blends**

| Material | Formulation (phr) |
|---|---|
| PVC | 50 |
| ENR | 50 |
| Tribasic lead sulfate | 2 |
| TMPTA | 0–5 |

### Blend Preparations

The PVC and the stabilizer were premixed at room temperature in a tabletop high-speed mixer at 1200 rpm for 10 min. Melt blending was carried out at 150°C with a 50 rpm rotor speed in a Brabender Plasticorder (model PL 2000) with a mixing cam attachment. When the desired temperature was reached, the ENR was charged into the mixing chamber and mixed for 1 min. The PVC compound was then added, and the blending was continued for a further 9 min.

The blends obtained from the Brabender Plasticorder were then compression molded into 1 mm thick sheets under a pressure of 14.7 MPa at 150°C for 3 min. The sheets were immediately cooled between two plates of a cold press at 25°C. Dumbbell-shaped test pieces were cut from these sheets in accordance with BS6746.

### Irradiation

The molded sheets and dumbbell test pieces were irradiated using a 3-MeV electron-beam accelerator at a dose range of 0–200 kGy. The acceleration energy, beam current, and dose rate were 2 MeV, 2 mA, and 10 kGy/pass, respectively.

### Gel Fraction

The gel fraction was determined by extraction in tetrahydrofuran (THF) at 50 ± 2°C. The blends were solvent extracted with THF for 24 h, and the extracted samples were dried to constant weight. The gel fraction was calculated as

$$\text{gel fraction} = (W/W_0) \times 100 \quad (1)$$

where $W$ and $W_0$ are the weight of the dried sample after extraction and the weight of the sample before extraction, respectively.

### Measurement of Tensile Properties

The tensile strength (TS) and modulus at 100% elongation (M100) were measured with a Toyoseiki Strograph-RI using a crosshead speed of 50 mm/min. Altogether, eight samples were used for

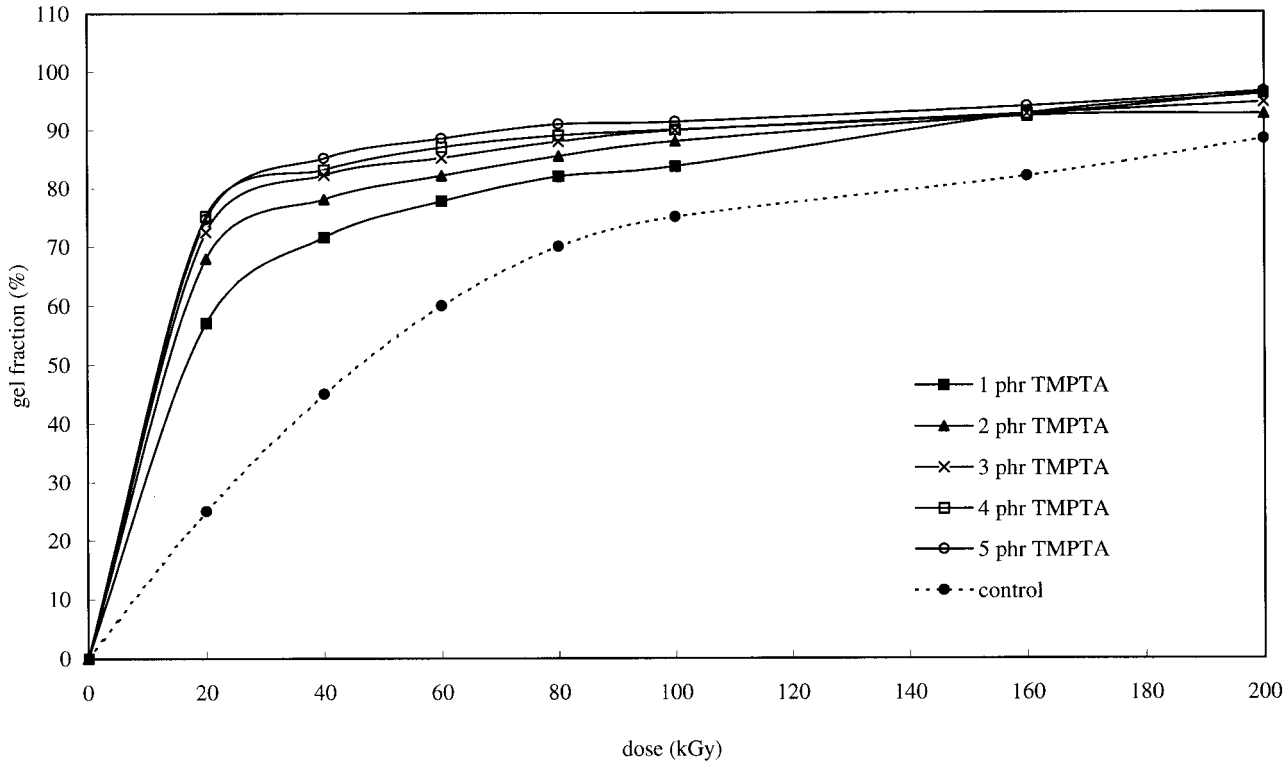



**Figure 1** The effect of TMPTA on the gel fraction of the PVC/ENR blend at various irradiation doses.

the tensile test and an average of six results was taken as the resultant value.

### Hardness

The Shore A hardness test was carried out according to ASTM D2240-89 using the Zwick 7206 hardness tester.

### Dynamic Mechanical Analysis

Dynamic mechanical analysis was performed using a Perkin–Elmer DMA-7e in the temperature–time scan mode with a parallel plate attachment. The measurements were carried out at a heating rate of 10°C/min over a temperature range of −50 to 120°C and a frequency of 1 Hz.

### Scanning Electron Microscopy (SEM)

Selected samples were manually fractured at liquid-nitrogen temperature. The fractured surface was then sputter coated with gold and examined using a Philips 515 scanning electron microscope.

## RESULTS AND DISCUSSION

Generally, the extent of irradiation-induced crosslinking of polymers can be estimated from gel fraction determination.[28] Thus, the gel fractions of the blends were determined to elucidate the irradiation-induced crosslinking. The results are plotted in Figure 1. Figure 1 shows that the crosslinking efficiency increased with the increase in TMPTA level. Such an observation was expected because the acrylate employed in this study is well known as a reactive additive that forms crosslink bridges by an irradiation-induced free-radical mechanism.[29,30] However, further increases in the dose above 40 kGy have less effect in the presence of TMPTA. A similar observation was also noted for the control (0 phr TMPTA) above 80 kGy. Comparing the gel fraction achieved at 40 kGy made it obvious that the rate of increase was relatively high up to 3 phr TMPTA. The increase was marginal after 3 phr TMPTA. These observations could be attributed to the reduced chain mobility due to the formation of a 3-dimensional network structure upon irradiation. As the TMPTA concentration was increased, the amount of gel formed also increased as a consequence of the faster rate of the 3-dimensional network formation, causing restricted mobility at the latter stage. Because of the bulky structure of the TMPTA molecule, which is shown

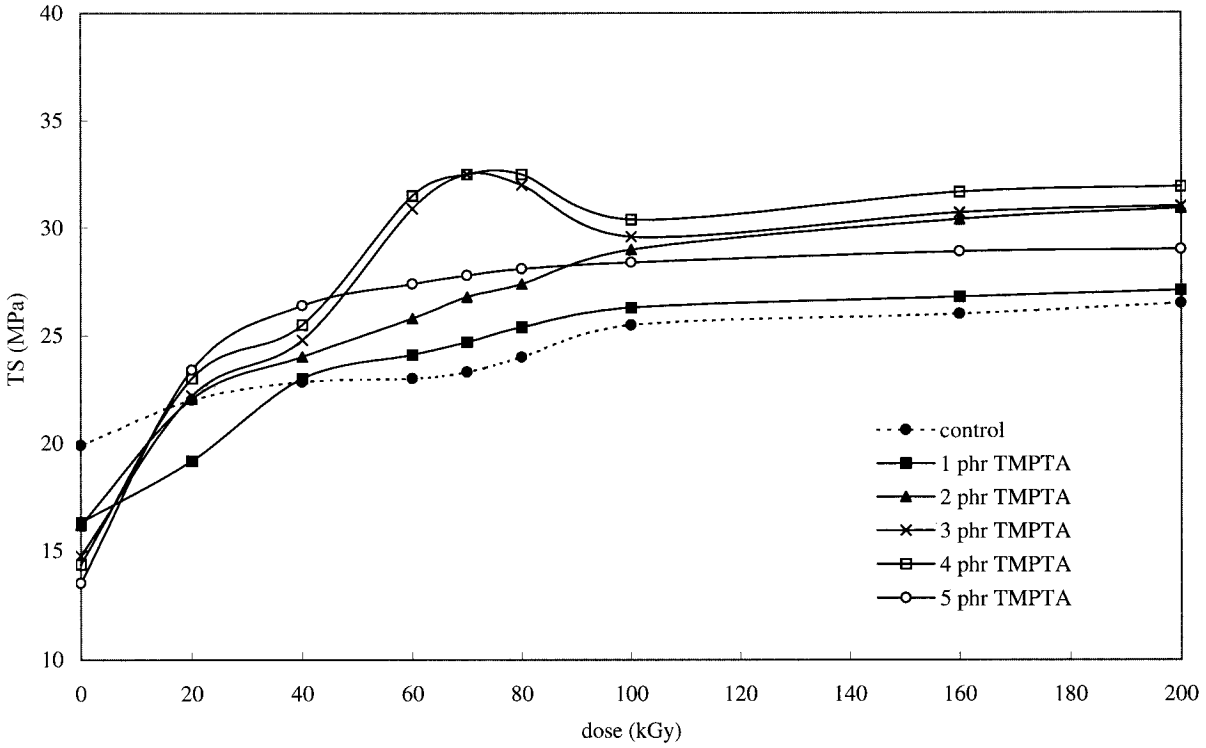
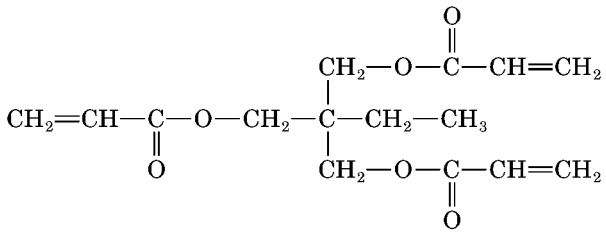



**Figure 2** The effect of TMPTA on the tensile strength of the PVC/ENR blend at various irradiation doses.

in the structure below, it is likely that these became sterically hindered.



This observation is in perfect agreement with Bowner et al.[31] who showed that the crosslinking rate in PVC, especially during the early stages of irradiation, was proportional to the monomer concentration. The breakdown of the excess crosslinks formed, which may occur simultaneously with crosslinking at higher irradiation doses, could also account for the above trend. This aspect is further demonstrated along with tensile and hardness results.

### Effects of Irradiation Dose on TS

The effects of the irradiation dose on the TS of the blends in the presence of TMPTA are shown in Figure 2. At 0 kGy the blends showed a decline in the TS with an increasing level of TMPTA. This was attributed to the plasticizing effect of the monomer. The effect of such additives, which may function as plasticizers, is well described by Czvikovszky.[30] Upon irradiation up to 40 kGy the TS was gradually increased with the increase in TMPTA loading. This trend of results was expected because the TMPTA accelerates irradiation-induced crosslinking, as observed from gel fraction data. However, above 40 kGy the blends containing 3 and 4 phr TMPTA reached an optimum TS at 70 kGy. Beyond this point, subsequent irradiation yields reduced the TS. This was believed to be associated with the embrittlement of the blends, which was due to a high extent of crosslinking. Similar observations were also noted by Thomas et al.[32] while studying $\gamma$ irradiated blends of PVC and thermoplastic copolyesters. Similarly, beyond 40-kGy irradiation the addition of TMPTA above 4 phr caused a reduction in the TS. This suggested that beyond 4 phr TMPTA the network structure breaks into a smaller network (microgel) with irradiation above 40 kGy; thus, the TS drops. Sujit et al.[33] also reported a similar trend in electron-beam

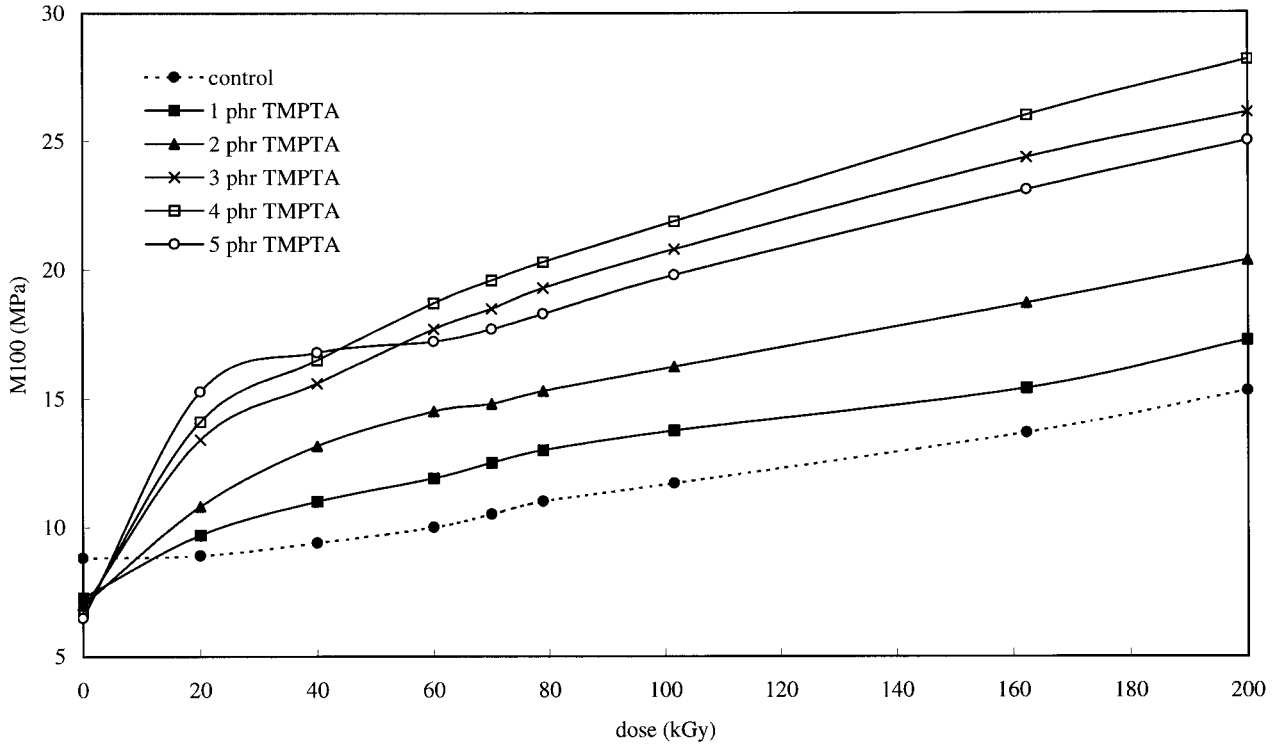



**Figure 3** The effect of TMPTA on the modulus at 100% elongation of the PVC/ENR blend at various irradiation doses.

crosslinking of ethylene vinyl acetate using trimethylolpropane trimethacrylate, and they arrived at the same conclusion. It may be therefore appropriate to infer that the fall in the TS with irradiation of highly crosslinked blends is associated with embrittlement that is attributable to excessive crosslinks and formation of a smaller network.

## Modulus

The effect of TMPTA loading on the modulus of the blend with increasing irradiation dose is illustrated in Figure 3. The drop in M100 with the addition of TMPTA at a 0-kGy irrradiation dose further supported our previous observation in which the TMPTA acted as a plasticizer. Upon irradiation the M100 increased with the level of TMPTA up to 4 phr. This was expected because the TMPTA accelerated the irradiation-induced crosslinking. The lower M100 observed with the addition of 5 phr TMPTA beyond a 40-kGy irradiation dose was in good agreement with the results on the TS. The modulus directly depends on the number of closed loops in the network, which is a perfect network (a network with more chain ends).[34] The chain ends in the network, which may be formed upon irradiation and retained in the gel, would not affect the measured gel fraction but would reduce the modulus. Thus, the lower modulus observed with the addition of 5 phr TMPTA implied that a more open network structure resulted with irradiation above 40 kGy. Therefore, the modulus confirmed the breakdown of the network structure explained in previous sections.

## Elongation at Break

Plots of the elongation at break versus the irradiation dose for the blends with increasing TMPTA content are shown in Figure 4. Generally, increasing the irradiation dose and TMPTA content caused a substantial reduction in the elongation at the break of the blend. Such a decline was expected because the rubber became increasingly brittle as a consequence of the increase in crosslink density with irradiation and TMPTA level. Therefore, the reduction in the elongation at break could be attributed to the reduced segmental mobility of the rubber chains, which resulted from the formation of 3-dimensional net-

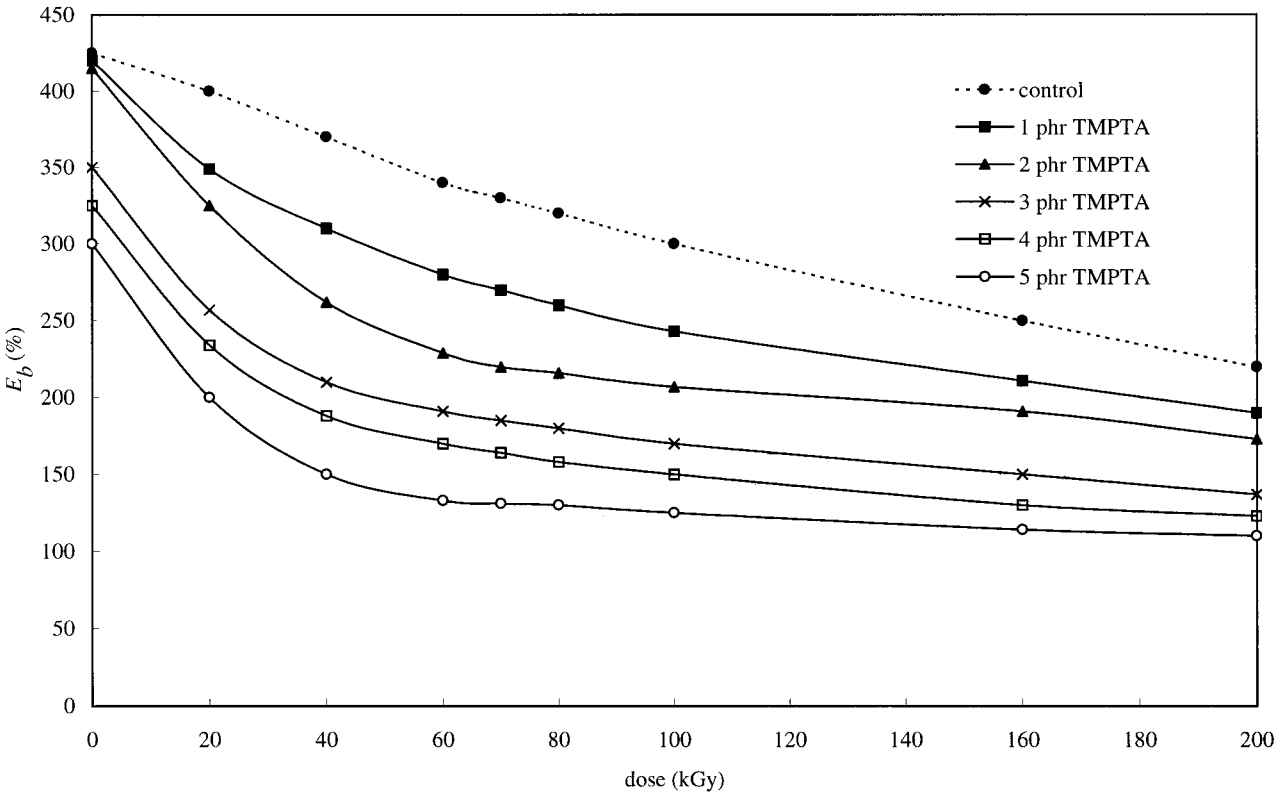



**Figure 4** The effect of TMPTA on the elongation at break ($E_b$) of the PVC/ENR blend at various irradiation doses.

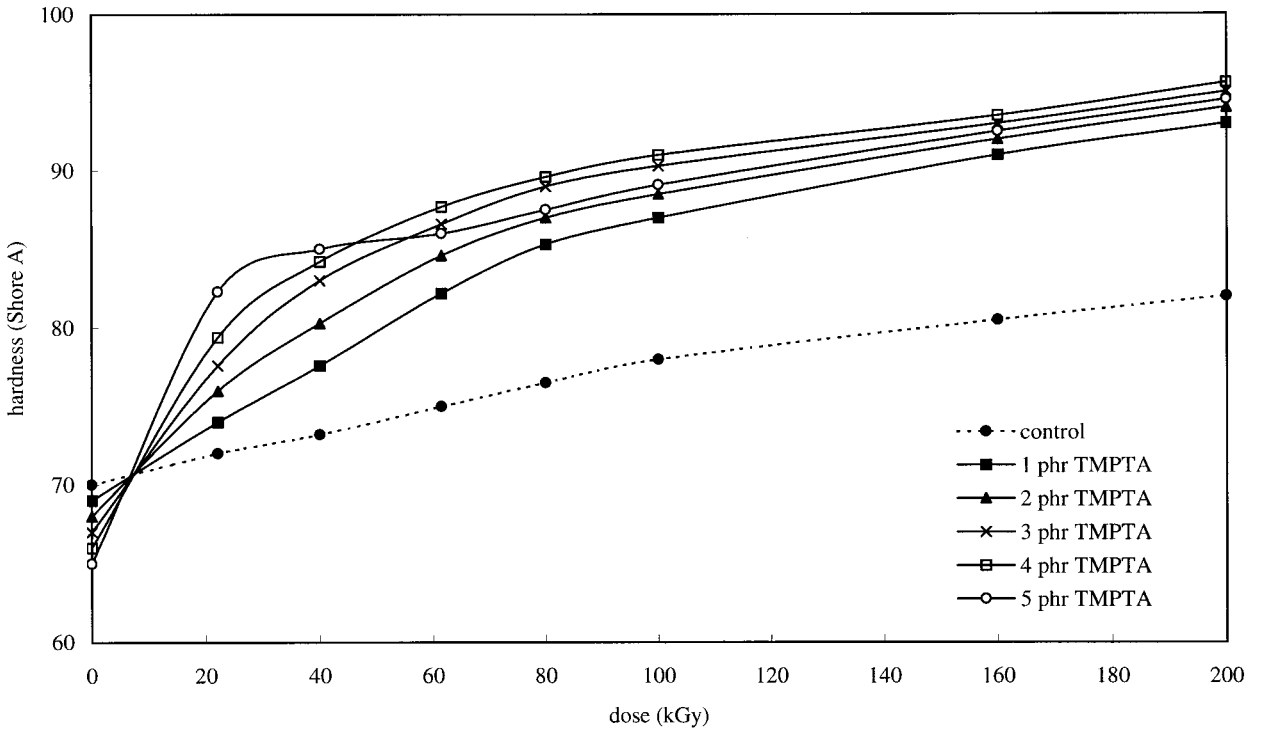



**Figure 5** The effect of TMPTA on the hardness of the PVC/ENR blend at various irradiation doses.

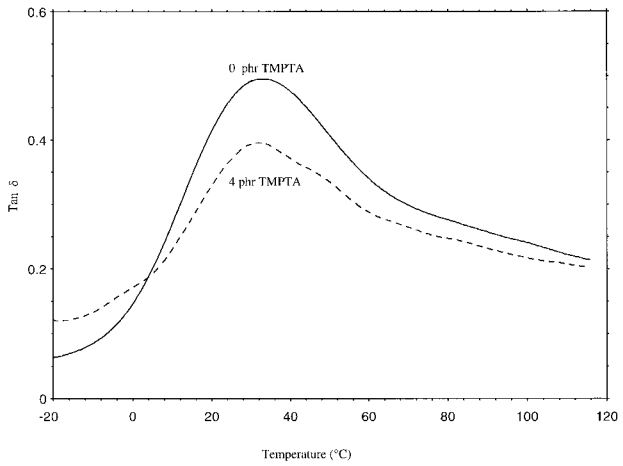
works. However, the plots at 0 kGy in Figure 4 show the addition of more than 2 phr TMPTA caused a considerable reduction in the elongation at break. The decrease could be due to the high plasticization effect, because the concentration of PVC was much less than 50%. A similar decrease was also observed in 50/50 PVC/ENR blends with the addition of epoxidized soya oil and di-2-ethylhexylphthalate.[35] It is also apparent from Figure 4 that the sample containing 5 phr TMPTA did not exhibit a similar trend, as observed for other properties studied. It showed a continuous decline, despite its change in network structure due to the breakdown of the network. Again such a decline was expected because the elongation at break invariably decreased, regardless of whether crosslinking or scission was predominant.[24]

## Hardness

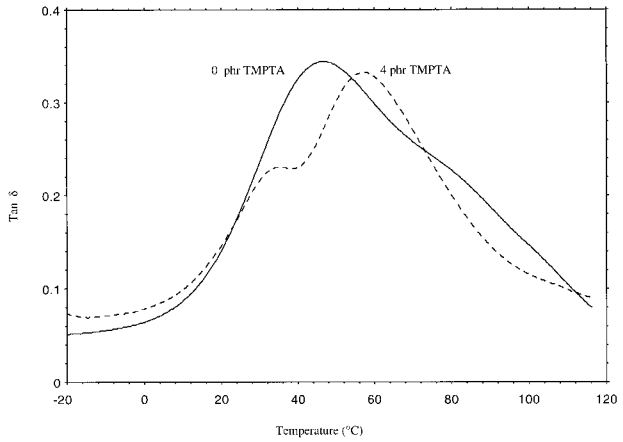
The variation in the hardness of the blends with the irradiation dose upon the addition of TMPTA is shown in Figure 5. The hardness exhibited a trend similar to the modulus. The irradiation-induced crosslinking in polymers is often reflected by the hardness of the irradiated sample.[36] Thus, the hardness further confirmed the observations on the modulus.

## Glass-Transition Temperatures

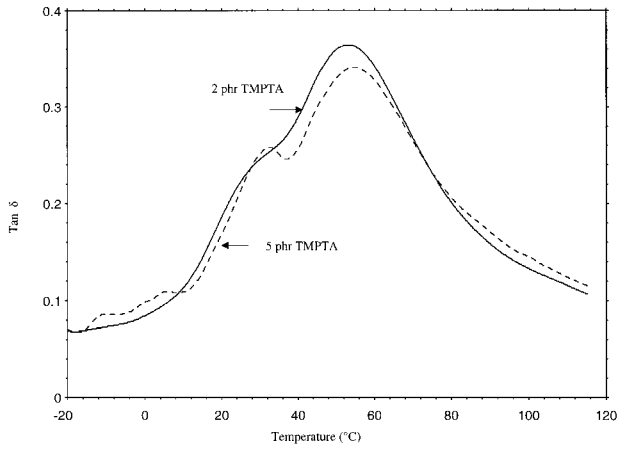
The tan δ curves of unirradiated samples and samples irradiated at 200 kGy are shown in Figure 6(a,b), respectively. Here the glass-transition temperatures ($T_g$) values were taken as the temperatures that correspond to the tan δ peak because they coincide with the $T_g$ values determined by other methods, for example, free volume studies.[37] Only the control (0 phr TMPTA) and the blend with 4 phr TMPTA were chosen for comparison purposes. The presence of a single tan δ peak in the dynamic mechanical analysis results is an indication of a homogeneous or good dispersion.[38,39] Numerous findings of a single $T_g$ for PVC/ENR blends were reported.[22,40–42] Figure 6(a) shows a single peak, implying the blends are miscible and homogenous. The reduction in $T_g$ with the addition of 4 phr TMPTA confirmed the plasticizing effect of the additives prior to irradiation. Similar effects on the dynamic mechanical properties are observed with plasticizers.[43] On comparing Figure 6(a,b) it is apparent that the tan δ peak shifts to higher temperature with irradiation. Such shifting was believed to be attrib-




(a)




(b)




(c)

**Figure 6** The effect of TMPTA on the temperature dependence of the tan δ on the PVC/ENR blend: (a) unirradiated (0 and 4 phr TMPTA), (b) irradiated at 200 kGy (0 and 4 phr TMPTA), and (c) irradiated at 200 kGy (2 and 5 phr TMPTA).

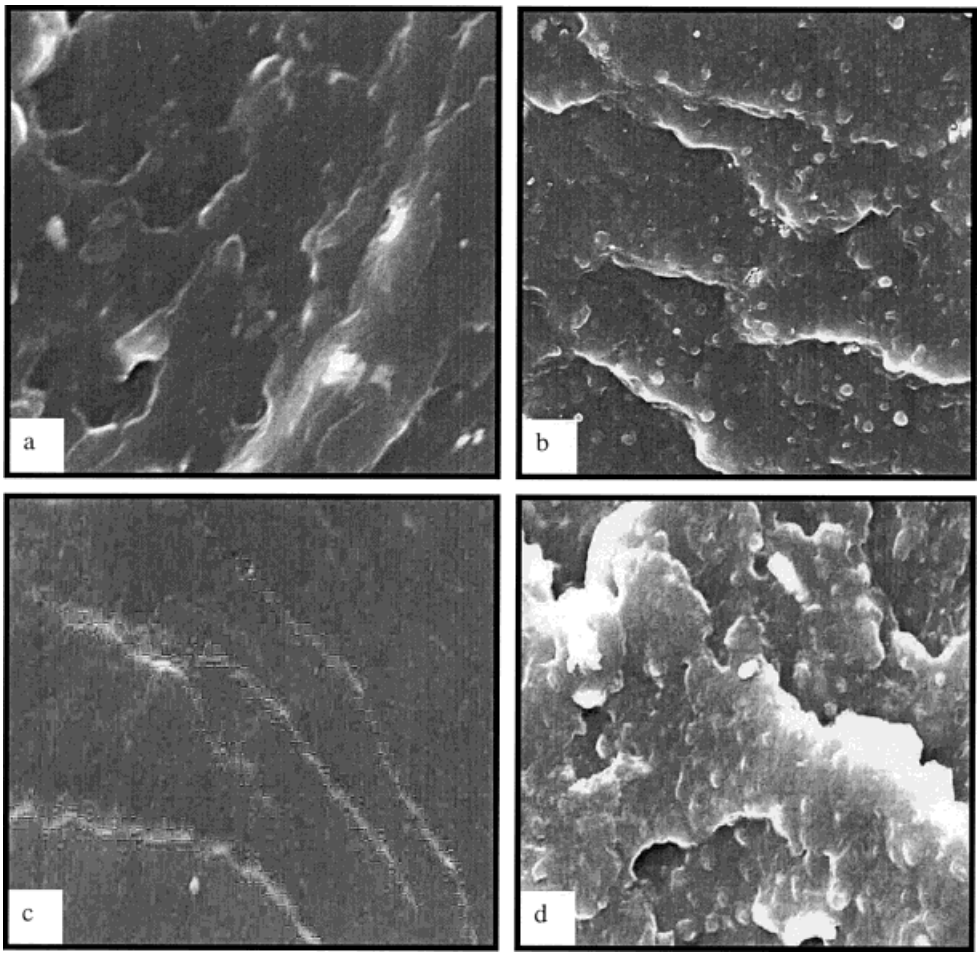



**Figure 7** SEM micrographs of cryogenically fracture surfaces of PVC/ENR blends; original magnification ×2000. (a) Control, unirradiated; (b) control, irradiated at 200 kGy; (c) 4 phr TMPTA, unirradiated; and (d) 4 phr TMPTA, irradiated at 200 kGy.

uted to the irradiation-induced crosslinking, because crosslinks would result in a reduction in free volume of the chain segments. The acceleration of crosslinking by the addition of the TMPTA is evident from the higher $T_g$ exhibited in Figure 6(b) in the presence of TMPTA.

Another noteworthy point was the presence of a shoulder below the $T_g$ upon irradiation of the blend in the presence of TMPTA. This could indicate the existence of microheterogeneity due to different segmental environments. In the homogenous polymers the shape of the dispersion does not change, and only the position of the dispersion shifts with irradiation. The formation of a microgel as a consequence of the breakdown of the network structure upon irradiation of the blend with the 4 phr TMPTA could account for the observed microheterogeneity. The obvious development of this shoulder at 5 phr TMPTA compared to the blend with 2 phr TMPTA, as shown in Figure 6(c), provided further evidence of the effect of TMPTA concentration on the microgel formation. From another perspective, such a shoulder may be associated with motion of the side groups,[44] implying that grafting of TMPTA could also have occurred upon irradiation of the blends in the presence of TMPTA. However, regardless of the type of network formed, the role of TMPTA in the acceleration of crosslinking and enhancement in the mechanical properties and the $T_g$ was apparent.

## SEM Studies

The photomicrographs in Figure 7 show the morphological development occurring as a consequence of irradiation for the control and the blend containing 4 phr TMPTA. From Figure 7(a) it is

obvious that the fracture surface of the blend in the absence of TMPTA showed discontinuous fracture paths over the entire surface. A more deformed surface with cavities is also shown in Figure 7(a). On the other hand, the surface of the unirradiated blend in the presence of TMPTA exhibited more continuous and longer cracks with no cavities [Fig. 7(c)]. According to Cornes and Howard,[45] formation of cavities is one characteristic of ductile failure. Thus, it is evident that the addition of TMPTA imparted a plasticizing effect to the blends. This correlates well with the lower mechanical properties and $T_g$ observed with the increase in TMPTA content prior to irradiation.

The fracture surfaces of the irradiated samples [Fig. 7(b,d)] appeared to be rougher and more brittle compared to the unirradiated surfaces [Fig. 7(a,c)]. Close examination of the irradiated blend surfaces showed the presence of small droplets, confirming the existence of microgels. Finally, the fracture surfaces of the irradiated sample in the presence of TMPTA [Fig. 7(d)] showed more irregularities at the edges of the cracks and more crack branching (fracture planes located at different heights). The development of these fracture patterns was attributed to the presence of excessive crosslinks and formation of microgels upon irradiation. The microgels arrested the propagation of the growing crack.[46] Thus, high energy was expended for the crack propagation. The existence of such a microgel region of high crosslink density was also noted by Morgan et al.[47] while working on crosslinking of epoxy resin in the presence of several crosslinking agents.

The surface of the irradiated control showed more regular cracks. Crack branching was completely absent, implying a lower degree of crosslinking to resist the crack propagation. This was reflected by the lower strength of the blend achieved upon irradiation with the absence of TMPTA.

## CONCLUSIONS

Our studies revealed that the addition of TMPTA can provide excellent enhancement in irradiation-induced crosslinking in PVC/ENR blends. The TS results proved that the optimum dose is achieved with the addition of 3 to 4 phr TMPTA. The decline in TS and M100 with the addition of 5 phr TMPTA (above 40 kGy) implied the formation of a more open network at higher TMPTA levels. The enhancement in brittleness due to a high extent of crosslinking upon the addition of TMPTA was evident from the shifting in the $T_g$ and SEM examination. The formation of microgels upon irradiation and the further tendency of the blend to exhibit heterogeneity with the addition of TMPTA was also discussed.

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