

# Novolac Resin–Poly(ethyl methacrylate) Interpenetrating Polymer Networks: Morphology and Mechanical and Thermal Properties

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**ABSTRACT:** Full interpenetrating networks (IPNs) and semi-IPNs of Novolac (phenolic) resin and poly(ethyl methacrylate) (PEMA) were prepared by the sequential mode of synthesis. These were characterized with respect to their mechanical properties, that is, ultimate tensile strength (UTS), percentage elongation at break, modulus, and toughness. Thermal properties were studied by DSC and thermogravimetric analysis (TGA). The morphological features were studied through polarizing light microscopy (PLM). The effects of variation of the blend ratios on the above-mentioned properties were examined. There was a gradual decrease of modulus and UTS with consequent increases in elongation at break and toughness for both types of IPNs with increasing proportions of PEMA. An inward shift and lowering (with respect to pure phenolic resin) of the glass-transition temperatures of the IPNs with increasing propor-

tions of PEMA were observed, thus indicating a plasticizing influence of PEMA on the rigid and brittle matrix of crosslinked phenolic resin. The TGA thermograms exhibit two-step degradation patterns. Although there was an apparent increase in thermal stability at the initial stages, particularly at lower temperatures, a substantial decrease in thermal stability was observed in the regions of higher temperatures. The surface morphology as revealed by PLM clearly indicates two-phase structures in all the full and semi-IPNs, irrespective of PEMA content. The matrix–PEMA domain interfaces are quite sharp at higher concentrations of PEMA. © 2003 Wiley Periodicals, Inc. *J Appl Polym Sci* 90: 412–420, 2003

**Key words:** interpenetrating networks (IPNs); crosslinking; glass transition; thermogravimetric analysis (TGA); blends

## INTRODUCTION

Polymer blends encompass many different kinds of materials containing two or more polymer components and most exhibit phase separation to a greater or lesser degree. Interpenetrating polymer networks (IPNs) are a new class of polymer blends in network form, in which the possibility of phase separation has been arrested to a great extent by suitably engineering the morphologies of the participating components. Here, one polymer is synthesized or crosslinked in the immediate presence of the other and they are at least partially interlaced on a molecular scale but not covalently bonded to each other and cannot be separated unless chemical bonds are broken.<sup>1–7</sup> When both networks are crosslinked, the morphology is fixed and well defined, and the associated properties do not vary much. The properties are dependent on the two-phase morphology that develops during polymerization/crosslinking processes of either phase. The morphology is dependent on crosslink density and the

sequence of formation of the two networks.<sup>8–10</sup> In the sequential technique, the continuous network dictates the properties, whereas the simultaneous process results in a material with the smallest degree of phase separation, and hence the highest degree of interpenetration.<sup>11</sup> The IPNs, regardless of how they are formed, can offer a wide spectrum of properties ranging from toughened elastomers to high-impact plastics by the judicious selection of the participating polymers. The degree of network interlocking between the components has a measurable effect on various mechanical properties, such as modulus, hardness, and ultimate tensile strength (UTS), which in turn depend on whether either component of the networks is crosslinked (semi-IPN) or whether both of them are crosslinked.<sup>12–15</sup> Increased network interlocking is expected to improve compatibility.

Phenol–formaldehyde is a widely known rigid, brittle thermosetting resin. Various toughening agents are incorporated into phenolics to reduce the brittleness.<sup>16–19</sup> The present study focused on improving the properties of the phenolic resin by blending another softer thermoplastic, poly(ethyl methacrylate) (PEMA), by the IPN technique. The rubber-toughening influence of the plastomeric PEMA on the continuous matrix of phenolic resin was investigated as a function of PEMA content.

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## EXPERIMENTAL

### Materials

Novolac, the precursor of the crosslinked phenolic thermosetting resin mixed with 10% (of the Novolac resin weight) of hexamethylene tetramine (HEXA), was procured from Hindustan Adhesives (Kolkata, India) and used without further modification. Ethyl methacrylate (EMA) from Fluka (Buchs, Switzerland) was purified by washing first with a 2% aqueous sodium hydroxide (NaOH) solution and then by thorough and repeated washings with distilled water (to make alkali free, as tested by litmus paper) and dried over fused calcium chloride ( $\text{CaCl}_2$ ), after which it was finally vacuum distilled. Benzoyl peroxide ( $\text{Bz}_2\text{O}_2$ ) from B.D.H. India was purified by repeated crystallization from chloroform. 2-Ethyl-2-(hydroxymethyl)-1,3-propanediol trimethacrylate (Aldrich Chemical, Milwaukee, WI), without any modification, was used as comonomer and crosslinker for PEMA.

### IPN synthesis

A weighed amount of purified EMA was placed in a test tube and thoroughly mixed with 2% by weight (based on EMA) of recrystallized  $\text{Bz}_2\text{O}_2$ . The Novolac resin (premixed with HEXA in the proportion required for its complete curing) was weighed in a glass jar to maintain a suitable ratio with the EMA as weighed earlier. The contents of the test tube were then poured into the jar and mixed thoroughly and uniformly until the mixture turned almost to a paste, although with an increase in EMA content the consistency of the paste became diluted. The resulting mass was then allowed to mature for about 2 h. The paste was then transferred into a positive type compression-sheet mold, which was preheated to 80°C. The mold was then closed and placed on the lower platen of the hydraulic press. The press was then closed with a mild pressure to keep the mold airtight and to ensure that no air was entrapped into the sheet. This condition was maintained for 0.5 h to allow the acrylic polymerization to initiate and propagate to a certain extent. Once the stipulated time period for the acrylic polymer formation was over, the temperature of the mold was increased to 150°C. The pressure was then increased to 5 tons/cm<sup>2</sup> and the mold was kept under such conditions for another 0.5 h, which ensured complete crosslinking of the phenolic resin and complete polymerization of EMA as well. The mold was then removed from the press in hot condition and opened cautiously so that there was no distortion and warpage of the sheet. Samples for testing were cut from the sheet after maturing for 7 days. In the case of full IPNs, the comonomer crosslinker was added (2% w/w with respect to the EMA monomer taken) before the addition of Novolac resin. All other steps remained unaltered.

### Measurements

#### Tensile properties

An Instron Universal Testing Machine (Model 4204) was used for measuring the tensile properties, such as UTS, percentage elongation at break (%EB), modulus, and toughness. The ASTM D638 method was followed. A crosshead speed of 5 mm/min was maintained. All testing was conducted at room temperature. The toughness of the IPN samples was determined from the area under the load versus elongation plot. The samples were visually inspected before measurements and were found to be free from pores or nicks. The data reported are averages of at least six measurements and the typical scattering range of the results was  $\pm 5\%$ .

#### Thermal properties

TGA thermograms were obtained on a Perkin-Elmer Delta Series TGA 7 thermogravimetric analyzer (Perkin Elmer Cetus Instruments, Norwalk, CT), under nitrogen atmosphere at a heating rate of 20°C/min. The samples ranging between 6 and 10 mg in weight were used for the TGA. A Du Pont 2100 instrument (Du Pont, Boston, MA) was used for the DSC studies. DSC scans were taken at the heating rate of 10°C/min under a continuous flow of nitrogen.

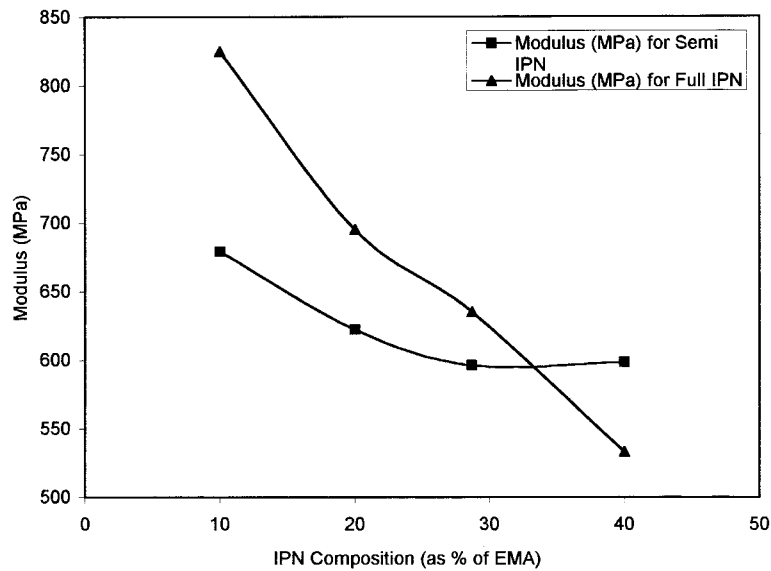
#### Morphology

Phase morphology was examined in a trinocular polarizing light microscope from Krüss (Optronic, Germany), with a magnification of  $\times 40$ . They were about 0.5 mm in thickness.

## RESULTS AND DISCUSSION

### Mechanical behavior

The various mechanical properties of the semi- and full IPNs of the phenol formaldehyde (PF)-PEMA system are compared as a function of blend compositions in Figures 1–5. The relative changes in the mechanical properties of these blends, in relation to the pure participating polymers (i.e., Novolac resin and pure PEMA), are also depicted in the same figures (the broken lines indicating the range not studied in the present work). The inset figures indicate the corresponding changes in the mechanical parameters over the ranges of concentrations under study. The mode of changes in modulus and UTS with variations in PEMA content, as shown in Figures 1 and 2, indicate the plasticizing influence of the plastomeric PEMA on the rigid, hard three-dimensional network of phenolic-matrix irrespective of the types of IPN. However, it is also evident that the full IPNs always have much higher moduli and UTS values compared with those

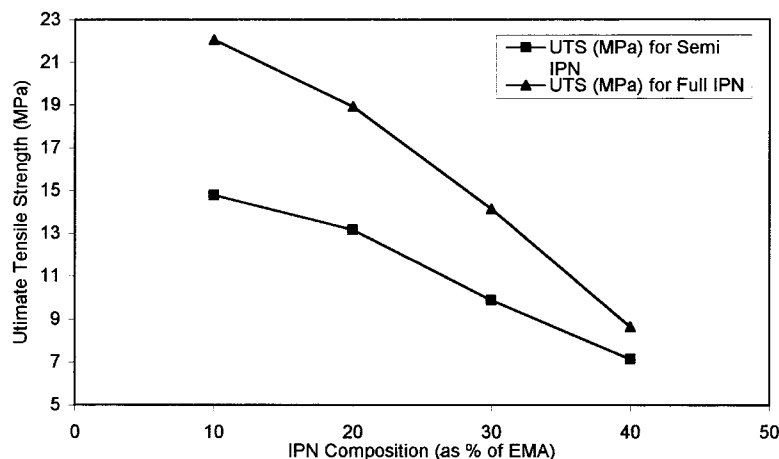


**Figure 1** Variation of modulus with IPN composition (as % EMA) for semi- and full IPN systems.

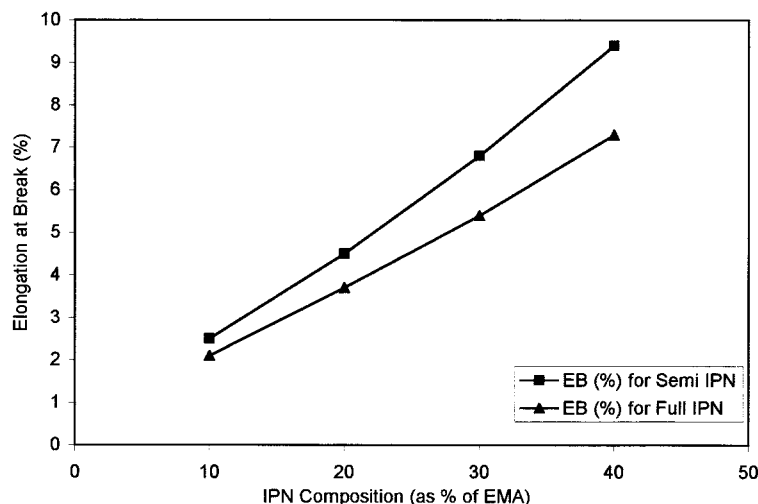
of semi-IPNs over the entire range of concentrations of PEMA studied. It is interesting to note that the marked differences in these two mechanical parameters exhibited by the full and semi-IPNs of identical compositions at the lower ranges of PEMA gradually narrowed down at higher acrylic content. It is observed that both the semi- and full IPNs have undergone tremendous decreases in their corresponding mechanical parameters within only 10% of PEMA incorporation. The decreases, however, are much smaller in the later stages of PEMA incorporation. The decreasing trend of the UTS and moduli may be attributed to the following reasons: (1) the increasing number of relatively weaker stress concentrators offered by the PEMA moieties, and thus gradually decreasing the effective cross-sectional area that bears the load<sup>20</sup>; (2) a reduction in the possibility of complete curing of the phenolic matrix by shielding its reactive sites with the dispersed PEMA domains, and thus leading to a de-

crease in crosslink density, a phenomenon that might have exerted a plasticizing action on the ultimate network system. The influence of crosslinking of the dispersed PEMA domains on these two strength properties is reflected in the mechanical curves of the full IPNs because they always lie above those for the semi-IPNs. Within the level of 10% of PEMA incorporation the influence of crosslinked PEMA in the full IPN is hardly reflected over that in semi-IPNs.

At higher concentrations of PEMA it is expected that the relatively higher free volume of the randomly coiled long polymer chains of PEMA in a semi-IPN enable it to be interpenetrated or threaded more with the subsequently formed crosslinked network of phenolic resin than what would have been possible in a corresponding full IPN, where the tighter crosslinked domains of PEMA restrict the mobility of the chains and reduce the degree of interpenetration. It is presumed that the effect of crosslinking in the dispersed



**Figure 2** Variation of ultimate tensile strength with IPN composition (as % EMA) for semi- and full IPN systems.

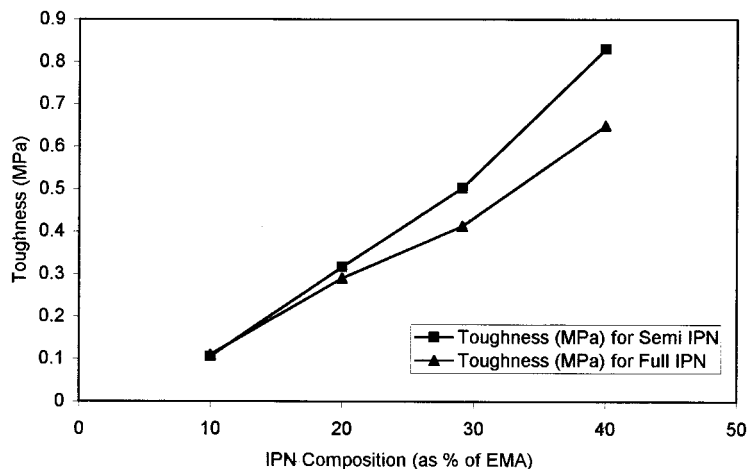


**Figure 3** Variation of percentage elongation at break with IPN composition (as % EMA) for semi- and full IPN systems.

phase in a full IPN is somewhat counterbalanced by the possibility of extensive interpenetration in a semi-IPN. This has narrowed down the differences in the mechanical properties of the full and semi-IPNs at higher levels of PEMA incorporation. The small differences in the mechanical properties in the full and semi-IPNs can be attributed to this interpenetration or threading.<sup>21</sup> Concerning both UTS and modulus it was observed that the semi- and full IPNs exhibit hardly any differences at higher ranges of PEMA incorporation and approach the properties of pure PEMA, given that gross phase separation might be expected to take place in these regions and the properties are dominated by PEMA itself.

As expected, both the %EB and toughness undergo an increase with increasing proportions of PEMA in the semi- and full IPN systems, respectively (shown in Figs. 3 and 4, which also include the corresponding values for the pure polymers). The plasticizing effect imparted by the PEMA domains, along with an increase in path length for the crack propagation attrib-

uted to the presence of a multitude of stress concentrators (PEMA moieties), can be accounted for by the observed increase in the %EB. Consequently, the energy expended in traversing a greater path will be much higher compared to that of pure phenolic resin. Thus the toughness may be expected to increase with increasing proportions of PEMA.<sup>2,20</sup> The influence of crosslinking of the dispersed domains in the full IPN is once more reflected in the mode of changes in %EB and toughness, respectively. The semi-IPNs offer the means for a crack to propagate through the uncrosslinked PEMA domains because it elongates more easily without causing any rupture other than through the restricted elongation before rupture in a full IPN. Thus it may be assumed that more energy is required for causing rupture in a semi-IPN compared to that required in a full IPN. Furthermore, it may be noted from the diagrams that both the %EB and toughness of the different semi-IPNs, particularly at higher levels of PEMA incorporation, appear to increase somewhat disproportionately in relation to those shown at lower



**Figure 4** Variation of toughness with IPN composition (as % EMA) for semi- and full IPN systems.

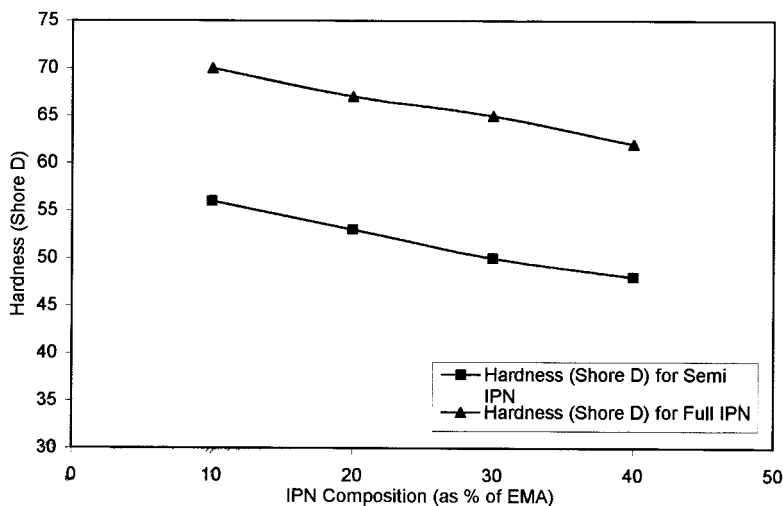


Figure 5 Variation of hardness with IPN composition (as % EMA) for semi- and full IPN systems.

acrylic concentrations. This can possibly be attributed to the fact that, as the content of *in situ* polymerized PEMA increases, its randomly coiled long chains become involved in increasing degrees of interpenetration through labile physical linkages, thus increasing the toughness as the propagating cracks, particularly at the interfaces of the matrix and PEMA domains, expend much higher energy in overcoming the resistances offered by these labile crosslinks. Furthermore it may be assumed that, at still higher ranges of PEMA incorporation, the %EB and toughness values of both the semi- and full IPNs approach the corresponding values of pure PEMA only.

Figure 5, which depicts the comparative studies of the variation of hardness with composition, indicates a slow and gradual decrease in hardness with increasing proportions of PEMA. The hardness values of the full IPNs, as expected, lie above those for the semi-IPNs because of the increased hardness of PEMA domains attributed to crosslinking. However, it is interesting to note that, compared to the other bulk mechanical properties discussed above, the influence of crosslinking of the dispersed PEMA moieties on the surface hardness, even at such a low level of crosslinker concentration, appears to be significant.<sup>2,22-24</sup>

### Thermal properties

#### Thermogravimetric analysis

TGA analyses [Fig. 6(a) and (b)] of both the semi- and full IPN systems appear to exhibit results characteristic of such systems.

The onset of degradation of the different IPN systems appears to remain almost unaffected when compared to that of pure phenolic resin. This holds true in both semi- and full IPNs. However, the onset is marginally accelerated with increasing proportions of PEMA.

Once the temperature reaches 230°C, the IPN system appears to behave in a different manner and both

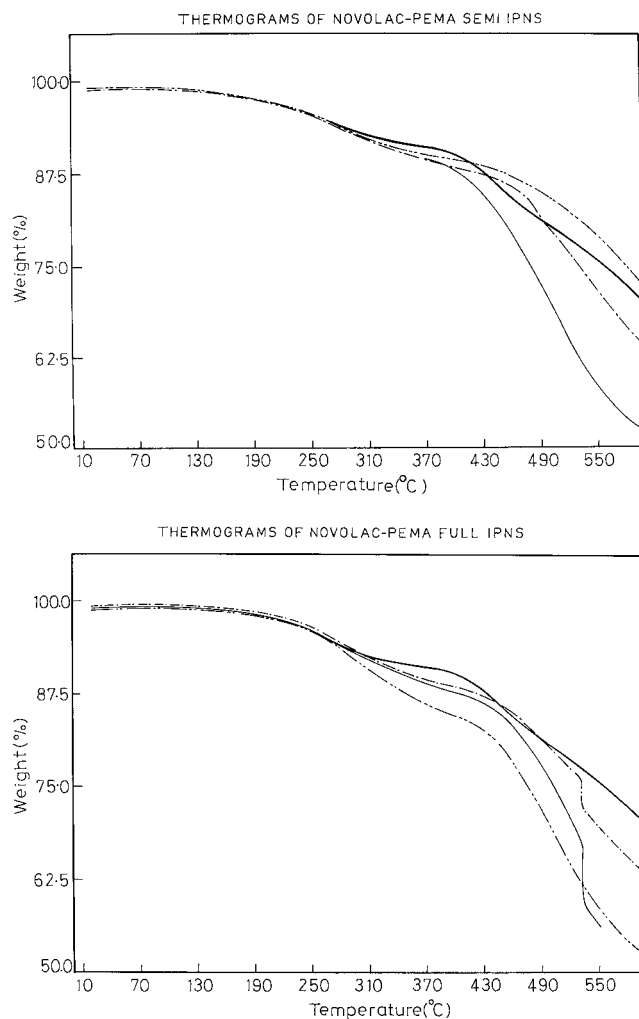
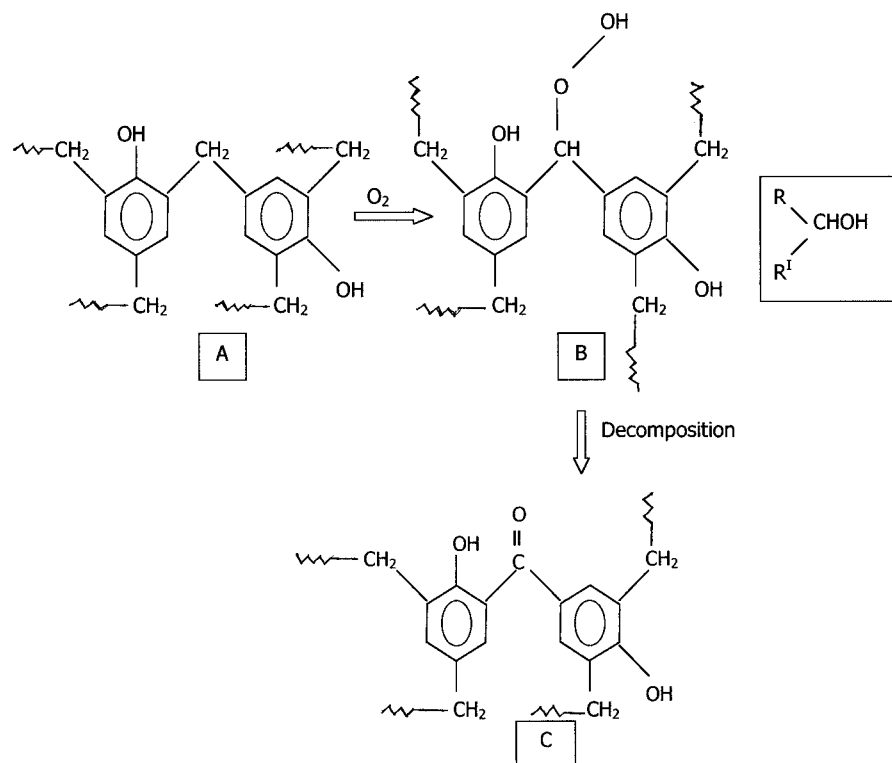


Figure 6 (a) TGA for semi-IPN system: pure PF (—); PF : PEMA, 90 : 10 (— - —); PF : PEMA, 80 : 20 (- - -); PF : PEMA, 60 : 40 (—). (b) TGA plots for full IPN system: pure PF (—); PF : PEMA, 90 : 10 (- - -); PF : PEMA, 80 : 20 (—); PF : PEMA, 60 : 40 (— - -).

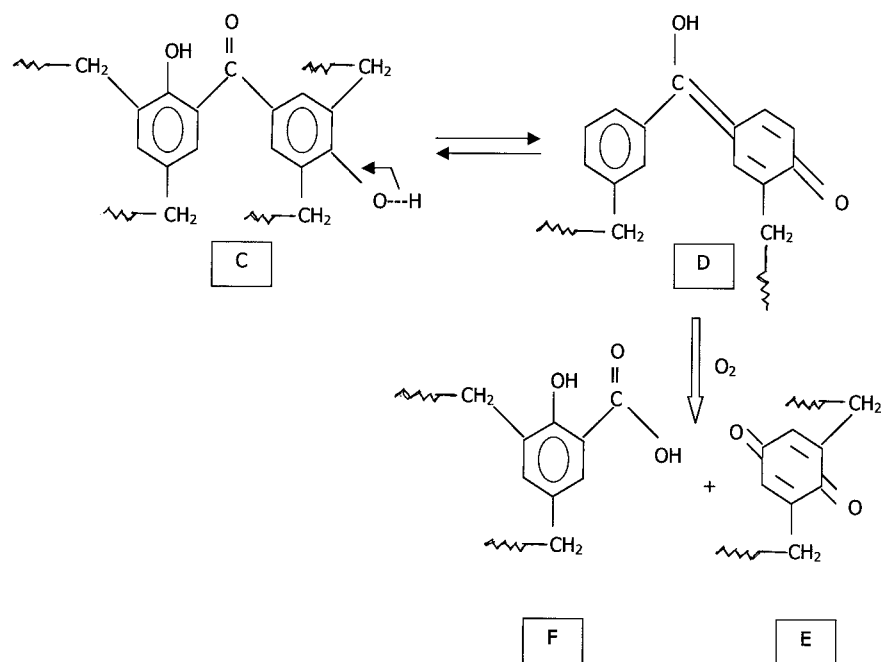


Scheme 1

semi- and full IPN systems exhibit marginally increased stability with respect to the pure phenolic resin. Within the semi-IPNs, the stability was found to undergo an increase with increasing proportions of PEMA within the range of concentrations of PEMA studied between 230 and 270°C. In full IPNs, however,

the thermogravimetric results appear to indicate a marginal decrease as the PEMA content increases.

Beyond 270°C, both semi- and full IPNs degrade at a much faster rate than the pure phenolic and clearly exhibit a decreased thermal stability at elevated temperatures considered in our study. Here again, the



Scheme 2

semi-IPNs appear to be more thermally stable than the full IPNs. Both types of IPN follow a similar pattern of decreasing thermal stability with increasing proportions of PEMA content.

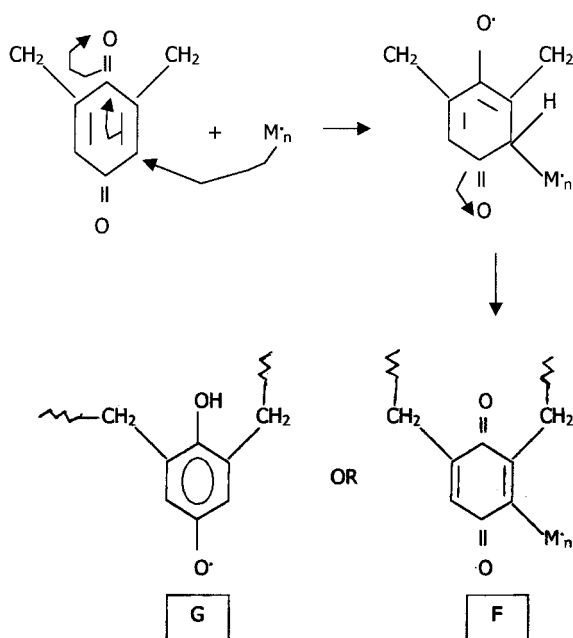
From the lower-temperature studies<sup>25,26</sup> the course of degradation of a phenolic resin was found to be primarily oxidation at the bridging methylene linkages. This initial step in the oxidation route of phenolic decomposition is assumed to take place as shown in **Scheme 1**.

Upon further oxidation it has been reported<sup>27</sup> that sterically hindered phenols are oxidized to quinone-type structures. Oxidative degradation thus proceeds further as shown in **Scheme 2**.<sup>28</sup>

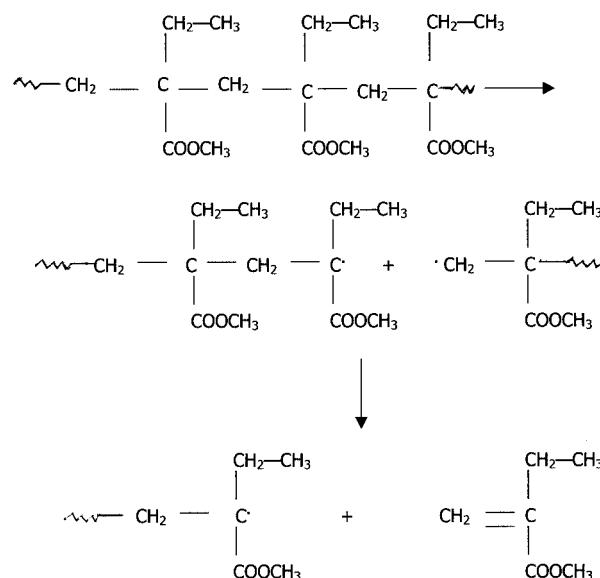
It is quite well known that the poly(alkyl methacrylates) undergo depolymerization by an unzipping reaction mechanism yielding almost 100% monomer. See **Scheme 3**.<sup>29</sup>

Thus it is obvious that the two polymers involved in IPN formation undergo degradation by two different noninterfering mechanisms in a manner similar to their modes of formation, which involve a step growth condensation reaction for the formation of phenolics and fast-chain growth addition polymerization for the formation of PEMA.

Thermal degradation of the phenolic resin, resulting in the formation of quinonoid structure, is a slow process and occurs in a stepwise manner. However, the statistically small number of such structures formed within the temperature region of 230–270°C can inhibit the free-radical-initiated depropagation of PEMA macro radicals by simply scavenging them and thus conferring stability, as shown in the following scheme<sup>30</sup>:

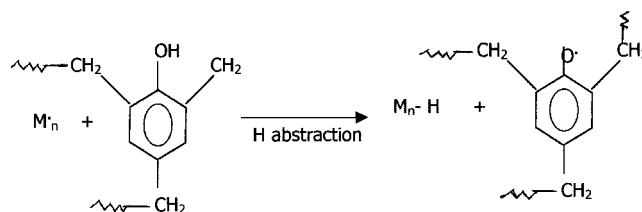


The radical G can further retard the depropagation of PEMA macro radicals by simply coupling with it. This



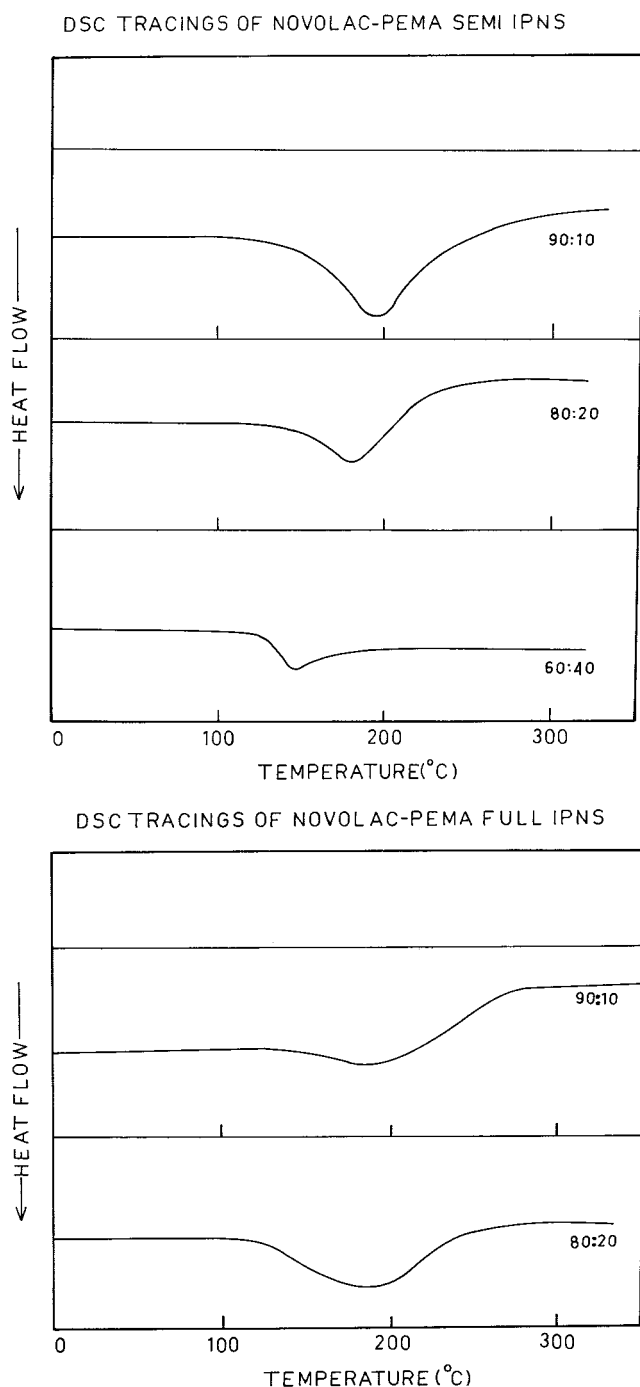
**Scheme 3**

retardation may further be enhanced because of the presence of poly alkyl ring substituted phenols and may be given as follows:



At further elevated temperatures the rate of formation of EMA monomer becomes high as does also the comparatively slower generation of the quinonoid structure (E). The quinonoid species is depleted slowly but steadily from the system, according to the above mechanism (F), and thus the forward degradation of phenolic is increasingly favored, ultimately leading to a faster process of degradation. It may further be postulated that the species (G) formed at intermediate stages may be coupled with products like (F) and the reaction gets faster. The polymer fragment joined to the quinonoid moiety may further depropagate at elevated temperatures, leading to an overall destabilization of the system.

The comparatively higher thermal stability of the semi-IPNs over that of the corresponding full IPNs may be attributed to the effect of interpenetration, which has even offset the effect of covalent crosslinks present in PEMA moieties. It may also be presumed that the crosslinker, besides generating the chemical crosslinks, may become involved in copolymer formation with the EMA monomer and the ultimate copolymer having lower thermal stability than that of the linear homopolymers.



**Figure 7** (a) DSC plots for semi-IPN systems. (b) DSC plots for full IPN systems.

#### DSC studies

The DSC tracings of the representative samples of both semi- and full IPN systems are depicted in Figure 7(a) and (b).

The progressively increasing plasticizing influence of relatively softer PEMA phase domains on the rigid, hard, and brittle continuous matrix of phenolic resin is quite evident, irrespective of the nature of IPN systems (i.e., whether it belongs to semi-IPNs or full IPNs). Full IPNs, however, exhibit higher  $T_g$  values than those

of the corresponding semi-IPNs. This is attributed to the fact that the first formed crosslinked network of PEMA has very limited and restricted mobility compared to that of the linear uncrosslinked PEMA moieties present in semi-IPNs. It is presumed that the increased chain mobility of the linear PEMA molecules is expected to hinder the crosslinking process of the subsequently formed network of phenolic that led to the creation of more free volume/empty holes for the resulting semi-IPN systems and hence the lowering of  $T_g$  values.

The extent of phase mixing gradually decreases with increasing proportions of PEMA. This can be inferred from the nature of the spread in the  $T_g$ . The width of the span over which the endothermic enthalpy change occurs gradually decreases. It is quite interesting to note, however, that full IPNs have a relatively higher extent of phase mixing compared to that of semi-IPNs of almost identical composition. This may possibly be attributable to the presence of comonomer crosslinker of the PEMA that, besides forming intermolecular crosslinks among the PEMA chains, may form some homopolymers that lead to better phase mixing. The DSC traces of the two full IPNs of different compositions under study, however, do not vary much in this regard.

#### Morphology

Polarizing light micrographs of both full and semi-IPNs are exhibited in Figure 8.

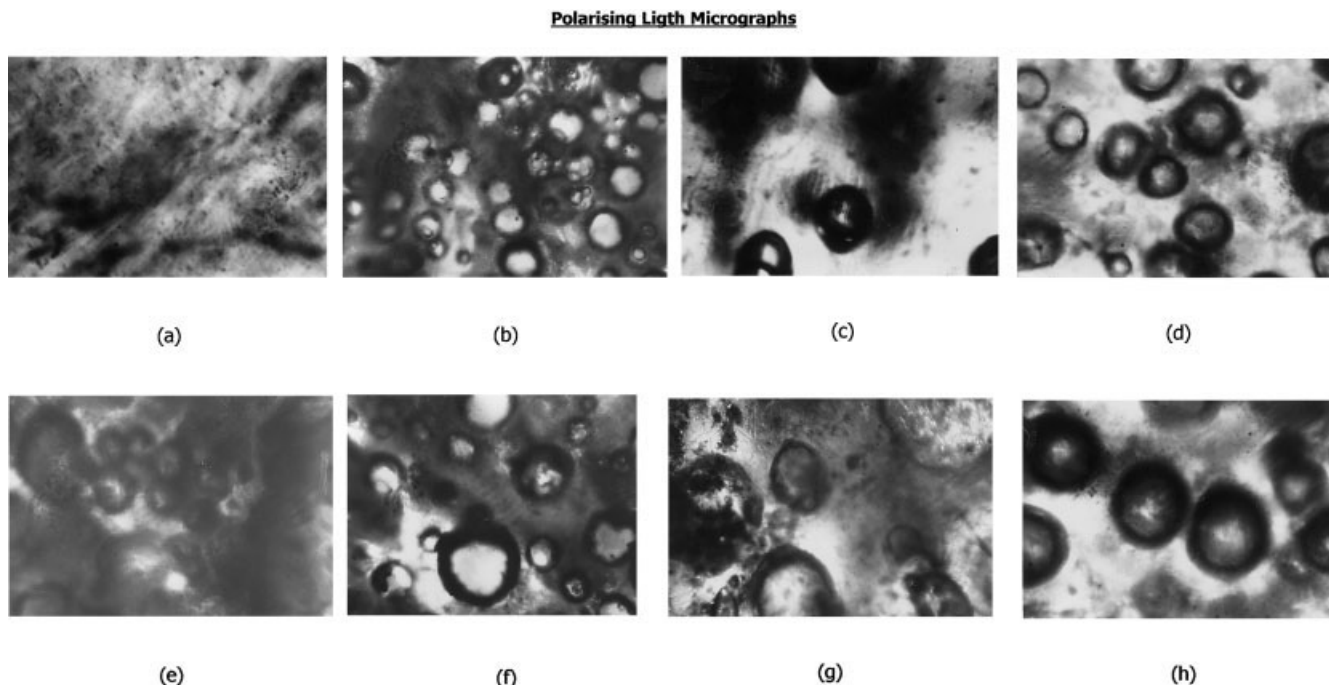
Here the bright PEMA phase is found to be dispersed quite irregularly in a Novolac-rich dark phase.<sup>31</sup> It is also evident that the phase separation starts at an early stage of PEMA incorporation in both cases of semi- and full IPNs, and such development of IPN morphology occurs according to the TTT (time-temperature-transformation) scheme of Kim et al.<sup>32</sup>

In the present case the sequence followed in IPN formation suggests that the curative (hexamethylene tetramine) containing Novolac powder becomes coated with peroxide (initiator) containing EMA monomer (in the semi-IPN) and also with comonomer crosslinker (in the full IPN). The first formed PEMA, along with unreacted monomer, is phase separated and gels, thus forming a fixed domain after which the slow crosslinking of Novolac resin takes place.

At a lower level of EMA concentration the PEMA domains appear to be entrapped in the continuous network of Novolac resin. At higher levels of PEMA incorporation, the PEMA phase appears to exhibit a tendency toward formation of a continuous phase in which the Novolac-rich phase, with a somewhat occluded PEMA domain, appears to be dispersed with some intrinsic connectivities. Thus it appears to develop a bicontinuous phase structure.<sup>33</sup>

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**Figure 8** Polarizing light micrographs for semi- and full IPN systems (magnification  $\times 40$ ): (a) pure PF; (b) PF : PEMA, 90 : 10 (full); (c) PF : PEMA, 80 : 20 (full); (d) PF : PEMA, 70 : 30 (full); (e) PF : PEMA, 90 : 10 (semi); (f) PF : PEMA, 80 : 20 (semi); (g) PF : PEMA, 70 : 30 (semi); (h) PF : PEMA, 60 : 40 (semi).

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