# Studies on Curing and Thermal Behavior of Diglycidyl Ether of Bisphenol-A and Benzoxazine Mixtures

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**ABSTRACT:** This article describes the synthesis and characterization of benzoxazine monomers prepared by reacting the mixture of amine (aniline [A], 3-chloroaniline [C], *o*-toluidine [T]) and phenol (bisphenol-A [B], phenol [P]) with formaldehyde [F]. The benzoxazine monomers prepared by reacting B and F with A, C, and T have been designated as BAF, BCF, and BTF, respectively. Structural characterization of benzoxazines was done using FTIR, ^{1}H NMR, and elemental analysis. The curing behavior of benzoxazine monomers was investigated by differential scanning calorimetry in the presence of diglycidyl ether of bisphenol-A (DGEBA). In all the samples, the molar ratio of benzoxazine monomer:DGEBA, was varied as 1 : 0, 3 : 1, 1 : 1, and 1 : 3. The peak exotherm temperature ($T_p$) was lowest in all the samples having molar ratio of 3 : 1 benzoxazine:DGEBA and highest in samples having molar ratio of 1 : 3. The heat of polymerization ($\Delta H$) was found to be maximum during curing of mixture of DGEBA and benzoxazine monomer prepared from phenol, aniline and formaldehyde (PAF). Thermal stability of benzoxazines:DGEBA mixture cured isothermally was evaluated by recording thermogravimetric traces in nitrogen atmosphere. The char yield was highest for a mixture having benzoxazine : DGEBA in the ratio of 3 : 1. © 2007 Wiley Periodicals, Inc. J Appl Polym Sci 106: 3327–3334, 2007

**Key words:** benzoxazines; diglycidyl ether of bisphenol-A; curing of polymer; thermal properties; thermosetting resins

## INTRODUCTION

In the recent years, the development of benzoxazine-based family of phenolic resins prepared by reacting phenols, primary amines, and formaldehyde has attracted significant attention. As a result of the ring-opening polymerization, polybenzoxazines overcome many shortcomings associated with the traditional phenolic resins such as releasing condensation by-products and using strong acids as catalysts, while retaining good thermal properties and flame retardance of phenolics.[1] Also, like epoxies and polyimide, polybenzoxazines have excellent mechanical properties and molecular design-flexibility. Benzoxazine resins undergo near-zero volumetric changes or expansion polymerization.[2]

Polybenzoxazine is an attractive class of phenolic resins and can be used as the matrix component of high-performance materials because of its stiffness, high-temperature stability, and a very wide range of molecular design flexibility.[3–5] Such resins combine the properties of epoxy and phenoxy resin and hence found application where higher temperature resistance and processability are important. The mechanical properties of crosslinked benzoxazine resins[6] suggest that, for some applications, the improvement of toughness will be useful. By the incorporation of low $T_g$ components, flexural and impact properties of resins are expected to be improved. Polymeric materials play a vital role in electronic packages as a result of their ease of processing, low cost, low dielectric constant, adhesive properties, etc. Moreover, many properties of polymers can be easily improved or altered by preparing polymer blends or composites.[7,8] As a result, plastic packages occupy ∼ 80–90% of all integrated circuit packages.[9–13] It appears that copolymerization of polybenzoxazine precursor with an epoxy may allow the network structure to achieve a higher crosslink density. Phenols have been shown to react with epoxy resins at elevated temperature.[14] The use of compositions consisting of both polyfunctional benzoxazine and reactive polyamines for coating and encapsulating was documented in the patent literature recently. The reaction involved opening the benzoxazine rings with primary or secondary amines.[15,16]

The article describes the synthesis and characterization of benzoxazine monomers of varying structure obtained by reacting phenol or bisphenol-A with primary amine (aniline, 3-chloroaniline or *o*-toluidine) and formaldehyde. Blending of DGEBA

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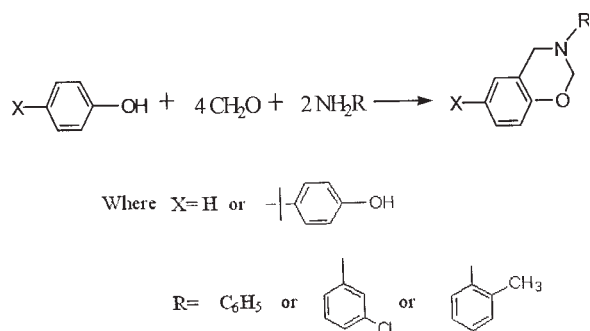
Scheme 1 Synthesis of benzoxazine monomers.

(diglycidyl ether of bisphenol-A) with varying amounts of benzoxazine monomers was done to investigate the effect of blend composition on the curing characteristics. The curing behavior was investigated using differential scanning calorimetry (DSC). Thermal stability of blends that were cured isothermally was evaluated using dynamic thermogravimetry analysis (TGA) under nitrogen atmosphere.

## EXPERIMENTAL

### Materials

DGEBA (Grade LY556; epoxy equivalent 177) was purchased from Hindustan Ciba Geigy. Bisphenol-A (B), phenol (P), *o*-toluidine (T), 3-chloroaniline (C) [all from Aldrich], aniline (A) [Merck], and formalin (F) [Merck] were used as received. Acetone, 1,4 dioxane, and methanol were purchased from CDH and used without further purification.
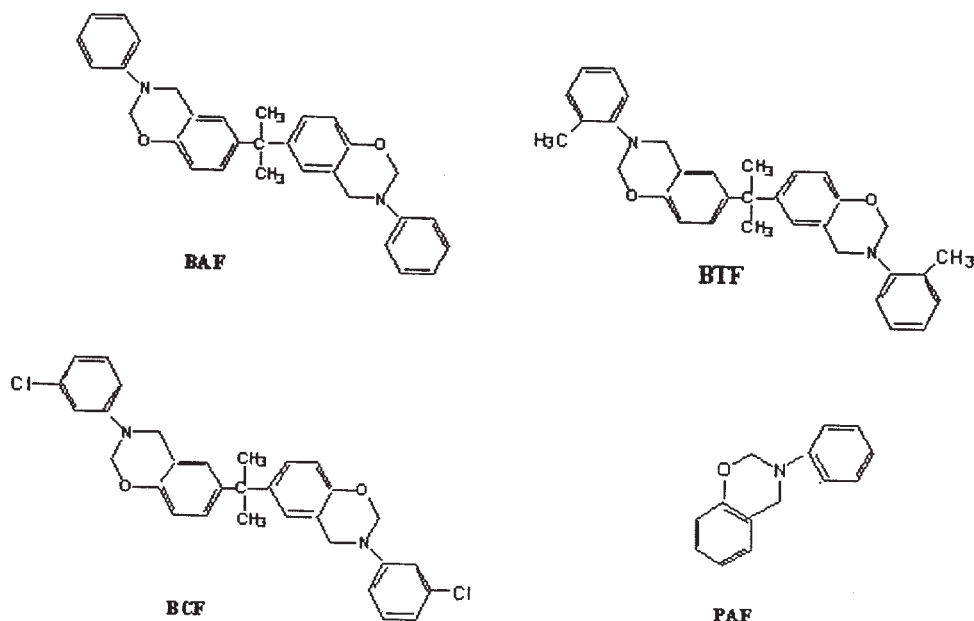
## Synthesis of benzoxazine monomers

Benzoxazines of varying structures were prepared by reacting formalin (0.1 mol) with mixture of aniline (0.05 mol) and phenol (0.025 mol), according to the following reaction scheme (Schemes 1 and 2):

The structure and the letter designation of benzoxazine monomers synthesized by reacting formaldehyde with phenol and aniline (PAF); bisphenol-A and aniline (BAF); bisphenol-A and *o*-toluidine (BTF); bisphenol-A and 3-chloroaniline (BCF) are given below.

## Procedure

In a 250-mL round-bottomed flask, fitted with a magnetic stirrer and reflux condenser, 0.1 mol of formalin was mixed with 20 mL of 1,4 dioxane, and the mixture was cooled (<10°C) using ice bath. The mixture was stirred using magnetic stirrer and after attaining this temperature, required amount of amine solution (0.05 mol dissolved in 5 mL of dioxane) was added slowly while stirring. Stirring was continued for another 10 min followed by the addition of phenol (0.025 mol dissolved in 25 mL of dioxane). The temperature was then raised to 100°C and refluxed for another 6 h. The solvent was removed by vacuum distillation. The ether solution was washed several times with water to eliminate any unreacted formalin and dried over sodium sulphate. Evaporating the ether resulted in a viscous liquid at room temperature.

Purification of the monomer was accomplished by repeatedly washing the ether solution with 3N



Scheme 2 Structure of benzoxazine monomers.

| Sample designation | Amount (g) | | | | |
|---|---|---|---|---|---|
| | DGEBA | BAF | PAF | BTF | BCF |
| BAF-13 | 1.28 | 0.58 | – | – | – |
| BAF-11 | 0.43 | 0.58 | – | – | – |
| BAF-31 | 0.43 | 1.73 | – | – | – |
| BTF-13 | 1.28 | – | – | 0.61 | – |
| BTF-11 | 0.43 | – | – | 0.61 | – |
| BTF-31 | 0.43 | – | – | 1.84 | |
| BCF-13 | 1.28 | – | – | – | 0.66 |
| BCF-11 | 0.43 | – | – | – | 0.66 |
| BCF-31 | 0.43 | – | – | – | 1.99 |
| PAF-13 | 1.28 | – | 0.26 | – | – |
| PAF-11 | 0.43 | – | 0.26 | – | – |
| PAF-31 | 0.43 | – | 0.79 | – | – |

NaOH solution to eliminate any phenolic structures in the monomer followed by washing with water till neutral. Finally, the solvent was removed under vacuum.

### Structural characterization of benzoxazine monomers

Structural characterization of benzoxazine monomers was done using FTIR, ^{1}H NMR spectroscopic techniques, and by elemental analysis. Elemental analysis was carried out using EURO EA 3000 elemental analyzer. IR spectra were recorded as thin film on a KBr plates using Shimadzu FTIR 8700 spectrophotometer. ^{1}H NMR was recorded on a Bruker 300 MHz spectrometer using CDCl$_3$ as solvent and tetramethyl silane as an internal standard.

### Curing studies

TA 2100 thermal analyzer having 910 DSC module was used for recording DSC traces at a heating rate of 10°C/ min under nitrogen atmosphere (flow rate 60 cm$^3$/min). About 5 ± 2 mg of sample was used in each experiment. For curing studies, the samples were prepared by mixing BAF : DGEBA in the molar ratio of 1 : 0, 1 : 1, 3 : 1, 1 : 3, and 0 : 1 without using any solvent. The mixture was then heated to about 80°C in a Teflon boat for 2 h. It has been designated as BAF, BAF-11, BAF-31, BAF-13, and BAF-0. The first numeral digit represents the amount of BAF and second digit represents the amount of DGEBA. BAF and BAF-0 represents the neat benzoxazine monomer and DGEBA respectively.

Similarly, the samples of PAF, BTF, and BCF have been designated. DSC scans of isothermally cured samples were also recorded to determine the effect of network structure on the glass transition temperature. The details of sample preparation along with their designation was given in Table I.

### Thermal stability

Thermal stability of the samples cured isothermally by heating at (160 ± 10)°C for BAF/BCF : DGEBA mixture and at (180 ± 10)°C for PAF/BTF : DGEBA mixture for 3 h in an air oven was evaluated by recording TG/DTG traces in nitrogen atmosphere (flow rate 60 cm$^3$/min). A Rheometric Scientific thermal analyzer having TG1500 + module was used for recording TG/DTG traces at a heating rate of 20°C/ min. Sample size of 10 ± 2 mg in powder form were used in each experiment.

## RESULTS AND DISCUSSION

The benzoxazine monomers were viscous liquids of yellow color and became transparent solid on heating. The color of benzoxazine monomers and DGEBA mixture after heating was orange.

### Characterization of the benzoxazine monomers

Infrared spectroscopic studies

Figure 1 shows the FTIR spectrum of BCF. In the FTIR spectra of all benzoxazine monomers (BAF, BCF, and BTF), the characteristic absorption band due to benzoxazine ring was present at 1498 ± 5 cm$^{-1}$ (1,2,4-trisubstituted benzene ring). In the FTIR spectra of all the monomers, the bands observed at 1159 ± 2 cm$^{-1}$, 1030 ± 3 cm$^{-1}$, and 1234 ± 7 cm$^{-1}$ are due to symmetrical C—N—C, symmetrical C—O—C and asymmetrical C—O—C stretching, respectively. The broad peak around 1597–1660 cm$^{-1}$ implies the C=C stretching of benzene ring.

^{1}H NMR

In the ^{1}H NMR spectra of BAF, BCF, and BTF, methyl protons of isopropylidene were observed at δ 1.6 ppm, which was absent in PAF. The characteris-
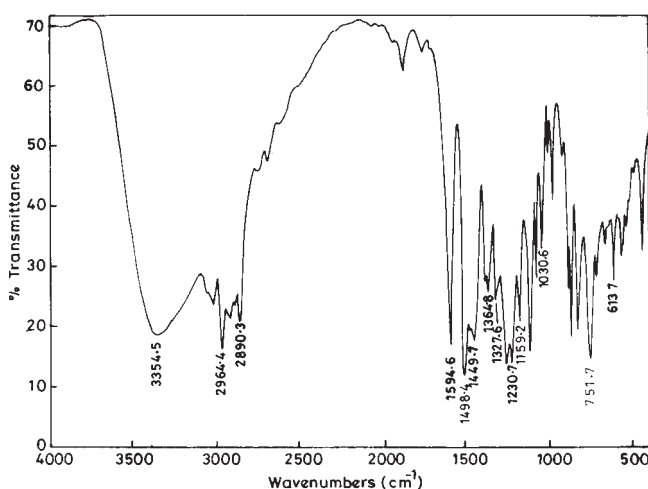


**Figure 1** FTIR spectrum of benzoxazine monomer BCF.

tic peaks due to methylene of (O—CH$_2$—N) and (Ar—CH$_2$—N) of oxazine ring were observed at δ 5.3 and δ 4.3 ppm, respectively, in the $^1$H NMR spectra of all the monomers. The aromatic protons were observed at δ 6.4–7.2 ppm. The signal observed at δ 2.4 ppm in case of monomer BTF is due to the methyl group attached to the benzene ring. The integration was used to calculate the number of protons thus confirming the structure of monomers. $^1$H NMR spectra of (a) BAF, (b) PAF, (c) BCF, and (d) BTF are shown in Figure 2.

## Elemental analysis

Results of elemental analysis are summarized in Table II. The calculated values of C, H, and N agreed well with the experimentally observed values. Thus, the structure of benzoxazines synthesized in this work was confirmed.
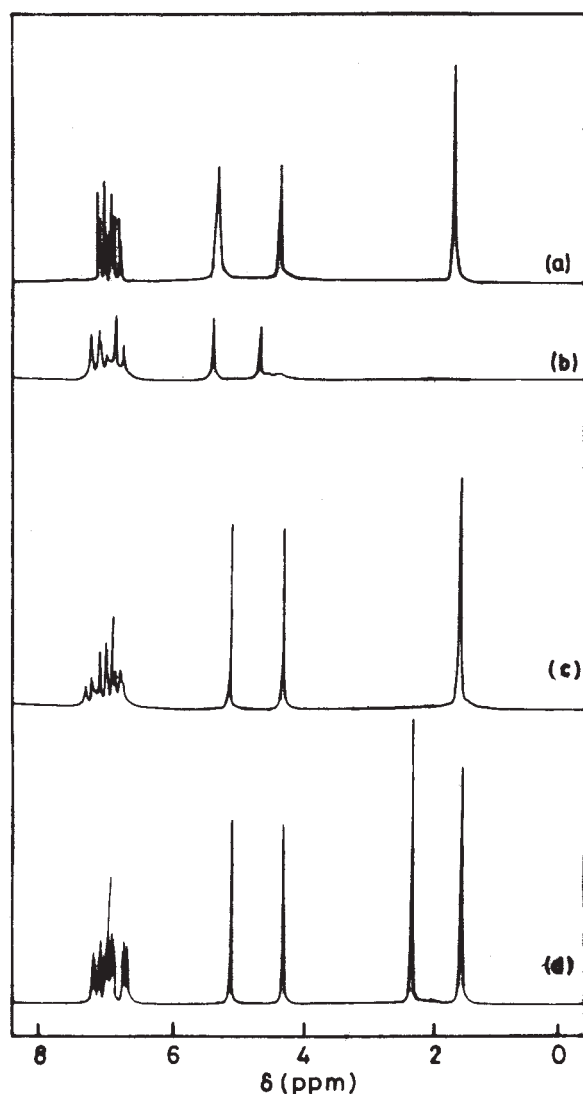
**TABLE II**
**Results of CHN Analysis of Benzoxazines Monomers**

| Sample designation | Carbon (%) | Hydrogen (%) | Nitrogen (%) |
|---|---|---|---|
| BAF | 80.1 (80.5) | 5.9 (6.5) | 5.7 (6.1) |
| PAF | 79.3 (79.6) | 6.0 (6.2) | 6.2 (6.6) |
| BTF | 80.2 (80.8) | 6.4 (6.9) | 5.4 (5.7) |
| BCF | 69.8 (70.2) | 5.0 (5.3) | 5.1 (5.3) |

The numerical values within parenthesis represent calculated values.

## Curing studies

DSC instrument was calibrated for temperature as well as for $\Delta H$ values using indium metal. This metal is used for the calibration of instrument. The curing behavior of benzoxazine monomers and the



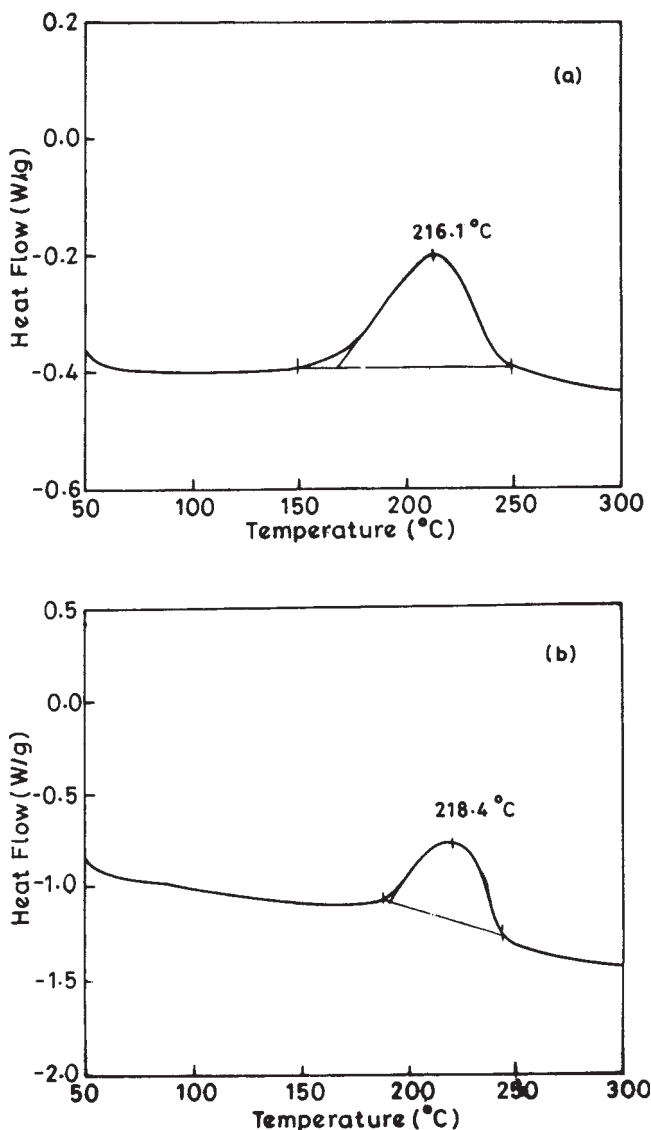**Figure 2** $^1$H-NMR spectra of (a) BAF, (b) PAF, (c) BCF, and (d) BTF.



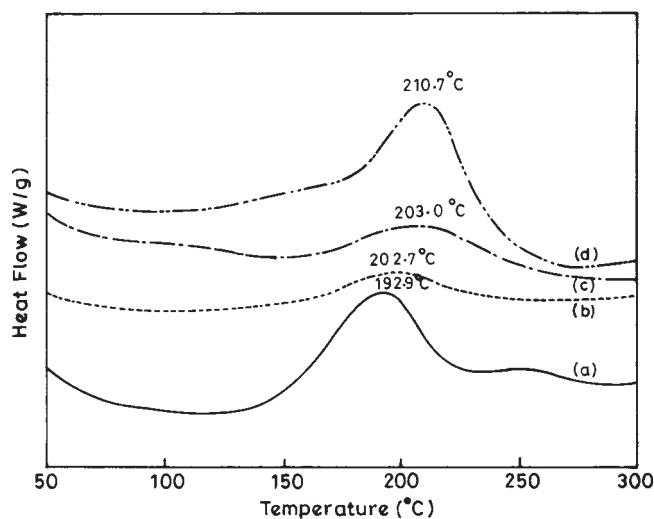**Figure 3** DSC scans of (a) BAF and (b) BTF at heating rate 10°C/min.

**Figure 4** DSC scans of benzoxazine : DGEBA mixtures in the molar ratio of 3 : 1 at a heating rate 10°C/min (a) BAF-31, (b) BCF-31, (c) BTF-31, and (d) PAF-31.

mixture of benzoxazines and DGEBA were investigated by recoding DSC traces. Figure 3(a,b) shows the DSC scans of benzoxazine monomers (BAF and BTF) recorded at a heating rate of 10°C/min. In the DSC scan of DGEBA alone, no exothermic transition was seen in the temperature range of 50–350°C, thereby indicating the absence of curing or crosslinking. In the DSC scans of benzoxazines mixed with varying amounts of DGEBA, an exothermic transition associated with crosslinking or curing was characterized by noting the following parameters:

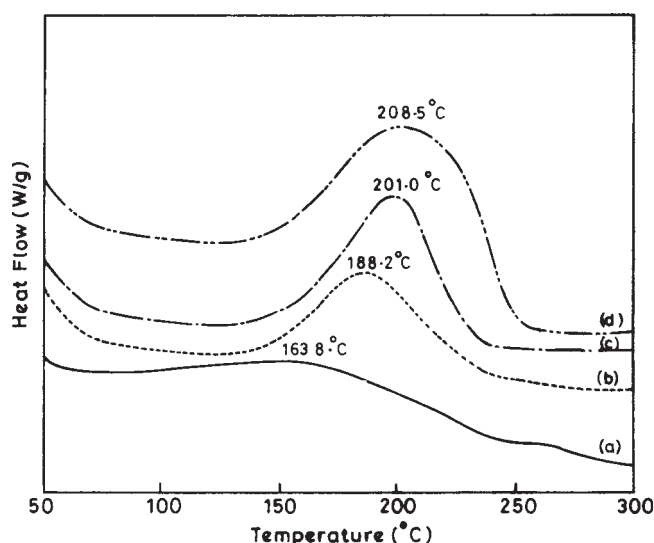$T_i$ is the kick-off temperature, where the curing starts.



**Figure 5** DSC scans of benzoxazine : DGEBA mixtures in the molar ratio of 1 : 1 at a heating rate 10°C/min (a) BCF-11, (b) BAF-11, (c) BTF-11, and (d) PAF-11.
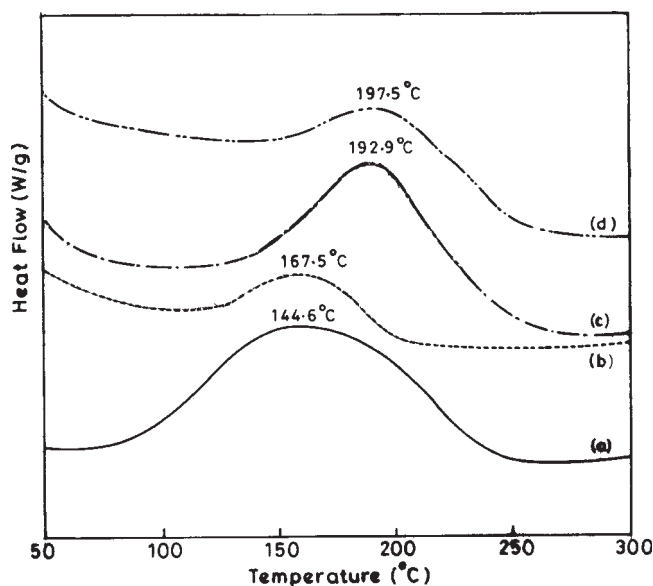


**Figure 6** DSC scans of benzoxazine : DGEBA mixtures in the molar ratio of 1 : 3 at the heating rate 10°C/min (a) BCF-13, (b) BAF-13, (c) PAF-13, and (d) BTF-13.

$T_{onset}$ the temperature where the first detectable heat is released. It was obtained by extrapolation of the steepest portion of curve.

$T_p$ the temperature of peak position of exotherm.

$T_f$ the temperature of the end of curing exotherm.

$\Delta H$ the heat of curing, calculated by measuring the area under the exothermic transition.

In the DSC scans of benzoxazine monomers, a broad exothermic transition was observed. The curing temperatures were dependent upon the structure of phenol or amine used in the preparation of benzoxazine monomers. DGEBA alone was also heated in an air oven at 80°C for 2 h and then DSC scan was recorded. No curing exotherm was observed in the DSC scan of DGEBA. It remains as a viscous liquid only. Comparing the curing behavior of BAF with PAF, it clearly showed that the curing temperatures were higher in case of PAF when compared with BAF, but $\Delta H$ value showed an opposite trend. The lower curing temperature in case of BAF could be due to the presence of isopropylidene group, which may be responsible for the increased mobility. Comparing the curing behavior of BAF, BCF, and BTF, the following trend in $T_i$, $T_{onset}$, and $T_p$ was observed.

$$BCF < BAF < BTF$$

On the other hand, $T_f$ was highest in BCF and lowest in BTF. Higher values of $T_f$ in case of BCF could be done to decreased nucleophilicity as it has elec-

**TABLE III**
**DSC Scans of DGEBA in Presence of Benzoxazine Monomers under Nitrogen Atmosphere (Heating Rate = 10°C/min)**

| Resin designation | Molar ratio of resin : epoxy | $T_i$ (°C) | $T_{onset}$ (°C) | $T_p$ (°C) | $T_f$ (°C) | $\Delta H$ (J/g) | $T_g$ °C |
|---|---|---|---|---|---|---|---|
| BAF | 1 : 0 | 150.0 | 166.9 | 216.1 | 249.2 | 54.9 | 264.6 |
| BAF-31 | 3 : 1 | 137.9 | 150.4 | 192.9 | 231.4 | 57.5 | 245.6 |
| BAF-11 | 1 : 1 | 128.1 | 147.3 | 188.2 | 243.0 | 60.1 | 220.7 |
| BAF-13 | 1 : 3 | 105.9 | 117.7 | 167.5 | 195.5 | 17.5 | 203.4 |
| PAF | 1 : 0 | 175.2 | 181.5 | 221.2 | 288.1 | 116.7 | 225.0 |
| PAF-31 | 3 : 1 | 111.6 | 165.2 | 210.7 | 270.0 | 115.4 | 226.4 |
| PAF-11 | 1 : 1 | 137.9 | 155.2 | 208.5 | 255.9 | 112.1 | 174.9 |
| PAF-13 | 1 : 3 | 118.2 | 149.9 | 192.9 | 278.2 | 158.3 | 101.5 |
| BTF | 1 : 0 | 175.1 | 181.2 | 218.4 | 244.0 | 31.4 | 224.1 |
| BTF-31 | 3 : 1 | 157.4 | 162.9 | 203.0 | 256.3 | 34.6 | 262.5 |
| BTF-11 | 1 : 1 | 136.1 | 156.2 | 201.0 | 241.7 | 73.9 | 235.4 |
| BTF-13 | 1 : 3 | 141.8 | 149.0 | 197.5 | 267.9 | 57.5 | 229.1 |
| BCF | 1 : 0 | 138.2 | 142.0 | 211.7 | 260.2 | 140.5 | 228.6 |
| BCF-31 | 3 : 1 | 157.1 | 162.2 | 202.7 | 251.7 | 18.0 | 224.9 |
| BCF-11 | 1 : 1 | 85.2 | 89.9 | 163.8 | 244.5 | 44.7 | 154.6 |
| BCF-13 | 1 : 3 | 64.9 | 89.9 | 144.6 | 244.5 | 131.6 | 102.3 |

tron-withdrawing groups, whereas in BTF the presence of electron releasing groups may be responsible for this kind of behavior. The incorporation of benzoxazine in different ratio was expected to affect the curing behavior and the thermal stability of the cured network. Therefore, various benzoxazine, such as PAF, BAF, BTF, and BCF were used to investigate systematically the curing behavior in the presence of varying molar ratios of DGEBA. Figure 4–6 show DSC scans of benzoxazines in the presence of varying amounts of DGEBA and the results of DSC scans are summarized in Table III. Curing temperatures of benzoxazines, i.e., $T_i$, $T_{onset}$, $T_p$, and $T_f$ decreased with increasing amount of DGEBA. This shows that the addition of DGEBA activates the polymerization or crosslinking of benzoxazines. On the other hand, curing of BCF was retarded in the presence of 0.25 mol of DGEBA (i.e., sample BCF-31) where all the curing temperatures were higher when compared with BCF. Retardation of ring opening polymerization of benzoxazines by epoxies and activation by phenolic resins has been reported by Ishida and coworkers.[16] $\Delta H$ values showed no definite trend as a function of molar ratios. These results clearly show that crosslinking or ring-opening polymerization of benzoxazine monomers is dependent on the structure of benzoxazine monomers and the blend composition.

The mixtures of DGEBA having varying amounts of benzoxazine monomers of varying structure were isothermally cured by heating in an air oven [(160 ± 10)°C for BAF, BCF : DGEBA, and at (180 ± 10)°C for PAF, BTF : DGEBA mixtures for 3 h] to determine the glass transition temperature. In the DSC scans of isothermally cured mixtures, no exothermic transition was seen thereby indicating the complete polymerization or crosslinking reaction, however a shift in the baseline was observed in the DSC scans
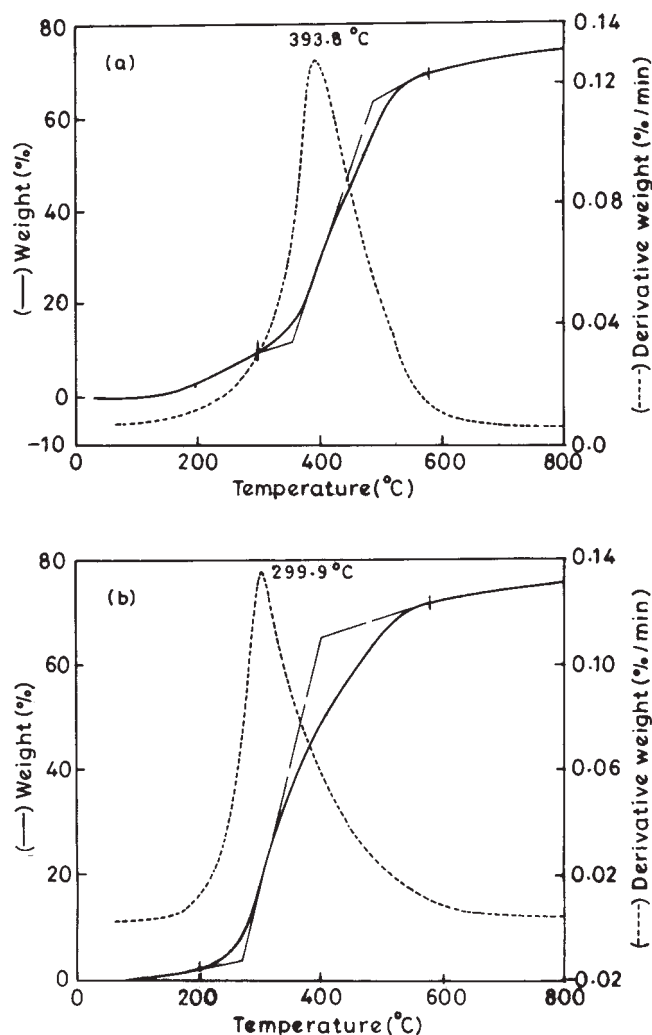


**Figure 7** TG/DTG traces of isothermally cured benzoxazine monomers in nitrogen atmosphere at a heating rate 20°C/min (a) PAF and (b) BAF.
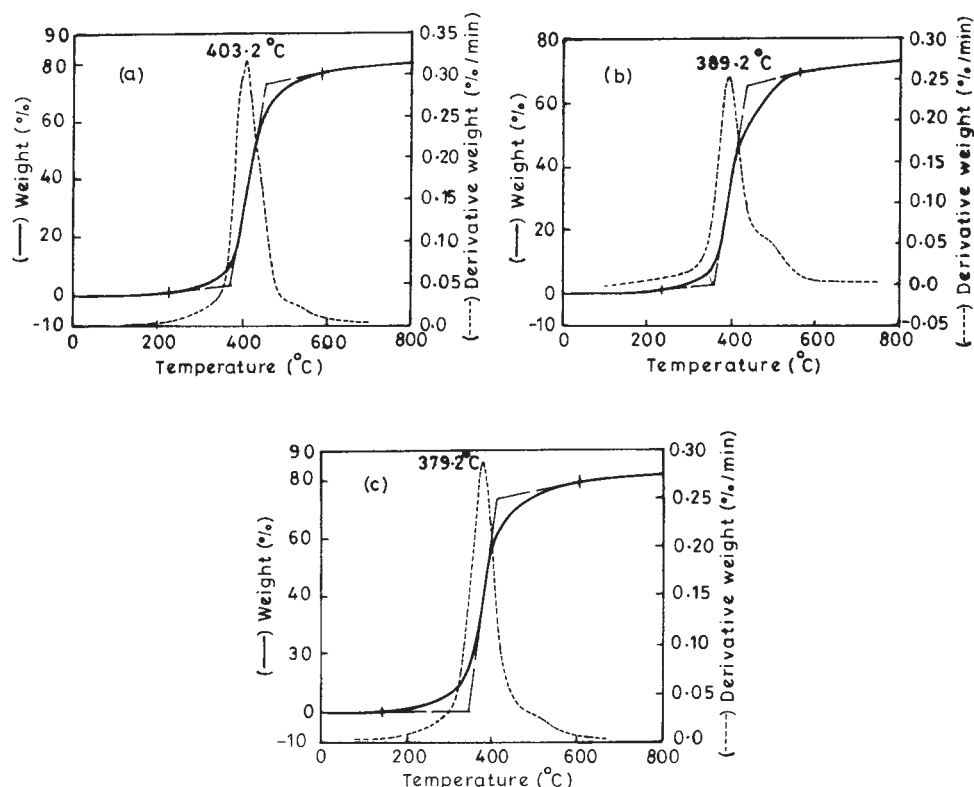
**Figure 8** TG/DTG traces of isothermally cured benzoxazine : DGEBA mixtures in nitrogen atmosphere at a heating rate 20°C/min (a) BAF-13, (b) PAF-11, and (c) BTF-31.

of cured samples. The glass transition temperature was noted as midpoint inflexion and the results are summarized in Table III.

In all the samples, $T_g$ of benzoxazines decreased upon mixing with DGEBA and it decreased with increasing amounts of DGEBA except BTF-31 where it was higher when compared with BTF. This could be due to the decrease in the crosslink density with

increasing amounts of DGEBA. In all the samples, $T_g$ of benzoxazines decreased upon mixing with DGEBA and it decreased with increasing amounts of DGEBA except in BTF-31 where it was higher when compared with BTF. This could be due to the decrease in the crosslink density with increasing amounts of DGEBA. In all the samples, the maximum value of $T_g$ was obtained at the molar ratio of

**TABLE IV**
**Thermal Behavior of DGEBA in Presence of Benzoxazine Monomers under Nitrogen Atmosphere**
**[Heating rate = 20°C/min]**

| Resin designation | Molar ratio of resin : epoxy | IDT (°C) | $T_{max}$ (°C) | FDT (°C) | %Char yield at 800°C | LOI (%) |
|---|---|---|---|---|---|---|
| BAF | 1 : 0 | 268.5 | 299.9 | 400.5 | 24.3 | 27.2 |
| BAF-31 | 3 : 1 | 322.4 | 360.2 | 403.6 | 26.4 | 28.1 |
| BAF-11 | 1 : 1 | 340.9 | 379.6 | 425.7 | 24.0 | 27.2 |
| BAF-13 | 1 : 3 | 372.6 | 403.2 | 447.1 | 20.2 | 25.6 |
| PAF | 1 : 0 | 354.9 | 393.8 | 483.9 | 25.7 | 27.8 |
| PAF-31 | 3 : 1 | 355.0 | 376.3 | 406.6 | 30.1 | 29.5 |
| PAF-11 | 1 : 1 | 357.5 | 389.2 | 431.9 | 26.9 | 28.3 |
| PAF-13 | 1 : 3 | 362.2 | 407.1 | 448.5 | 21.0 | 25.9 |
| BTF | 1 : 0 | 272.5 | 322.9 | 425.0 | 14.6 | 23.3 |
| BTF-31 | 3 : 1 | 347.4 | 379.2 | 412.1 | 18.4 | 24.9 |
| BTF-11 | 1 : 1 | 359.2 | 394.2 | 436.7 | 17.4 | 24.5 |
| BTF-13 | 1 : 3 | 363.2 | 403.2 | 439.7 | 20.3 | 25.6 |
| BCF | 1 : 0 | 224.0 | 291.8 | 344.7 | 25.7 | 27.8 |
| BCF-31 | 3 : 1 | 250.3 | 308.2 | 460.7 | 30.2 | 29.5 |
| BCF-11 | 1 : 1 | 335.4 | 381.6 | 412.0 | 19.4 | 25.3 |
| BCF-13 | 1 : 3 | 379.6 | 410.7 | 538.9 | 23.3 | 26.8 |

3 : 1 (benzoxazines:DGEBA). Glass transition temperature is dependent on the rigidity of the polymer backbone as well as on the crosslink density. Increase in $T_g$ with increasing amount of benzoxazine monomer could be due to the formation of more compact network structure. In benzoxazine monomers alone, the following trend was observed:

$$BAF > BCF > PAF \sim BTF$$

On the basis of structure, one would have expected the highest $T_g$ in PAF, but discrepancy observed could be due to the hindrance to propagation. Higher $T_g$ in BCF could be due the increased polar–polar interactions due to the presence of polar —Cl group.

Thermal stability

Figure 7(a,b) shows the TG/DTG traces of benzoxazine monomer cured isothermally, i.e., samples of PAF and BAF. The relative thermal stability of cured resin was evaluated by comparing initial decomposition temperature (IDT), the temperature of maximum rate of weight loss ($T_{max}$), the final decomposition temperature (FDT), and the percentage char yield at 800°C. Figure 8 shows TG/DTG traces of isothermally cured mixtures of DGEBA in the presence of varying amounts of benzoxazine, i.e., samples (a) BAF-13, (b) PAF-11, and (c) BTF-31. The results of TG/DTG scans are summarized in Table IV. The lowest value of char yield was in BTF and highest in PAF-31 and BCF-31. Benzoxazine monomers after isothermal curing leave behind a char yield of (25 ± 1)% in BAF, PAF, and BCF, whereas BTF showed a minimum char yield of 14.6% at 800°C.

In all samples, char yield increased with the addition of small amount of DGEBA (i.e., in samples BAF-31, PAF-31, BTF-31, and BCF-31). Further increase in the amount of DGEBA resulted in a decrease in the char yield. IDT and $T_{max}$ increased with increasing amounts of DGEBA in the all of the formulations.

Char yield can be used as a criterion for evaluating limiting oxygen index (LOI) of the resins in accordance with Van Krevelen and Hoftyzer equation.[17]

$$LOI = 17.5 + 0.4\, CR$$

where CR is the char yield.

All the samples had LOI values calculated based on their char yield was higher than 28. On the basis of LOI values, such materials can be classified as self-extinguishing resin.

CONCLUSIONS

From these results, the following conclusion can be made:

1. Curing behavior of DGEBA in the presence of benzoxazine monomers was found to be dependent on the structure as well as on the molar ratio of DGEBA : benzoxazine.
2. Curing temperature of benzoxazine decreased upon the incorporation of DGEBA.
3. Thermal stability of the isothermally cured resin was found to be dependent on the molar ratio of DGEBA : benzoxazine.
4. Char yield was highest in samples having DGEBA:benzoxazine ratio of 1 : 3.
5. Glass transition temperature increased with increasing moles of benzoxazine and gave mixture values at a ratio of 3 : 1.

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