

Synthesis and Characterization of Epoxy/Amine Terminated Amide-Imide-Imide Blends

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ABSTRACT: A new amine terminated amide-imide (ATAI) was synthesized by the polycondensation reaction of tetrimide dicarboxylic acid containing bulky *m*-chloro phenyl pendant with *p*-phenylene diamine. The structure of all the prepared compounds were confirmed by FTIR, ¹H-NMR and ¹³C-NMR techniques. This new diamine was then used to cure epoxy resin namely diglycidyl ester of bisphenol-A and the cure reaction was studied by Differential scanning calorimetry. The cured blends show better thermal properties. The *T_g* of the epoxy resin was increased from 134°C to 156°C on addition of 6% of ATAI. The DMA results indicate that the polymer blend with 8% ATAI composition has

higher storage modulus compared to 3% and 6% ATAI composition. The polymer blends with 3% and 6% ATAI composition have higher crosslinking density and lower intersegmental and intrasegmental friction coefficients than 8% ATAI composition. In the DMA curves an increase in the peak half-width was observed with increasing ATAI composition, indicating the possibility of the existence of more than one phase with increasing ATAI concentration. © 2008 Wiley Periodicals, Inc. *J Appl Polym Sci* 108: 2001–2009, 2008

Key words: blends; crosslinking; DSC; flame retardance; mechanical properties

INTRODUCTION

High performance polymers are a class of polymers finding application in advanced composites, electronics, aerospace industries, etc. Advanced composites have become established as efficient high-performance structural material and their use is expanding rapidly. Up to now epoxy based composites have dominated the technology owing to their many attractive properties. Since their introduction in the late 1940s, epoxy resins have found widespread use in coatings, flooring, sealants, grouts, and adhesive applications. Epoxy resin systems are so versatile because of the large number of potential epoxy resin and curing agent combinations each giving a different molecular structure to the final thermoset material. However, certain combinations of physical properties continue to offer significant challenges to epoxy formulators. For instance, low temperature cure coatings are readily formulated with specialty amine curing agents like amine adducts, Mannich bases and phenalkamines. Unfortunately, in high solids or solventless formulations, the working potlife is usually less than desired for field applications (1–2 h max.). Highly flexible yet tough materials for coatings,

flooring, grouts, adhesives, and sealants are another difficult challenge for epoxy formulators. The general experience has been that enhancing one property produces unacceptable losses in several other properties.¹ Achieving an acceptable balance of these properties for a specific application is the goal of every formulator.

Increasing the molecular weight of the epoxy resin or curing agent while maintaining the same number of reactive sites per molecule increases viscosity significantly and lowers heat resistance, while providing only modest increase in toughness. The incorporation of flexible backbone segments can impart high degrees of elongation and lower stiffness to epoxy-amine systems. The use of plasticizers to develop highly flexible formulations has generally been unsatisfactory with epoxies. Plasticizers generally do not provide sufficiently high levels of flexibility and bring about large reductions in tear resistance, chemical resistance, heat resistance and adhesion properties.

Blending of polymers is an effective way to obtain materials with specific properties. In recent years, the feasibility of toughening highly crosslinked networks with high modulus and high *T_g* thermoplastics has been investigated. Results of thermoplastic toughening of epoxy resins, such as polysulfone (PS),^{2,3} polyethersulfone (PES),^{4–6} poly(ether ether ketone) (PEEK),⁷ polyimide (PI)^{8,9} and polyetherimide (PEI)^{10,11} show that the improvement in fracture toughness is achieved without the expense of modulus at elevated temperature. Generally, modifying epoxy resins with

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polyimide (or) imido compounds^{12,13} provided a convenient approach of enhancing the thermal stability of epoxy resins. The present study describes a new curing agent that provides highly flexible, tough epoxy systems while maintaining standard epoxy performance levels in other properties such as adhesion and water resistance. This curing agent is an amine terminated amide-imide-type hardener that can be used with epoxy resins.

EXPERIMENTAL

Materials

2,6-Dimethyl aniline (SRL), *p*-amino benzoic acid (SRL), 3-chloro benzaldehyde, HCl (E-Merck), *p*-phenylene diamine (SRL), pyridine and anhydrous calcium chloride were purchased from Qualigen chemicals (India). Anhydrous calcium chloride was dried under reduced pressure at 150°C for 6 h prior to use. 4,4',3,3'-Benzophenonetetracarboxylic acid dianhydride was purchased from Lancaster and used as such. Triphenyl phosphite (TPP) was purchased from E-Merck and used as such. A commercial epoxy resin (diglycidyl ether of bisphenol A) of equivalent weight 180 was used in this study (Huntsman, India), DDM hardener (HT972).

Monomer synthesis

Synthesis of BADCM

The synthesis of BADCM (bis(4-amino-3,5-dimethyl phenyl)3-chloro phenyl methane) was carried out using the following procedure. A three-necked flask equipped with a reflux condenser, a N₂ inlet and a bent inlet for dry HCl was charged with 2,6-dimethyl aniline (0.04 m, 4.84 g). Dry HCl (0.045 m, 5 mL) was passed into the reaction vessel for 30 min. The solid obtained was dissolved in required

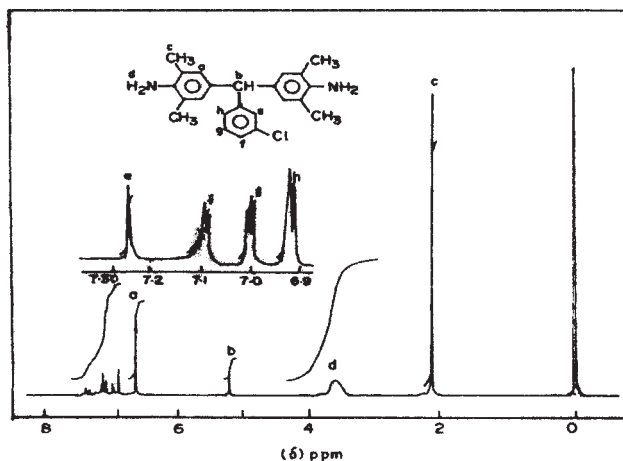


Figure 2 ¹H-NMR spectrum of diamine.

amount of DMF. To this solution, *m*-chlorobenzaldehyde (0.018 m, 2.52 g) was added dropwise. After complete addition, the reaction mixture was stirred at room temperature for 1 h. Then the temperature was slowly raised and maintained at 120°C for 6 h. The reaction mixture was cooled and the pH brought to 7, the solid product obtained was filtered, washed repeatedly with methanol and dried in vacuum oven at 60°C for 12 h¹⁴; m.p. 143°C, yield 92%.

IR [(KBr) cm⁻¹ (Fig. 1)] 3451 and 3380 (asymmetric and symmetric stretching vibrations of N—H group), 1613 (N—H bending vibration), 2960 and 2914 (C—H stretching of CH₃). Absence of peak at 1670 shows the absence of aldehydic C=O.¹⁵

¹H-NMR (CDCl₃, 400 MHz, δ, Fig. 2) 2.12 (s, 12H) CH₃, 7.23–7.28 (t, 1H), 6.98–7.1 (d, 2H), 7.2 (s, 1H), 3.7 (s, 4H), 6.6 (s, 4H), 5.5 (s, 1H).

¹³C-NMR (Fig. 3, ppm), 17.8, 55.2, 122, 126, 127.6, 129.1, 129.2, 129.4, 133.6, 133.9, 140, 147.

Elemental analysis data calculated for C₂₃H₂₅N₂Cl, C 75.70%, H 6.89%, N 7.67%; found C 75.58%, H 6.81%, N 7.73%.

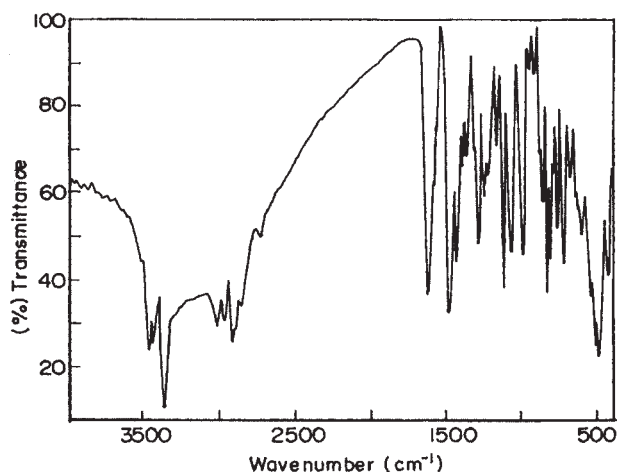


Figure 1 FTIR spectrum of diamine.

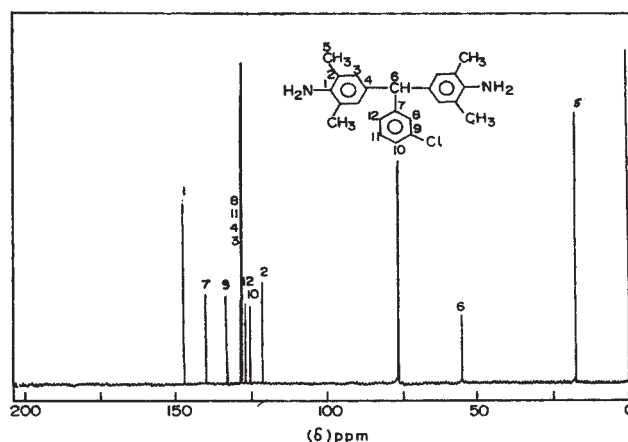


Figure 3 ¹³C-NMR spectrum of diamine.

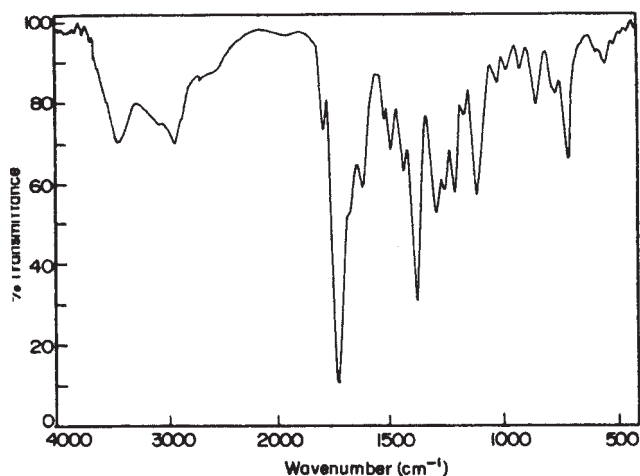


Figure 4 FTIR spectrum of diacid.

Synthesis of imide containing dicarboxylic acid (BBMCA)

The tetrimide diacid BBMCA (bis-4-(3,3',4,4'-benzophenone tetracarboximido,3,5-dimethyl phenyl, 3-chloro phenyl methane) benzoic acid) was prepared by using the following procedure. A three-necked

150-mL RB flask equipped with nitrogen inlet and reflux condenser was charged with a solution of 2.912 g (8 mmol) of diamine [bis(4-amino-3,5-dimethyl phenyl) 3'-chloro phenyl methane] and 2.192 g (16 mmol) of *p*-ABA in 20 mL of NMP. To this 3.488 g (16 mmol) of BPTDA was added in one portion. The mixture was stirred at room temperature for 2 h. The exchange reaction of amic acid was carried out during this long stirring and the product with lowest free energy (diacid) was prepared. About 35 mL of toluene was then added and the mixture was refluxed for about 3 h. The water formed in the reaction was distilled off azeotropically using a Dean-Stark trap. At the end of the reaction, the residual toluene was distilled off under reduced pressure. After cooling, the obtained solution was trickled into water and the precipitated product was collected by filtration and dried in vacuum at 100°C for 12 h to get diacid. The purity of the diacid was checked from the integral ratios of protons; m.p. -320°C (by DSC).

IR (KBr, cm^{-1} , Fig. 4); 3444 (acid $-\text{OH}$ stretching vibration), 1779 (imide, asymmetric $\text{C}=\text{O}$ stretching), 1722 (acid $\text{C}=\text{O}$ stretching and symmetric imide

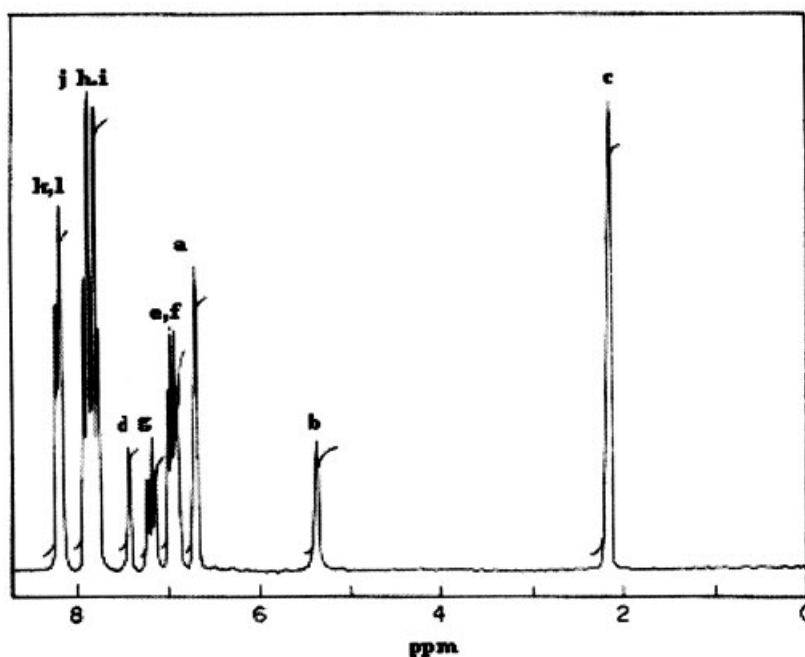
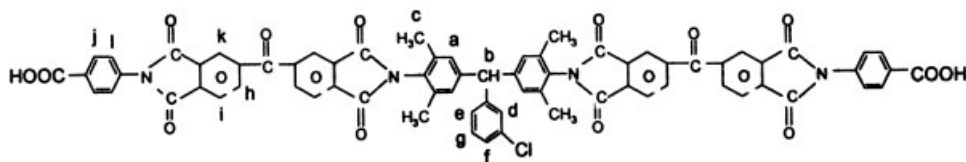
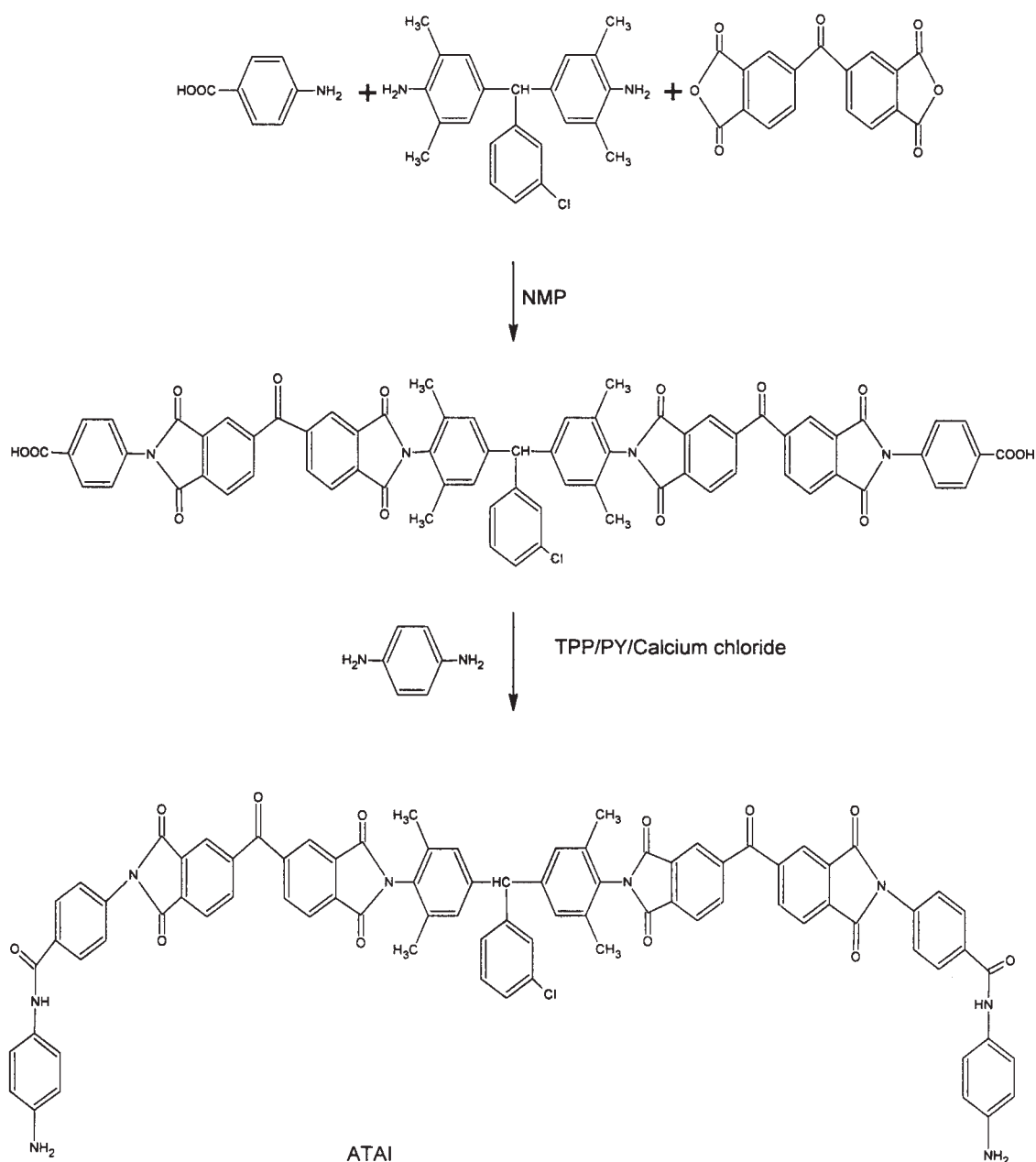


Figure 5 ^1H -NMR spectrum of diacid.



Scheme 1 Preparation of amine terminated amide imide (ATAI).

C=O stretching), 1372 (imide-imide ring vibration, axial) 2927 ($-\text{CH}$ stretching of $-\text{CH}_3$ group), 1111 (imide-imide ring vibration, transverse) and 719 cm^{-1} imide ring deformation.¹⁶

¹H-NMR (400 MHz, DMSO-*d*₆, Fig. 5) 8.19 δ (H_j, Hl, d, 8H), 7.88 δ (Hk, Hh, Hi, dd, 12H), 6.90 δ (Ha, s, 4H), 5.40 δ (Hb, s, 1H), 2.15 δ (Hc, s, 12H), 7.08–7.14 δ (He, Hf, dd, 2H) 7.28 δ (H_g, t, 1H), 7.40 δ (Hd, s, 1H).

The elemental analysis results also confirm the formation of diacid. The found values are very close to the calculated ones.

Synthesis of amine terminated amide-imide (ATAI)

A two-necked 250-mL RB flask equipped with reflux condenser and an addition funnel was charged with a solution of 12.1 g (0.01 mol) of BBMCA and 3.24 g (0.03 mol) of *p*-phenylene diamine in 5 mL of NMP. To this about 3 g of anhydrous calcium chloride, 6 mL of TPP and 10 mL of pyridine were added and refluxed for 3 h. As the reaction proceeded the viscosity of the reaction increased and an additional 3 mL NMP was added. The obtained viscous solution was trickled into excess methanol with vigorous stirring. The product thus obtained was filtered,

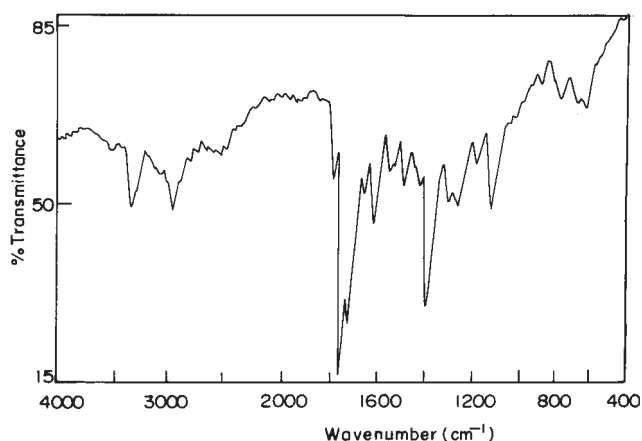


Figure 6 FTIR spectrum of ATAI.

washed thoroughly with methanol and dried under vacuum at 100°C for 12 h. Yield 82%.

Preparation of cured epoxy resin

The cured epoxy resins were obtained by thermal curing of epoxy resins with DDM, and prepared amine-terminated amide-imide-imide (in different compositions) as curing agents. Epoxy resin was mixed with DDM and prepared diamine and degassed. The degassed resin was then poured into the preheated mold. The resin was cured at 120°C for 2 h, 150°C for 2 h, and finally at 180°C for 2 h. Finally, it was subjected to post-curing at 220°C for 2 h after demolding.

Measurements

Infrared spectra were recorded on a Nicolet Fourier transform infrared spectrometer using KBr pellets. ¹H-NMR and ¹³C-NMR spectra were obtained on a Jeol Ex-400 spectrometer (400 MHz) using DMSO-*d*₆ and CDCl₃ as solvents and the chemical shifts were calibrated by using 1% TMS as reference. Elemental analyses were carried out with a Perkin-Elmer model 2400. Thermogravimetric data were obtained on a Dupont 2100 in flowing Nitrogen at a heating rate of 20°C/min. Differential scanning calorimetry

TABLE I
Curing Characteristics of the ATAI-Epoxy Cured System

Resin composition	Cure onset temperature (°C)	Peak temperature (°C)	Final temperature (°C)
Epoxy/DDM/ATAI 3%	78	160	219
Epoxy/DDM/ATAI 6%	92	162	220
Epoxy/DDM/ATAI 8%	99	163	221

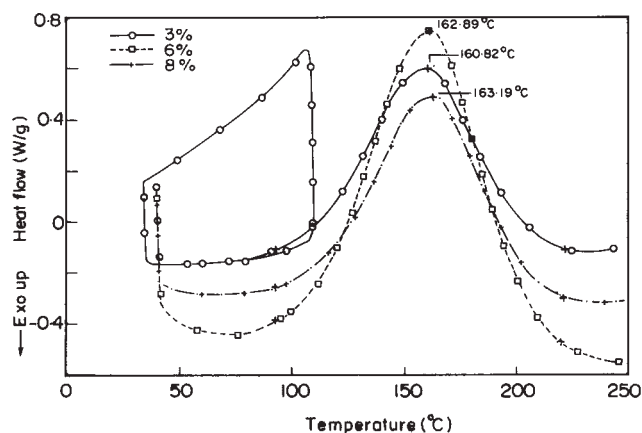


Figure 7 Cure behavior of epoxy/DDM/ATAI blends.

(DSC) analysis was performed on a Dupont model 910 Differential scanning calorimeter using 3 mg of the sample crimped in aluminum pans at a heating rate of 10°C/min and a flow rate of 40 mL/min. The mechanical properties of the cured epoxy/DDM/ATAI resins were tested using universal testing machine with the cross head speed of 2 mm/min. The specimen for tensile test (ASTM D638) was prepared with the length 150 mm, width 25 mm and thickness 3 mm. The flexural (ASTM D790) and impact (ASTM D256) tests were carried out with the specimen of length 125 mm and width 13 mm. The shore D hardness test was carried out as per the ASTM D 2240. The Dynamic mechanical analysis was carried out with a NETZSCH DMA 242 Dynamic mechanical analyzer in a three point bending mode using different frequencies (2, 5, 10, 20 Hz). Rectangular specimens of 55 mm length, 10 mm width, and 3 mm thickness were used for this study.

RESULTS AND DISCUSSION

ATAI synthesis

The amine terminated amide-imide-imide was prepared by the polycondensation reaction of BMCA with *p*-phenylene diamine (1 : 2) in NMP using TPP and pyridine as catalysts (Scheme 1). The structure of the prepared diamine was confirmed by FTIR

TABLE II
Thermal Properties of the ATAI-epoxy Cured System

S. no.	Polymer composition	Decomposition temperature (°C)	Char yield %
1	Epoxy/DDM ^a	310 ^a	18 ^a
2	Epoxy/DDM/ATAI 3%	376	57
3	Epoxy/DDM/ATAI 6%	387	65
4	Epoxy/DDM/ATAI 8%	373	78

^a Reported value.²⁰

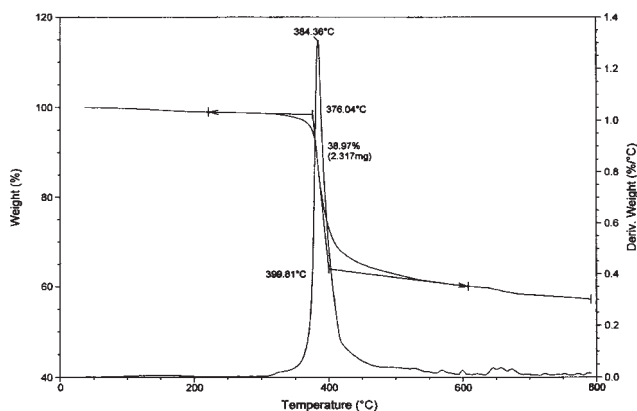


Figure 8 Thermogram of epoxy/DDM/ATAI 3% blend.

technique. This prepared diamine was then blended with epoxy/DDM resin in three different compositions (3%, 6%, and 8%) and poured into the pre-heated mold. The blend was then cured at 120°C for 2 h, 150°C for 2 h and finally at 180°C for 2 h. It was then demolded and finally post cured at 220°C for 2 h. The FTIR spectrum of the amine terminated amide-imide-imide is given in Figure 6. The broad absorption around 3463 cm^{-1} and 3380 cm^{-1} corresponds to *N*-H symmetric and asymmetric stretching of NH_2 group and the absorption band around 1617 cm^{-1} is due to *N*-H bending vibration. The monomers show absorptions around 2970 and 2921 cm^{-1} due to C-H stretching vibration of CH_3 group. The diamine shows sharp absorption at 1722 and 1777 cm^{-1} due to symmetric and asymmetric stretching vibration of the imide ring. The amide group absorption is found at 1682 cm^{-1} confirming the formation of the desired product. The $^1\text{H-NMR}$ spectrum of ATAI clearly shows a singlet at 2.16 δ for 12 methyl protons, a singlet at 5.2 δ for one methyne proton. It also shows a singlet at 3.9 δ for 4 amine protons and another singlet at 10.61 δ for the two amide protons. All the aromatic protons appear as a multiplet around 6.92–8.46 δ

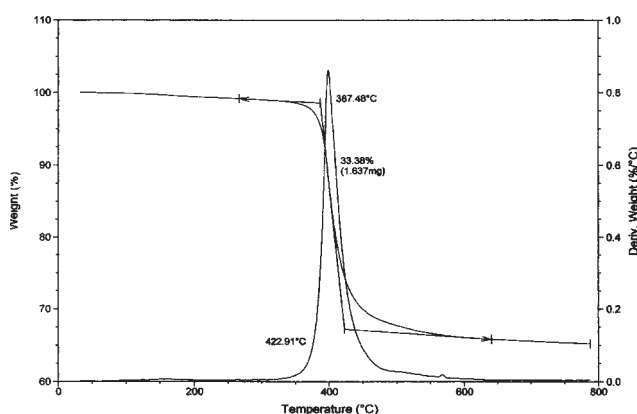


Figure 9 Thermogram of epoxy/DDM/ATAI 3% blend.

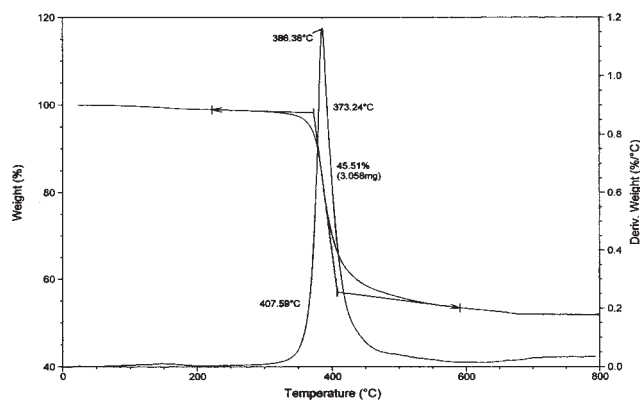


Figure 10 Thermogram of epoxy/DDM/ATAI 3% blend.

Cure studies

The curing reaction of PAI-epoxy was studied using differential scanning calorimetry. The data obtained from DSC scans are furnished in Table I. The DSC thermogram of the blends is shown in Figure 7. All the ATAI-epoxy-cured systems show a single exothermic peak in the temperature range of 160–163°C. This may be due to the simultaneous reaction of major secondary amine and minor primary hydrogen atoms with epoxide.^{17–19} From the typical DSC thermogram (at 10°C/min) the cure onset temperature (T_i), peak exotherm temperature (T_p), and temperature of completion of curing (T_f) were obtained.

Thermogravimetric analysis

The thermal stability of the cured resins was studied using thermogravimetric analysis. The results are tabulated in Table II and the thermograms are shown in Figures 8–10. The thermograms of the cured samples reveal that they all degrade in a single step and start decomposing at around 370°C. Degradation takes place at a faster rate between 400°C and 600°C. The results also show that the thermal stability of the resin increases with increase in ATAI percentage. However, the cured resin with 8% of ATAI shows a slight decrease in thermal stability.

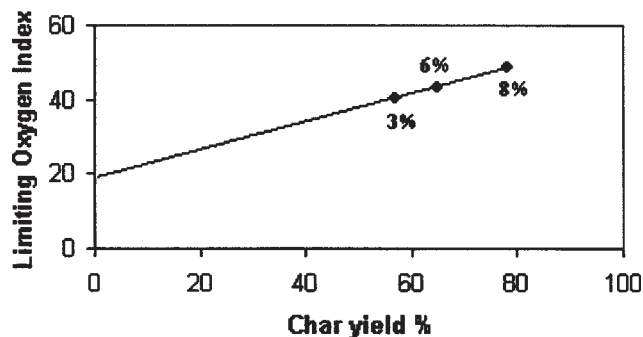


Figure 11 Plot of char yield versus LOI.

TABLE III
Mechanical Properties of Epoxy/DDM/ATAI Cured System

ATAI composition (%)	Tensile strength ^a (MPa)	Elongation ^a (%)	Tensile modulus ^a (GPa)
0	64.73, 66.00 (65.37)	4.98, 4.85 (4.92)	1.30, 1.36 (1.33)
3	62.46, 62.41 (62.44)	5.10, 5.18 (5.14)	1.22, 1.20 (1.21)
6	56.10, 55.68 (55.89)	5.40, 5.32 (5.36)	1.038, 1.04 (1.04)
8	54.77, 55.33 (55.05)	5.36, 5.38 (5.37)	1.02, 1.03 (1.03)

^a In parenthesis are average values.

This may be attributed to the presence of uncured ATAI in the matrix. At this composition the dissolution of ATAI in epoxy was partial.

Flame retardancy

It can be seen from the TGA curves that the thermal degradation rates of the resin are high only in the high temperature region (400–600°C). Apart from this, the cured ATAI/epoxy resins exhibit increased char yield with increasing amide-imide-imide content. Increasing char formation could limit the production of combustible gases, decrease the exothermicity of the pyrolysis reaction, and decrease the thermal conductivity of the resin consequently to limit the resin's flammability.

The char yield of these cured resins was also found to be high in the range of 57–78% indicating the high fire resistance property of the resin. This can be explained by the presence of chlorine moiety in the hardener which retards ignition. HCl vapors are formed during ignition which acts as a blanket between the surface and atmosphere depriving the surface of oxygen, and thus inhibits combustion.²¹ Furthermore, in the solid state, some halogen acids catalyze char formation.

From the Figure 11 it is clear that the flame resistance increases with increase in concentration of amide-imide-imide content. The flame retardancy of this cured epoxy resin was also confirmed by their limiting oxygen index value. The LOI value was calculated by using Krevelen's equation and the obtained value was plotted against char yield.²²

$$\text{LOI} = 17.5 + 0.4(\sigma)$$

TABLE IV
Mechanical Properties of Epoxy/DDM/ATAI Cured System

ATAI composition (%)	Flexural strength (MPa)	Impact strength ^a (J/cm)	Hardness
0	98.2	0.96, 0.90 (0.93)	83
3	96.6	1.16, 1.26 (1.21)	81
6	91.7	1.60, 1.66 (1.63)	82
8	90.0	1.80, 1.90 (1.85)	83

^a In parenthesis are average values.

where σ is the percentage of char yield. The curve thus obtained was linear showing that the LOI increases with increasing char yield. Thus, the developed epoxy-amide-imide-imide resin can be considered as a better flame retardant material than neat epoxy.

Mechanical properties

Mechanical properties of epoxy/DDM/ATAI blends of various compositions were studied. Tensile properties of epoxy/DDM/ATAI were studied as per ASTM D-638 (Table III). Addition of ATAI hardener into the epoxy matrix decreases the tensile strength, modulus and flexural strength. Composition having 3% ATAI hardener has the highest tensile strength and modulus. Beyond this composition, as the percentage of ATAI was increased a decrease in tensile strength was observed. This behavior may be attributed to the lower reactivity of NH₂ group present in the new hardener (as a result of electron withdrawing groups present) compared with that present in DDM. This behavior may also be attributed to the fact that the ATAI is itself a thermoplastic oligomer which when used as hardener for epoxy resin in place of DDM decreases the crosslink density. The increase in % elongation supports this observation. A similar trend was observed with respect to flexural strength also. The tensile elongations of various compositions were found to be in the range of 5.10–5.38% indicating that the incorporation of thermoplastic ATAI increases the elongation property of the thermoset. The 3% composition has the highest

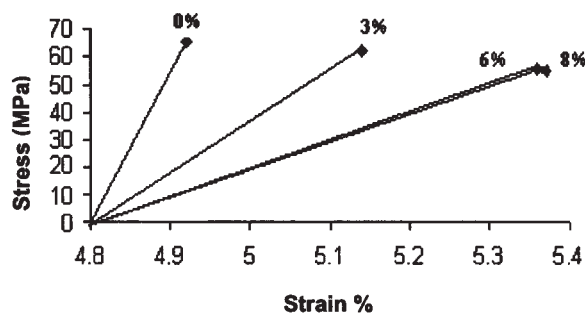


Figure 12 Stress-strain curves for the epoxy/DDM/ATAI resin systems.

TABLE V
Dynamic Mechanical Properties of Epoxy/DDM/ATAI Cured System

ATAI composition (%)	Tan δ peak temperature ($^{\circ}$ C) at 20 Hz	Peak half-width (mm)	Storage modulus E' (Mpa)	Tan δ
0	134	50	3816 (104 $^{\circ}$ C)	0.265
3	155	45	3081 (124 $^{\circ}$ C)	0.217
6	156	47	3547 (125 $^{\circ}$ C)	0.213
8	143	49	3680 (112 $^{\circ}$ C)	0.242

value, and % elongation values increase with increase in the hardener content. Impact strength of the epoxy resin was increased from 0.96 to 1.16 J/cm on addition of 3% of hardener. Further increase of hardener content increases the impact strength. Hardness values were not found to be affected much on addition of various amounts of hardener to the epoxy matrix (Table IV).

The stress-strain curve for the epoxy/DDM and epoxy/DDM/ATAI resin systems are given in Figure 12. It can be seen that the area under the stress-strain curve for 6% and 8% ATAI composition are the highest and 3% ATAI composition has smaller area under the stress-strain curve indicating that the addition of ATAI increases the toughness of the epoxy system and with increasing ATAI content the toughness increases up to 6% beyond that it remains constant. This conclusion is also supported by the impact strength data.

Dynamic mechanical analysis

The characteristics of the new polymer blends provided by the Dynamic Mechanical Analysis are also of prime interest in this study. Table V summarizes the results of DMA analysis. Figures 13–16 summarize the temperature dependence of the loss factor tan δ for the epoxy/DDM/ATAI blends based on three different composition of ATAI. The blends with 3% and 6% ATAI possess higher T_g at approximately 155 $^{\circ}$ C and 156 $^{\circ}$ C respectively. This is because the crosslinking hindered the polymer segmental

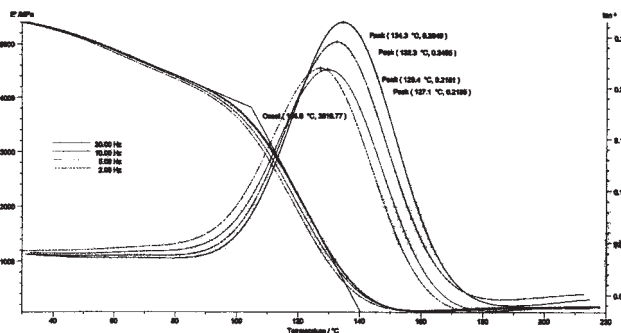


Figure 13 DMA curve of epoxy/DDM cured system.

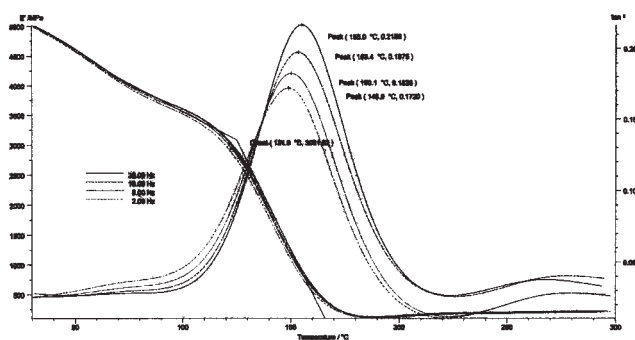


Figure 14 DMA curve of epoxy/DDM/ATAI 3% cured system.

motion and thus required a higher temperature for the onset of segmental motion of the polymer chains. As molecular motions became more and more restricted, the amount of energy that could be dissipated throughout the polymer specimen decreased dramatically. Therefore, the loss factor peak positions of the polymer blends shifted to higher temperatures. The tan δ intensities also diminished. Meanwhile, a significant broadening of the α relaxation was observed. The blend with 8% ATAI shows the lowest T_g value of 143 $^{\circ}$ C. The height and the area of the tan δ peak associated with the α relaxation is related to the crosslinking density,^{23,24} impact resistance and toughness^{25–27} of a material. Compared with the polymer blend with 8% ATAI, the polymer blend with 3% and 6% ATAI composition exhibited considerably lower tan δ value because of higher crosslinking densities and higher intersegmental and intrasegmental friction coefficients in the polymer blend. The height and area under the tan δ curve gives an indication of the total amount of energy that can be absorbed by the material. As the ATAI composition was increased from 3% to 8% a broadening of tan δ peak was observed from 47 to 49 mm. A large area under the tan δ curve indicates a high degree of molecular mobility, which translates into better damping properties. This means that the material can better absorb and dissipate energy.

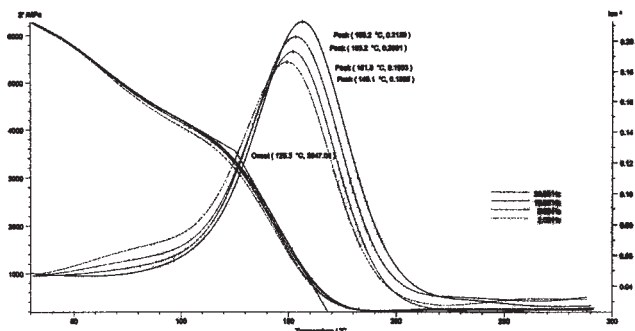


Figure 15 DMA curve of epoxy/DDM/ATAI 6% cured system.

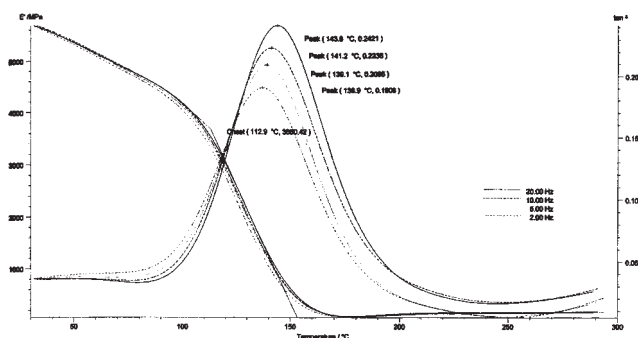


Figure 16 DMA curve of epoxy/DDM/ATAI 8% cured system.

Figures 13–16 also show the temperature dependence of the storage modulus E' for the epoxy/DDM/ATAI polymer blends. The polymer blend with 8% ATAI exhibits considerably higher storage modulus than those with 3% and 6% ATAI. The blend with 8% ATAI composition show a storage modulus drop at lower temperature (112°C) indicating soft character whereas those with 3% and 6% ATAI show a modulus drop at considerably higher temperature of 125°C indicating hard character.

The single loss factor peak apparently indicates that these polymer blends possess a single homogeneous phase. The shape of $\tan \delta$ peak may be used as a convenient indicator for the morphological state of the phases within the polymer blend. The broadness of a relaxation indicates the complexity of the polymer blend. Although only one damping peak was observed for these polymer blends, their $\tan \delta$ peaks do tend to become broader with increasing ATAI content, which is indicated by the increase in half width of $\tan \delta$ damping peaks. Such a phenomenon suggests an increasing possibility of the existence of more than one phase with increasing ATAI concentration.

CONCLUSIONS

A new amine terminated amide-imide-imide was synthesized by the polycondensation reaction of the tetrimeric diacid containing bulky *m*-chloro phenyl pendant group and *p*-phenylene diamine in 1 : 2 molar ratio. Blending of this diamine (in different proportion with DDM) with epoxy resin yields cured

blends with good thermal stability and flame retardance properties. Addition of this diamine increases the toughness of the epoxy system. The polymer blends with 3% and 6% ATAI show lower storage modulus because of their higher crosslink density. However, increased percentage of ATAI leads to phase separation. Thus, the prepared compound with terminal $-\text{NH}_2$ group and amide linkage can act as an efficient curing agent for epoxy systems.

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