

Research Article

Preparation and Characterization of Chain-Extended Bismaleimide/Carbon Fibre Composites

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This paper presents an experimental study of the influence of the addition of aromatic diamine (MDA) to bismaleimide (BMI) resin on the crystallinity, solubility, melting temperature, and mechanical properties of BMI/carbon composites. The modified BMI was prepared through the chain extension with MDA via Michael addition reaction with molar ratio of 3 : 2 (BMI : MDA). Both modified and unmodified BMI were characterised for chemical structure, crystallinity, melting temperature, mechanical property, and morphology and fracture behaviour using FTIR, XRD, DSC, UTM, and SEM, respectively. The FTIR results revealed the formation of polymeric chain due to the broad N-H absorption. The modified resin was semicrystalline in nature having low melting temperature and hence showed good processibility. The modification of BMI resulted in decrease of pores and increase of tensile, flexural, and impact properties of the composites. Also, SEM studies of the tensile fractured specimens revealed that modification of BMI resulted in improved resin/fibre interfacial strength.

1. Introduction

Constant development in the area of temperature resistant thermosets has been witnessed in recent years. Thermosetting polyimides, in particular, aromatic bismaleimides (BMIs) are gaining wide acceptance by the aerospace/aircraft, electric/electronics industries because they have good thermal stability at elevated temperatures and in wet environments [1–5]. The need for bismaleimide arose because of the poor hot/wet performance of epoxies. And, BMI systems are able to be fabricated using epoxy-like conditions and are capable of performing at temperatures up to 230°C. Polyimides have excellent mechanical properties, high T_g (up to 450°C) and long term stabilities [6–8].

The BMIs can be self-polymerized through their reactive maleic double bonds which result in highly cross-linked brittle polyimides leading to premature failure. This disadvantage has been overcome by introducing an appropriate chemical structure between two polymerizable maleimide groups. Another successful approach is the use of chain-extended high-molecular-weight prepolymers, which were

prepared by the Michael addition reaction of BMIs and diamines [9, 10]. However, it is generally not desirable to use a highmolecular-weight polymer solution as a matrix precursor for fiber-reinforced composites, because at high solids content the solution is too viscous to handle; at low solids content, there is usually insufficient resin pick up in one pass in the prepregging operation [11]. Another one biggest drawbacks in the use of BMIs and polyimides is still processing difficulty due to their highly aromatic chemical structures. Meanwhile, BMI resins suffer from brittleness that leads to composite microcracking. Polyimides also have the toughness limitations and poor hydrolytic stabilities.

Many researchers have attempted to overcome the brittle nature of BMI via modification of its chemical structure by adding other chemical groups [12–15]. Another successful approach is the use of chain-extended high-molecular-weight prepolymers prepared by the Michael addition reaction of BMIs and diamine [16–21]. The Michael addition reaction between diamines and BMIs are a successful method of reducing the brittleness of BMIs and the reaction will result in the chain extended BMI amine adduct and the

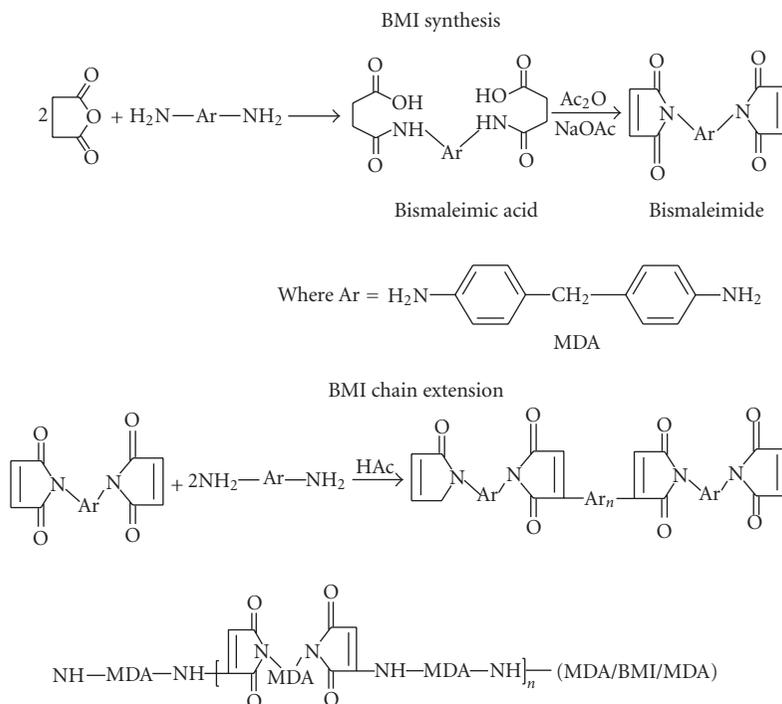


FIGURE 1: BMI synthesis and chain extension of BMI with MDA.

reaction proceeds as BMI can react with bis-nucleophilic species to form the cross-linking structure via Michael-Addition reaction. Dithiols and diamines are the favoured bis-nucleophiles because they have high basicity [21–23].

If chain-extended BMI prepolymers are used in place of BMIs, they reduce the brittleness due to the larger polymer segments available for internal molecular motions after cross-linking. Although many investigators [16–21] have studied MDA-modified BMI systems, little attention has been paid to certain areas like the crystallinity studies of the developed modified resins the cured resin system and its composites. Also, the MDA/BMI/Carbon composite and their mechanical properties compared with unmodified BMI/Carbon composites are not addressed in open literature. Thus, a systematic investigation is required for understanding the modification process starting from the spectroscopic analysis and its influence of chain extension with MDA on the properties such as crystallinity, processibility, morphology, and mechanical properties of the resin and the carbon composites of the modified resin composites.

The objective of this paper was to synthesise chain-extended BMI with MDA and characterise the modified BMI resin and its composites for spectroscopy, morphology, crystallinity, and mechanical properties.

2. Experimental

2.1. Materials Used. The BMI, MDA, and dimethyl formamide (DMF) were used as matrix resin, chain extender, and solvent, respectively. The carbon fabrics (204 gsm, plain weave, supplied by M/s CD Interglass Germany) was used as the reinforcement for the composites.

2.2. BMI Synthesis and Modification. BMI synthesis and chain extension was carried out at ABR Organics Hyderabad following the chemical reaction scheme shown in Figure 1. All solvents and reagents were commercial and of analytical grade and used as received. An amount of 1.0 mol of appropriate aromatic diamine, (4,4'-Diaminodiphenylmethane (MDA)) was dissolved in N,N-Dimethyl formamide (DMF), in a three-necked flask equipped with a magnetic stirring bar and fitted with a thermometer, a dropping funnel, and a reflux condenser with a CaCl₂ drying tube, Maleic anhydride (2.1 mol) as DMF solution, was added dropwise over a period of 10 min at RT. The solution was maintained at 50°C for 2 h in a thermoregulated bath. Cyclization of aminic acid was carried out by adding excess amount of fused sodium acetate and acetic anhydride mixture to the formed yellow solution. The temperature of the reaction mixture was maintained at 50°C for another 2 h, and then the solution was poured into an ice bath. The precipitate was collected by filtration, repeatedly washed with cold water, and dried at 60°C in a vacuum oven (Figure 1). MDABMI was purified by recrystallization from a methanol-chloroform (1 : 1 v/v) mixture.

The chain extension process involves the Michael addition type reaction with an aromatic diamine (Methylenedianiline, MDA) in m-cresol solvent. MDA (2.0 mol) as m-cresol solution was added drop-wise to the m-cresol solution of BMI (3.0 mol) at room temperature, containing a catalytic amount of glacial acetic acid. The reaction mixture was stirred for 96 hours in an oil bath at 100–105°C, till a viscous solution was obtained. The solution was then poured into ethanol and the precipitate was collected by filtration. The synthesized polymeric resin was washed repeatedly with ethanol and dried at 60°C in a vacuum oven.

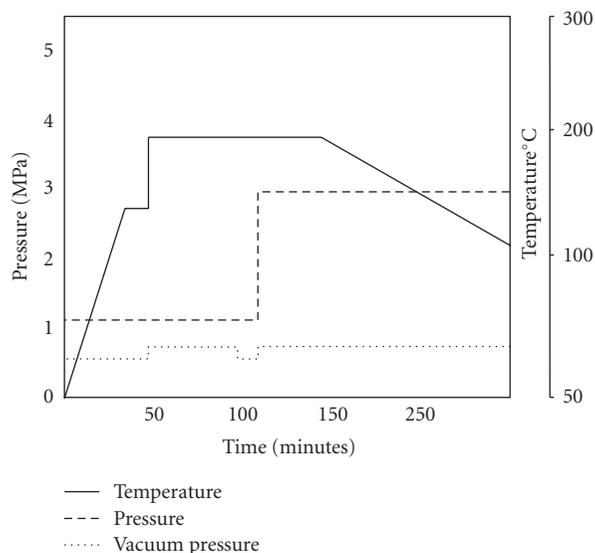


FIGURE 2: Optimum processing conditions for BMI/Carbon Composites.

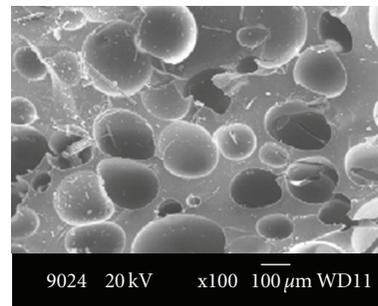
2.3. Preparation of Prepregs and Fabrication of Composite Laminates. Before prepregging, carbon fabrics were exposed to 400°C in air for removing the coated epoxide sizing, which would otherwise chemically degrade during high temperature post cure conditions. The prepregs were prepared by dissolving modified BMI in 45%–50% DMF solution, which was used to impregnate carbon fabrics and then dried in an oven at 160°C for 10 minutes.

Thirteen plies of prepregs were placed in a metal mould $240 \times 120 \times 3$ mm with the application of vacuum pressure. The mould was placed in a hot press at 120°C and the curing temperature cycle shown in Figure 2 was applied.

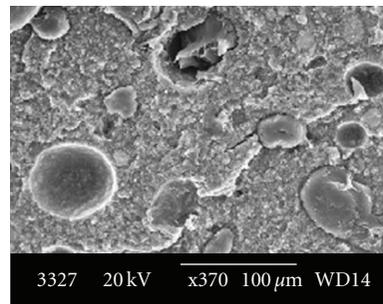
- (1) Increase the temperature from 120 to 180°C in 30 min at 4 MPa pressure.
- (2) Dwell (Cure) at 180°C for 2 hours.
- (3) Reduce the temperature from 180°C to 100°C and remove specimen from the mould.
- (4) Dwell (Post cure) at 250°C for 16 hours in the oven.

2.4. Characterisation. The chemical structure of the modified BMI resin was studied using FTIR spectroscopy. The samples for FTIR analysis were mixed with KBr powder, pressed in to pellets and introduced into Perkin Elmer spectrum GX4 fourier transform infrared (FTIR) spectrophotometer at a resolution of 4 cm^{-1} in an optical range of $400\text{--}4000 \text{ cm}^{-1}$. Thermal characterization of the modified resin was studied using Mettler 823, Differential Scanning Calorimeter (DSC) calibrated with an indium standard. A stream of Nitrogen at a flow rate of 20 ml/min was used to purge the DSC cell. The experiment was conducted at a heating rate of $5^\circ\text{C}/\text{min}$ and on line DSC thermograms were obtained.

The crystallinity of the specimens was studied by Panalytical X'pert high score plus XRD machine. The scan speed of the machine was $0.35^\circ/\text{min}$ and stepsize (2θ) was 0.0170.



(a)



(b)

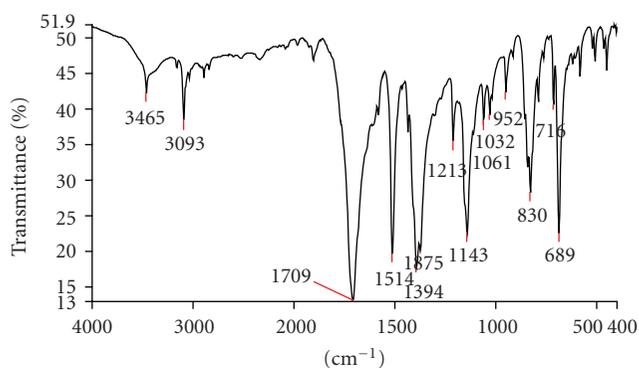
FIGURE 3: Microstructural surfaces of (a) cured unmodified BMI, and (b) cured modified BMI, respectively.

The specimens were tested for tensile properties as per ASTM D 3039 at a strain rate of 2 mm/min. The flexural test was conducted as per ASTM D 790 and the impact test as per ASTM D 256–88. The sample size was selected as five for mechanical testing and the average values are presented. The morphology of the resin and the tensile fracture analysis of the composites were studied using JEOL JSM840A-(Japan) Scanning Electron Microscope (SEM).

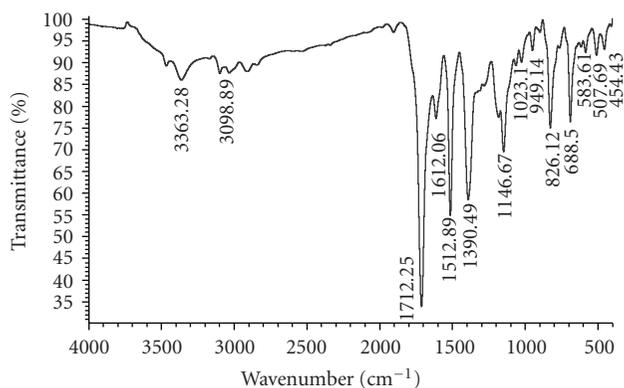
3. Results and Discussin

3.1. Morphological Studies. Morphological features of unmodified and modified cured resins are shown in Figures 3(a) and 3(b), respectively. The micrographs of unmodified BMI showed the presence of micro pores and white stripes indicating brittle phase, where as the micrographs of modified BMI showed very little micro pores and amorphous structure which may be due to the dense chemical links caused by interaction of MDA with BMI.

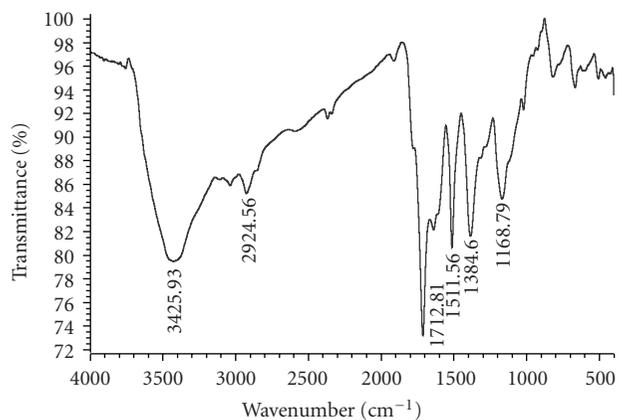
3.2. Characterization of Chain-Extended Bismaleimide Using FTIR. Figures 4(a), 4(b), and 4(c) show the spectra of unmodified, modified, and fully cured modified BMI systems, respectively. Figure 4(a) shows the characteristic absorption peaks of the imides at $1715 (\pm 10) \text{ cm}^{-1}$. The other important peaks are 1393 cm^{-1} and 1146 cm^{-1} which are due to the CNC groups present in the resin. A few more peaks are seen as expected due to C–H, CH_2 , and aromatic C–H, groups. In Figure 4(b), the formation of polymeric chain was observed by the formation of broad



(a)



(b)



(c)

FIGURE 4: FTIR Spectrum of (a) Unmodified BMI, (b) modified BMI and (c) modified cured BMI, respectively.

N-H absorption at 3378 cm^{-1} . The characteristic carbonyl absorption band of imides was observed at 1712 cm^{-1} . The expected double bond frequency of the maleimido end groups was overshadowed by the strong aromatic and carbonyl absorptions. The bands due to CNC and C-N stretching were observed at 1513 cm^{-1} and 1393 cm^{-1} , respectively. The bands most frequently utilized are imide absorption bands $\text{N } 1380 \pm 10 \text{ cm}^{-1}$ (C-N stretching) and the strongest band that occurs at 1720 cm^{-1} (C=O symmetrical stretching). The spectrum of the cured resin in which the

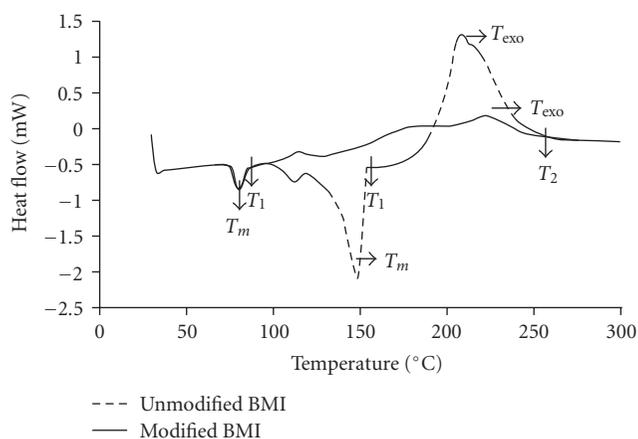


FIGURE 5: Comparison of the modified and unmodified BMI traces.

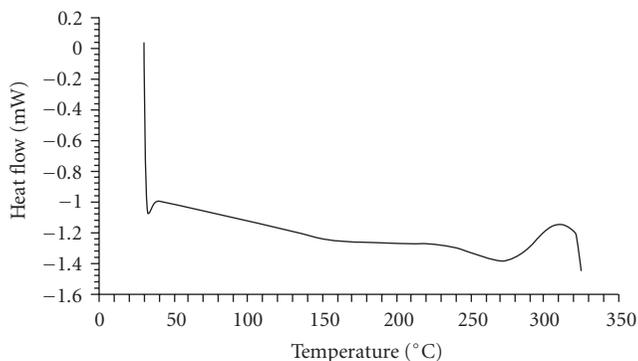


FIGURE 6: DSC thermogram of cured BMI.

absorption pattern in the range of $1000\text{--}500 \text{ cm}^{-1}$ showed reduced number of peaks indicating the cure completion of the BMI chains. In the same spectrum of Figure 4(c), a broad peak is seen at 3426 cm^{-1} which is due to the NH stretching vibration. The widening of the spectra may be due to the texture of the whole polymeric resin as a result of curing. A tentative assignment of the main absorption bands of FTIR is presented in Table 1

3.3. Cure Profile of the Unmodified and Modified Resin by DSC. The neat and the modified resin are evaluated for curing and glass transition temperature by DSC studies shown in Figure 5. In the case of the pure BMI uncured resin, the material displays transition temperature around 145°C followed immediately by two sharp melting peaks and a single cure exotherm from $180^{\circ}\text{C}\text{--}300^{\circ}\text{C}$. However, with the inclusion of the modifier MDA, the cure process becomes more complicated and multiple cure exotherms are observed as seen in Figure 5 and in the single endotherm is observed at $90^{\circ}\text{C}\text{--}95^{\circ}\text{C}$ followed by the curing exotherm. The shifting of the endotherm can be explained by the inclusion of the flexible and MDA which interferences with the close packing of the long-chain basic bismaleimide leading to the chain extension and shifting of the melting peak to lower temperature.

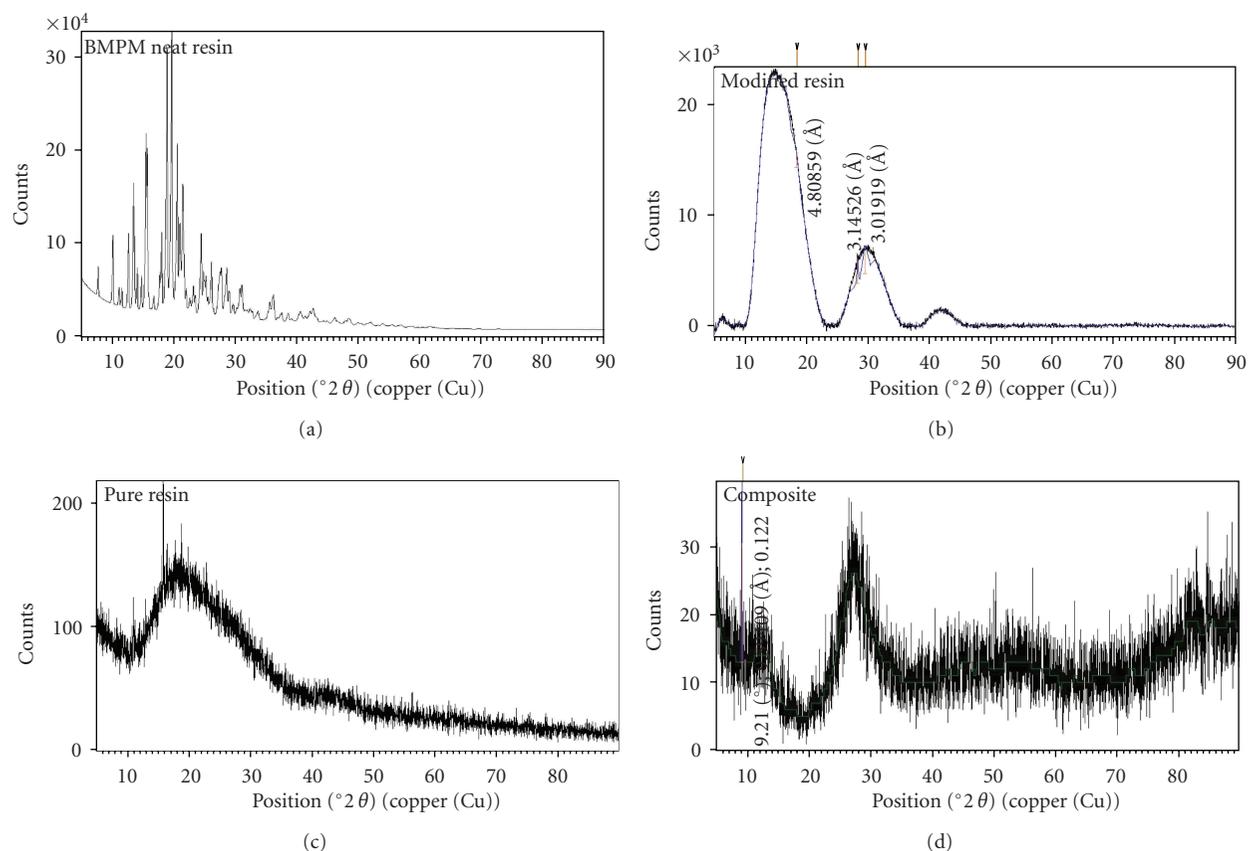


FIGURE 7: XRD spectrum for (a) unmodified BMI resin, (b) modified BMI resin, (c) modified cured BMI and (d) modified BMI/carbon composites.

The polymerization initial temperatures of these Bismaleimides are in the small temperature range. This indicates that the reactivity of double bonds of bismaleimide was not obviously affected by the chain length and the structure of Bismaleimide resins. However, the range between T_m and T_i (ΔT) is enlarged to 75°C from 5°C by insertion of flexible linkages and bulky group in to the structure of Bismaleimide resin with MDA. This means that they will have a large processing temperature range between their melting points and polymerization initial temperature, at which the matrix has good fluidity and wettability for fibre reinforced composite.

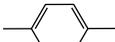
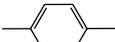
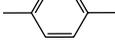
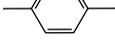
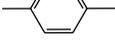
From the DSC thermogram of the modified cured resin system (Figure 6), T_g was found to be 260°C .

From the DSC traces (Figure 5) endothermic peak temperature (T_m), exothermic peak temperature (T_{exo}), onset of curing reaction (T_1), and completion of curing reaction (T_2) for both the BMI and modified BMI are determined and the same is presented in Table 2.

3.4. Crystallinity Studies of the Resin and the Composite by XRD. The crystallinity of the resin and the composites were examined by wide angle X ray diffraction. In the XRD analysis, the crystallinity parts give sharp narrow diffraction peaks and the amorphous component gives a broad peak (halo). However, excess of crystallinity causes brittleness

[22, 23]. From the first diffractogram Figure 7(a) which corresponds to unmodified BMI, it is seen that the material is highly crystalline having sharp narrow diffraction peaks in the region of $2\theta = 10$ to 40° showing that the neat resin is brittle. It is expected that electronic polarization occurs between electron-rich diamine and electron-deficient dianhydride regions in aromatic bismaleimide chains. Intermolecular charge polarization occurs when a group in one chain donates some of its electron density to an electron-deficient group in another chain. This phenomenon is called "charge transfer complexation". Due to the stiffness of linear rigid-rod and segmented rigid-rod polyimides, chain-chain interactions can occur over several consecutive repeat units. Consequently, the segments become aligned along their axes. It is believed that this alignment contributes to the formation of short-range order and crystallinity in the neat bismaleimide resin system as evidenced by the X-ray diffractogram in Figure 7(a). In Figure 7(a), the strong and weak intensities showed the prominent crystal planes and the subordinate crystal planes, respectively. The peak pattern is related to the structure and number of planes of diffraction. For the modified BMI resin the crystallite size has reduced as evident from FWHM (full width at half maximum) of the unmodified BMI as shown in Figure 7(b). The modified BMI resin powder diffractogram gives a broad peak showing that the BMI in the material is of

TABLE 1: Tentative assignment of the main absorption bands of FTIR.

Unmodified BMI	Intensity	Tentative assignment	Modified BMI	Intensity	Tentative assignment
3464	w	C=O		No C=O group	
	NH ₂ group not present		3389	m	NH ₂ Asymmetrical
3101	m	C-H maleimide		No C-H maleimide	
3038	w				
2900	vw	CH ₂	2925	vw	CH ₂
1709	vs	C=O	1712	vs	C=O
		asymmetrical stretching			asymmetrical stretching
1602	w		1613	w	
1511	vs		1513	m	
		Benzene ring			Benzene ring
1393	vs	-C-N-C- stretching	1393	s	C-N-C stretching
1146	vs	-C-N-C- maleimide	1148	s	C-N-C maleimide
1030	m		1025	w	
949	m		950	w	
833	s		827	s	
		maleimide			maleimide
688	s		689	m	
586	m	CH	586	m	CH

Vs—very strong, s—strong, m—medium, w—weak, and vw—very weak

TABLE 2: Results of the DSC analysis of the BMI Resin.

Resin	T_m^a (°C)	T_1^b (°C)	ΔT^c (°C)	T_{exo}^d (°C)	T_2 (°C) ^e	ΔH^f (J/g)
BMPM	165	170	5	241	268	226.61
BMPM/MDA	90	165	75	230	270	116.58

^a T_m , Melting point, ^b T_1 , polymerization initial temperature, ^c ΔT , $T_1 - T_m$, ^d T_{exo} , polymerization peak temperature, ^e T_2 , cure completion temperature, ^f ΔH , Enthalpy of polymerization from T_i to 300°C

TABLE 3: Mechanical properties of the developed composites.

CFRP laminate	Tensile strength Mpa	Flexural strength Mpa	Impact strength (kJ)
Modified BMI	554.46	615.38	0.21
Unmodified BMI	423.39	374.05	0.1333

semicrystalline nature as the chain structure has changed due to the inclusion of MDA leading to a semicrystalline form. This observation is reasonable because the presence of noncoplanar conformation of the MDA unit and bulky phenyl groups on the diphenylmethylen linkage decreased the intermolecular forces between the polymer chains, causing a decrease in crystallinity. However, the incorporation of the MDA substituent on the BMI unit effectively reduced the packing of the polymer chains. From the diffractogram it can

be inferred that the modifier has nicely reacted with the resin, thus reducing the number of peaks. The crystallite size has reduced, as evident from the increase in half-width of the peaks. In other cases, (Figures 7(c) and 7(d)) the materials have become amorphous due to the curing reaction and the introduction of the amorphous carbon fibres in to the system.

3.5. Mechanical Performance. Tensile, flexural, and impact properties of the specimens are presented in Table 3. The BMI modified by chain extender MDA increased the tensile strength of the laminates upto 31% compared to that of the unmodified BMI. In tensile loading, mainly voids act as defect sites that initiate crack and premature failure, resulting in lowering of the tensile properties [24]. The voids are due to the poor compatibility of the resin with the reinforcement fibres after curing [25]. Chain extender MDA has increased

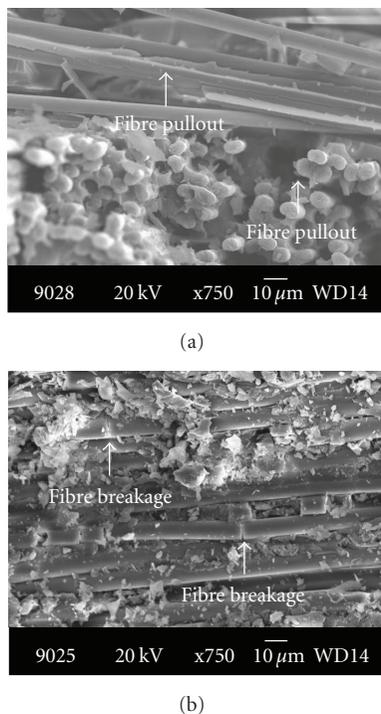


FIGURE 8: Fracture surface of (a) unmodified BMI/carbon laminates and (b) Modified BMI/carbon laminates, respectively.

the compatibility between the BMI and the carbon fibres resulting in increase of the tensile strength.

The flexural strength of the laminates was found to increase to a drastic amount of 64.52% for the modified BMI compared to that of the unmodified BMI. This difference can be explained by the lower degree of crystallinity of the modified resin and its laminates compared to unmodified one which is evident by the XRD analysis as shown in Figure 7. The greater the crystallinity in the polyimide, the lower the solubility and the processibility. It has been hypothesized that crystals act as physical crosslink sites which inhibit dissolution. In case of the modified resin-based composite, the inclusion of the chain extender reduced the crystalline/brittle nature to a semi crystalline/amorphous form and resulted in the increase in flexural strength of the composite.

The impact strength which is a matrix-dominated property was increased by around 50% for the modified BMI composite compared to that of the unmodified BMI composite. Unmodified BMI laminates have the rigid aromatic units and arranged tightly by the short methylene linkages make the BMI highly brittle and hence lower the impact strength. The chain extender improves processibility and toughness of BMI because of the increase in chain length, which tends to improve the toughness of the BMI. On the other hand, BMI reacts with bis-nucleophilic species to form the crosslink structure via Michael-addition reaction. MDA are the favoured bis-nucleophilics because they have high basicity and can be engaged in the nonstoichiometric Michael addition reaction between BMI and aromatic MDA as supported by the FTIR analysis (Figure 4).

3.6. Fractography. Figures 8(a) and 8(b) show the tensile fracture surfaces of unmodified BMI/carbon composites and modified BMI/carbon composites, respectively. Unmodified composites show greater degree of fibre pullout (Figure 8(a)), which is due to the poor interfacial strength. The modified composites showed only fibre breakage due to better interfacial strength. The resin-fibre bonds have broken and lumps of resin are observed in amorphous form, Figure 8(b).

4. Conclusions

Modified BMI was synthesised using BMI and chain extender MDA (BMI:Extender = 3:2). Modified BMI/Carbon composite specimens were prepared. The specimens were characterised for crystallinity, chemical structure, and thermal and mechanical behaviour. Based on the experimental results the following conclusions were drawn.

From the FTIR results, the formation of polymeric chain was observed by the presence of broad N-H absorption band. Modification of BMI showed several advantages. They are: decrease in the number of pores, increase of matrix/fibre interfacial strength, better processibility, and transformation to semicrystalline structure in resin and amorphous structure in the composites, significant increase of tensile, flexural and impact properties. These improvements suggest that modification of BMI is a successful method to enhance the applicability of BMI and its composites for several high-end areas.

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