

Curing behavior and processability of BMI/3-APN system for advanced glass fiber composite laminates

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ABSTRACT: The prepolymers containing bismaleimide (BMI) and 3-aminophenoxyphthalonitrile (3-APN) were prepared through simple solution prepolymerization, and the corresponding curing behaviors and processability were investigated by differential scanning calorimetry and dynamic rheological analysis. The results showed that the processability of the prepolymers could be controlled by temperature and time on processing, also depended on the relative content of 3-APN and BMI. The possible curing reactions of the prepolymers were studied by Fourier transform infrared spectroscopy, which involved the Michael addition between BMI and 3-APN and self-polymerization of BMI or 3-APN. The resulting polymers displayed high thermo-oxidative stabilities ($T_{5\%} > 425\text{ }^{\circ}\text{C}$) and good adhesion capability. Furthermore, BMI/3-APN systems were employed to prepare BMI/3-APN/glass fiber (GF) composite laminates and their morphological, mechanical, and electrical stable properties were also investigated. The BMI/3-APN/GF laminates exhibited the improvement of the mechanical properties (the maximum flexural strength is 633.5 MPa and flexural modulus is 38.7 GPa) compared with pristine BMI/GF laminates because of the strong interfacial adhesions between GF and matrices, which was confirmed with SEM observations. This study provides a concise strategy for diversifying the preparation of BMI/3-APN prepolymers to obtain advanced GF composite laminates with various properties which have potential applications in industrial manufacture or electronic circuit, and so on. © 2016 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* **2016**, *133*, 43640.

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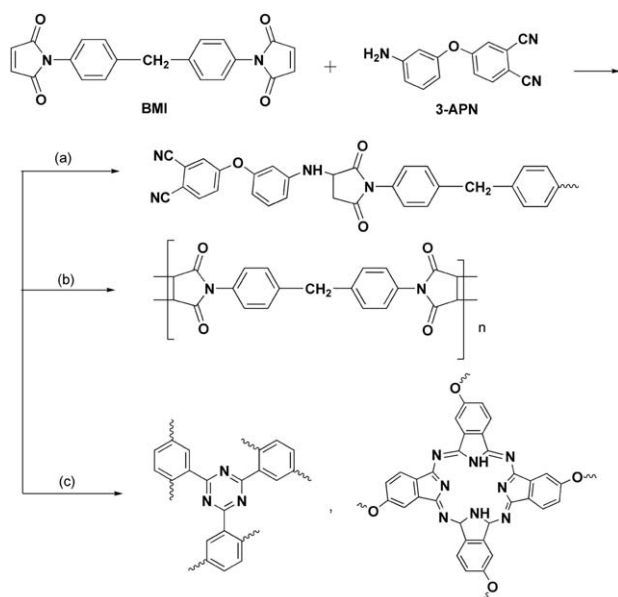
INTRODUCTION

Over the past few decades, high-performance thermosets have attracted much attention because of their wide applications in a variety of areas, such as electronics, aerospace, and other industrials. Bismaleimides (BMI) represent an important class of thermosetting resin^{1,2} and have been extensively accepted as matrix resins which dominate over the other polymer matrices due to their high performance-to-cost ratio.³ They can undergo self-addition reactions to achieve highly cross-linked networks without the formation of volatile byproducts, offering excellent thermal and oxidative stability, superior thermomechanical properties, and good flame retardance.⁴ However, the pure BMI resins are brittle as a result of the high cross-link density, poor solubility, poor processability, and narrow processing window and are rarely used directly.

Many investigations have focused on overcoming these problems. Thermoset/thermoset blend is a promising approach due

to the synergistic effect exists in inter-cross-linked or interpenetrated polymer networks, producing considerable improvements in some properties.^{5–9} For example, the modified resin systems based on BMI and epoxy have shown appropriate properties which improve the toughness and process of BMI resin and the thermal properties of epoxy.^{7,8} Although most methods have been developed to improve various properties, there are very few successful examples based on BMI as adhesives.^{5,10,11}

Phthalonitriles as high-temperature-resistant polymers have received increasing attention.^{12,13} It can undergo thermal polymerization to obtain a class of high-performance resins, and the polymerization occurs through the cyano groups by an addition mechanism to afford heterocyclic cross-linked products, such as triazine and phthalocyanine.^{14,15} It is worth mentioning that there is a small amount of cyano residue after thermal polymerization through our previous research,^{16,17} and it is well known that the cyano group as “strongly polar” can improve the adhesive properties. Therefore, we speculated that introducing



Scheme 1. The possible reactions between BMI and 3-APN.

phthalonitrile into BMI would be high-temperature-resistant adhesives with other excellent properties.

A large variety of addition of the active double bond of BMI has been reported, mainly depends on the active hydrogen atoms from the amino or hydroxyl groups. Some representative examples which involve the addition reaction between diamine or allyl-type compounds and BMI have been the focus of research.^{18,19} Based on the abovementioned, we intended to introduce an amino in phthalonitrile as bridge group connected to BMI. The phthalonitrile monomer containing an amino group (3-aminophenoxypthalonitrile) was synthesized via nucleophilic substitution reaction.²⁰ In this article, we report the *in situ* addition of 3-APN to BMI resins in different proportions. The possible reactions process between 3-APN and BMI were presented in Scheme 1. Differential scanning calorimetry (DSC) and dynamic rheological analysis (DRA) were used to establish the curing behaviors and processability of the blends. The thermostability and adhesive properties of the cured resins were discussed. Furthermore, BMI/3-APN system was applied to BMI/3-APN/glass fiber (GF) composite laminates through hot-pressed method. The corresponding mechanical, dielectric, and adhesive properties of BMI/3-APN/GF composite laminates were also studied.

EXPERIMENTAL

Materials

Unless otherwise noted, all reagents were obtained from commercial suppliers and used without further purification. The monomer 3-APN was synthesized by our previous work.²⁰

Preparation of BMI/3-APN Prepolymers

A mixture containing 3-APN and NMP was placed to a three-neck flask equipped with a mechanical stirrer and condenser pipe in the oil bath. The solution was then heated at 200 °C for 2 h. After cooling to 150 °C, BMI was added to the solution and stirred for 30 min while the molar ratios of BMI and 3-

APN were 1:1, 1:2, and 1:3, respectively. The reaction mixture was cooled to ambient temperature. Then it was poured into deionized water, accompanying by a large precipitated solids. The precipitate was filtered off, washed with deionized water, and dried in a vacuum oven overnight at 80 °C.

Curing of the BMI/3-APN Prepolymers

The BMI/3-APN prepolymers were placed on the folded aluminum mold, and then heated in an oven at 180 °C for 2 h, 220 °C for 2 h, 260 °C for 2 h, 300 °C for 2 h, and 320 °C for 2 h, respectively.

Preparation of Lap Shear Strength Test Specimens

The lap shear strength test specimens of BMI/3-APN were prepared according to ISO 4587-2003. The steel plates were polished with emery paper and ultrasonic cleaned with alcohol. The test specimens mentioned above should be gummed at least two times to achieve a coating thickness of around 0.15–0.2 mm. Two pieces of metal sheets gummed with BMI/3-APN prepolymer coating were stuck together in such a manner as to obtain an overlap of 12.5 mm and used to ensure suitable pressure of 0.1–0.5 MPa during the curing process. Finally, the single lap-joint specimen fixed with a fixture was then placed into a vacuum oven heated at 180 °C for 2 h, 230 °C for 2 h, and 250 °C for 2 h.

Preparation of BMI/3-APN/GF Composite Laminates

As-prepared BMI/3-APN prepolymer viscous solution was used directly to impregnate the GF fabric. The GF fabric (10 × 10 cm²) was brush-coated with the viscous solution obtained above and dried at room temperature for 24 h. It was designed to give a prepreg of 35% polymer matrix and 65% GF by weight. Ten layers of GF prepreg fabric were placed in a stainless-steel mold and hot-pressed under a pressure of 20 MPa at 180 °C for 2 h, 220 °C for 2 h, 260 °C for 2 h, 300 °C for 2 h, and 320 °C for 2 h, respectively.

Characterization

DSC analysis was performed by TA Instruments Modulated DSC-Q100 at a heating rate of 10 °C/min and a nitrogen flow rate of 50 mL/min. DRA was performed using TA Instruments Rheometer AR-G2 with a frequency of 1 Hz at different temperatures in air. The samples (0.5–1 g) were melted between 25-mm-diameter parallel plates with an environmental testing chamber of the rheometer. FTIR spectra were recorded with Shimadzu FTIR8400S Fourier Transform Infrared spectrometer in KBr pellets between 4000 and 400 cm⁻¹ in air. Thermal gravimetric analysis (TGA) was performed on a TA Instruments TGA Q50 with a heating rate of 20 °C/min (under nitrogen or air) and a purge of 40 mL/min. Mechanical properties of the composite laminates were investigated on a SANS CMT 6104 Series Desktop Electromechanical Universal Testing Machine. Dielectric properties (dielectric constant and dielectric loss) are monitored according to ASTM D150 using a TH2819A precision LCR meter (Tonghui Electronic Co., Ltd.). Experiments are performed at frequencies from 100 Hz to 200 kHz at 25 °C. The morphology of the fractured surfaces of the composites was observed by SEM (JSM25900LV) operating at 5 kV.

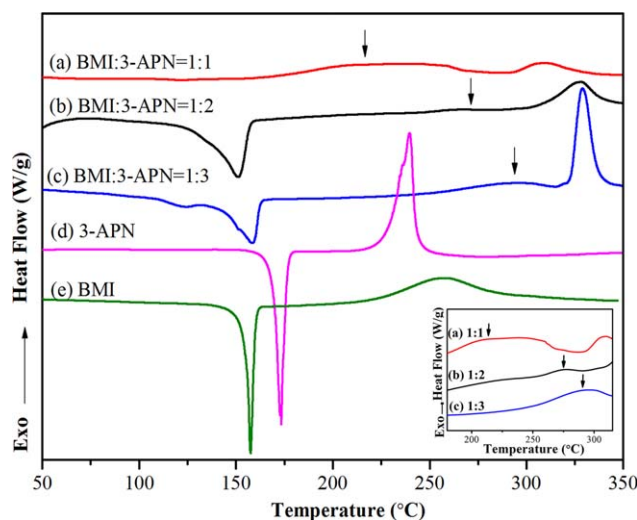


Figure 1. DSC curves of BMI/3-APN prepolymers with various molar ratios: (a) 1:1, (b) 1:2, (c) 1:3, (d) 3-APN, and (e) BMI. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

RESULTS AND DISCUSSION

Curing Behaviors of BMI/3-APN Prepolymers

The curing behaviors of BMI/3-APN prepolymers were studied by DSC analysis. The corresponding DSC curves were presented in Figure 1 and the main data were summarized in Table I. For pristine BMI, melting occurs about 157.7 °C and homopolymerization is started at 220.5 °C and continues up to 304.3 °C. The curing mechanism of BMI is known to be self-polymerization via the double bonds of the maleimide groups [Scheme 1(b)].²¹ While for pristine 3-APN, there is an endothermic peak at 173.4 °C which belongs to its melting peak and the exothermic peak occurs at 239.6 °C which was attributed to the curing peak of cyano group. When BMI and 3-APN were blended in various molar ratios to obtain BMI/3-APN prepolymers, the samples showed a weak and broad polymerization exotherm spanning ranging from 168.4 to 252.3 °C, resulting from the polymerization of the BMI double bonds [Scheme 1(b)] and Michael addition of the primary amines to double bond in the maleimide groups [Scheme 1(a)].²² As the Michael addition and self-polymerization of BMI take place at a lower temperature than the polymerization of phthalonitrile,²³ a second exotherm peak appear at higher temperature (>300 °C) which belong to the polymerization of cyano group.²⁴ Based on the above results, the

Table I. Thermal Properties of BMI/3-APN Prepolymers

BMI/3-APN	T_m (°C)	T_i (°C)	T_p (°C)	T_p' (°C)	ΔH_1 (J/g)	ΔH_2 (J/g)
1:1	-	168.4	215.9	309.8	64.6	21.9
1:2	151.2	239.2	277.5	327.5	19.5	48.6
1:3	158.6	252.3	290.4	329.0	15.5	74.3
BMI	157.7	220.5	258.0	-	87.4	-
3-APN	173.4	229.8	-	239.6	90.3	-

T_m , melting point; T_i , initial curing temperature; T_p , peak curing temperature; T_p' , curing peak temperature of nitrile.

prepolymers exhibit wider processing window which contribute to the improvement of the processing property.

Characterization of BMI/3-APN Polymers

To further understand the curing procedure of BMI/3-APN, the BMI/3-APN prepolymer (the molar ratio = 1:2) was chosen as an example. The obtained BMI/3-APN polymers heated at 180 °C, 260 °C, and 320 °C were investigated by FTIR spectra, as shown in Figure 2. The corresponding characteristic absorption peaks of BMI and 3-APN are marked. In Figure 2, for 3-APN, the intensity characteristic absorption band at 3459 and 3366 cm^{-1} represents the stretching vibrations of the primary amine ($-\text{NH}_2$) and 2237 cm^{-1} is assigned to the stretching vibrations of cyano groups ($-\text{CN}$).²⁴ While for BMI, the peaks observed at 837 cm^{-1} and 685 cm^{-1} are corresponded to the stretching vibrations of $\text{C}=\text{C}$ and 3054 cm^{-1} is the stretching vibrations of $\text{C}=\text{C}-\text{H}$.²⁵ With the increase of temperature, the characteristic absorption peaks of primary amine and $\text{C}=\text{C}$ were disappeared simultaneously indicated the possible reaction between BMI and 3-APN [shown in Scheme 1(a,b)], while the polymerization of the BMI double bonds could not be excluded in this process. Notably, the absorption band at 3340 cm^{-1} is attributed to stretching vibration of secondary amine $\text{N}-\text{H}$, which further confirmed the addition reaction between BMI and 3-APN.¹⁵ Meanwhile, the intensity absorption band at 2237 cm^{-1} assigning to cyano groups became lower with the increase of temperature, suggested the high polymerization degree of cyano groups. However, it is clearly seen that a small amount of cyano group was existed which is agreed with the previous reports.^{16,17} Additionally, triazine rings (1360 cm^{-1}) or phthalocyanine rings (1010 cm^{-1}) were formed through polymerization of cyano groups.¹⁵ Therefore, the FTIR spectra of the polymers confirmed the aforementioned curing process, including additional reaction between BMI and 3-APN, homopolymerization of BMI and polymerization of $-\text{CN}$ groups.

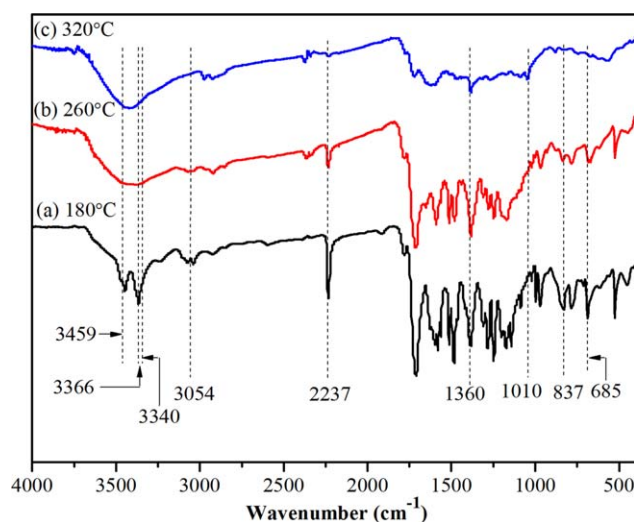


Figure 2. FTIR spectra of BMI/3-APN = 1:2 on curing procedure: (a) 180 °C, (b) 260 °C, and (c) 320 °C. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

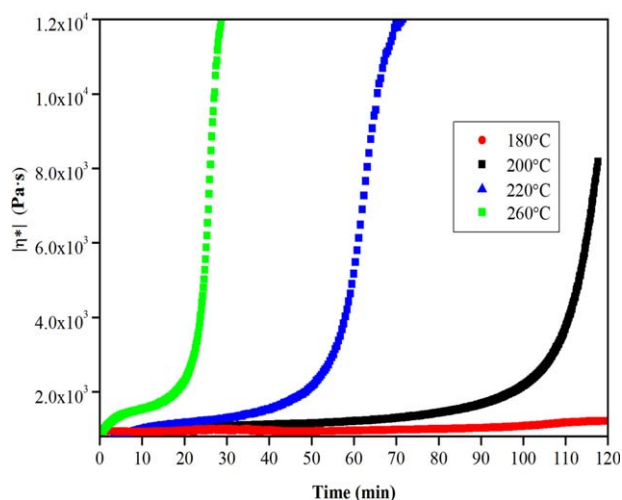


Figure 3. Time sweep viscosity curves of the BMI/3-APN = 1:2 prepolymer at different temperatures. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Processability of BMI/3-APN Prepolymers

The viscosity changes are very important for studying the processability of prepolymer. The complex melt viscosity changes accompanying the curing reaction were monitored by viscosity studies, as shown in Figures 3 and 4. To determine the processing temperature and time for BMI/3-APN prepolymer, the viscosity change of the BMI/3-APN prepolymer (the molar ratio = 1:2) was measured as a function of time at different temperatures. In Figure 3, the viscosity was low before the curing reaction occurred and then rapidly increased after the curing reaction started.²⁶ Moreover, the increasing rate of viscosity became faster with the temperature increases. It suggested that high temperature has a great tendency to accelerate the polymerization reaction of BMI/3-APN prepolymers. Combined with Table II, it can be seen that the increase of the viscosity for BMI/3-APN prepolymers took a rather long or short time at 180, 200, and 260 °C. Thus, 220 °C was a proper processing temperature for BMI/3-APN prepolymers.

For comparison, isothermal viscosity measurements based on different proportions of BMI/3-APN prepolymers were investigated. As depicted in Figure 4, it can be observed that the increasing trend of complex viscosity was coincident with the increasing content of BMI. It was suggested that the content of BMI could accelerate the polymerization. It may be because the Michael addition and self-polymerization of BMI are more

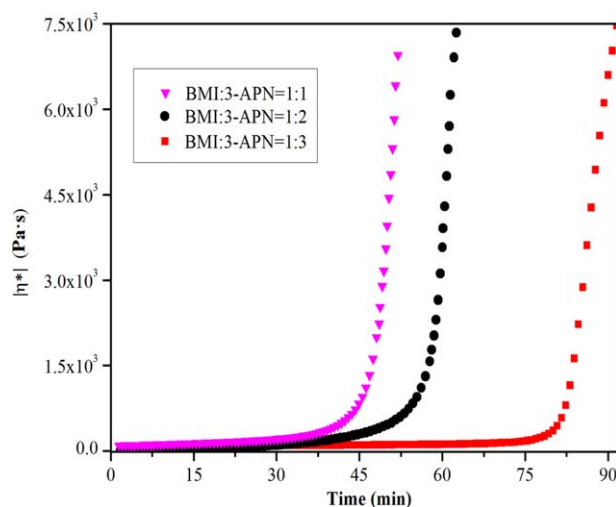


Figure 4. Time sweep viscosity curves of BMI/3-APN prepolymers with various molar ratios at 220 °C. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

prone to occur at low temperatures. Overall, results revealed that the blends showed a wide processing window and the proper polymerization rate, which have important applications in resin infusion processes or resin transfer molding.

Thermostability of BMI/3-APN Polymers

Thermal stability of the cured BMI/3-APN polymers under N₂ atmosphere was evaluated from TGA (Figure 5) and the main results were summarized in Table III. As can be seen from Figure 5, compared with BMI and 3-APN, the thermostability of BMI/3-APN polymers decreased slightly. However, there was no observable weight loss under 380 °C which indicated that all the polymers still have good thermal stability. In addition, the char yield (Y_c) of the BMI/3-APN is significantly higher than those of 3-APN and BMI as well as the values increases as the content of 3-APN increases. The previous works demonstrated that the high Y_c value would give the resin system excellent flame retardant properties because the thick charred layer is an excellent thermal barrier that could delay and prevent thermal degradation.²⁷ The temperature of the maximum degradation rate (T_{max}) of each polymer is about 450 °C, demonstrating that the polymers have similar degradation processes. In addition, the $T_{5\%}$ and $T_{10\%}$ values of the polymers are closely related to the content of BMI. It may be attributed to the formation of phthalocyanine rings or triazine rings with higher thermal stability through the cross-linking of the cyano groups.¹⁵ From the

Table II. Gel-Time of BMI/3-APN Prepolymers with Various Molar Ratios at Different Temperatures

Molar ratios BMI/3-APN	Temperatures (°C)	Initial viscosity (Pa·s)	Viscosity of gel-time (Pa·s)	Gel-time (min)
1:1	220	18.01	950.2	39
1:2	180	1.22	-	-
	200	8.72	2075.2	105
	220	28.6	1356.1	51
	260	67.7	1738.1	18
1:3	220	16.91	886.3	78

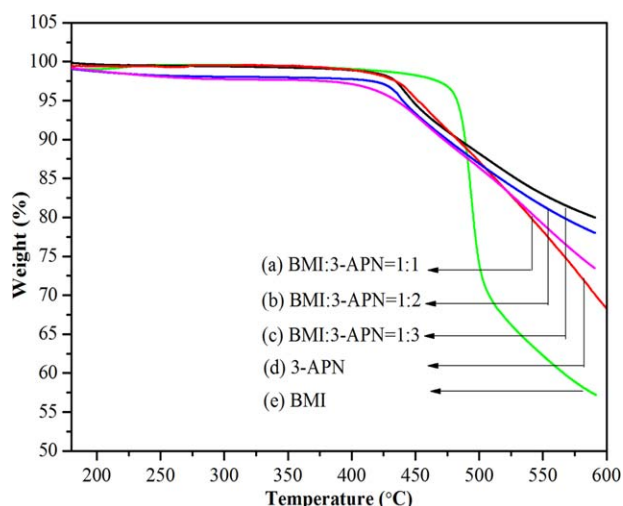


Figure 5. TGA curves of BMI/3-APN polymers with various molar ratios under N_2 atmosphere: (a) 1:1, (b) 1:2, (c) 1:3, (d) 3-APN, and (e) BMI. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

above, we can come to the conclusion that all BMI/3-APN polymers show good thermostability.

Adhesive Property of BMI/3-APN Polymers

From FTIR spectra, it is seen that there is a small amount of cyano residue after thermal polymerization. We speculated the residue cyano group may improve the adhesive properties. Thus, shear strength of the adhesive-bonded single lap-joint specimens are studied. The molar ratio of BMI and 3-APN was investigated to achieve optimum bonding performance. As shown in Figure 6, the lap shear strength increased from 3.2 to 5.3 MPa at room temperature which depend on the increase of BMI content. The highest shear strength is up to 5.3 MPa when the molar ratio of BMI:3-APN = 1:1. Compared with BMI and 3-APN, BMI/3-APN polymers showed the improvement of adhesive property. It may be explained by the formation of cross-linking structure through the copolymerization behavior of BMI and 3-APN to obtain the used adhesive sample of a strong and durable bond.²⁸ On the other hand, a small amount of cyano residue after thermal polymerization could improve

Table III. Thermal Stabilities of BMI/3-APN Polymer with Various Molar Ratios

Molar ratios BMI/3-APN	$T_{5\%}$ (°C)	$T_{10\%}$ (°C)	T_{max} (°C)	Y_c (wt %)
BMI	487.8	493.4	498.7	57.1
1:1	426.3	465.5	453.2	74.8
1:2	436.5	478.0	450.0	77.3
1:3	446.3	486.4	447.2	79.1
3-APN	446.6	508.1	462.5	68.1

$T_{5\%}$, the temperatures at weight loss of 5%.

$T_{10\%}$, the temperatures at weight loss of 10%.

T_{max} , the temperatures of the maximum degradation rate.

Y_c , char yield at 600 °C.

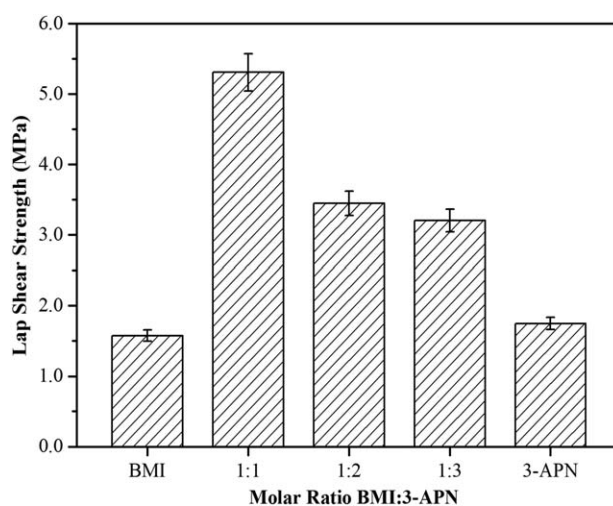


Figure 6. Lap shear strength of BMI/3-APN bonded joints with various molar ratios.

the adhesive properties. In all, results indicated that the BMI/3-APN resin may be used as adhesive.

SEM of BMI/3-APN/GF Composite Laminates

In the previous parts, BMI/3-APN resin with good thermal stability has been obtained. Meanwhile, it has been proven that it may be as adhesive. Then BMI/3-APN system was applied to BMI/3-APN/GF composite laminates. The investigations of curing behaviors and processability based on BMI/3-APN blends can provide us with effective molding procedures to prepare BMI/3-APN/GF composite laminates. First, the SEM images of the interface structures based on BMI/3-APN/GF composites laminates at the molar ratio of BMI:3-APN = 1:1 are investigated. In Figure 7, the bonding interface between the resins and GF fabric was indicated by red arrows. It is clearly showed that the GF fabric was homogeneously covered by the resins. What's more, there are strong interfacial adhesions between fiber and

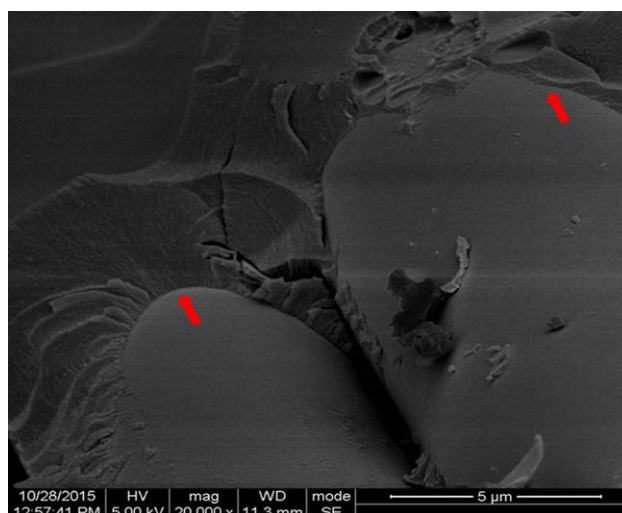


Figure 7. SEM images of interface structures of BMI/3-APN/GF composite laminates. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

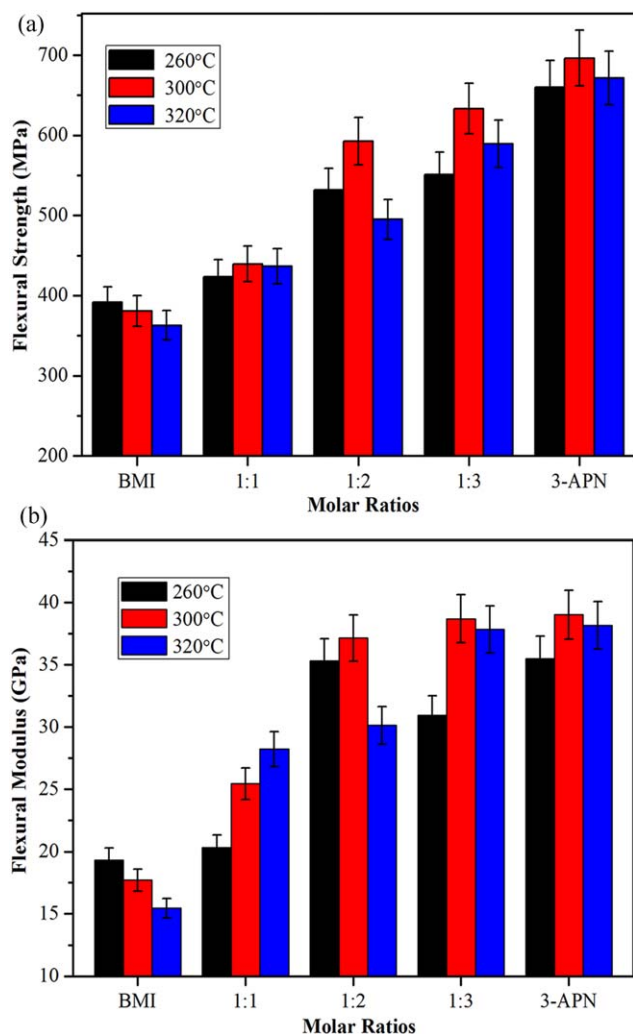


Figure 8. Mechanical property of various molar ratios of BMI/3-APN/GF composite laminates at different temperatures: (a) flexural strength and (b) flexural modulus. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

resin matrix as expected. These suggest that BMI/3-APN/GF composite laminates may lead to other excellent properties.

Mechanical Property of BMI/3-APN/GF Composite Laminates

The mechanical property of BMI/3-APN/GF composites with the various content of 3-APN and different post-treatment temperatures are shown in Figure 8(a,b). The three-point bend test was used to determine the flexural strength and modulus of the composites. From Figure 8, for pristine BMI, brittleness is a major hazard, leading to inferior mechanical property. However, in BMI/3-APN system, it is clearly observed that the flexural strength and flexural modulus increased with the increase of 3-APN, and the maximum flexural strength (633.5 MPa) and flexural modulus (38.7 GPa) are obtained when the BMI/3-APN molar ratio is 1:3. It was reported that the enhancement of flexural strength and modulus may be attributed to the network structure of high cross-linking density.²⁹ In BMI/3-APN system, the synergistic effects of the self-polymerization of 3-APN that formed phthalocyanine rings or triazine rings, Michael addition reaction between primary amine and

imide ring, and homopolymerization of BMI would affect the mechanical property of the composites. On the other hand, different post-treatment temperatures also show different results. After heated to 300 °C, all the molar ratios of the composite laminates show better mechanical property than the other temperatures. Results indicated that BMI/3-APN/GF composite laminates exhibit the enhancements of mechanical properties due to the strong interfacial adhesions between GF and matrices, which is consistent with the SEM observation.

Electrical Properties of BMI/3-APN/GF Composite Laminates

Figure 9 shows the dependence of dielectric constant and dielectric loss on frequency for various molar ratios of BMI/3-APN/GF composite laminates at room temperature. The dielectric properties of cured polymers usually depend on the orientation and relaxation of dipoles which is accompanied by the movement of polymer chain segments.³⁰ The BMI/3-APN/GF composite laminates show a good stability on its dielectric constant over a wide frequency range from 1 kHz to 200 kHz. It is worth mentioning that the

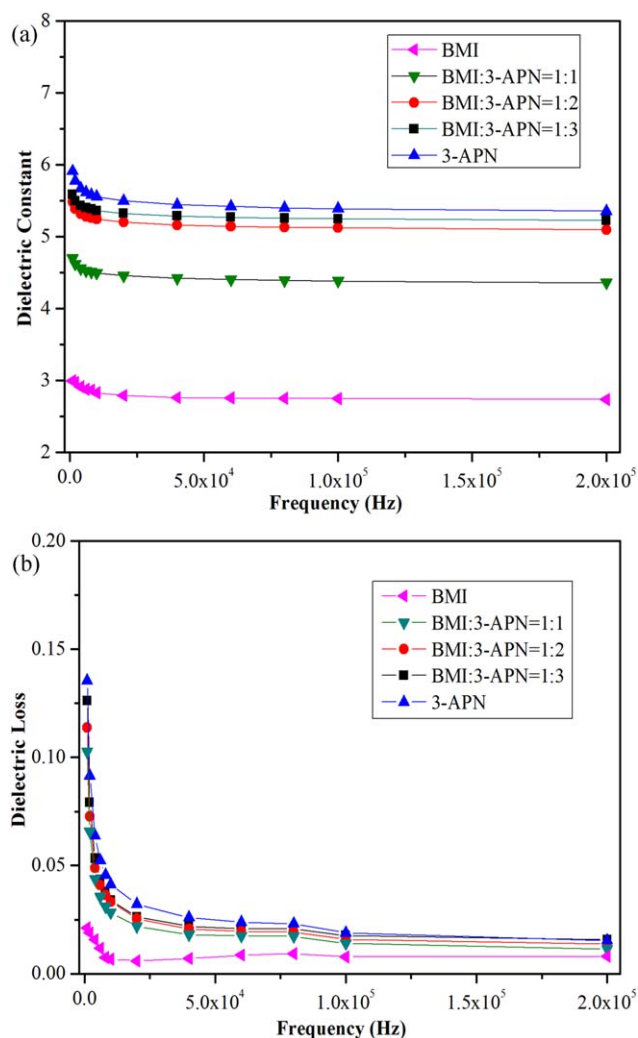


Figure 9. Dependence of dielectric constant and dielectric loss on frequency for various molar ratios of BMI/3-APN/GF composite laminates: (a) dielectric constant and (b) dielectric loss. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

electrical properties on composite laminates are affected by the content of BMI. For the BMI/3-APN molar ratio is 1:1, the dielectric constant and dielectric loss at 100 kHz are 4.38 and 0.017, respectively. These are much lower than those of 3-APN composite laminates, which may be attributed to the cross-linking structure of the resins. As mentioned above, the involved Michael addition reaction between BMI and 3-APN formed a large molar volume to a molecule and noncoplanar structures, which are beneficial in reducing the dielectric constant of the molecule.³¹ Therefore, the wide frequency and dielectric stability of BMI/3-APN/GF composite laminates may be attractive for practical applications.

CONCLUSIONS

The BMI/3-APN prepolymers were prepared through solution prepolymerization. The curing behaviors and processability of BMI/3-APN prepolymers were investigated. The possible curing reactions were discussed. Results demonstrated that the curing reactions involved the Michael addition between the primary amines of 3-APN and double bond in BMI, self-polymerization of BMI, and the polymerization of phthalonitrile. The processability of the prepolymers could be controlled by temperature and time on processing. The components of the polymers depended on the relative content of 3-APN and BMI. As discussed above, the polymers possess thermo-oxidative stabilities, high char yield in nitrogen, and adhesion capability. The GF-filled BMI/3-APN composite laminates were prepared and their mechanical and dielectric properties were investigated. The SEM showed the strong interfacial adhesion between fiber and resin matrix which may be attributed to the cross-linking structures. The BMI/3-APN/GF composite laminates have the maximum flexural strength (633.5 MPa) and modulus (38.7 GPa) when the BMI/3-APN molar ratio is 1:3 and exhibit a good stability on its dielectric constant over a wide frequency range. These characteristics could enable the BMI/3-APN/GF composites to apply in industrial manufacture or electronic circuit, and so on. In conclusion, this method can be used in future for rapid screening of various properties of prepolymers with different proportions to obtain advanced GF composite laminates.

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