Properties of phthalonitrile monomer blends and thermosetting phthalonitrile copolymers

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Abstract

Binary blends of biphenyl phthalonitrile and the $n = 4$ oligomeric phthalonitrile were prepared and characterized by differential scanning calorimetry and rheology studies. The blended phthalonitriles also were polymerized from the melt in the presence of bis[4-(4-aminophenoxy) phenyl]sulfone and the dynamic mechanical and thermal properties of the resulting copolymers were investigated. The properties of the phthalonitrile blends and copolymers were compared with those of the neat polymers. The phthalonitrile blends were found to have larger processing windows relative to that observed for the biphenyl phthalonitrile. The size of the processing window depended on the $n = 4$ phthalonitrile content in the blend. Dynamic mechanical measurements and thermogravimetric analysis showed that temperatures up to 425 °C were necessary to completely cure the phthalonitrile copolymers. The dynamic mechanical measurements also revealed that the fully-cured phthalonitrile copolymers did not soften or exhibit a glass transition temperature upon heating to 450 °C. Thermogravimetric analysis showed that the phthalonitrile copolymers exhibited excellent thermal stability along with long-term thermo-oxidative stability.
Published by Elsevier Ltd.

Keywords: Phthalonitrile blends; Thermosetting resin; Processability

1. Introduction

Besides the phenylethynyl-terminated polyimides [1], the phthalonitriles are one of the leading candidates for use in composite structures where thermally stable, high performance resins are required. The phthalonitriles constitute a class of high-temperature polymers that has a number of exceptional properties such as high glass transition temperatures (T_g), outstanding thermal and thermo-oxidative stability, excellent mechanical properties, good moisture resistance and superior fire resistance [2–12]. In addition, phthalonitrile (PN) monomers and oligomers possess a low complex melt viscosity (0.01–1 Pa s) which enables facile processing by the cost-effective, non-autoclavable processing techniques such as resin transfer molding, resin infusion molding and filament winding.

The combination of good processability and excellent high-temperature properties makes the phthalonitriles attractive for many military and civilian advanced technological applications.

4,4′-Bis(3,4-dicyanophenoxy)biphenyl (biphenyl PN) (Fig. 1(a)) was the first phthalonitrile-based, high-temperature resin reported [2,3]. Subsequently, other phthalonitrile monomers were developed with a variety of linkages incorporated between the terminal phthalonitrile units [4–6,10,11]. To achieve enhanced processability while maintaining high thermo-oxidative stability, the oligomeric $n = 4$ phthalonitrile ($n = 4$ PN) monomer (Fig. 1(b)) was recently designed and synthesized with *meta*-substituted, aromatic ether linkages [12].

The biphenyl PN and $n = 4$ PN monomers differ in a number of important ways. Biphenyl PN monomer is a pure material with a relatively high degree of crystallinity and a high melting point (∼233 °C). In contrast, the $n = 4$ PN monomer

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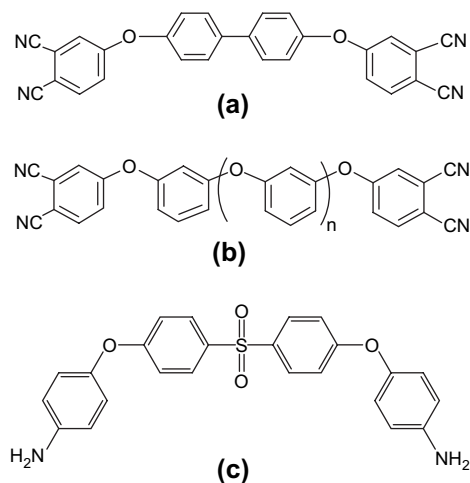*E-mail address:* dawn.dominguez@nrl.navy.mil (D.D. Dominguez).

Fig. 1. Chemical structures of (a) 4,4′-bis(3,4-dicyanophenoxy)biphenyl, (b) oligomeric phthalonitrile monomers and (c) the curing additive, bis[4-(4-aminophenoxy)phenyl]sulfone (*p*-BAPS).

is an oligomeric mixture of components with variable length of multiple aromatic ether linkages interconnecting the terminal phthalonitrile units. As such, the $n = 4$ PN monomer has a lower degree of crystallinity and a much lower melting point ($\sim 40\ ^{\circ}$C). Another significant difference between the monomers is their rigidity. The biphenyl PN exhibits greater rigidity due to the presence of the *para*-substituted biphenyl group in the backbone whereas the $n = 4$ PN oligomer has the flexibility introduced in the *meta*-catenated aromatic ether spacer.

Both biphenyl PN and the oligomeric $n = 4$ phthalonitrile monomers are commercially available and are currently being evaluated in industry. For practical applications, some shortcomings of these resin systems have been identified. These include a small processing window, defined as the temperature difference between the melting point of the phthalonitrile and the cure temperature, for the biphenyl PN ($\sim 20-30\ ^{\circ}$C) and the high temperature ($425\ ^{\circ}$C) required to fully cure the $n = 4$ PN. Another drawback of these resins is their brittleness.

The formation of blends and copolymers is one way of modifying the polymer properties when an individual material does not quite meet all the requirements for a specific application [13−19]. As part of an ongoing effort to develop useful high-temperature polymeric materials, biphenyl PN and the oligomeric $n = 4$ PN were blended and thermally polymerized and the properties of the resulting crosslinked copolymers were evaluated. The objective of this effort was to determine whether the aforementioned limitations of the individual resins could be overcome by blending the components and forming the corresponding copolymers without sacrificing the processability and the necessary high-temperature properties. The results of the study on the thermal and rheological behavior of biphenyl PN/$n = 4$ PN blended monomers and the thermal and dynamic mechanical properties of biphenyl PN/$n = 4$ PN copolymers with varying oligomeric $n = 4$ PN contents are presented in this paper.

2. Experimental

2.1. Materials

4,4′-Bis(3,4-dicyanophenoxy)biphenyl and $n = 4$ oligomeric phthalonitrile monomers were used as-received from Dow Chemical Co. and JFC Technologies, respectively. Bis[4-(4-aminophenoxy)phenyl]sulfone (*p*-BAPS, Fig. 1(c)) was acquired from ChrisKev Co. *p*-BAPS was dried overnight at 175 $^{\circ}$C under vacuum before use.

2.2. Preparation of PN blends, prepolymers, polymers and copolymers

PN blends were prepared by mixing predetermined concentrations of biphenyl PN monomer and the $n = 4$ PN oligomer in powdered form under ambient conditions. The compositions of the physical mixtures were 75:25, 50:50 and 25:75 in which the numbers represent the weight percentages of the biphenyl PN monomer and the $n = 4$ PN oligomer, respectively. B-stage resins (prepolymers) of the biphenyl PN/$n = 4$ PN blends and the $n = 4$ PN oligomer were prepared in air by adding 5 mol% *p*-BAPS curing additive to each melt at 250−265 $^{\circ}$C with vigorous stirring. After 15−20 min, the reaction between the diamine and the phthalonitrile end groups was quenched by rapidly cooling the melt to room temperature. The prepolymers were pulverized for use in viscosity studies. Rectangular solid samples of the biphenyl PN and the $n = 4$ PN polymers and copolymers were prepared in aluminum molds with cavity dimensions 65 mm $\times$ 13 mm $\times$ 4 mm. Samples (3 g) of a neat phthalonitrile or a PN blend were initially melted and degassed for several hours. After the initial degassing, *p*-BAPS (5 mol%) was added to the melt with stirring for about 5 min. The molten mixture was then further degassed and heated for another 3−5 h until gelation occurred. The gelled samples were thermally cured by heating in an oven at 250 $^{\circ}$C for 1 h, 325 $^{\circ}$C for 3 h, 350 $^{\circ}$C for 2 h and 375 $^{\circ}$C for 8 h under an atmosphere of argon. The samples were then removed from the molds and some freestanding samples were postcured in the oven at temperatures up to 425 $^{\circ}$C under an atmosphere of argon. Cured (375 $^{\circ}$C) and postcured (425 $^{\circ}$C) polymers and copolymer samples were either sanded to a thickness of approximately 2 mm for the dynamic mechanical measurements or pulverized for thermogravimetric studies.

2.3. Characterizations

Samples for differential scanning calorimetric (DSC) analysis were prepared by thoroughly mixing biphenyl PN monomer and the $n = 4$ PN oligomer in dry powdered form under ambient conditions. For DSC cure studies, *p*-BAPS curing additive (5 mol%) was thoroughly stirred into the solid phthalonitrile monomer mixtures at room temperature. DSC data were recorded from 0 to 400 $^{\circ}$C on samples (1−2 mg) in sealed aluminum pans using a TA Instruments 2920 DSC at heating and cooling rates of 10 $^{\circ}$C min^{-1} and a nitrogen flow rate of 100 mL min^{-1}. The T_{g} was taken as the temperature of the

inflection point in the heat flow versus temperature curve. DSC analysis was also used for cure studies of the biphenyl PN/ $n = 4$ PN blends with 5 mol% of the aromatic amine curing additive p-BAPS.

Dynamic viscosity measurements were performed on a TA Instruments AR-2000 rheometer in conjunction with an environmental testing chamber for temperature control. The rheological behavior of the biphenyl PN/$n = 4$ PN blends was determined in air after melting the powdered samples between the 40 mm diameter parallel plates in the test chamber of the rheometer. The melted samples were heated from 70 to 350 °C at a heating rate of 3 °C min^{-1}. The complex melt viscosity of phthalonitrile prepolymers was also monitored at 200 °C in air as a function of time. In both cases, viscosity measurements were made at low strain values (2.5×10^{-4}) and a frequency of 1 Hz. The rheometer was used with torsion fixtures to measure the storage modulus (G') and damping factor ($\tan\delta$) of rectangular PN polymer and copolymer samples (dimensions 50 mm × 12 mm × 2 mm) in nitrogen over the temperature range from 40 to 450 °C. To determine the range of the linear viscoelastic region, measurements of G' were first made as a function of strain at room temperature and a frequency of 1 Hz. Using a strain of 2.5×10^{-4} selected from within the linear viscoelastic region, an oscillatory temperature ramp of 3 °C min^{-1} was used to determine G' and $\tan\delta$ at a frequency of 1 Hz. Normal force control was utilized throughout the tests to keep the samples taut.

Thermal and thermo-oxidative stability studies were performed on pulverized polymer and copolymer samples (15–20 mg). Thermogravimetric analysis was carried out from room temperature to 1000 °C using a TA Instruments Q50 thermogravimetric analyzer (TGA) at a heating rate of 10 °C min^{-1} in an atmosphere of nitrogen or air at a flow rate of 100 mL min^{-1}. Long-term thermo-oxidative aging studies were also conducted on pulverized polymer and copolymer samples (75–80 mg). Polymer and copolymer samples for the aging studies were pre-dried in the TGA chamber by heating from room temperature to 150 °C where they were held for 60 min to remove the absorbed moisture. After drying, the samples were heated to 260 °C in the TGA at a heating rate of 10 °C min^{-1} and their weight at zero time was recorded. Isothermal aging of the samples was achieved by maintaining the samples at 260 °C for 200 h in air flowing at a rate of 50 mL min^{-1}.

3. Results and discussion

The melting and crystallization behaviors of the biphenyl PN/$n = 4$ PN blends were studied by DSC. Curves generated from the initial DSC heating scans of the blends are plotted in Fig. 2(a). DSC curves acquired on the individual phthalonitrile monomers are also included in the figure. Neat biphenyl PN and $n = 4$ PN monomers each exhibit a single characteristic melting transition. The peaks of these transitions occurred at 233 and 41 °C, respectively. Similarly, only one melting peak was evident for each blend suggesting the existence of one predominate type of crystal species. The peak melting
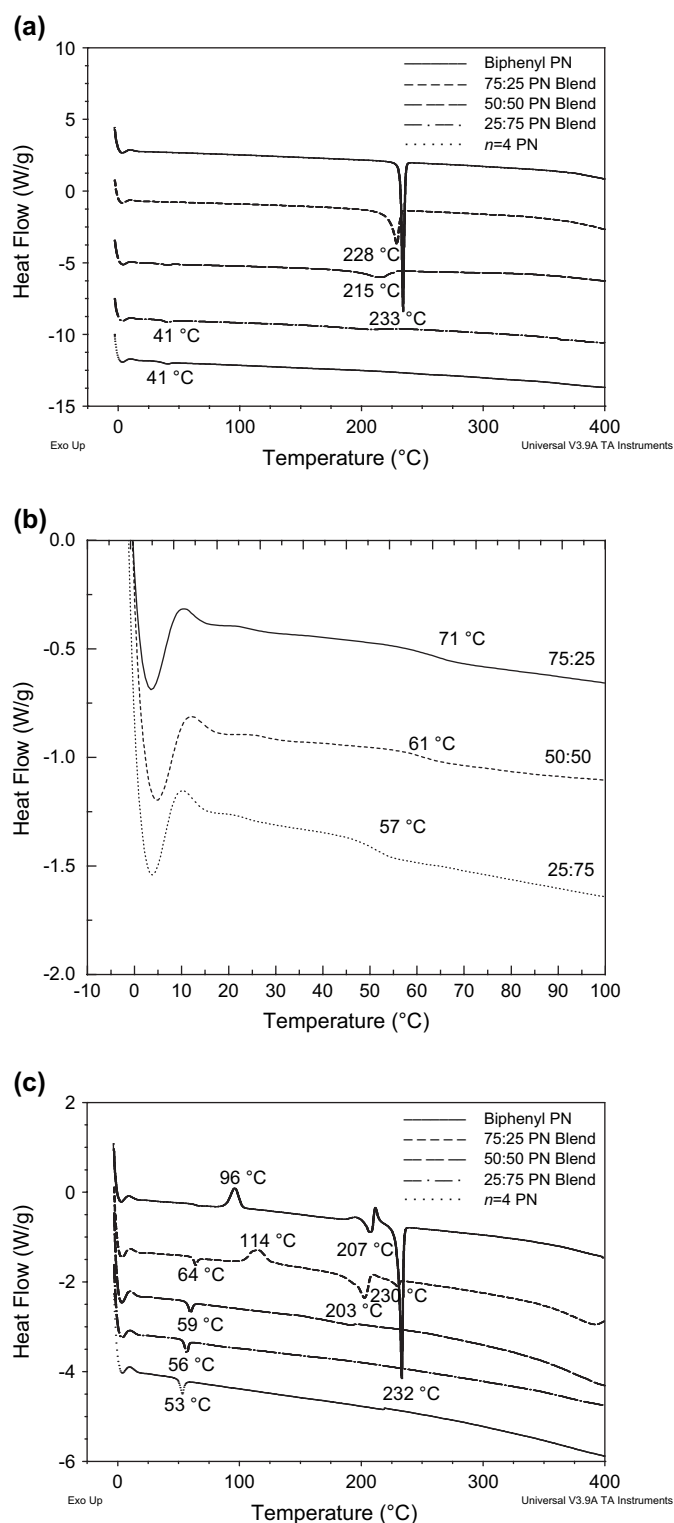
Fig. 2. DSC traces of the biphenyl PN/$n = 4$ PN blends, the neat biphenyl PN monomer and the $n = 4$ PN oligomer generated during: (a) initial heating, (b) 2nd heating immediately after cooling in the DSC and (c) 3rd heating after sitting under ambient conditions for one week. (The scans generated on cooling the samples in the DSC following the initial heating are not shown.)

temperatures for the 75:25 and 50:50 biphenyl PN/$n = 4$ PN blends were 228 and 215 °C, respectively. For these blend compositions, the melt temperature depression and the degree

of crystallinity, determined from the melt enthalpies, appeared to be related to the $n = 4$ PN content. In contrast, the peak melting temperature for the 25:75 biphenyl PN/$n = 4$ PN blends was identical to that of the $n = 4$ PN oligomer. In this case, it seems that the biphenyl PN concentration was not sufficiently high to affect the melting temperature of the blend.

On cooling, no crystallization peaks were apparent in the DSC scans which indicated that the phthalonitriles had remained in the amorphous phase. When the samples were immediately reheated in the DSC (second heating), only a single T_g was seen for each blend and the T_g values decreased as the $n = 4$ PN content in the blends increased (Fig. 2(b)). A single, composition-dependent T_g value for a blend generally indicates good miscibility in the amorphous phase [13,16]. For the 75:25, 50:50 and 25:75 biphenyl PN/$n = 4$ PN blends, the T_g values were 71, 61 and 57 °C, respectively. The biphenyl PN/$n = 4$ PN blends and the neat phthalonitrile samples were reheated in the DSC for a third time after sitting on the bench top for one week under ambient conditions. Curves from these third DSC heating scans are presented in Fig. 2(c). A single endothermic transition was again observed for the 25:75 and 50:50 biphenyl PN/$n = 4$ PN blends and the neat $n = 4$ PN monomer indicating that recrystallization or partial recrystallization of these samples had occurred gradually during the week. The peaks of these endothermic transitions occurred at higher temperatures (53–59 °C) than they did in the initial DSC scans. The higher temperatures reflected either a change in crystallinity or miscibility of the phthalonitriles blends in the amorphous phase. In contrast, DSC scans of the 75:25 biphenyl PN/$n = 4$ PN blends and the neat biphenyl PN sample each exhibited multiple endotherms and one recrystallization peak (exotherm) with a maximum intensity at 114 or 96 °C. The appearance of the additional transitions in the DSC traces of these more highly crystalline samples (the 75:25 biphenyl PN/$n = 4$ PN blends and the neat biphenyl PN) indicated that some recrystallization and phase separation had occurred when the samples were left undisturbed for a week.

The reaction of the biphenyl PN/$n = 4$ PN blends with 5 mol% p-BAPS was studied by DSC analysis. For the study, homogeneous mixtures of the phthalonitrile monomer blends and the curing additive were obtained by thoroughly stirring the powdered materials at room temperature. Curves generated by heating the blends with the curing additive in the DSC are presented in Fig. 3. For comparison, curves generated from mixtures of the neat phthalonitrile monomers with the curing additive are included in the figure. Characteristic melting transitions for the $n = 4$ PN oligomer and the biphenyl PN monomer were observed on heating the phthalonitrile blend/p-BAPS mixtures. These endothermic transitions were centered at 39–40 and 202–221 °C, respectively. Within the blended mixtures, the peak temperature attributed to the biphenyl PN seemed to depend on the blend composition whereas the peak temperature attributed to the $n = 4$ PN was not affected. The melting transition for p-BAPS (183–184 °C) was only seen in the DSC traces of the 75:25 biphenyl PN/$n = 4$ PN blends and the biphenyl PN. The phthalonitrile blend/p-BAPS mixtures also
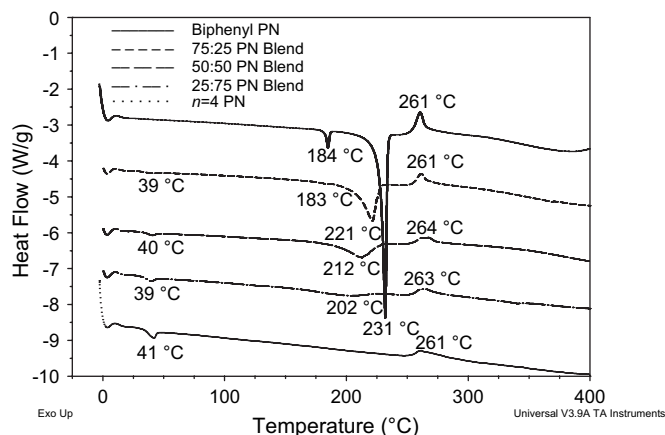

Fig. 3. DSC traces by heating the biphenyl PN/$n = 4$ PN blends, the neat biphenyl PN monomer and the $n = 4$ PN oligomer each mixed with 5 mol% p-BAPS.

exhibited a small exotherm centered at 261–264 °C due to the initial reaction of the phthalonitrile(s) with the diamine curing additive. The exotherm maximum was independent of the blend composition. The DSC data revealed that the biphenyl PN/$n = 4$ PN blends have larger processing windows (40–60 °C) than that of the biphenyl phthalonitrile and that the size of the processing window is increased with larger $n = 4$ PN contents. Thus, the melt viscosity of the blends and the rate of the phthalonitrile polymerization reaction can be more easily controlled when larger $n = 4$ PN concentrations are used.

The rheological behavior of a material is a key factor in determining its processability. Therefore, the variation of the melt viscosity of the biphenyl PN/$n = 4$ PN blends was determined as a function of temperature from 70 to 350 °C and compared with that measured for the neat phthalonitriles. The results, presented in Fig. 4, revealed that the complex viscosity of the blends was intermediate between those of the neat biphenyl
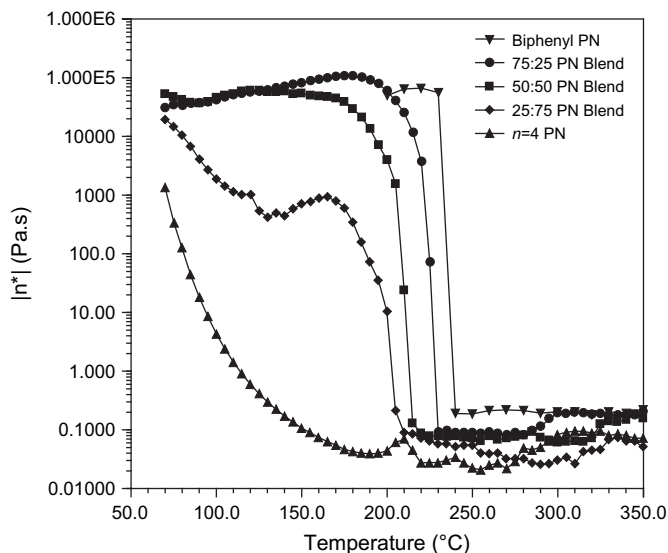

Fig. 4. Complex viscosity (η^*) as a function of temperature for the biphenyl PN/$n = 4$ PN blends, the neat biphenyl PN monomer and the $n = 4$ PN oligomer.

and $n = 4$ phthalonitriles at temperatures up to about 225 °C. As shown in the figure, biphenyl PN displayed a rapid decrease in viscosity on melting at 233 °C. In contrast, the viscosity of the oligomeric $n = 4$ PN melt gradually decreased in the temperature range from 70 °C up to approximately 175 °C. This behavior was expected for the pure material and the oligomeric mixture. Above 235 °C, a minimum melt viscosity of 0.01–1 Pa s was observed for the neat phthalonitriles. The temperature where the biphenyl PN/$n = 4$ PN blends began to melt decreased with an increase in the $n = 4$ PN content. The 75:25 and 50:50 PN blends appeared to melt in one step whereas the PN blend with the higher $n = 4$ PN content (25:75) melted in two steps. A minimum melt viscosity of around 0.1 Pa s was observed for all the blends at temperatures above 225 °C. The low melt viscosity of the blends at the higher temperatures is evidence of their good melt stability.

Complex viscosity changes that accompany the phthalonitrile polymerization reaction were also investigated by performing isothermal rheometric measurements at 200 °C on prepolymers formed in the presence of 5 mol% *p*-BAPS. In Fig. 5, prepolymer viscosity is shown as a function of time for prepolymers prepared from the blends and from the oligomeric $n = 4$ PN prepolymer. The data revealed that the higher the $n = 4$ PN content, the slower the viscosity increase. This result was expected since the blends richer in biphenyl PN have a greater tendency to react or cure at a faster rate. The rheological behavior of the blends indicates that there is a processing temperature advantage for these materials over that of the neat biphenyl PN.

Rheometric measurements were conducted on the phthalonitrile copolymers formed from curing the various biphenyl PN/$n = 4$ PN blends with 5 mol% *p*-BAPS up to maximum temperatures of 375 and 425 °C. From these studies, the influence of different thermal treatments on the dynamic mechanical properties of copolymers was evaluated. Fig. 6(a)–(b) and (c)–(d) compares the storage modulus (G') and damping
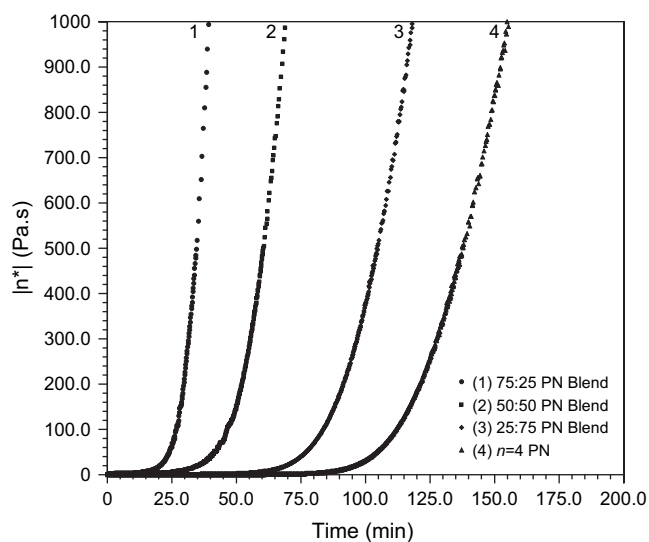
factor (tan δ) curves as a function of temperature for copolymers cured up to 375 and 425 °C, respectively. G' and tan δ plots generated on biphenyl PN and $n = 4$ PN polymers cured under identical conditions are included in the figures.

After curing to a maximum temperature of 375 °C, the storage moduli of the PN polymers and the biphenyl PN/$n = 4$ PN copolymers were 1200–1600 MPa at 40 °C. When heated from 40 to 450 °C, only small changes occurred in the modulus and the intensity of the tan δ peak for the biphenyl PN which indicated that this material was fully cured. In contrast, the changes observed for the $n = 4$ PN polymer were larger which revealed that a higher temperature was needed to complete the phthalonitrile cure. The copolymers exhibited storage moduli decreases and tan δ increases intermediate between those of the two phthalonitrile polymers (Fig. 6(a) and (b)). The extent of the changes in the storage moduli and tan δ increased as the $n = 4$ PN content in the copolymer increased. The variation in the dynamic mechanical properties of the copolymers cured to 375 °C reflected changes in the copolymer rigidity resulting from differences in crosslinking density. The tan δ versus temperature plots for the copolymers cured to 375 °C showed only one relaxation peak for each curve with the peak temperature independent of the copolymer $n = 4$ PN content. The estimated T_g, defined as the peak temperature of the tan δ versus temperature plots, for the $n = 4$ PN polymer and biphenyl PN/$n = 4$ PN copolymers was 225–230 °C.

Following an additional heat-treatment up to 425 °C, no viscoelastic transitions indicative of a T_g were apparent in the storage moduli and tan δ versus temperature plots of the $n = 4$ PN polymer and biphenyl PN/$n = 4$ PN copolymers (Fig. 6(c) and (d)). This behavior revealed that stable crosslinked networks had formed which hindered the segmental motion of the polymer/copolymers. At 40 °C, the storage moduli of the polymer/copolymer were 1200–1300 MPa. Upon heating to 450 °C, only gradual decreases in the storage moduli were observed that were attributed to stress relaxation of the polymer/copolymer network. Thus, the $n = 4$ PN polymer and all the copolymers exhibited dynamic mechanical behavior characteristic of a glassy material up to 450 °C. These data showed that a higher cure temperature (425 vs. 375 °C) was needed to complete the cure of the phthalonitrile copolymers. At 450 °C, the fully-cured copolymers exhibited the storage moduli of 700–1000 MPa. The rheometric measurements revealed that the copolymers retain 60–80% of their structural properties when heated up to 450 °C for short time periods.

The thermal and thermo-oxidative stability of phthalonitrile copolymers that had been cured up to maximum temperatures of 375 and 425 °C were determined by TGA analysis. The results of these studies are presented in Fig. 7(a) and (b), respectively. The temperatures displayed in the figures are the degradation temperatures where the copolymers exhibited a 5% weight loss. The char yields for the copolymer samples heated to 1000 °C are also noted in the figures. For example, after curing up to 375 °C, the copolymers formed from the 75:25, 50:50 and 25:75 biphenyl PN/$n = 4$ PN blends exhibited 5% weight loss at 550, 535 and 493 °C, respectively, and char yields of 74, 72 and 63% when heated to 1000 °C
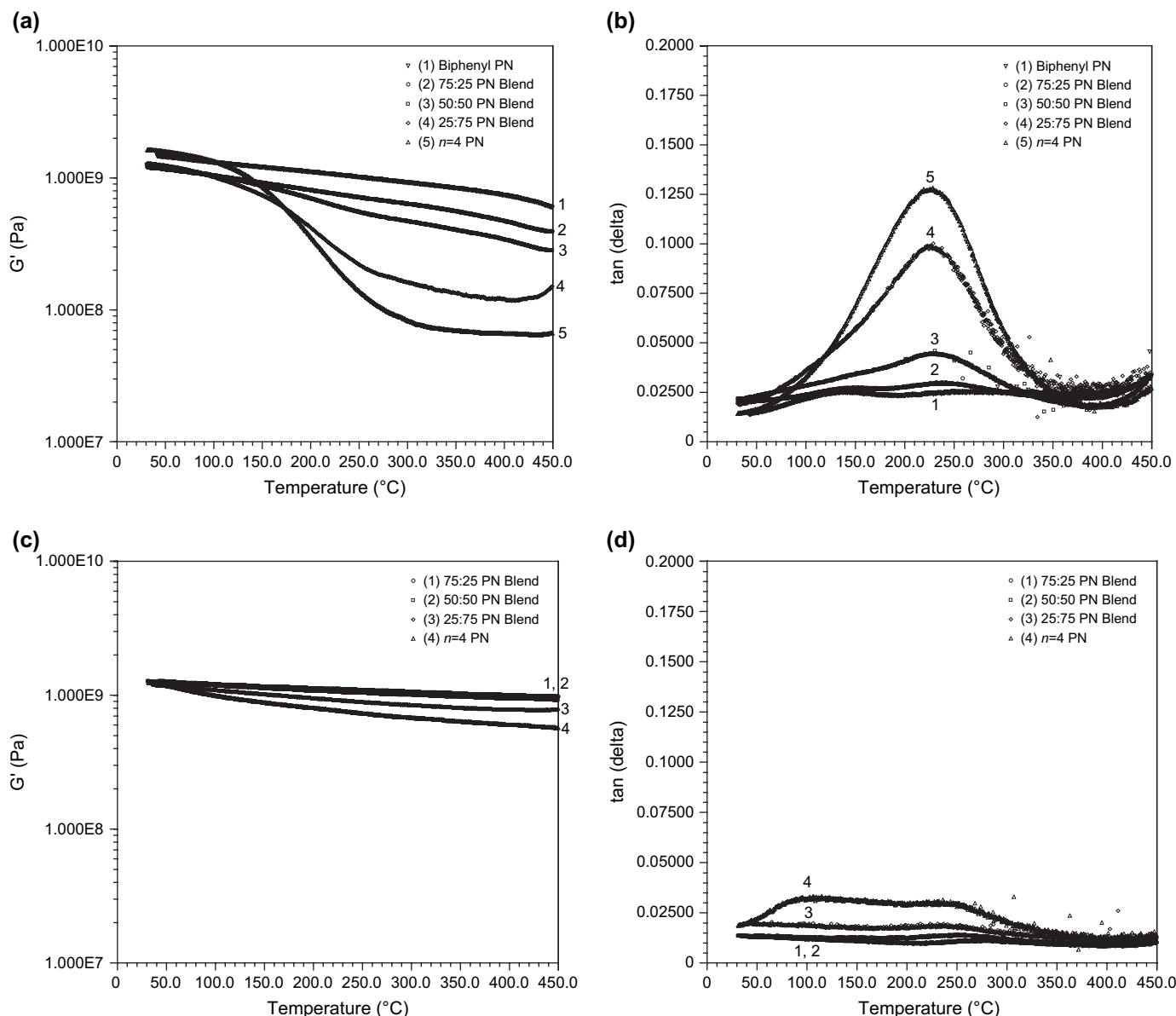

Fig. 5. Complex viscosity (η^*) at 200 °C as a function of time for prepolymers of the biphenyl PN/$n = 4$ PN blends and the $n = 4$ PN oligomer.

(a)

(b)

(c)

(d)

Fig. 6. Dynamic mechanical properties of the biphenyl PN/$n = 4$ PN blends, the neat biphenyl PN monomer and the $n = 4$ PN oligomer as a function of the maximum cure temperature: (a) G' after cure at 375 °C, (b) tan δ after cure at 375 °C, (c) G' after cure at 425 °C and (d) tan δ after cure at 425 °C.

in a nitrogen atmosphere. When heated in air, the copolymers showed 5% weight loss at 580, 559 and 512 °C, respectively. These data show the dependence of the thermal and thermo-oxidative stability on the $n = 4$ PN content in the copolymers. The decrease in copolymer stability with an increase in the $n = 4$ PN content was attributed to the corresponding decrease in the crosslinking density. When heated up to 425 °C, the degradation temperatures of the copolymers increased to 573−577 °C and the char yields improved to 77% when heated to 1000 °C under an atmosphere of nitrogen. The results show the dependence of copolymer thermal stability on the maximum processing temperature.

The long-term thermo-oxidative stability of the completely cured phthalonitrile copolymers was assessed by isothermally aging the powdered samples in the TGA chamber at 260 °C with an atmosphere of flowing air. For comparison, fully-cured

biphenyl and $n = 4$ phthalonitrile polymers were also aged under identical conditions. The data revealed that the phthalonitrile copolymers retained 96−97% and 91−94% of their initial weight after aging in air at 260 °C for 100 and 200 h, respectively. Similarly, weight retentions of 98−100% and 95−99% were observed for the biphenyl and $n = 4$ phthalonitrile polymers after aging for 100 and 200 h, respectively. Clearly, these data indicate that all the phthalonitrile polymers and copolymers exhibit excellent thermo-oxidative stability at 260 °C.

4. Conclusions

A series of biphenyl PN/$n = 4$ PN blends and phthalonitrile copolymers with varying $n = 4$ PN contents were prepared and characterized. The melting behavior of the blends and the degree of crystallinity observed for each blend were
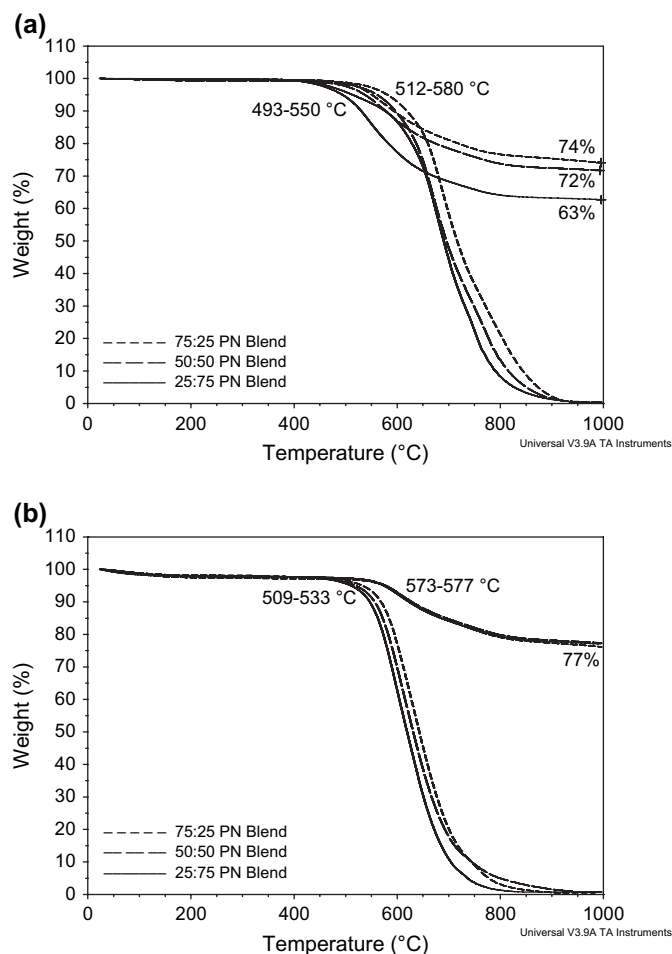
Fig. 7. Thermal and thermo-oxidative stability of the biphenyl PN/$n = 4$ PN blends as a function of maximum cure temperature: (a) 375 °C and (b) 425 °C.

related to the $n = 4$ PN content. It was determined that the phthalonitrile blends exhibit larger processing windows than that of biphenyl PN and that the size of the processing windows were also related to the $n = 4$ PN content in the blend. As such, the viscosity of the melt and the rate of the phthalonitrile polymerization can be more easily controlled for the phthalonitrile blends as a function of the curing additive concentration and temperature compared to the biphenyl PN. The cure of the phthalonitrile copolymers was investigated by

determining the dynamic mechanical properties and the thermal stability of the copolymers as a function of the maximum cure temperature. The thermal studies showed that a cure temperature of 425 °C was necessary for complete conversion to the thermosetting copolymers. This required temperature for the complete cure of the copolymers was higher than that (375 °C) required for the cure of the biphenyl PN polymer because of the longer spacers between the reactive end groups in the copolymers. The fully-cured phthalonitrile copolymers did not soften or exhibit a glass transition temperature upon heating to 450 °C. The fully-cured phthalonitrile copolymers also displayed excellent thermal stability along with long-term thermo-oxidative stability.

Acknowledgements

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