

## Mechanical and Thermal Properties of Dimer Acid Based Nylon 636/Nylon 66 Copolymers

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**ABSTRACT:** Dimer acid (DA)-based nylon 636/nylon 66 copolymers were synthesized by *in situ* polymerization. The effects of incorporating nylon 66 on the mechanical and thermal properties were characterized by means of intrinsic viscosity determination, attenuated total reflection (ATR)-Fourier transform infrared (FTIR) spectroscopy, X-ray diffraction (XRD), differential scanning calorimetry, thermogravimetric analysis, and mechanical testing. The results show that the intrinsic viscosity of the copolymers ranged from 1.0 to 2.1 dL/g, depending on the content of nylon 66. The incorporation of nylon 66 into DA-based nylon 636 had no significant effect on the values of the glass-transition temperature, melting temperature, temperature at 50% weight loss, or temperature at the maximum rate of decomposition, but the crystallization temperature, crystallinity degree, and extrapolated onset temperature increased. ATR-FTIR spectroscopy and XRD demonstrated that with increasing nylon 66 mass, the content of  $\alpha$ - and  $\beta$ -crystal forms would increase accordingly. The mechanical test data revealed that with increasing nylon 66 concentration, the tensile strength of the copolymers increased, and the flexural strength and flexural modulus first increased and then decreased. However, the notched Izod impact strength decreased. © 2013 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* **2014**, *131*, 39845.

**KEYWORDS:** biomaterials; mechanical properties; polyamides; thermal properties

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### INTRODUCTION

Nowadays, replacing petroleum-based raw materials with renewable resources is a major concern from both economic and environmental viewpoints.<sup>1</sup> Vegetable oils, which are renewable resources, are expected to be an ideal alternative chemical feedstock for the development of new environmentally friendly materials because they are present in abundance throughout the world and have the particularity of containing several active chemical sites, such as double bonds, allylic moieties, ester groups, and atoms in the  $\alpha$  position of the ester groups, and can be used for polymerization purposes.<sup>2–5</sup> Nevertheless, thermosets based on vegetable oils are largely developed on account of the high functionality of triglycerides,<sup>6,7</sup> and thermoplastic materials have attracted much attention in recent years.<sup>8–11</sup>

Dimer acids (DAs), which are environmentally friendly and commercially available products, are obtained by the polymerization of  $C_{18}$  acids, such as oleic and linoleic acids, and have characteristics of a low price, being liquid at room temperature, noncrystallinity, solubility in hydrocarbons, being biodegradable, and being reproducible. DA-based nylons, because of their special molecular structure based on DA, display relatively good performance.<sup>10,12</sup> However, they are still far from conventional

polyamide materials, and research work that is focused on enhancing polymer properties has developed. An effective approach for improving the thermal and mechanical properties of DA-based nylons is its association with another compound or the incorporation of fibers in the matrix.<sup>1,13–16</sup> Cavus and Gürkaynak<sup>13</sup> used the monofunctional reactant to modify dimer acid based polyamides (DAPAs), and they concluded that the formation of interchain hydrogen bonds and the density of the amide groups remarkably affected the crystallization and the physical properties. Wang et al.<sup>15</sup> studied the influence of adipic acid on the morphology and thermal properties and determined the way adipic acid affected the thermal degradation properties. Hablot et al.<sup>1</sup> studied the morphology and thermal and viscoelastic behavior of DAPAs reinforced with highly refined cellulose fibers. Matadi et al.<sup>17</sup> reported the compressive dynamic behavior of DAPA and DAPA composites with different cellulose fibers.

As of today, few studies have been carried out to introduce short nylon to DA-based nylon 636. The aim of this study was to examine the mechanical and thermal behaviors of the DA-based nylon 636/nylon 66 copolymers with different nylon 66 contents to prepare the products whose properties were similar to current nylon products.

## EXPERIMENTAL

### Materials

The DA commercially obtained from Liancheng Baixin Science and Technology Co., Ltd. (China) was a viscous liquid at room temperature with a monomer/dimer/trimer ratio of 1.0:98.0:1.0, an acid value of 195.0 mg of KOH/g, and an iodine value of 42.6 g of I<sub>2</sub>/100 g. 1,6-Hexanediamine was supplied by Jiangsu Yonghua Fine Chemical Co., Ltd. (China), and nylon 66 salt was purchased from Huafo Group Co., Ltd. (China). The catalyst was sodium hypophosphate, which was diluted to 5 wt % with distilled water, and was obtained from Xilong Chemical Co., Ltd. (China).

### Sample Preparation

DA-based nylon 636/nylon 66 copolymers were prepared by *in situ* polymerization.

First, the weighed DA, 1,6-hexanediamine, nylon 66 salt, and sodium hypophosphate solution were introduced into a 2-L autoclave equipped with a mechanical stirrer, a gas inlet, and a gas outlet. Then, the atmospheric oxygen present in the autoclave was removed with repeated rinsing under pressure with nitrogen. Second, the mixture was heated to 90°C and kept there for 0.5–1.5 h. Third, the mixture was again heated to 250–260°C while the pressure was increased to 1.3–1.5 MPa; these conditions were kept constant for 0.5 h to achieve polymerization. Afterward, the pressure began to level off within 1 h, and the reaction proceeded further at atmospheric pressure for 1 h. Finally, the polymerization was kept *in vacuo* for 1 h to obtain the resulting copolymers. The pure nylon 66 was also prepared by a similar method.

After the completion of the polymerization, the copolymers were cooled and granulated. The prepared DA-based nylon 636/nylon 66 copolymers containing 30, 40, 50, 60, and 70 wt % nylon 66 were abbreviated as N-30, N-40, N-50, N-60, and N-70, respectively.

### Characterization

The intrinsic viscosity was used to represent the molecular masses of the copolyamides. They were determined from the measurements taken on solution (concentration (*c*) = 0.25 mg/50 mL in *m*-cresol) of the copolymers with an Ubbelohde viscometer at 25 ± 0.1°C.

Attenuated total reflection (ATR)–Fourier transform infrared (FTIR) spectroscopy was used to estimate the structure of the copolymers. The ATR spectra were recorded at room temperature in the wave-number range from 4000 to 550 cm<sup>-1</sup> at a resolution of 4 cm<sup>-1</sup> and 32 scans per run with a Nicolet 8700 FTIR spectrograph.

Differential scanning calorimetry (DSC) measurements were carried out with a PerkinElmer Pyris Diamond DSC under a nitrogen atmosphere. The samples were rapidly heated up to 300°C at a rate of 80°C/min and kept there for 10 min to get rid of the thermal history. Then, the samples were cooled to -60°C at a rate of 10°C/min and heated again to 300°C at the same rate. The glass-transition temperatures (*T<sub>g</sub>*'s) were obtained at the midpoint of the heat-capacity changes. The crystallization temperature (*T<sub>c</sub>*) and the melting temperature

(*T<sub>m</sub>*) were determined from the maximum of *T<sub>c</sub>* and *T<sub>m</sub>* peak, respectively. The values of *T<sub>m</sub>* were determined on the second heating scan.

Thermogravimetric analysis (TGA) measurements were performed on a PerkinElmer Pyris 1 TGA instrument under a nitrogen flow. The samples were heated up to 600°C at 10°C/min from room temperature. The characteristic degradation temperatures were observed from the maximum of the derivative thermogravimetry (DTG) curves. All of the samples were dried *in vacuo* at 80°C for 12 h before analysis.

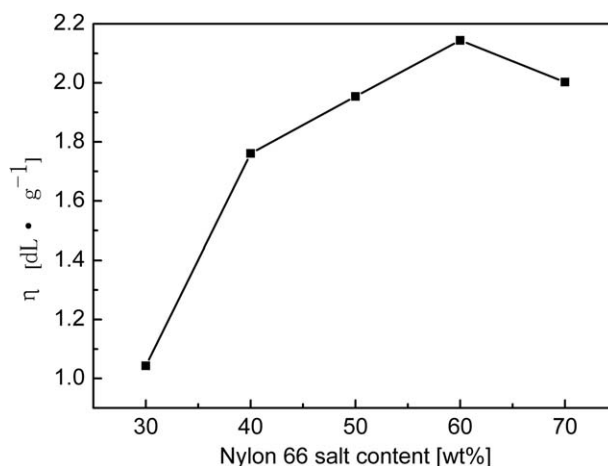
The X-ray diffraction (XRD) patterns were obtained with a Bruker D8 Advance diffractometer. The data was collected at room temperature in a range of angles (2θ's) from 5 to 50° and with a step of 0.02° at 2 s/step, under an acceleration voltage of 40 kV and a tube current of 30 mA with Cu Kα radiation (λ = 1.5406 Å). The lattice spacing of polyamides was calculated with Bragg's law.<sup>10</sup>

The tensile tests were tested with an Instron CMT5254 machine at 23 ± 2°C according to ASTM D 638 at 50 mm/min. For the flexural test, rectangular specimens were cut with dimensions of 100 × 10 × 5 mm<sup>3</sup> according to ASTM D 790 and were tested at a crosshead speed of 5 mm/min. The Izod notched impact strength was measured on an impact testing machine (CSI-137C) at 23 ± 2°C according to ASTM D 256. The average values were calculated on the basis of more than five tests to minimize the testing error.

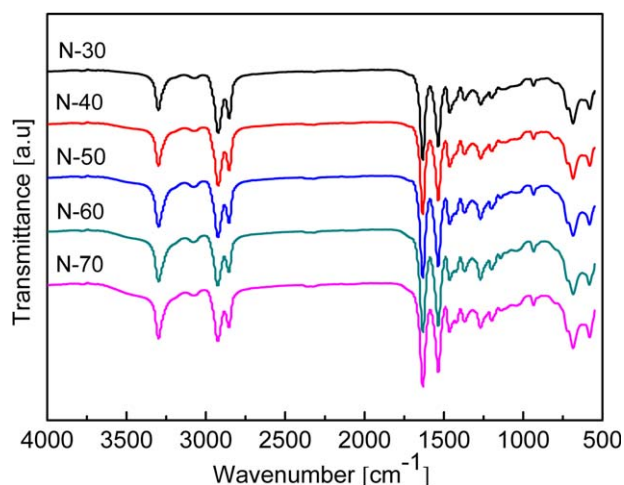
## RESULTS AND DISCUSSION

### Intrinsic Viscosity

Figure 1 shows the effect of nylon 66 on the intrinsic viscosity. The measured intrinsic viscosity showed that the molecular masses of the products first increased and then decreased with increasing content of nylon 66. The intrinsic viscosities of nylon 66, prepared under technological conditions similar to those of nylon 636, were 0.46 and 2.5 dL/g, respectively. Because the polymerization rate of nylon 66 salt was faster than that of nylon 636 salt, the copolymerization rate thus became greater with the incorporation of nylon 66 salt and resulted in the



**Figure 1.** Effect of the nylon 66 content on the intrinsic viscosity ( $[\eta]$ ) of the DA-based nylon 636/nylon 66 copolymers.



**Figure 2.** ATR-FTIR spectra of the DA-based nylon 636/nylon 66 copolymers. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

molecular masses of copolymers, which were reflected by the greater intrinsic viscosity.<sup>18</sup> However, self-polymerization might have occurred in the presence of an excessive amount of nylon 66 salt and resulted in the hindrance of the block polymerization reflected in N-70. The intrinsic viscosities of the HDA-based nylon copolymers are greater than DA-based copolymers with the same content of nylon 66 salt.<sup>19</sup> This could account for the improved purity of HDA during hydrogenation and purifi-

**Table I.** Thermal Properties and Crystalline Characteristics of the Copolymers

Sample	$T_g$ (°C)	$T_c$ (°C)	$\Delta H_c$ (J/g)	$T_m$ (°C)	$\Delta H_m$ (J/g)	$\Delta H_m^0$ (J/g)	$X_c$ (%)
N-30	21.2	179.5	9.19	253.0	5.16	200.4	2.6
N-40	21.2	187.8	17.20	255.6	12.66	201.2	6.3
N-50	21.4	199.4	22.50	257.9	21.19	202.0	10.5
N-60	21.2	214.1	26.58	256.4	25.73	202.8	12.7
N-70	21.2	213.1	36.68	257.2	32.78	203.6	16.1

cation process. The HDA could build longer chain copolymers due to its higher purity and fewer double bonds.

#### ATR-FTIR Spectroscopy Analysis

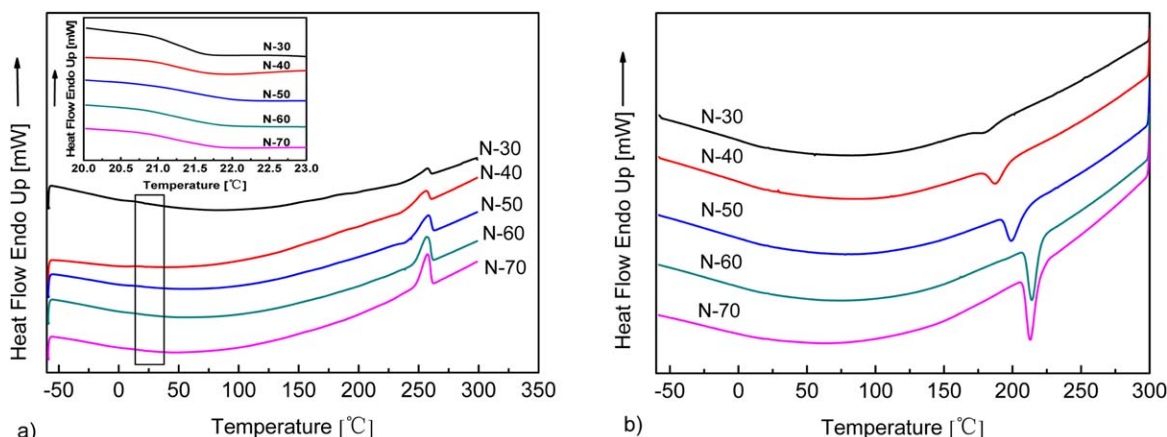
The ATR-FTIR spectra of the DA-based nylon 636/nylon 66 copolymers are shown in Figure 2. The bands around  $3299\text{ cm}^{-1}$  were assigned to the hydrogen-bonded and N-H stretching vibrations. The bands around  $2923\text{ cm}^{-1}$  were attributed to N-H in-plane bending vibrations.<sup>12</sup> The peaks around  $1632\text{ cm}^{-1}$  were due to C-O stretching vibrations (amide I). The bands due to N-H bending and C-N stretching vibrations (amide II) were around  $1536\text{ cm}^{-1}$  and were attributed to the  $\alpha$  form of various nylons according to Arimoto<sup>20</sup> and Bradbury and Elliott.<sup>21</sup> The bands around  $1417\text{ cm}^{-1}$  were originated from the scissoring vibration of the  $\text{CH}_2$  units.<sup>22</sup> The bands that appeared around  $1273\text{ cm}^{-1}$  were assigned to the C-N-H coupling vibration (amide III). The peaks around  $935/688\text{ cm}^{-1}$ , which were assigned to amide IV/V mode, were typical bands of the planar zigzag structure of the  $\alpha$  form, and the characteristic bands at  $582\text{ cm}^{-1}$  due to the C-O out-of-plane bending vibrations of amide VI were attributed to  $\alpha$ - and  $\beta$ -mixture forms.<sup>23–26</sup> The intensity of these main absorption bands increased slightly with the addition of nylon 66; this was in agreement with the intrinsic viscosity. The results indicate that with the enhancement of the intensity of these main absorption bands, the contents of  $\alpha$ - and  $\beta$ -crystal forms increased accordingly.

#### Thermal Properties

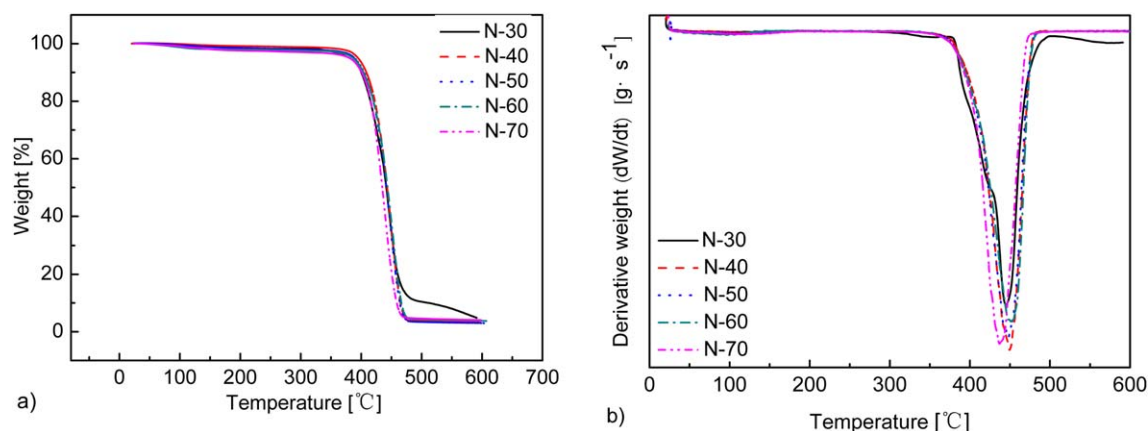
Table I presents the main values determined by DSC of the copolymers with increasing nylon 66 contents. We determined the different enthalpy change of crystallization ( $\Delta H_c$ ) and enthalpy of change of melting ( $\Delta H_m$ ) from the corresponding peaks. The percentage crystallinity ( $X_c$ ) could be estimated with eq. (1), where  $\Delta H_m^0$  is the theoretical value which is calculated for the 100% crystalline polyamides:<sup>27,28</sup>

$$X_c = \frac{\Delta H_m}{\Delta H_m^0} \times 100 \quad (1)$$

The second heating scans of the DSC thermograms of N-30, N-40, N-50, N-60, and N-70 are shown in Figure 3(a). It was obvious that there was only one peak for the melting and



**Figure 3.** DSC diagram for N-30, N-40, N-50, N-60, and N-70: (a) second heating scan and (b) crystallization curves. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]



**Figure 4.** (a) TGA and (b) DTG curves for N-30, N-40, N-50, N-60, and N-70, dW/dt is the reaction rate, which indicates the speed of weight change. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

crystallization curves; this confirmed that nylon 66 was completely miscible in the DA-based nylon 636. The  $T_g$  values of the copolymers are shown in illustrations. With increasing amounts of nylon 66, the  $T_g$  of the copolymers changed very little but were higher than that of the neat DA-based nylon 636, which was reported to be  $-9.63^\circ\text{C}$  in the literature.<sup>10</sup> The lower  $T_g$  could be explained by the fact that the DA possessed rings and aliphatic-grafted chains, which added the conformational disorder free volume; thus, with increasing short-chain nylon, the polymer mobility decreased. The  $T_m$  values of the copolymers were higher than the neat DA-based nylon 636 reported in the literature at  $81^\circ\text{C}$  because with the incorporation of nylon 66, the flexible chain had a lower ratio.<sup>10</sup> With increasing nylon 66 content, the values of  $T_m$  of these showed no significant variation; this could imply that their crystalline structures were much the same. But the  $T_m$  values of the copolymers were higher than those of HDA-based copolymers,<sup>19</sup> this could be explained given the formation of conjugated structures resulting in poor segmental activity. Figure 3(b) shows the crystallization curves of the copolymers. The  $\Delta H_m$  values of the DA-based copolyamides increased significantly with nylon 66 contents from 30 to 70%, which reflected the  $X_c$  values.

Figure 4 shows the TGA and DTG curves of the copolymers. The extrapolated onset temperature ( $T_{\text{onset}}$ ), temperature at 50% weight loss ( $T_{50\%}$ ), temperature at the maximum rate of decomposition ( $T_{\text{max}}$ ), and the residue at  $600^\circ\text{C}$  are listed in Table II. We observed that  $T_{\text{onset}}$  showed no obvious change with the addition of nylon 66. The  $T_{50\%}$  and  $T_{\text{max}}$  values were relatively constant for all of the copolymers.

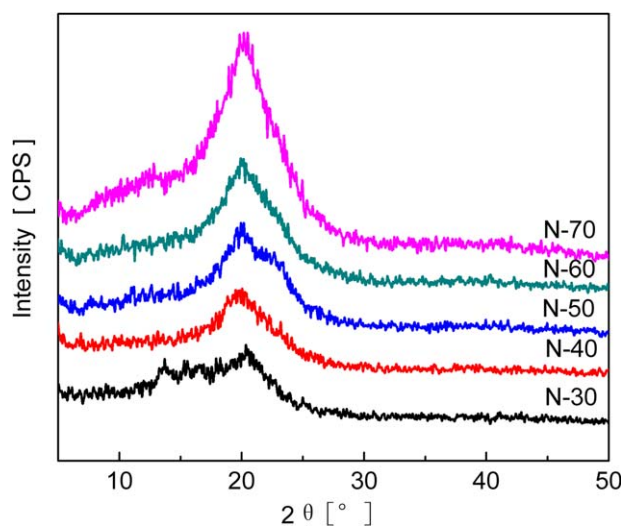
**Table II.** TGA and DTG Results for the Copolymers

Sample	$T_{\text{onset}}$ ( $^\circ\text{C}$ )	$T_{50\%}$ ( $^\circ\text{C}$ )	$T_{\text{max}}$ ( $^\circ\text{C}$ )	Residue at $600^\circ\text{C}$ (%)
N-30	415.7	441.5	444.8	4.9
N-40	420.3	442.9	449.4	3.4
N-50	417.3	441.5	447.9	3.0
N-60	413.8	443.6	451.1	3.8
N-70	412.4	434.9	436.8	4.0

The  $T_{\text{max}}$  values of the copolymers rose with increasing nylon 66 content from 30 to 40%. This indicated that the thermal stability improved. It could be explained that the formation of highly combining azo double-bond C–N groups was due to tautomerism and group change.<sup>29</sup> In addition, the elimination polymerization occurred between reactive amine terminal groups and carboxyl groups, which could enhance the thermal stability.<sup>30</sup> The decrease in the thermal stability of the copolymers with increasing nylon 66 content from 40 to 70% was attributed to the formation of cyclopentanone and caprolactam at  $350\text{--}420^\circ\text{C}$ . The results indicate that the DA-based nylon 636/nylon 66 copolymers were more thermally stable than the pure DAPA.<sup>10</sup> The residue at  $600^\circ\text{C}$  of DA-based nylon copolymer was higher than for HDA-based copolymer,<sup>19</sup> this also indicated that the purity of HDA was higher.

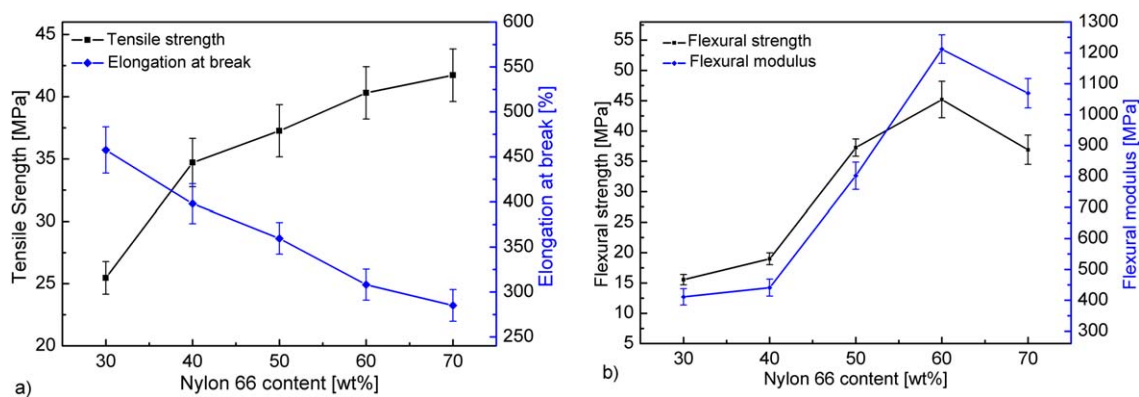
#### XRD Analysis

XRD diagrams of copolymers are shown in Figure 5. All of the samples exhibited a very strong diffraction signal at  $2\theta = 20^\circ$ ,



**Figure 5.** XRD patterns of the DA-based nylon 636/nylon 66 copolymers, CPS indicates counts per second. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]



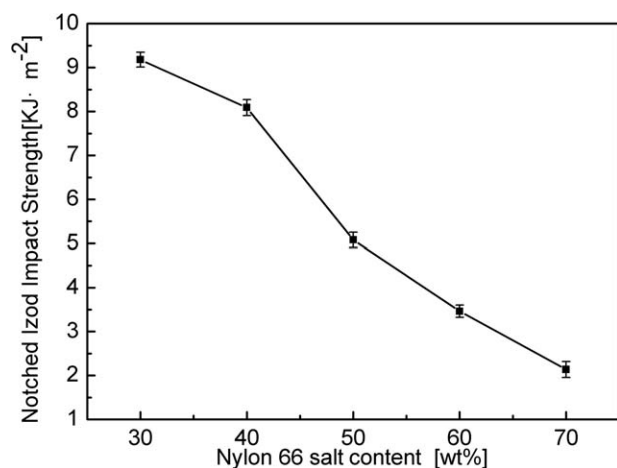


**Figure 6.** (a) Tensile strength and elongation at break and (b) flexural strength and flexural modulus for N-30, N-40, N-50, N-60, and N-70. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

corresponding to a distance of about 0.44 nm. The additional weak signal observed at  $2\theta = 24^\circ$ , which corresponded to distance of about 0.37 nm, was very weak for N-40 and N-50 and was hardly visible for N-30, N-60, and N-70. The two peaks at about  $2\theta = 20$  and  $24^\circ$  represented the projected interchain distance within a hydrogen-bonded sheet and the intersheet distance, respectively.<sup>31</sup> This phenomenon may have indicated that with the incorporation of nylon 66 into DA-based nylon 636, strong intermolecular hydrogen bonds formed; this led to the molecules assembling to form a more spectacular and varied morphology. The weak signals appeared at about  $2\theta = 11.8\text{--}14.4^\circ$  and could be designated as the  $\beta$  phase.<sup>32,33</sup> We also observed a weak peak around  $2\theta = 21^\circ$ , which represented the  $\gamma$  phase for N-30, N-40, and N-50. The results indicate that these were in agreement with the FTIR results. But compared with HDA-based nylon copolymer, the coacervation structure stability of DA-based copolymers was relatively poor.<sup>19</sup>

### Mechanical Properties

Figures 6 and 7 shows the effect of nylon 66 on the mechanical properties. The tensile strength of the copolymers was evidently improved with nylon 66 mass but improved slightly from 40 to 70 wt %. The elongation at break showed a reverse trend, which decreased, except in N-70, as shown in Figure 6(a). This result was not only due to the long main chain of DA, which caused a



**Figure 7.** Notched Izod impact strength for the nylon copolymers.

big deformation, but also to the intermolecular hydrogen bond, which was dominant in coacervation structure with the addition of nylon 66. The flexural strength and flexural modulus revealed nonlinear increase trends with the increase in concentration of nylon 66, as shown in Figure 6(b). We could see that the flexural strength and flexural modulus of the copolymers increased slowly when we introduced small amounts of nylon 66. In the initial stage of the induction of nylon 66, the coacervation structure was controlled by van der Waal's forces, which existed in the amorphous phase. With increasing nylon 66 content, strong intermolecular hydrogen bonds formed, which led to a structural transition from a pseudo-hexagonal  $\gamma$  form to more stable  $\alpha$ - and  $\beta$ -mixture forms. Compared with the performance of pure nylon 66, the mechanical properties of the copolymers were lower. Because nylon 66 was linked by short chains, a small intermolecular steric hindrance resulted in the high degree of polymerization, which was reflected in the high tensile strength and flexural strength. The results were in accordance with the intrinsic viscosity.

We observed a significant reduction in the notched Izod impact strength upon the additions of nylon 66 salt. This indicated that the incorporation of nylon 66 resulted in a higher crystallinity. Because with an increasing nylon 66 salt content, the rigid molecular backbone of nylon 66 replaced the block DA, which contained two long alkyl chains and resulted in an increase in the cohesive energy density of the copolymers, and induced the molecular chain to aggregate to the conformational order and densification. Compared with HDA-based nylon copolymers, the mechanical properties of DA-based copolymers were worse.<sup>19</sup> This can be attributed to the absence of double bond structures and the higher purity of HDA, which led to higher polymerization degree.

### CONCLUSIONS

A series of DA-based nylon 636/nylon 66 copolymers was synthesized by *in situ* polymerization. The intrinsic viscosity of the prepared samples ranged from 1.0 to 2.1 dL/g and was influenced by the nylon 66 content. ATR-FTIR analysis showed that with the increase of nylon 66, the intensity of the main absorption bands increased slightly; this implied that with the enhancement of the intensity of these main absorption bands,

the content of  $\alpha$ - and  $\beta$ -crystal forms increased. The DSC studies showed that nylon 66 had no significant effect on the values of  $T_g$  and  $T_m$  of the copolymers. The TGA studies showed that nylon 66 had no notable effect on the values of  $T_{50\%}$  and  $T_{max}$ , but  $T_{onset}$  increased first and then decreased because their crystalline structures were much the same. The change in  $T_{onset}$  and  $T_{max}$  indicated that the DA possessed rings and long aliphatic grafted chains; this increased the conformational disorder. XRD and DSC displayed high organization in the copolymers with the incorporation of nylon 66. The incorporation of the short-chain nylon decreased the polymer mobility and led to the formation of a more stable crystal phase. The mechanical test data showed that with increasing nylon 66 concentration, the tensile strength of the copolymers and flexural strength increased, but the elongation at break and the notched Izod impact strength decreased. This indicated that the long main chain of DA caused large deformation, and intermolecular hydrogen bonding was dominant in coacervation structure. When nylon 66 was incorporated into the DA-based nylon, the strong intermolecular hydrogen bonds induced the molecular chains to assemble to a conformational order and to become more dense; this also manifested in the FTIR and XRD studies.

#### ACKNOWLEDGMENTS

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