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# Polycondensation Reaction of 4-(4í-*N*-1,8-Naphthalimidophenyl)-1,2,4-triazolidine-3,5-dione with Aliphatic Diacid Chlorides

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## ABSTRACT

4-(4í-Aminophenyl)-1,2,4-triazolidine-3,5-dione **(1)** was reacted with 1,8-naphthalic anhydride **(2)** in a mixture of acetic acid and pyridine (3:2) under refluxing temperature and gave 4-(4í-N-1,8-naphthalimidophenyl)-1,2,4-triazolidine-3,5-dione (NIPTD) **(3)** in high yield and purity. The compound NIPTD was reacted with excess acetyl chloride in *N,N*í-dimethylacetamide (DMAc) solution and gave 1,2-bisacetyl-4-[4í-(1,8-naphthalimidophenyl)]-1,2,4-triazolidine-3,5-dione **(4)** as a model compound. Solution polycondensation reactions of monomer 3 with succinyl chloride (SucC), suberoyl chloride (SubC) and sebacoyl chloride (SebC) were performed under conventional solution polymerization techniques in the presence of different catalysts and led to the formation of novel aliphatic polyamides. These novel polyamides have inherent viscosities in a range of 0.08-0.19 $dLg^{-1}$ in con. $H_2SO_4$ or *N,N*í-dimethylformamide (DMF) at 25°C. Some structural characterization and physical properties of these novel polymers are reported.

Key Words:
polyamides;
step-growth polymerization;
inherent viscosity;
4-(4í-*N*-1,8-naphthalimidophenyl)-1,2,4-triazolidine-3,5-dione;
thermogravimetric analysis (TGA).

## INTRODUCTION

Urazole derivatives are very interesting five-membered heterocyclic compounds which contain three nitrogen atoms and two carbonyl groups. They are prepared in five steps from corresponding carboxylic acids and in two steps from corresponding isocyanates. At position 4 one can have a wide variety of aliphatic as well as aromatic substituents. These heterocyclic compounds have two N-H protons. These protons are rather acidic. The urazole derived from the ene reaction of tria-

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zolinediones with alkenes and polydienes has one N-H proton, which seems to be very acidic. The acidity of this proton has been measured and is quite acidic with $pK_a$ of 4.71 which is almost the same as that of acetic acid [1]. The urazole compounds have potential to undergo *N*-acylation. 4-Substituted urazole was converted to 1-acyl derivatives by acylation reaction with a series of carboxylic acid anhydrides [2]. A simplified procedure for the *N*-acylation of oxazolidin-2-one chiral auxiliaries has also been reported [3].

Recently we have been able to take advantage of acidic N-H in a compound, 1-methyl-2,5-bis(4-phenylurazolyl) pyrrole and synthesize novel polymers via *N*-alkylation and *N*-acylation reactions [4,5]. Polymerization of compound 4-phenylurazole (PHU) with phosgene, terephthaloyl chloride and epichlorohydrin has been reported to give insoluble polymers [6]. Polymerization of compound PHU with aliphatic diacid chlorides gave soluble polyamides with inherent viscosity of 0.39 $dLg^{-1}$ [7]. Copolymerization of 4-cyclohexylurazole (CHU) with aliphatic diacid chlorides gave soluble polyamides [8]. We have also reported the reaction of PHU and other urazole derivatives with diisocyanates [9-14]. The resulting novel polyureas have inherent viscosities in a range of 0.04-0.23 $dLg^{-1}$ and some of them are photoactive materials as well as polymeric dyes.

Aromatic and aliphatic polyamides have been widely prepared and used. The purpose of this investigation was to examine the step-growth polymerization reactions of NIPTD as a heterocyclic monomer with aliphatic diacid chlorides. In the present paper we report on the successful polycondensation reaction where NIPTD as a novel monomer is used for the synthesis of new aliphatic polyamides containing heterocyclic moieties.

## EXPERIMENTAL

### Materials and Equipments

Reagents were purchased from Fluka Chemical (Buchs, Switzerland), Aldrich Chemical (Milwaukee, WI) and Riedel-deHaen AG (Seelze, Germany). 4-(4í-Aminophenyl)-1,2,4-triazolidine-3,5-dione **(1)** was prepared in 6 steps according to our previous work [15]. DMAc (*N,N*í-dimethylacetamide) was dried over BaO and then it was distilled under reduced pressure. Proton nuclear magnetic resonance $^1$H NMR (90MHz) and (500MHz) spectra were recorded on a Varian EM-390 (Varian Associates, Palo Alto, CA) and Bruker (Germany), Advance 500 instrument, respectively. Tetramethylsilane (TMS) was used as an internal reference. IR Spectra were recorded on a Shimadzu 435 IR spectrophotometer. Spectra of solids were carried out using KBr pellets.

All melting points were taken with a Gallenham melting point apparatus. Inherent viscosities were measured by standard procedure using a Cannon Fensk Routine viscometer. Thermal gravimetric analysis (TGA) data for polymers were taken on a Mettler TGA-50. Elemental analyses were performed by Malek Ashtar University, Tehran, I.R. Iran.

### Preparation of 4-(4í-*N*-1,8-Naphthalimidophenyl)-1,2,4-triazolidine-3,5-dione (NIPTD) (3)

Into a 100-mL round bottom flask, 4-(4í-aminophenyl) -1,2,4-triazolidine-3,5-dione **(1)** (3.00 g, 1.56 * $10^{-2}$mol) and 1,8-naphthalic anhydride **(2)** (3.094 g, 1.56 * $10^{-2}$ mol) were dissolved in a mixture of 16 mL of pyridine and 24 mL of acetic acid. The solution was refluxed for 5 h. The reaction mixture was concentrated to 5 mL and the resulting white solid was poured into 50 mL of water. The white solid was filtered off, and washed with 50 mL of water to give 2.93 g (98%) of white solid. Recrystallization from acetic acid gave white crystals mp 322°C (dec); IR (KBr): 3600 (m), 3470 (m), 3210 (m), 3070 (m), 3020 (m), 2910 (w), 2820 (w), 1773 (m), 1708 (s), 1657 (s), 1627 (m), 1587 (s), 1515 (s), 1462 (w), 1435 (s), 1378 (s), 1360 (s), 1315 (w), 1293 (w), 1265 (w), 1239 (s), 1190 (s), 1142 (w), 1122 (m), 1098 (m), 1080 (w), 1025 (m), 957 (m), 902 (s), 855 (m), 830 (m), 780 (s), 740 (m), 712 (w), 700 (w), 680 (w), 660 (w), 620 (m), 590 (m), 550 (w), 535 (m) $cm^{-1}$; $^1$H NMR (90 MHz, DMSO-$d_6$, TMS): $\delta$ 7.4-8.0 (m, 6H, Ar-H), 8.4-8.6 (m, 4H, Ar-H), 10.4 (s, br, 2H, N-H). Anal. Calcd for $C_{20}H_{12}N_4O_4$: C, 64.52%; H, 3.25%; N, 15.05%. Found: C, 64.14%; H, 3.62%; N, 15.18%.

### Preparation of 1,2-bis-Acetyl-4-[4í-(1,8-naphthalimidophenyl)]-1,2,4-triazolidine-3,5-dione (Model Compound 4)

Into a 25-mL round-bottom flask, acetyl chloride

(0.4216 g, 5.37 * $10^{-3}$ mol ) was added to a solution of NIPTD **3** (0.200 g, 5.37 * $10^{-4}$ mol ) in 1.5 mL of dry DMAc. The solution was stirred for 36 h at room temperature. The excess acetyl chloride was removed by aspirator and the residue was percipitated in 50 mL of water. The resulting solid was filtered off, dried to give 0.2108 g (86.0%) of off-white solid **4**. This solid was recrystallized from acetic acid, mp>300°C; IR(KBr): 3080 (m), 2906 (w), 2814 (w), 2017 (w), 1770 (m, sh), 1709 (s), 1656 (s), 1625 (m), 1585 (s), 1516 (s), 1435 (s), 1375 (s), 1357 (s), 1240 (s), 1192 (m), 898 (w), 850 (w), 825 (w), 781 (s), 736 (w) $cm^{-1}$. $^1H$ NMR (500 MHz, DMSO-$d_6$): δ 2.52 (s, 6H), 7.59-7.68 (m, br, 4H), 7.92-7.94 (distorted dd, 2H), 8.52-8.54 (distorted dd, 4H, $J_1$ = 7.60 Hz). Anal. Calcd for $C_{24}H_{16}N_4O_6$: C, 63.16%; H, 3.53%; N, 12.27%. Found: C, 63.71%; H, 3.64%; N, 12.51%.

### Polymerization of NIPTD with SucC

*Method 1*

Into a 25-mL round-bottom flask NIPTD (0.0948 g, 2.54*$10^{-4}$ mol) and pyridine (0.0410 mL, 5.09*$10^{-4}$ mol) were added to a solution of SucC (5) (0.0394 g, 2.54*$10^{-4}$ mol) in 0.5 mL of *N*-methyl-2-pyrrolidone (NMP). The solution was stirred for 3 h at -5°C, then for 16 h at room temperature. The viscous solution was precipitated in 30 mL of methanol. The solid was filtered off, dried to give 0.1006 g (75%) of polyamide PA1A. mp 282-290°C. The above polymerization was repeated, in the presence of triethylamine and dibutyltin dilurate as a catalyst, respectively.

*Method 2*

The above polymerization was repeated, but the reaction mixture was refluxed in NMP for 1 min in the presence of different catalysts.

IR(KBr): 3155 (w), 3078 (m), 2930 (w), 1720 (s), 1668 (s), 1587 (s), 1516 (s), 1417 (s), 1375 (s), 1356 (s), 1292 (s), 1238 (s), 1190 (m), 829 (m), 779 (s), 613 (m) $cm^{-1}$. $^1H$ NMR (500 MHz, DMSO-$d_6$): δ 2.51 (s, 4H), 7.53-7.62 (distorted dd, 4H), 7.92 (s, br, 2H), 8.53 (s, br, 4H), 10.57 (s, br, N-H). Anal. Calcd for $C_{24}H_{14}N_4O_6$: C, 63.44%; H, 3.10%; N, 12.33%. Found: C, 62.35%; H, 3.82%; N, 14.33%.

### Polymerization of NIPTD with SubC

*Method 1*

Into a 25-mL round-bottom flask NIPTD (0.1299 g, 3.49*$10^{-4}$ mol ) and pyridine (0.056 mL, 3.49 * $10^{-4}$ mol) was added to a solution of SubC **(6)** (0.0736 g, 3.49*$10^{-4}$ mol) in 1.30 mL of NMP. The solution was stirred for 3 h at -5°C, then for 22 h at room temperature. The viscous solution was precipitated in 50 mL of methanol. The solid was filtered off, dried to give 0.1658 g (81.5%) of pale-brown polyamide PA2A. mp 278-294°C. The above polymerization was repeated, in the presence of triethylamine and dibutyltin dilurate as a catalyst, respectively.

*Method 2*

The above polymerization was repeated, but the reaction mixture was refluxed in NMP for 1 min in the presence of different catalysts.

IR(KBr): 3080 (w), 2820 (w), 1749 (s), 1709 (s), 1664 (s), 1589 (m), 1518 (m), 1421 (m), 1375 (s), 1356 (s), 1238 (s), 1157 (w), 781 (m) $cm^{-1}$. $^1H$ NMR (500 MHz, DMSO-$d_6$): 1.24-1.66 (m, br,8H), 2.53 (s, overlapped with DMSO peak, 4H), 7.57-7.60 (d, 2H, $J$ = 12.5 Hz), 7.63-7.66 (d, 2H, $J$ = 12.5 Hz), 7.91-7.94 (distorted dd, 2H), 8.53-8.55 (distorded dd, 4H, $J_1$ = 7.60 Hz), 10.57 (s, br, N-H). Anal. Calcd. for $C_{28}H_{22}N_4O_6$: C, 65.88%; H, 4.34%; N, 10.97%. Found: C, 64.01%; H, 3.86%; N, 13.01%.

### Polymerization of NIPTD with SebC

*Method 1*

Into a 25-mL round bottom flask, NIPTD (0.1333 g, 3.58*$10^{-4}$ mol ) and pyridine (0.058 mL, 7.16*$10^{-4}$ mol) were added to a solution of SebC **(7)** (0.0856 g, 3.58*$10^{-4}$ mol) in 0.7 mL of NMP. The reaction mixture was stirred for 3 h at -5°C, then for 22 h at room temperature. The viscous solution was precipitated in 50 mL of methanol. The solid was filtered off, dried to give 0.1839 g (84.0%) of off-white polyamide PA3A mp 256-268°C. The above polymerization was repeated, in the presence of triethylamine and dibutyltin dilurate as a catalyst, respectively.

*Method 2*

The above polymerization was repeated, but the reaction mixture was refluxed in NMP for 1 min in the presence of different catalysts.

IR(KBr): 3173 (w), 3072 (m), 1753 (s), 1720 (s), 1662 (s), 1587 (s), 1518 (s), 1429 (s), 1375 (s), 1356 (s), 1292 (s), 1238 (s), 1190 (s), 1155 (s), 1022 (m), 898 (m), 829 (m), 779 (s), 586 (m), 532 (m) cm$^{-1}$. $^1$H NMR (500 MHz, DMSO-$d_6$): δ 1.24-1.69 (m, br, 12H), 2.52 (s, overlapped with DMSO peak, 4H), 7.55-7.68 (m, br, 4H), 7.91-7.95 (distorted dd, 2H), 8.52-8.55 (distorded dd, 4H, $J_1$ = 7.44 Hz). Anal. Calcd for $C_{30}H_{26}N_4O_6$: C, 66.91%; H, 4.86%; N, 10.40%. Found: C, 67.33%; H, 5.03%; N, 10.44%.

## RESULTS AND DISCUSSION

### Monomer Synthesis

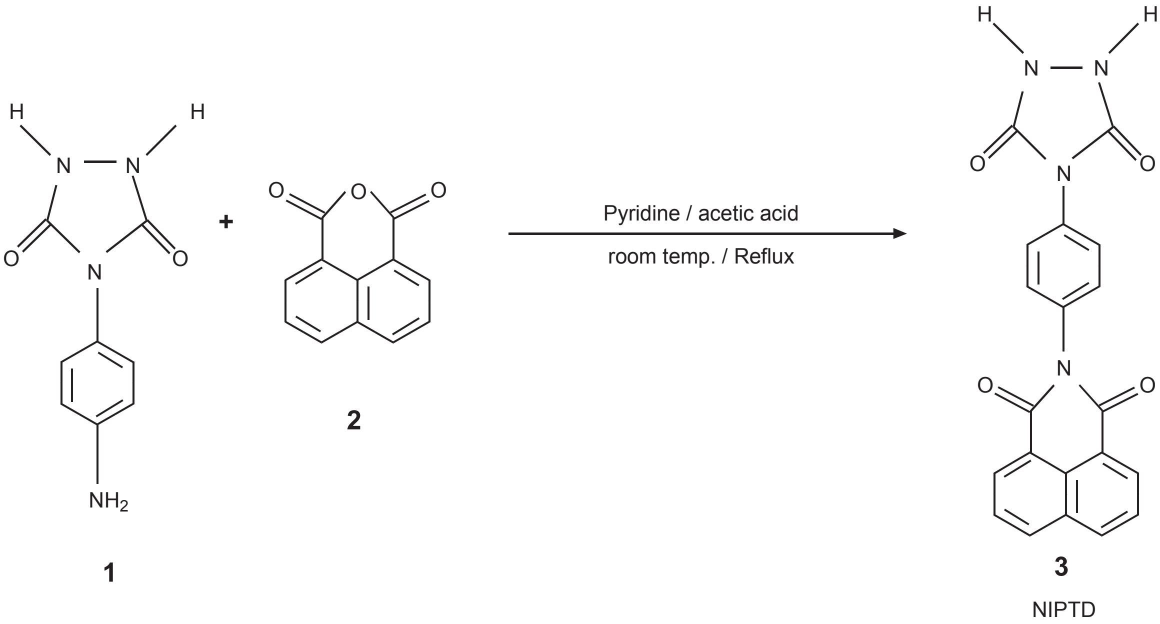
The monomer 4-(4í-*N*-1,8-naphthalimidophenyl)-1,2,4-triazolidine-3,5-dione (NIPTD) **(3)** was synthesized from the reaction of 4-(4í-aminophenyl)-1,2,4-triazolidine-3,5-dione **(1)** with 1,8-naphthalic anhydride **(2)** in a mixture of acetic acid and pyridine (3:2) under refluxing temperature (Scheme I). The purity of monomer **3** was checked by TLC which shows one spot in chloroform/methanol mixture (60:40) with $R_f$ = 0.54. The structure of this monomer was confirmed by IR, $^1$H NMR, UV-Vis spectra and elemental analysis.

### Model Compounds Studies

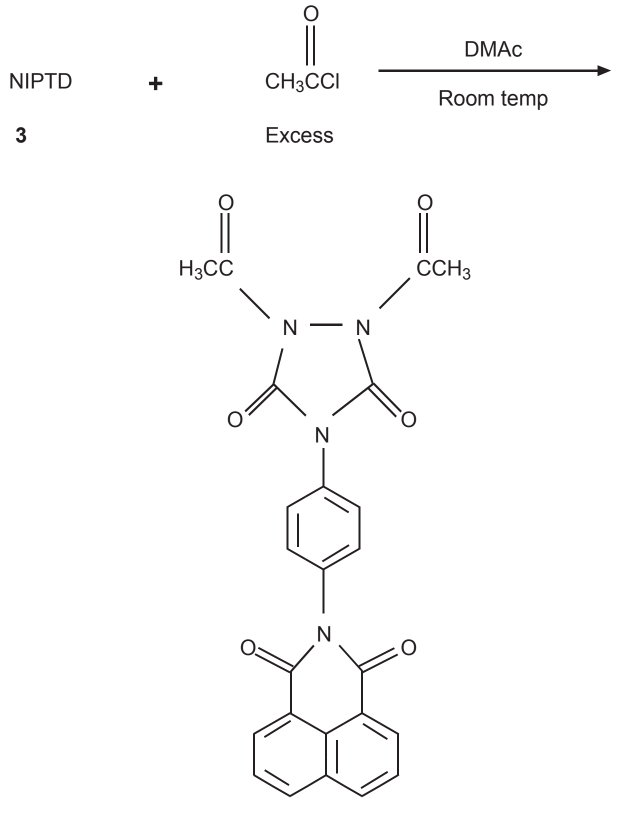
NIPTD **(3)** was allowed to react with excess acetyl chloride in DMAc solution at room temperature for 36 h and gave 1,2-bisacetyl-4-[4í-(1,8-naphthalimidophenyl)]-1,2,4-triazolidine-3,5-dione **(4)** in high yield (Scheme II). The compound **(4)** was characterized by IR, $^1$H NMR, UV-Vis spectra and elemental analysis. The IR spectrum of **4** does not show any peaks around 3350 cm$^{-1}$ for the N-H bond, this indicates that both N-H,s have been reacted with acetyl chloride. It also shows three peaks at 1770, 1709 and 1656 cm$^{-1}$ for the carbonyl groups. The first two peaks are characteristic pattern for the urazole moiety. The $^1$H NMR spectrum of **4** showed all peaks which are in agreement with the structure of compound **4** (**Figures** 1,2). The purity of this model compound **4** was checked by TLC which shows one spot in cyclohexane/ethylacetate mixture (20:80) with $R_f$ = 0.42.

### Polymerization Reactions

Because 1,2-bisacetyl-4-[4í-(1,8-naphthalimidophenyl)]-1,2,4-triazolidine-3,5-dione **(4)** as a model compound was synthesized in high yield and purity we became interested to perform this type of the reaction for the formation of the novel polyamides. Thus SucC **(5),** SubC **(6)** and SebC **(7)** were selected as diacid chlorides. The polymerization reaction of monomer **3** with these diacid chlorides were performed under conventional solution polymerization techniques at low as well as high temperture in the presence of different catalysts PA1-PA3 (Scheme III). The polymerization reac-

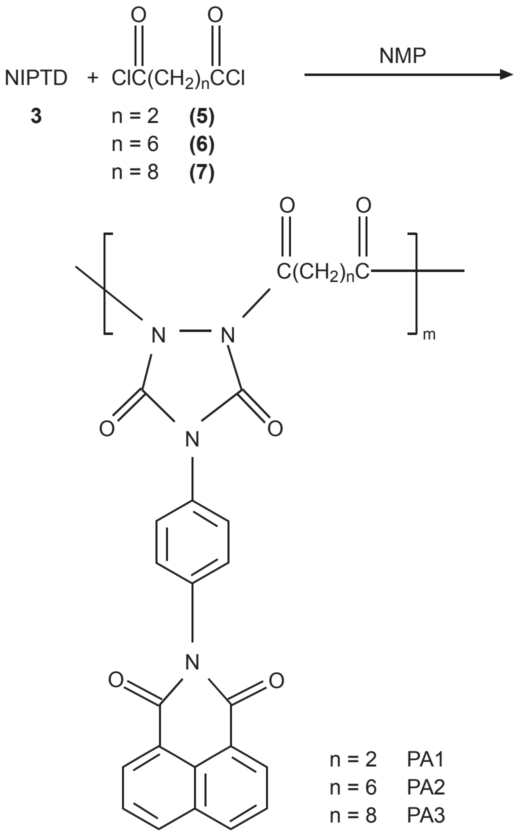


**Scheme I.** Synthesis of monomer **3**.



**4**

**Scheme II.** Synthesis of model compound **4**.



**Scheme III.** Polycondensation reactions of monomer **3** with aliphatic diacid chlorides.

tion of monomer **3** with SucC was carried out with two different methods. In method 1 the reaction mixture was kept at -5°C, then it was allowed to come to room temperture in the presence of pyridine, dibutyltin dilaurate and triethylamine, respectively. The resulting polyamides PA1A-PA1C have good inherent viscosity, but lower yield. In method 2 the reaction mixture was refluxed up for 1 min in NMP. The resulting

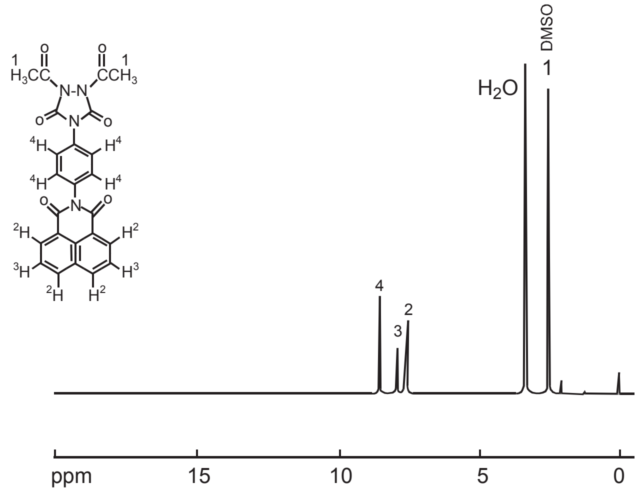


**Figure 1.** $^1H$ NMR (500 MHz) Spectrum of model compound **4** in DMSO-$d_6$ at room temperature.

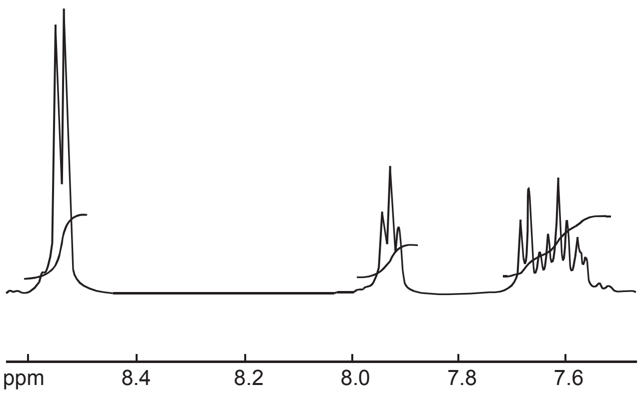


**Figure 2.** $^1H$ NMR (500 MHz) Spectrum of model compound 4 in DMSO-$d_6$ at room temperature. Expanded region for the aromatic protons ($\delta$ = 7.50-8.60 ppm).

**Table 1.** Reaction conditions for the polymerization of monomer NIPTD **(3)** with SucC **(5)** by different methods and some physical properties for PA1A-PA1F.

| Polymer | Method[a] | Catalyst | Yield (%) | Inherent viscosity (dL/g)[e] |
|---|---|---|---|---|
| PA1A | I | Py[b] | 75.0 | 0.19 |
| PA1B | I | TEA[c] | 73.0 | 0.14 |
| PA1C | I | DBTDL[d] | 68.0 | 0.10 |
| PA1D | II | Py | 81.0 | 0.12 |
| PA1E | II | TEA | 86.0 | 0.12 |
| PA1F | II | DBTDL | 83.0 | 0.09 |

(a) Method 1: reaction time: 3 h, -5°C; 16 h, R.T., Method 2: refluxing at 1 min; (b)Pyridine, (c)Triethylamine, (d)Dibutyltin dilaurate; (e)Measured at a concentration of 0.5 g/dL in $H_2SO_4$ at 25°C.

polyamides PA1D-PA1F have high yield and good inherent viscosity. Reaction conditions and some physical properties for PA1A-PA1F are summarized in Table 1. The resulting polymers were characterized by IR, $^1$H NMR, elemental analysis, UV-vis and TGA. The IR spectrum of polymer PA1 showed two peaks at 1720 and 1668 cm$^{-1}$ for the carbonyl groups. The $^1$H NMR spectrum (Figures 3,4) of polymer PA1 showed all peaks for aliphatic and aromatic as well as end group N-H protons. The elemental analysis result is also in good agreement with calculated percentages for carbon, hydrogen and nitrogen contents in polymer repeating unit.

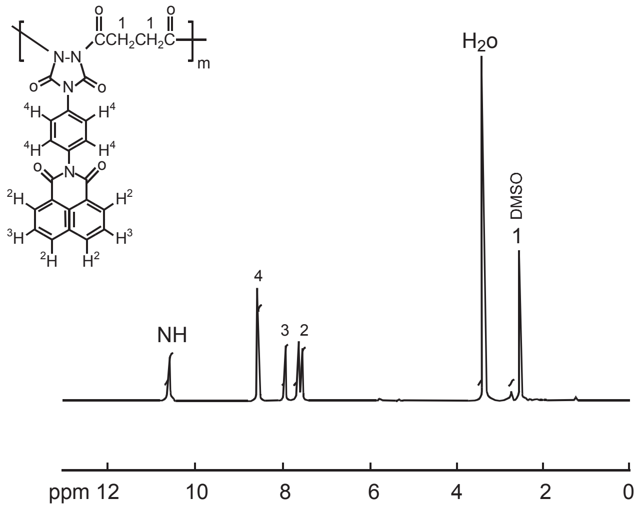


**Figure 3.** $^1$H NMR (500 MHz) Spectrum of PA1 in DMSO-$d_6$ at room temperature.

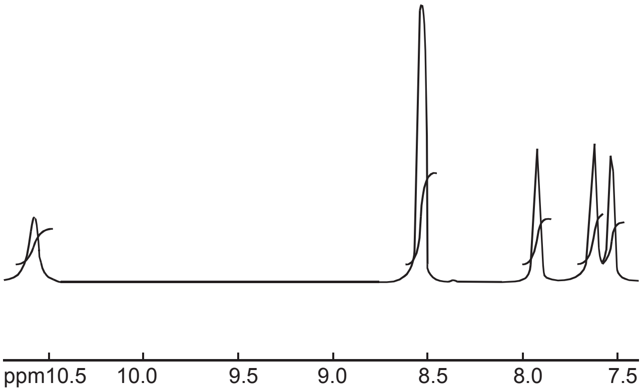


**Figure 4.** $^1$H NMR (500 MHz) Spectrum of PA1 in DMSO-$d_6$ at room temperature. Expanded region for the aromatic protons (δ = 7.40-10.70 ppm).

The polymerization reaction of monomer **3** with SubC was also carried out with two different methods (Table 2). We obtained comparable results of two methods.

The IR spectrum of polymer PA2 showed two strong peaks at 1749 and 1709 cm$^{-1}$ for the carbonyl groups which are related to the urazole moiety. The $^1$H NMR spectrum (Figures 5-7) of polymer PA2 showed peaks that confirm its chemical structure. The elemental analysis result are also in good agreement with calculated percentages for carbon, hydrogen and nitrogen contents in polymer repeating unit of PA2.

The polymerization reaction of monomer **3** with

**Table 2.** Reaction conditions for the polymerization of monomer NIPTD **(3)** with SubC **(6)** by different methods and some physical properties for PA2A-PA2F.

| Polymer | Method[a] | Catalyst | Yield (%) | Inherent viscosity (dL/g)[e] |
|---|---|---|---|---|
| PA2A | I | Py[b] | 81.5 | 0.16 |
| PA2B | I | TEA[c] | 72.0 | 0.13 |
| PA2C | I | DBTDL[d] | 80.0 | 0.14 |
| PA2D | II | Py | 69.5 | 0.14 |
| PA2E | II | TEA | 75.0 | 0.12 |
| PA2F | II | DBTDL | 82.0 | 0.11 |

(a)Method 1: reaction time: 3 h, -5°C; 22 h, R.T., Method 2: refluxing at 1 min; (b)Pyridine, (c)Triethylamine, (d)Dibutyltin dilaurate; (e)Measured at a concentration of 0.5 g/dL in $H_2SO_4$ at 25°C.

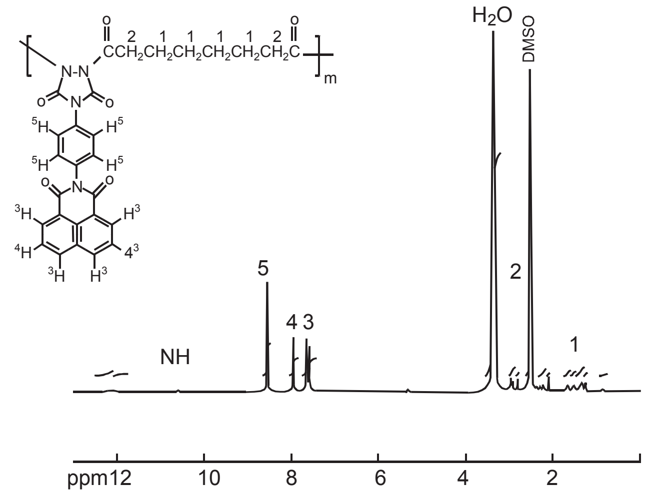


**Figure 5.** $^1$H NMR (500 MHz) Spectrum of PA2 in DMSO-$d_6$ at room temperature.

SebC was also performed with two different methods (Table 3). Here also we obtained comparable results of two methods.

The IR spectrum of polymer PA3 showed two strong peaks at 1753 and 1720 cm$^{-1}$ for the carbonyl groups which are related to the urazole moiety. The $^1$H NMR spectrum of polymer (Figures 8-10) PA3 showed peaks that confirm its chemical structure. The elemental analysis results are also in good agreement with calculated percentages for carbon, hydrogen and nitrogen contents in polymer repeating unit of PA3.

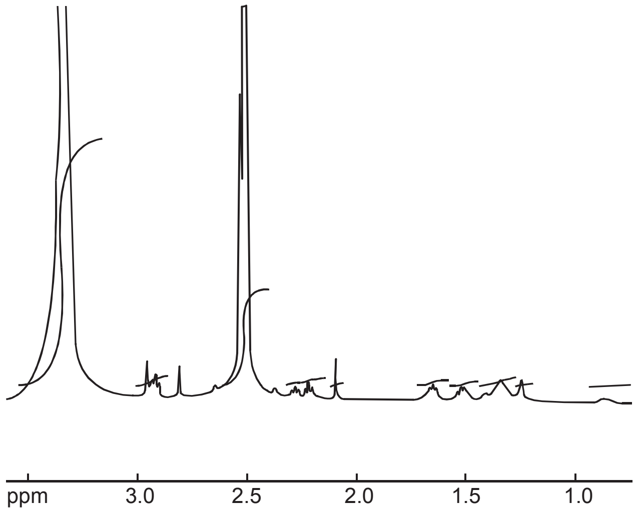


**Figure 6.** $^1$H NMR (500 MHz) Spectrum of PA2 in DMSO-$d_6$ at room temperature. Expanded region for the aliphatic protons (δ= 0.80-3.60 ppm).

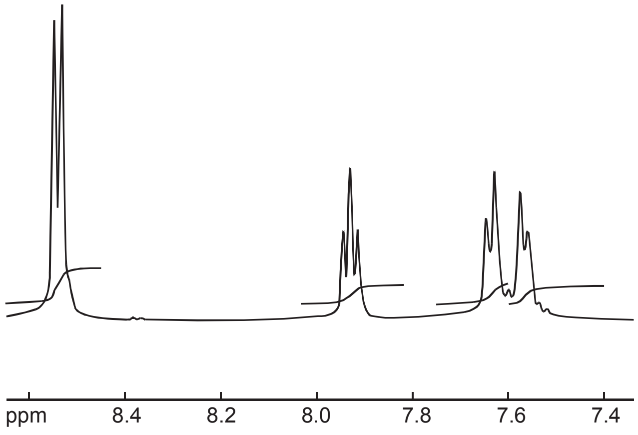


**Figure 7.** $^1$H NMR (500 MHz) Spectrum of PA2 in DMSO-$d_6$ at room temperature. Expanded region for the aromatic protons (δ = 7.40-8.60 ppm).

The UV-Vis spectra of the monomer **3**, model compound **4**, polyamides PA1, PA2 and PA3 were recorded in DMF and the data are shown in Table 4. It is apparent that the wavelength of maximum absorptions are related to the naphthalene moieties in the compounds. All of these compounds show almost similar UV-Vis spectra pattern.

The polymers PA1 PA2 and PA3 are soluble in organic solvents such as NMP and $H_2SO_4$, partial soluble in DMF, DMSO, DMAc and are insoluble in solvents such as water, methanol, acetone, cyclohexane and chloroform.

**Table 3.** Reaction conditions for the polymerization of monomer NIPTD **(3)** with SebC **(7)** by different methods and some physical properties for PA3A-PA3F.

| Polymer | Method$^a$ | Catalyst | Yield (%) | Inherent viscosity (dL/g)$^e$ |
|---|---|---|---|---|
| PA3A | I | Py$^b$ | 84.0 | 0.15$^e$ |
| PA3B | I | TEA$^c$ | 79.0 | 0.12$^e$ |
| PA3C | I | DBTDL$^d$ | 73.5 | 0.08$^f$ |
| PA3D | II | Py | 75.0 | 0.14$^e$ |
| PA3E | II | TEA | 70.5 | 0.12$^e$ |
| PA3F | II | DBTDL | 82.0 | 0.12$^e$ |

(a)Method 1: reaction time: 3 h, -5°C; 22 h, R.T., Method 2: refluxing at 1 min; (b)pyridine; (c)triethylamine; (d)dibutyltin dilaurate; (e)Measured at a concentration of 0.5 g/dL in $H_2SO_4$ at 25°C; (f)Measured at a concentration of 0.5 g/dL in DMF at 25°C.

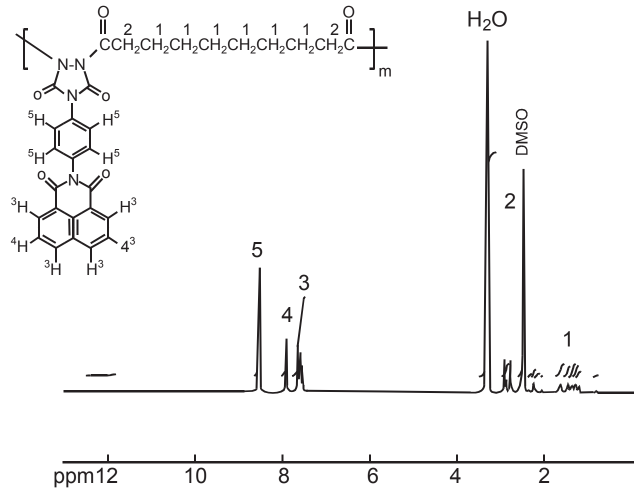


**Figure 8.** $^1$H NMR (500 MHz) Spectrum of PA3 in DMSO-$d_6$ at room temperature.

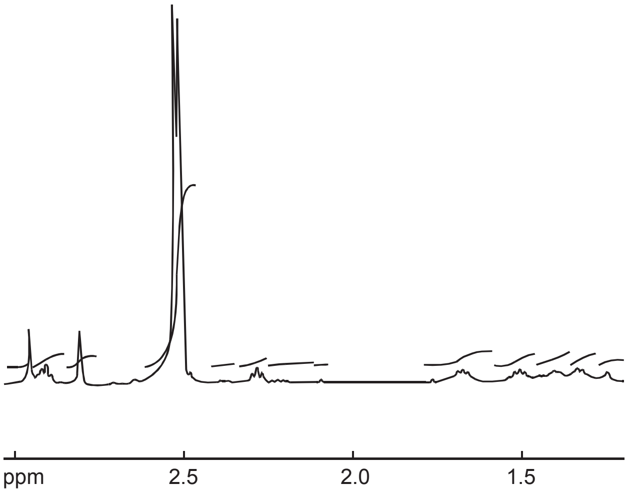


**Figure 9.** $^1$H NMR (500 MHz) Spectrum of PA3 in DMSO-$d_6$ at room temperature. Expanded region for the aliphatic protons ($\delta$ = 1.20-3.00 ppm).

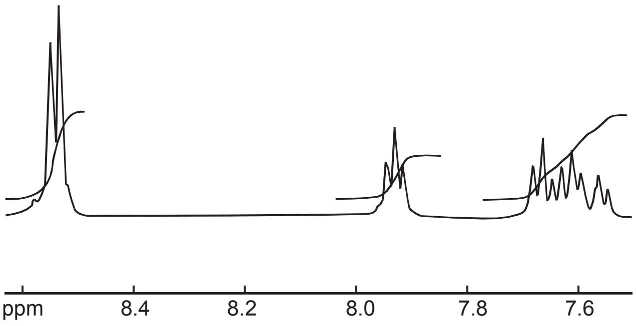


**Figure 10.** $^1$H NMR (500 MHz) Spectrum of PA3 in DMSO-$d_6$ at room temperature. Expanded region for the aromatic protons ($\delta$ = 7.50-8.60 ppm).

**Table 4.** UV-Vis spectra of monomer **3**, model compound **4** and polymers PA1-PA3 in DMF.

| Code | Colour | $\lambda_{Max}$(A$\varepsilon$, M$^{-1}$ cm$^{-1}$) |
|---|---|---|
| Compound **3** | White | 265 ( 0.193, 3596)<br>336 ( 0.428, 7963)<br>348 ( 0.383, 7128) |
| Compound **4** | Off white | 334 ( 0.939, 12795)<br>263 ( 0.498, 6785)<br>349 ( 0.804, 10955) |
| PA1 | Pale brown | 334 ( 0.877, 19937)<br>263 ( 0.374, 8493)<br>351 ( 0.731, 16606) |
| PA2 | Pale brown | 334 ( 0.721, 18633)<br>263 ( 0.519, 13419)<br>348 ( 0.610, 15752) |
| PA3 | Off white | 334 ( 0.929, 19976)<br>263 ( 0.622, 13378)<br>351 ( 0.756, 16252) |

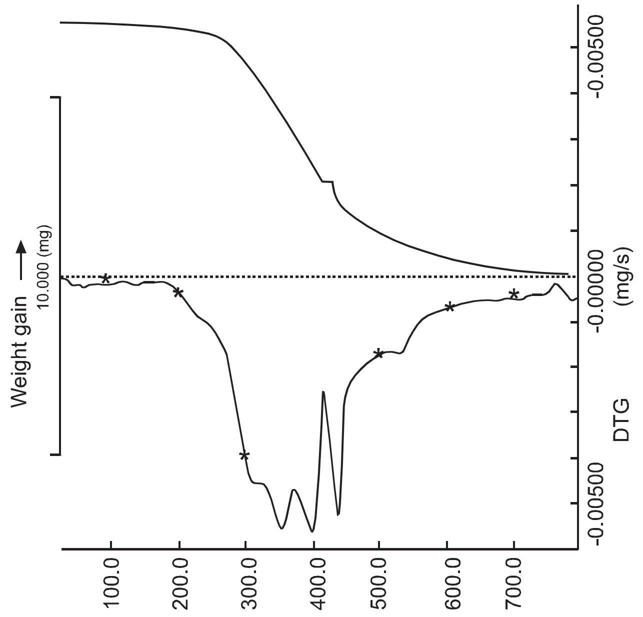


**Figure 11.** TGA/DTG of PA1 with a heating rate of 10°C/min in nitrogen atmosphere.

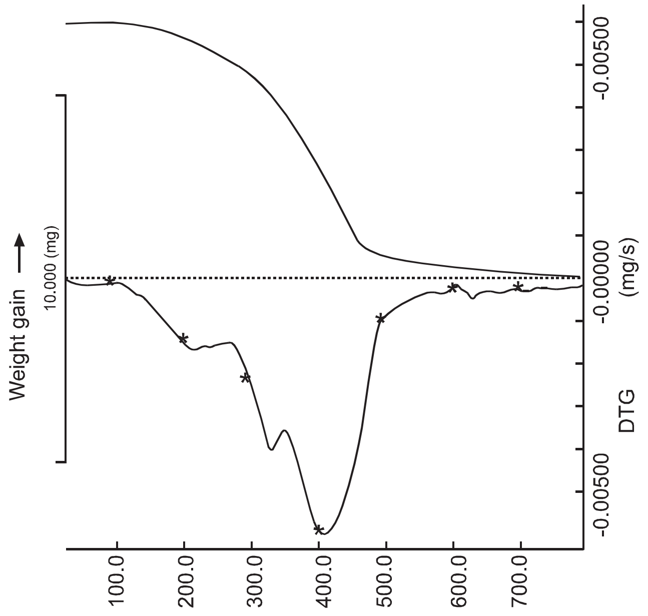


**Figure 12.** TGA/DTG of PA3 with a heating rate of 10°C/min in nitrogen atmosphere.

### Thermal Properties

The thermal behaviour of polyamides PA1, PA2 and PA3 were measured by thermogravimetric analysis (TGA) at a rate of 10°C/min in nitrogen atmosphere. An examination of the data reveals that all of the above polyamides are thermally stable up to 200°C. Some weight losses below 150°C are due to water or solvent loss. The temperatures of 5% and 10% weight loss together with char yield at 600°C for these polyamides are 265°C, 253°C, 200°C, 310°C, 315°C, 255°C, 29.38%, 14.34% and 15.10%, respectively. (Figures 11,12) show thermograms of PA1 and PA3.

## CONCLUSION

This investigation has shown that NIPTD **(3)** is an interesting monomer for the polycondensation reactions. This compound has two acidic N-H groups and it can readily react with acetyl chloride. Thus compound **(3)** can act as a bifunctional monomer and its polymerization reaction with aliphatic diacid chlorides gave novel polyamides with naphthalene containing urazole linkages having inherent viscosities of 0.08-0.19 dL/g, which roughly correspond to molecular weight of 4000 to 10000 g/mol. Although all catalysts used in this polymerization reactions are useful, but pyridine seems to be more effective. We are currently using this method for the synthesis of novel polymers and modification of polymers. Since these polyamides have naphthalene moieties, they have potential to be used as photoactive materials.

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