Polymer Bulletin 57, 611–621 (2006)
DOI 10.1007/s00289-006-0604-5

Polymer Bulletin

Step-Growth Polymerization of 4-(1-Naphthyl)-1,2,4-triazolidine-3,5-dione with Diisocyanates

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Received: 29 March 2006 / Revised version: 12 May 2006 / Accepted: 15 May 2006

Summary

We have synthesized 4-(1-naphthyl)-1,2,4-triazolidine-3,5-dione (**4-NTD**) in high yield and purity from 1-naphthyl isocyanate and used it as a new monomer for polymer synthesis. A series of novel naphthalene-containing polyureas have been successfully prepared from **4-NTD** with hexamethylene diisocyanate (**HMDI**), isophorone diisocyanate (**IPDI**) and toluene-2,4-diisocyanate (**TDI**) in *N,N*-dimethylacetamide (DMAc) or *N*-methylpyrrolidone (NMP) solution in the presence of pyridine, triethylamine or dibutyltin dilurate as catalysts. Some physical properties and structural characterization of these novel polyureas are reported. Fluorimetric studies of the new monomer, model compound as well as polymers were also investigated.

Introduction

Polyureas are a class of polymers which has a –NH-CO-NH- functional group; they can make hydrogen bonds from different sides. They are polyamide of carbonic acid and are tough, high melting and suitable for fiber applications [1]. They can be tailor-made to obtain properties which lead to versatile applications such as coating systems for waterproofing and corrosion protection [2].

Chromophoric polymers have been extensively explored in the last decade due to their potential technological application in electronic and opto-electronic devices [3-4]. Naphthalene structure as a chromophore gave special features such as rigid, bulky, moisture resistance and low coefficient of thermal expansion to polymer. Naphthalene has also been incorporated into the backbones of many polymers including polyesters, polyamides, bismaleimides and etc. The incorporation of naphthalene as a pendant group in a polymer chain has resulted in decreased crystallinity and enhanced solubility and thermal stability [5]. In order to improve the heat resistance of phenyl polyimide, some naphthyl polyimides have been developed, such as naphthalene pendant group.

Urazole derivatives are very fascinating five-membered heterocyclic compounds, which at position 4 can provide a wide variety of aliphatic as well as aromatic substituents. Urazole is an important chemical reagent in the laboratory and in industry. Industrially urazole is used in the manufacture of automobile air bags, as a blowing agent in plastics, in the production of herbicides [6], in the production of antifungal compounds and in polymeric materials [7]. It is also used in the production of anti-tumor drugs [8-9] and as a stabilizer in milk. Urazole is used as a laboratory reagent for synthesizing novel heterocyclic [10-13] and organometallic compounds.

As part of our continuing efforts in developing heterocyclic polymers containing urazole units [14-16], the present study describes the synthesis of a new urazole, 4-(1-naphthyl)-1,2,4-triazolidine-3,5-dione (**4-NTD**) containing naphthalene as chromophore group and its application for the synthesis of novel photoactive polyureas.

Experimental

Materials

Reagents were purchased from Fluka Chemical (Buchs, Switzerland), Aldrich Chemical (Milwaukee, WI) and Riedel-deHaen AG (Seelze, Germany). DMAc (*N,N*-dimethylacetamide) was dried over BaO and then was distilled under reduced pressure.

Equipments

Proton nuclear magnetic resonance ^{1}H-NMR (300MHz) spectra were recorded on a Bruker (Germany), Avance 300 instrument. Multipilicities of proton resonance were designated as singlet (s), triplet (t), and multiplet (m). FT-IR spectra were recorded on JASCO-680 (Japan) spectrophotometer. Spectra of solids were carried out using KBr pellets. Vibrational transition frequencies are reported in wave number (cm^{-1}). Band intensities are assigned as weak (w), medium (m), shoulder (sh), strong (s) and broad (br). All melting points were taken with a Gallenham melting point apparatus. Inherent viscosities were measured by standard procedure using a Cannon-Fenske Routine viscometer. Elemental analyses were performed by Research Institute of petroleum Industry, Tehran, I.R. Iran. Fluorescence and UV-Vis spectra were recorded on a JASCO FP-750 spectrofluorometer, and JASCO, V-570, UV/VIS/NIR spectrophotometer, respectively. Mass spectra were recorded on a Fisons Trio 1000 spectrometer. Thermal Gravimetric Analysis (TGA) data for polymers were taken on a Stanton-650 TGA under N_2 atmosphere by the Research Institute of Polymer and Petrochemical of Iran (IPPI).

Preparation of 1-ethoxycarbonyl-4-(1-naphthyl) semicarbazide

Into a 250-mL round bottom flask was placed (5.04 g, 2.98×10^{-2} mol) of 1-naphthyl isocyanate in 100 mL of dry toluene. The clear solution was cooled in an ice bath then a solution (3.10 g, 2.98×10^{-2} mol) of ethyl carbazate in 40 mL of dry toluene was added dropwise. A white solid formed and then turned to a past, 120 mL of toluene was added and the mixture was stirred at room temperature for 20 h. The white precipitated was collected, dried under vacuum at 80°C to give 7.97 g (98%). m.p. 188°C (dec.). IR (KBr): 3307 (m), 2988 (m), 1759 (s), 1655 (s), 1635 (m), 1606 (m), 1558 (m), 1520 (m), 1444 (w), 1384 (w), 1356 (w), 1302 (w), 1222 (s), 1204 (s), 1058 (w), 1033 (w), 946 (w), 899 (w), 818 (m), 697 (w), 582 (w), 527 (w) cm^{-1}.

Preparation of 4-(1-naphthyl)-1,2,4-triazolidine-3,5-dione

Into a 100-mL round-bottom flask was placed (6.72 g, 2.45 × 10^{-2} mol) 1-naphthyl semicarbazide **(2)**, 20 mL of 4 M KOH and a magnetic bar. The mixture was heated between 60-70°C for 3 h. The clear solution was cooled and 20 mL of water was added. The yellow clear solution was acidified with conc. HCl (20 mL) to about pH = 2. Then 50 mL of water was added, the white precipitated was collected, dried under vacuum at 80°C for 20 h to give 5.45 g (98%). mp 248°C. IR (KBr): 3188 (m), 3053 (s), 2819 (m), 1766 (w), 1671 (s), 1635 (m), 1599 (w), 1509 (w), 1473 (s), 1448 (s), 1229 (m), 1115 (m), 830 (w), 798 (s), 779 (s), 770 (s), 678 (m), 642 (m), 553 (w), 538 (w), 479 (w), 418 (w) cm^{-1}; ^{1}HNMR (300 MHz, DMSO-*d6*): δ 7.52-7.67 (m, 5H, Ar-H), 8.02-8.10 (m, 2H, Ar-H), 10.61 (s, 2H, N-H) ppm. Mass (EI), m/z: 227 ([M^+], 100%), 169 (97%), 141 (28%), 114 (34%).

Preparation of 1,2-bis-(n-propylamidocarbonyl)-4-(1-naphthyl)-1,2,4-triazolidine-3,5-dione (model compound 4)

Into a 25-mL round-bottom flask, *n*-propylisocyanate (0.749 g, 8.81 × 10^{-3} mol) was added to a solution of **4-NTD (3)** (0.100 g, 4.40 × 10^{-4} mol) in 0.5 mL of dry DMAc. The solution was stirred for 72 h at room temperature. The excess *n*-propylisocyanate was removed by aspirator and the residue was percipitated in 30 mL of water. The resulting solid was filtered off, dried to give 0.167 g (95.5%) of white solid **4**. This solid was recrystallized from mixture of water and methanol, m.p. 249°C (dec). IR(KBr): 3414 (m), 3177 (m), 3055 (m), 1768(m), 1730 (s), 1674 (s), 1600 (w), 1539 (w), 1509 (w), 1471 (s), 1447 (s), 1229 (m), 1115 (m), 799 (s), 779 (s), 770 (s), 642 (m), 538 (w), 477 (w), 418 (w) cm^{-1}; ^{1}H NMR (300 MHz, DMSO-*d6*): δ 0.86 (t, 6H, J = 7.40 Hz, CH_3), 1.51 (m, 4H, CH_2), 3.17 (m, 4H, N-CH_2), 7.62-7.78 (m, 6H, Ar-H, N-H), 7.82-8.16 (m, 3H, Ar-H) ppm. Anal. Calcd for $C_{20}H_{21}N_5O_4$: 60.44% C; 5.83% H; 17.62% N. Found: 59.93% C; 5.88% H; 16.78% N. Mass (EI), m/z: 397 (1%), 311 (3%), 227 (100%), 169 (58%), 141 (15%).

Polymerization of 4-NTD with hexamethylene diisocyanate (HMDI)

The **PU**s were prepared by the following general procedure (using polymer **PU1** as an example).

Method I: Into a 25-mL round-bottom flask **4-NTD** (0.107 g, 4.7 × 10^{-4} mol) and triethylamine (0.06 mL, 4.7 × 10^{-4} mol) were added to a solution of **HMDI** (**5**) (0.0792 g, 4.47 × 10^{-4} mol) in 0.5 mL of DMAc. The solution was stirred for 2 h at room temperature, 4 h at 60°C and then 6 h at 90°C. During of this period 0.6 mL of DMAc was added. The viscous solution was precipitated in 30 mL of methanol. The solid was filtered off, dried to give 0.1713 g (92%) of polyurea **PU1B**. m.p. 231°C (dec). The above polymerization was repeated, in the presence of pyridine and dibutyltin dilurate as a catalyst, respectively. The reaction was also carried out in NMP as a solvent.

Method II: The above polymerization was repeated, but the reaction mixture was refluxed in DMAc or NMP for one min. in the presence of different catalysts.

IR(KBr): 3367 (m), 3077 (w), 2931 (m), 2871 (m), 1791 (m), 1746 (s), 1613 (s), 1539 (s), 1468 (s), 1419 (s), 1366 (s), 1259 (s), 1121 (m), 775 (s) cm^{-1}. ^{1}H-NMR (300 MHz, DMSO-d_6): δ 1.30-1.51 (m, br, 8H, CH_2), 3.24-3.48 (m, 4H, CH_2), 7.58-7.61 (m, br, 4H, Ar-H), 7.77 (s, N-H), 7.95-8.06 (m, br, 3H, Ar-H). Anal. Calcd for $C_{20}H_{21}N_5O_4$: 60.75% C; 5.35% H; 17.71% N. Found: 59.03% C,5.92% H, 17.92% N.

The other **PU**s were prepared with a similar procedure.

Polymer PU2:

IR(KBr): 3329 (m), 3061 (w), 2955 (s), 2925 (s), 2854 (m), 1785 (m), 1735 (s), 1673 (s), 1550 (s), 1468 (s), 1420 (s), 1307 (m), 1253 (m), 775 (s), 644 (w), 418 (w) cm^{-1}. ^{1}H-NMR (300 MHz, DMSO-d_6): δ 0.82-1.06 (m, br, 11H, CH_3, CH_2), 1.23 (s, br, 4H, CH_2), 2.73 (m, br, 2H, CH_2), 2.95 (s, 1H, CH), 5.60-5.81 (m, br, N-H), 7.42-8.25 (m, 9H, Ar-H, N-H), 12.12 (s, br, N-H).

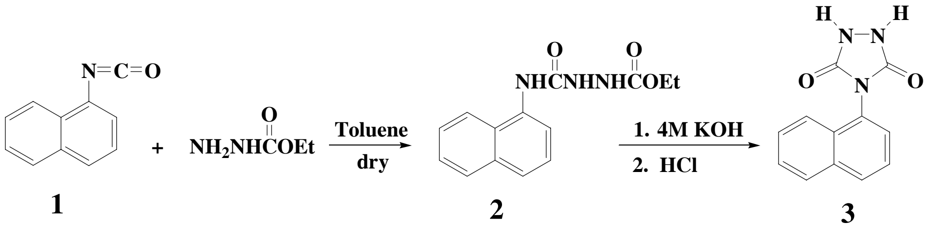
Polymer PU3:

IR(KBr): 3291 (s, br), 3061 (w), 2927 (w), 2856 (w), 1783 (s), 1735 (s), 1599 (s), 1599 (s), 1469 (s), 1424 (s), 1217 (s), 776 (s), 641 (m) cm^{-1}. ^{1}H-NMR (300 MHz, DMSO-d_6): δ 2.13-2.25 (m, 3H, CH_3), 7.07-8.26 (m, br, 10H, Ar-H), 9.68 (m, N-H), 12.37 (s, br, N-H).

Results and Discussion

Monomer synthesis

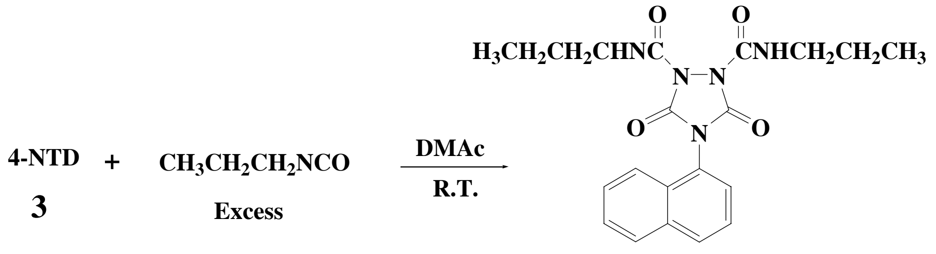
4-NTD (**3**) was synthesized in two steps starting from 1-naphthyl isocyanate. Isocyanate was reacted with ethyl carbazate in dry toluene and 1-ethoxycarbonyl-4-(1-naphthyl) semicarbazide was obtained in quantitative yield. Cyclization of compound 1-ethoxycarbonyl-4-(1-naphthyl) semicarbazide with 4 M KOH solution gave urazole (Scheme 1). The purity of the monomer **3** was checked by TLC. The structure of the urazole was also confirmed by IR, ^{1}H-NMR, UV-Vis, flurimetric, mass spectra and elemental analysis.



Scheme 1. Synthesis of monomer **3**.

Model Compound Study

4-NTD was allowed to react with excess *n*-isopropylisocyanate in DMAc solution at room temperature for 72 h and gave 1,2-bis-(*n*-propylamidocarbonyl)-4-(1-naphthyl)-1,2,4-triazolidine-3,5-dione **(4)** in high yield (Scheme 2). The compound **4** was characterized by IR, ^{1}H-NMR, UV-Vis, flurimetric, mass spectra and elemental analysis. The IR spectrum of **4** show a medium peak at 3414 cm^{-1} for the N-H bond. It also shows two peaks at 1768 and 1674 cm^{-1} for the carbonyl groups which are related to the urazole moiety. The ^{1}H-NMR spectrum of **4** showed all peaks which are in agreement with the structure of compound **4** (Figure 1). The purity of this model compound **4** was checked by TLC.



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Scheme 2. Synthesis of model compound **4**.

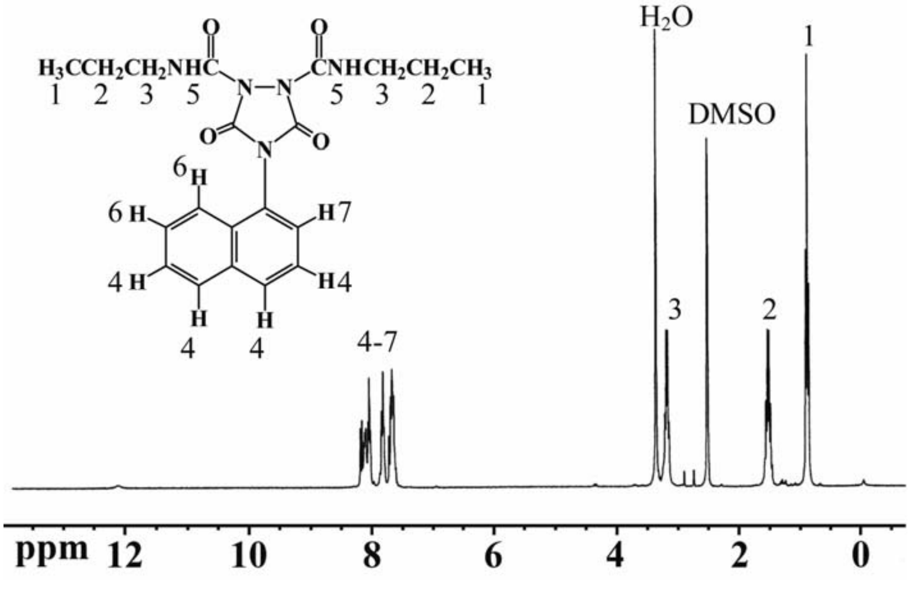
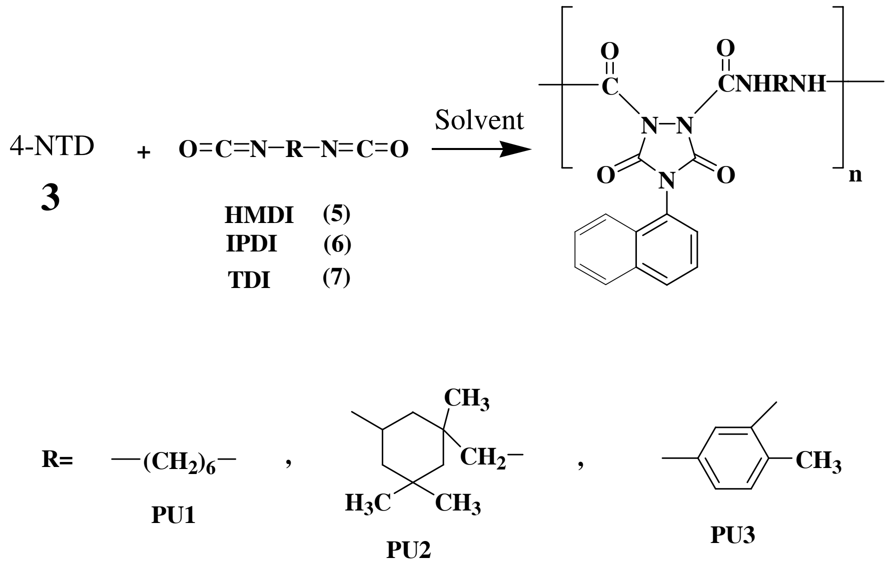


Figure 1. ^{1}H-NMR (300 MHz) Spectrum of model compound **4** in DMSO-d_6 at rt.

Polymerization Reactions

Because 1,2-bis-(*n*-propylamidocarbonyl)-4-(1-naphthyl)-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 polyureas.

Thus **HMDI (5)**, **IPDI (6)** and **TDI (7)** were selected as diisocyanates. The polymerization reaction of the monomer **3** with these diisocyanates were performed under conventional solution polymerization techniques as well as high temperture in the presence of different catalysts which gave polyureas **PU1-PU3** (Scheme 3). The polymerization reaction of the monomer **3** with **HMDI** was carried out with two different methods.



Scheme 3. Polycondensation reactions of the monomer **3** with different diisocyanates.

In method **I** the reaction mixture was heated gradually from room temperature to $90^{o}C$ in the presence of pyridine, dibutyltin dilaurate and triethylamine, respectively. The resulting polyureas **PU1A-PU1F** have moderate inherent viscosity and high yeilds. In method **II** the reaction mixture was refluxed up for 1 min. in DMAc. The resulting polyureas **PU1G-PU1L** have high yields and moderate inherent viscosity. Reaction conditions and some physical properties for **PU1A-PU1L** are summerized in Table 1. The polymerization reactions for the methods **I** and **II** were also carried out in NMP as a solvent and the results are comparable, which indicates both DMAc and NMP are suitable solvents for this polycondensation reaction. The resulting polymers were characterized by IR, ^{1}H-NMR, elemental analysis, UV-Vis and TGA techniques. The IR spectrum of polymer **PU1** showed two peaks at 1791 and 1746 cm^{-1} for the carbonyl groups. The ^{1}H-NMR spectrum (Figure 2) of polymer **PU1** showed all peaks for aliphatic and aromatic as well as N-H end group. The elemental analysis result is also in good agreement with the calculated percentages for carbon, hydrogen and nitrogen contents in polymer repeating unit. A film of the polymer could be formed from solusion casting.

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

Although the polymerization reactions were performed in DMAc or NMP as a solvent, but we obtained better results in DMAc solution.

The IR spectrum of polymer **PU2** showed two strong peaks at 1785 and 1735 cm^{-1} for the carbonyl groups which are related to the urazole moiety. The ^1H-NMR spectrum of polymer **PU2** showed peaks that confirm its chemical structure.

The polymerization reaction of the monomer **3** with **TDI** was also performed with two different methods (Table 3). Here also we obtained comparable results of two methods.

The IR spectrum of polymer **PU3** showed two strong peaks at 1791 and 1735 cm^{-1} for the carbonyl groups which are related to the urazole moiety.

Table 1. Reaction conditions for the polymerization of monomer **4-NTD (3)** with **HMDI (5)** by different methods and some physical properties for **PU1A-PU1L**

Polymer	*Method*[a]	*Solvent*	*Refluxing time (min.)*	*Catalyst*	*Yield (%)*	η_{inh} *(dL/g)*[e]
PU1A	I	DMAc	-	Py[b]	84	0.18
PU1B	I	DMAc	-	TEA[c]	92	0.26
PU1C	I	DMAc	-	DBTDL[d]	89	0.43
PU1D	I	NMP	-	Py	85	0.29
PU1E	I	NMP	-	TEA	79	0.36
PU1F	I	NMP	-	DBTDL	77	0.26
PU1G	II	DMAc	1	Py	85	0.31
PU1H	II	DMAc	1	TEA	92	0.27
PU1I	II	DMAc	1	DBTDL	86	0.45
PU1J	II	NMP	1	Py	74	0.30
PU1K	II	NMP	1	TEA	92	0.30
PU1L	II	NMP	1	DBTDL	82	0.28

[a] **Method I**: gradual heating at different temperatures, **Method II**: Refluxing at 1 min.

[b] pyridine, [c] triethylamine, [d] dibutyltin dilaurate.

[e] Measured at a concentration of 0.5 g/dL in DMF at 25°C.

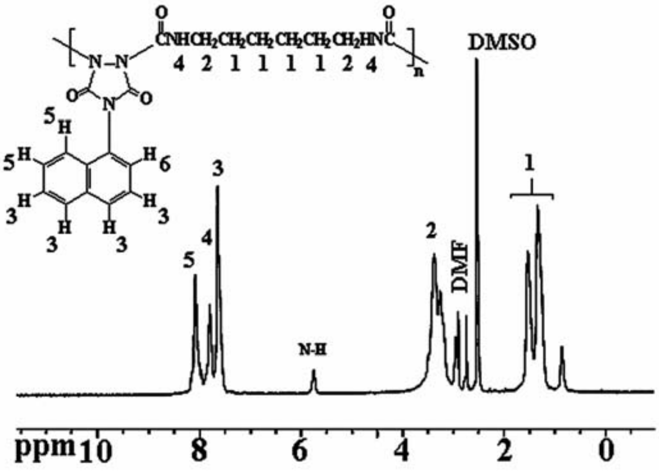


Figure 2. ^1H-NMR (300 MHz) Spectrum of **PU1** in DMSO-d_6 at rt.

Table 2. Reaction conditions for the polymerization of monomer **4-NTD (3)** with **IPDI (6)** by different methods and some physical properties for **PU2A-PU2L**

Polymer	*Method*[a]	*Solvent*	*Refluxing time (min.)*	*Catalyst*	*Yield (%)*	η_{inh} *(dL/g)*[e]
PU2A	I	DMAc	-	Py[b]	86	0.12
PU2B	I	DMAc	-	TEA[c]	74	0.11
PU2C	I	DMAc	-	DBTDL[d]	72	0.16
PU2D	I	NMP	-	Py	80	0.14
PU2E	I	NMP	-	TEA	68	0.08
PU2F	I	NMP	-	DBTDL	60	0.15
PU2G	II	DMAc	1	Py	81	0.17
PU2H	II	DMAc	1	TEA	72	0.14
PU2I	II	DMAc	1	DBTDL	61	0.19
PU2J	II	NMP	1	Py	71	0.10
PU2K	II	NMP	1	TEA	59	0.07
PU2L	II	NMP	1	DBTDL	66	0.15

[b] pyridine, [c] triethylamine, [d] dibutyltin dilaurate.

[e] Measured at a concentration of 0.5 g/dL in DMF at 25°C.

The UV-Vis spectra of the monomer **3**, the model compound **4**, polyureas **PU1**, **PU2** and **PU3** were recorded in *N,N*-dimethylformamide (DMF) and the data are shown in Table 4. It is apparent that the wavelength of maximum absorption are related to the naphthalene groups in the aforementioned compounds. All of these compounds show almost similar UV-Vis spectra pattern. Fluorescence spectra of the monomer **3**, the

Table 3. Reaction conditions for the polymerization of monomer **4-NTD (3)** with **TDI (7)** by different methods and some physical properties for **PU3A-PU3L**

Polymer	*Method*[a]	*Solvent*	*Refluxing time (min.)*	*Catalyst*	*Yield (%)*	η_{inh} *(dL/g)*[e]
PU3A	I	DMAc	-	Py[b]	89	0.26
PU3B	I	DMAc	-	TEA[c]	86	0.26
PU3C	I	DMAc	-	DBTDL[d]	85	0.35
PU3D	I	NMP	-	Py	90	0.22
PU3E	I	NMP	-	TEA	82	0.34
PU3F	I	NMP	-	DBTDL	90	0.31
PU3G	II	DMAc	1	Py	83	0.27
PU3H	II	DMAc	1	TEA	86	0.29
PU3I	II	DMAc	1	DBTDL	86	0.37
PU3J	II	NMP	1	Py	89	0.29
PU3K	II	NMP	1	TEA	70	0.34
PU3L	II	NMP	1	DBTDL	87	0.29

[a] **Method I**: gradual heating at different temperatures, **Method II**: Refluxing at 1 min.

[b] pyridine, [c] triethylamine, [d] dibutyltin dilaurate.

[e] Masured at a concentration of 0.5 g/dL in DMF at 25°C.

model compound **4**, polyureas **PU1**, **PU2** and **PU3** were also recorded in DMF and the data are shown in Table 5. For example, in case of the monomer **3**, the compound was excited at 282 nm, and emision fluorescence wavelengths were observed at 334, 355 and 375 nm, respectively.

All of these compounds show almost similar fluorescence spectra pattern**.** The fluorescent intensity of these compounds is good. The fluorescent intensity of **PU3** is higher than of **PU1** and **PU2**. It can be probably due to the rigidity enhancement of this polymer in compared with other two polymers. These fluorescent characteristic of monomer, model compound and polymers are due to naphthalene chromophore.

The solubility was determined by dessolving 5 mg of the polymers in 1 mL at room temperature. The polymers **PU1, PU2** and **PU3** are soluble in organic solvents such as NMP and in H_2SO_4, partial soluble in DMF, dimethylsulfoxide (DMSO), DMAc and are insoluble in solvents such as water, methanol, acetone, cyclohexane and chloroform.

Thermal Properties

The thermal behavior of polyureas **PU1I** and **PU3I** were measured by thermaogravimetric analysis (TGA) at a rate of 10°C/min in nitrogen atmosphere. An examination of the data reveals that polyureas **PU1I** and **PU3I** are thermally stable up to 200°C. The temperatures of 5% and 10% weight loss together with char yield at 600°C for these polyureas **PU1I** and **PU3I** are 214°C, 289°C, 14%, and 274°C, 312°C and 38%, respectively. Since **PU2**s have low inherent viscosity, it is expected to have low thermal stability. Figures 3 and 4 show thermograms of **PU1I** and **PU3I**.

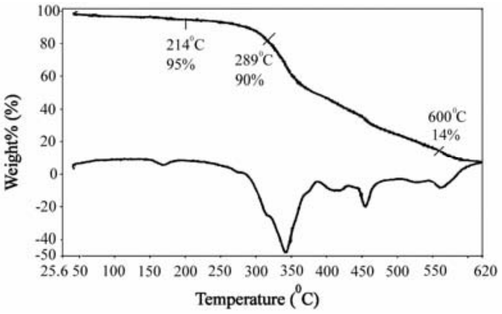


Figure 3. TGA/DTG of **PU1** with a heating rate of 10 °C/min in nitrogen atmosphere.

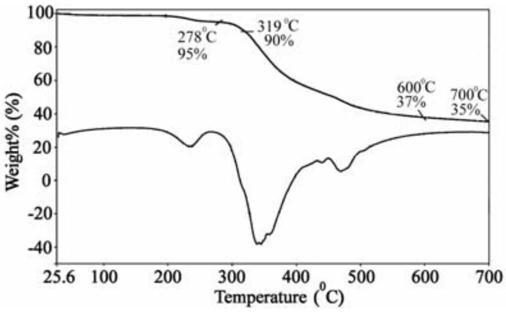


Figure 4. TGA/DTG of **PU3** with a heating rate of 10 °C/min in nitrogen atmosphere.

Table 4. UV-Vis spectra of the monomer **3**, the model compound **4** and polymers **PU1-PU3** in DMF

Code	Color	λ_{max}(A, ε M^{-1} cm^{-1})
Compound 3	White	263.5 (0.758, 3.8×10^{7})
Compound 4	White	281 (0.775, 4.1×10^{6}) 271 (0.701, 3.7×10^{6}) 260 (0.564, 3.0×10^{6})
PU1	White	281 (0.620, 3.2×10^{6}) 271.5 (0.560, 2.9×10^{6}) 260 (0.474, 2.5×10^{6})
PU2	White	294.5 (0.363, 2.1×10^{6}) 261.5 (0.731, 4.4×10^{6})
PU3	White	282 (0.635, 1.7×10^{7}) 272 (0.560, 1.5×10^{7}) 260 (0.512, 1.3×10^{7})

Table 5. Fluorescence spectra of the monomer **3**, the model compound **4** and polymers **PU1-PU3** in DMF

Code	C (M)	$\lambda_{Ex.}$ (nm)	$\lambda_{Em.}$ (nm)
Compound 3	1.98×10^{-8}	[a]268, 283 [b]278, 303, 326	[c]334, 355, 375 [d]363, 381
Compound 4	1.88×10^{-7}	[a]277, 320 [b]280, 304, 327	[c]336, 378 [d]331, 385
PU1	1.89×10^{-7}	[a]269, 283 [b]279, 300, 326	[c]336, 353, 382 [d]329, 380
PU2	1.66×10^{-7}	[a]270, 283 [b]279, 304, 326	[c]335, 377 [d]326, 381
PU3	3.73×10^{-8}	[a]283 [b]282, 301, 325	[c]340, 387 [d]326, 381

[a] $\lambda_{Em.}$ for scan emission spectrum = 282 nm.
[b] $\lambda_{Em.}$ for scan emission spectrum = 326 nm.
[c] $\lambda_{Ex.}$ for scan emission spectrum = 282 nm.
[d] $\lambda_{Ex.}$ for scan emission spectrum = 328 nm.

Conclusion

A new urazole **4-NTD** was prepared from 1-naphthyl isocyanate. This compound has two acidic N-H groups, and it can be readily reacted with *n*-propylisocyanate. Polymerization of this novel monomer with three different commercially available aliphatic and aromatic diisocyanates gave novel polyureas with naphthalene containing urazole linkages with inherent viscosities ranging from 0.07 to 0.45 dL/g, which roughly correspond to molecular weight of 5,000 to 15,000 g/mol. The resulting polyureas were prepared by different methods in the presence of different catalysts in different solvent such as DMAc or NMP. All of these methods gave interesting results. Since these polyureas have naphthalene moieties, they have potential to be used as photoactive materials.

Acknowledgements: We wish to express our gratitude to the Research Affairs Division Isfahan University of Technology (IUT), Isfahan, for partial financial support. Further financial support from Center of Excellency in Chemistry Research (IUT) is gratefully acknowledged.

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