

# Synthesis and Characterization of Novel Polyimides Containing Triazoles Units in the Main Chain by Click Chemistry

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**ABSTRACT:** In this study, some polyimides containing triazoles units in the main chain was prepared from the polymerization of dialkynes including imide linkages and diazides in the presence of Cu (I) catalyst in yield of 76.2–87.6%, with inherent viscosity of 0.37–0.53 dL g<sup>-1</sup>. The obtained polymers are soluble in polar aprotic solvents such as *N,N*-dimethylformamide (DMF), *N,N*-dimethylacetamide (DMAc), dimethyl sulfoxide (DMSO), and *N*-methyl-2-pyrrolidone (NMP). These polymers were characterized

using FT-IR, <sup>1</sup>H-NMR, and elemental analysis techniques. Their thermal stability was evaluated with thermogravimetric (TGA) analysis and differential scanning calorimetry (DSC) techniques under a nitrogen atmosphere which is indicative of their good thermal stability. © 2011 Wiley Periodicals, Inc. *J Appl Polym Sci* 124: 1757–1763, 2012

**Key words:** click chemistry; polyimides; polymerization; triazoles; diazo compounds

## INTRODUCTION

“Click” chemistry has attracted significant attention in recent years. It, established by Sharpless and co-workers,<sup>1</sup> refers to a group of reactions that are fast, simple to use, easy to purify, versatile, regiospecific, and that give high yields and near perfect fidelity in the presence of most functional groups.<sup>2–7</sup> Four major classifications of click reactions, including cycloadditions, nucleophilic ring-openings, carbonyl chemistry of the nonaldol type, and additions to carbon–carbon multiple bonds have been reported.<sup>1,5</sup> The most popular click chemistry reaction is the Huisgen dipolar cycloaddition reaction between an azide and an alkyne, leading to 1,2,3-triazole.<sup>7</sup> The reaction can be performed under mild experimental conditions using a copper (I) catalyst.<sup>8–12</sup> This reaction has found a vast number of applications in different research fields, such as material sciences,<sup>11,12</sup> polymer chemistry,<sup>13–21</sup> and pharmaceutical sciences.<sup>5,6,13–16</sup>

It has been extensively used in the synthesis of polymers with different composition and topology.<sup>8</sup> In spite of the importance of polyimides (PI), click

chemistry has not been widely employed for the preparation of these compounds. Polyimides exhibit a number of outstanding properties, such as excellent thermal and thermo-oxidative stability, solvent resistance, mechanical strength, low dielectric constants, and superior chemical resistance.<sup>22,23</sup> These polymers have a wide range of diverse and potential applications in several major technologies.<sup>24,25</sup>

The ability to manipulate and enhance the properties of PI is important to its widespread application. Following our investigation of the synthetic new polymers,<sup>26–30</sup> we wish to report the synthesis of some new thermal stable polyimides containing triazoles units in the main chain by click chemistry.

## EXPERIMENTAL

### Materials

*N*-Methyl-2-pyrrolidone (NMP, Merck) and pyridine (Py, Merck) were purified by distillation under reduced pressure over calcium hydride and stored over the 4Å molecular sieve. 3,3',4,4'-Benzophenone-tetracarboxylic-3,3',4,4'-dianhydride (**1a**) (Merck) and 4,4'-(hexafluoroisopropylidene)diphthalic anhydride (**1b**) (Aldrich) were recrystallized from the acetic anhydride. Reagent grade aromatic diamine (Aldrich) including 4,4'-diaminodiphenylsulfone (**4c**), 4,4'-diaminodiphenylether (**4d**) and 4,4'-diaminodiphenylmethane (**4e**) were recrystallized from water/ethanol

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(1: 1). 1,4-Phenylenediamine (**4f**) was purified by sublimation. CuBr (99%, Riedel-de Haën) was purified by stirring it with acetic acid, washed with methanol and ethanol, and finally dried under vacuum at 70°C. Propargylamine (99%, Aldrich) and sodium azide (99%, Merck) were used as received.

### Characterization

The FT-IR adsorption spectra were recorded on a JASCO 400D spectrophotometer using KBr pellets. Band intensities were assigned as weak (w), medium (m), and strong (s). Inherent viscosities of polymer solution ( $\eta_{inh}$  at a concentration of 0.5 g/dL) were measured by a standard procedure using a Cannon Fenske Routine viscometer at 25°C with dimethylformamide (DMF) as solvent. <sup>1</sup>H-NMR spectra were recorded on a Bruker Avance 500 MHz instrument (Bruker, Rheinstetten, Germany) using dimethylsulfoxide (DMSO-*d*<sub>6</sub>) as solvent. Thermal gravimetric analysis (TGA) and differential scanning calorimetry (DSC) were obtained by a Mettler Toledo TS0800GC1 instrument under nitrogen atmosphere at a heating rate of 5°C min<sup>-1</sup>. Melting points were measured in open capillaries with a Gallenkamp instrument. Elemental analyses were performed on a Heareus, CHN-O-RAPID analyzer.

### Synthesis of monomers

#### Preparation of diimides (**3a,3b**)

*N,N'*-Dipropargyl(3,3',4,4'-benzophenonetetracarboxylic-3,3',4,4')bisphthalimide (**3a**) and *N,N'*-dipropargyl(4,4'-hexafluoroisopropylidene)bisphthalimide (**3b**) were synthesized according to the published procedure for synthesis of **3b**.<sup>31</sup>

Diimide **3a**: Yellow solid; yield = 74.1%; m.p. (°C) = 179, FT-IR (KBr; cm<sup>-1</sup>): 3247 (m), 3069 (m), 2936 (m), 2125 (m), 1778 (s), 1718 (s), 1660 (m), 1482 (w), 1419 (m), 1390 (s), 1344 (m), 1289 (m), 1251 (m), 1181 (w), 1158 (w), 1115 (m), 1075 (w), 956 (w), 936 (w), 860 (w), 829 (w), 750 (w), 732 (m), 712 (m). <sup>1</sup>H-NMR (500 MHz, DMSO-*d*<sub>6</sub>),  $\delta$  (ppm), 8.20–8.09 (m, 6H, aromatic), 4.40 (d, 4H, *J*<sup>4</sup> = 2.45 Hz, CH<sub>2</sub>), 3.27 (t, 2H, *J*<sup>4</sup> = 2.45 Hz, ≡CH), <sup>13</sup>C-NMR (125 MHz, DMSO-*d*<sub>6</sub>),  $\delta$  (ppm), 193.15, 166.15, 166.12, 142.10, 136.07, 135.71, 132.84, 124.96, 124.58, 77.06, 72.39, 27.84. Anal. Calcd. for C<sub>23</sub>H<sub>12</sub>N<sub>2</sub>O<sub>5</sub> (396.36): C, 69.70%; H, 3.05%; N, 7.07%. Found: C, 69.34%; H, 2.92%; N, 6.91%.

#### Preparation of diazides **5(c-f)**

4,4'-Diazidodiphenylsulfone (**5c**), 4,4'-diazidodiphenylether (**5d**) and 4,4'-diazidodiphenylmethane (**5e**) were synthesized using the method reported

by Hergenrother et al.<sup>32</sup> and 1,4-diazidobenzene (**5f**) was prepared using the method reported by Hering.<sup>33</sup> Melting points and spectra analysis confirmed the synthesis of diazides.

### Polymer synthesis

The thermal [3 + 2] cycloaddition reactions were performed under nitrogen atmosphere. A 10 mL round-bottomed flask equipped with a magnetic stir bar was charged with diimide **3a** or **3b** (0.63 mmol), diazide **5(c-f)** (0.63 mmol), copper (I) bromide (0.04 mmol), and NMP (1 mL). The solution was heated at 50°C for 24 h. After cooling the reaction mixture, it was diluted with 20 mL tetrahydrofuran (THF) and passed through a short column of neutral alumina. The THF was removed under reduced pressure and the solution containing polymer was added to acetone, and the precipitate was collected by filtration and rinsed thoroughly with acetone. The desired polymers **6a(c-f)** and **6b(c-f)** were dried under vacuum for 24 h.

#### Polymer **6ac**

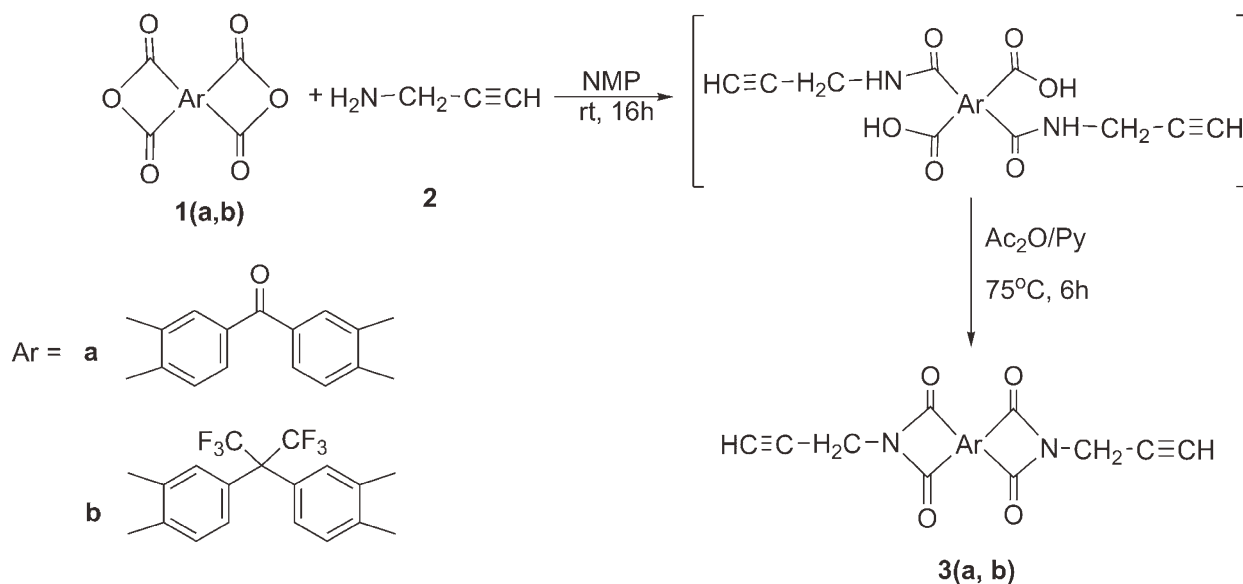
FTIR (KBr; cm<sup>-1</sup>): 3100 (m), 1776 (s), 1719 (s), 1593 (m), 1505 (m), 1435 (m), 1391 (m), 1332 (m), 1298 (m), 1246 (m), 1180 (m), 1156 (s), 1104 (m), 1044 (m), 987 (m), 840 (m), 768 (m), 716 (m). <sup>1</sup>H-NMR (500 MHz, DMSO-*d*<sub>6</sub>),  $\delta$  (ppm): 8.93 (s, 2H, triazole rings), 8.18–7.97 (m, 14H, aromatic), 4.96 (s, 4H, CH<sub>2</sub>). Anal. Calcd. for (C<sub>35</sub>H<sub>20</sub>N<sub>8</sub>O<sub>7</sub>S)<sub>n</sub> (696.66): C, 60.34%; H, 2.89%; N, 16.08%; S, 4.60%. Found: C, 60.21%; H, 2.78%; N, 15.95%; S, 4.53%.

#### Polymer **6ad**

FTIR (KBr; cm<sup>-1</sup>): 3070 (m), 1776 (s), 1719 (s), 1590 (m), 1507 (m), 1444 (m), 1391 (m), 1342 (m), 1294 (m), 1243 (s), 1186 (m), 1159 (m), 1104 (m), 1045 (m), 990 (w), 836 (m), 762 (w), 716 (m). <sup>1</sup>H-NMR (500 MHz, DMSO-*d*<sub>6</sub>),  $\delta$  (ppm): 8.75 (s, 2H, triazole rings), 8.19–7.14 (m, 14H, aromatic), 4.96 (s, 4H, CH<sub>2</sub>). Anal. Calcd. for (C<sub>35</sub>H<sub>20</sub>N<sub>8</sub>O<sub>6</sub>)<sub>n</sub> (648.59): C, 64.81%; H, 3.11%; N, 17.28%. Found: C, 64.53%; H, 2.98%; N, 16.97%.

#### Polymer **6ae**

FTIR (KBr; cm<sup>-1</sup>): 3066 (m), 1776 (s), 1718 (s), 1601 (m), 1518 (m), 1434 (m), 1390 (m), 1341 (m), 1292 (m), 1245 (m), 1157 (m), 1102 (m), 1045 (m), 990 (m), 845 (m), 793 (m), 716 (m). <sup>1</sup>H-NMR (500 MHz, DMSO-*d*<sub>6</sub>),  $\delta$  (ppm): 8.73 (s, 2H, triazole rings), 8.18–7.43 (m, 14H, aromatic), 4.94 (s, 4H, CH<sub>2</sub>), 4.08 (s, 4H, CH<sub>2</sub>–C<sub>6</sub>H<sub>4</sub>–). Anal. Calcd. for (C<sub>36</sub>H<sub>23</sub>N<sub>8</sub>O<sub>5</sub>)<sub>n</sub>



Scheme 1 Synthesis of diimide monomers 3(a,b).

(646.62): C, 66.87%; H, 3.43%; N, 17.33%. Found: C, 66.63%; H, 3.38%; N, 17.25%.

#### Polymer 6af

FTIR (KBr;  $\text{cm}^{-1}$ ): 3092 (m), 1777 (s), 1718 (s), 1598 (m), 1525 (m), 1433 (m), 1391 (m), 1344 (m), 1295 (m), 1247 (m), 1182 (m), 1158 (m), 1103 (m), 1045 (m), 987 (m), 842 (m), 764 (w), 716 (m).  $^1\text{H-NMR}$  (500 MHz,  $\text{DMSO}-d_6$ ),  $\delta$  (ppm): 8.91 (s, 2H, triazole rings), 8.18–7.68 (m, 14H, aromatic), 4.92 (s, 4H,  $\text{CH}_2$ ). Anal. Calcd. for  $(\text{C}_{29}\text{H}_{16}\text{N}_8\text{O}_5)_n$  (556.50): C, 62.59%; H, 2.90%; N, 20.14%. Found: C, 62.08%; H, 2.96%; N, 19.87%.

#### Polymer 6bc

FTIR (KBr;  $\text{cm}^{-1}$ ): 3100 (w), 1779 (s), 1724 (s), 1594 (m), 1505 (m), 1439 (m), 1392 (m), 1335 (m), 1255 (m), 1211 (m), 1155 (s), 1105 (m), 1043 (m), 987 (m), 843 (w), 773 (m), 719 (w).  $^1\text{H-NMR}$  (500 MHz,  $\text{DMSO}-d_6$ ),  $\delta$  (ppm): 8.90 (s, 2H, triazole rings), 8.19–7.88 (m, 14H, aromatic), 4.98 (s, 4H,  $\text{CH}_2$ ). Anal. Calcd. for  $(\text{C}_{37}\text{H}_{20}\text{F}_6\text{N}_8\text{O}_6\text{S})_n$  (818.67): C, 54.28%; H, 2.46%; N, 13.69%; S, 3.92%. Found: C, 54.02%; H, 2.39%; N, 13.61%; S, 3.87%.

#### Polymer 6bd

FTIR (KBr;  $\text{cm}^{-1}$ ): 3100 (m), 1779 (s), 1723 (s), 1607 (m), 1508 (m), 1439 (m), 1392 (m), 1340 (m), 1244 (s), 1212 (m), 1178 (m), 1141 (m), 1109 (m), 1045 (m), 990 (m), 838 (m), 771 (m), 720 (m).  $^1\text{H-NMR}$  (500 MHz,  $\text{DMSO}-d_6$ ),  $\delta$  (ppm): 8.74 (s, 2H, triazole rings), 8.07–7.13 (m, 14H, aromatic), 4.92 (s, 4H,  $\text{CH}_2$ ). Anal. Calcd. for  $(\text{C}_{37}\text{H}_{20}\text{F}_6\text{N}_8\text{O}_5)_n$  (770.61): C, 55.67%; H,

2.62%; N, 14.54%. Found: C, 55.52%; H, 2.53%; N, 14.36%.

#### Polymer 6be

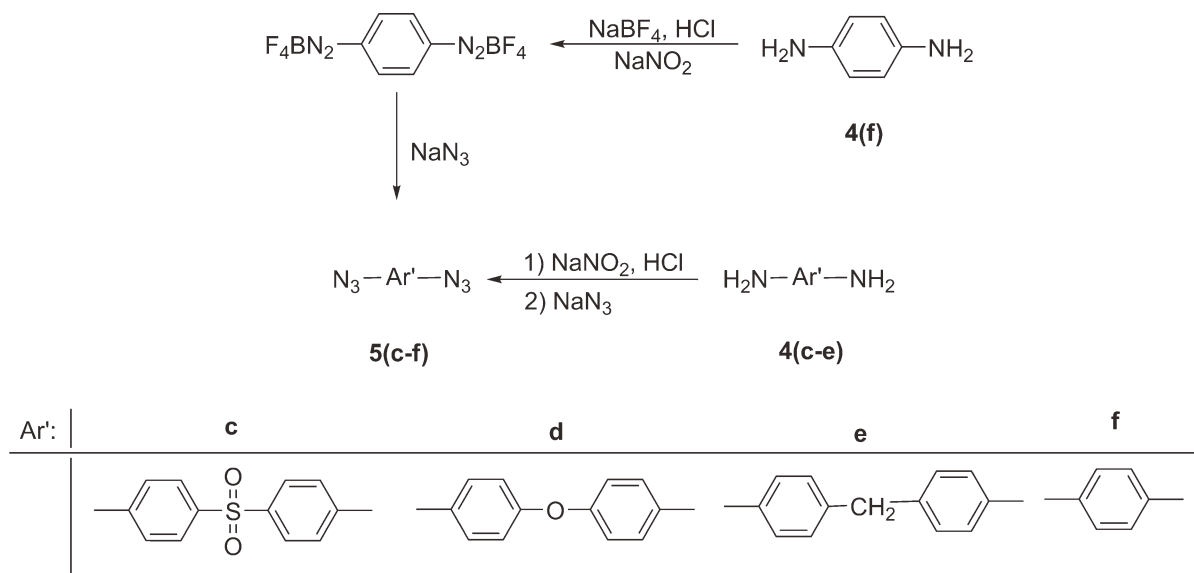
FTIR (KBr;  $\text{cm}^{-1}$ ): 3087 (m), 1779 (s), 1724 (s), 1600 (m), 1519 (m), 1438 (m), 1392 (m), 1345 (m), 1255 (m), 1211 (m), 1141 (m), 1110 (m), 1045 (m), 990 (m), 815 (w), 794 (w), 719 (w).  $^1\text{H-NMR}$  (500 MHz,  $\text{DMSO}-d_6$ ),  $\delta$  (ppm): 8.71 (s, 2H, triazole rings), 8.06–7.42 (m, 14H, aromatic), 4.90 (s, 4H,  $\text{CH}_2$ —triazole rings), 4.06 (s, 4H,  $\text{CH}_2$ — $\text{C}_6\text{H}_4$ —). Anal. Calcd. for  $(\text{C}_{38}\text{H}_{23}\text{F}_6\text{N}_8\text{O}_4)_n$  (768.63): C, 59.38%; H, 2.88%; N, 14.58%. Found: C, 59.19%; H, 2.75%; N, 14.49%.

#### Polymer 6bf

FTIR (KBr;  $\text{cm}^{-1}$ ): 3090 (m), 1780 (s), 1724 (s), 1615 (m), 1525 (m), 1439 (m), 1392 (m), 1346 (m), 1298 (m), 1211 (m), 1180 (m), 1141 (m), 1110 (m), 1043 (m), 986 (m), 844 (m), 771 (m), 720 (m).  $^1\text{H-NMR}$  (500 MHz,  $\text{DMSO}-d_6$ ),  $\delta$  (ppm): 8.87 (s, 2H, triazole rings), 8.05–7.70 (m, 14H, aromatic), 4.93 (s, 4H,  $\text{CH}_2$ ). Anal. Calcd. For  $(\text{C}_{31}\text{H}_{16}\text{F}_6\text{N}_8\text{O}_4)_n$  (678.51): C, 54.88%; H, 2.38%; N, 16.51%. Found: C, 54.79%; H, 2.31%; N, 16.75%.

## RESULTS AND DISCUSSION

Generally, aromatic polyimides have attracted much attention over the past years due to their thermal stability and vast number of applications. However, their processing is difficult. In this process, water is produced as a byproduct, which increases the bulk density, causes bulk shrinkage, and can produce

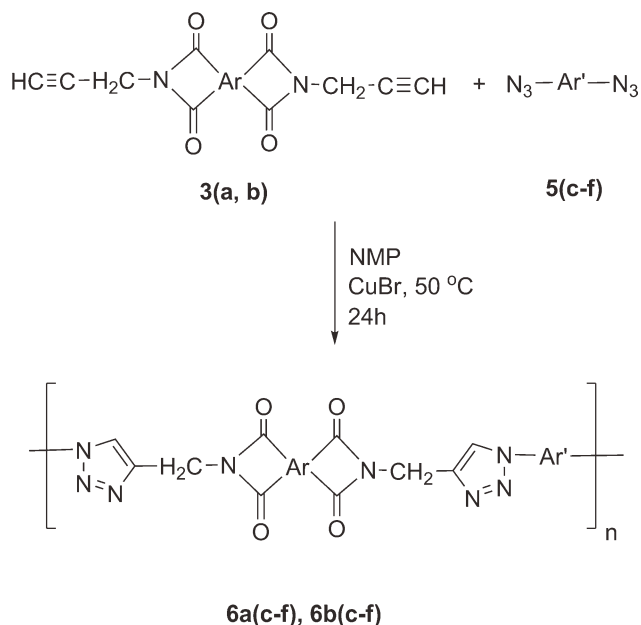


defects in the materials.<sup>34</sup> We examined the preparation of these polymers by click chemistry. These polyimides were prepared in two stages *via* the general reactions as shown in Schemes 1–3. The diimide monomers **3(a,b)** were prepared by addition of a tetracarboxylic acid dianhydride **1a** or **1b** to a solution containing two equivalents of propargylamine (**2**) and then cyclodehydration of the obtained amic acids by adding acetic anhydride and pyridine (2: 1) to the amic acid solutions at 75°C (Scheme 1).

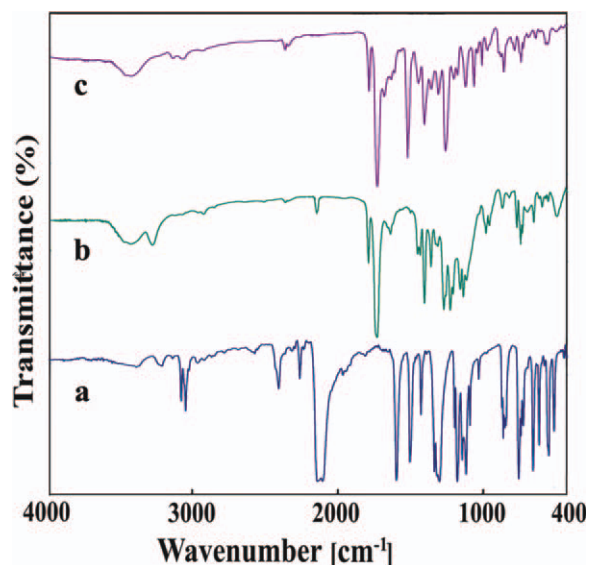
The formation of imide heterocycle in these monomers was confirmed by FT-IR. Cyclization of the amic acids was deemed complete because the N-H

and COOH bands of the amic acids were not observed. They were replaced by the imide groups: 1778 and 1718  $\text{cm}^{-1}$  for carbonyl bands and 1390  $\text{cm}^{-1}$  for the C–N absorption (Fig. 1). The diimide monomers **3(a,b)** were obtained in 74.1 and 76% yields, respectively.

The diamines **4(c-f)** were converted to 4,4'-diazidodiphenylsulfone (**5c**), 4,4'-diazidodiphenylether (**5d**), 4,4'-diazidodiphenylmethane (**5e**), and 1,4-diazidobenzene (**5f**) according to the reported procedures (Scheme 2). The formation of diazides **5(c-f)** was confirmed by the appearance of a strong band at 2100  $\text{cm}^{-1}$  (Fig. 1).



**Scheme 3** Synthesis of polymers **6a(c-f)** and **6b(c-f)**.



**Figure 1** FT-IR spectra of (a) diazide **5c**, (b) diimide **3b**, and (c) polyimide **6bc** [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://www.interscience.wiley.com)].

TABLE I  
Optimization of Reaction Conditions in the  
Synthesis of Polymer **6bc**

| Temperature (°C) | Time (h) | Yield (%) | $\eta_{inh}$ (dL g <sup>-1</sup> ) <sup>a</sup> |
|------------------|----------|-----------|---|
| RT               | 12       | –         | –   |
| RT               | 24       | –         | –   |
| RT               | 36       | –         | –   |
| RT               | 48       | –         | –   |
| 35               | 12       | 60.5      | 0.21  |
| 35               | 24       | 69.3      | 0.28  |
| 35               | 36       | 72.7      | 0.31  |
| 50               | 12       | 70.4      | 0.33  |
| 50               | 24       | 79.1      | 0.42  |
| 50               | 36       | 79.0      | 0.42  |

<sup>a</sup> Measured at a concentration of 0.5 g dL<sup>-1</sup> in DMF at 25°C.

The monomers were polymerized by “click” chemistry using CuBr catalyst in NMP under nitrogen atmosphere to produce the desired polymers **6a(c-f)**, **6b(c-f)** in relatively high yields (Scheme 3).

As shown in Table I, to optimize the polymerization conditions, the synthesis of polymer **6bc** was investigated at room temperature, 35°C, and 50°C. After 48 h, the desired polymer was not formed at room temperature. This polymerization was also carried out at 35°C and 50°C and we found that the best inherent viscosity and yield were obtained at 50°C in 24 h. The inherent viscosity of polyimide **6bc** after 24 h was better than that was obtained after 12 h, but there was not any difference between the obtained inherent viscosity and yield of polymer after 36 h in comparison with 24 h, so the shorter time was chosen as optimum time.

The disappearance of azide and (C≡C) alkyne bands indicates a virtually complete conversion of the diazide and diimide precursors into polyimide (Fig. 1).

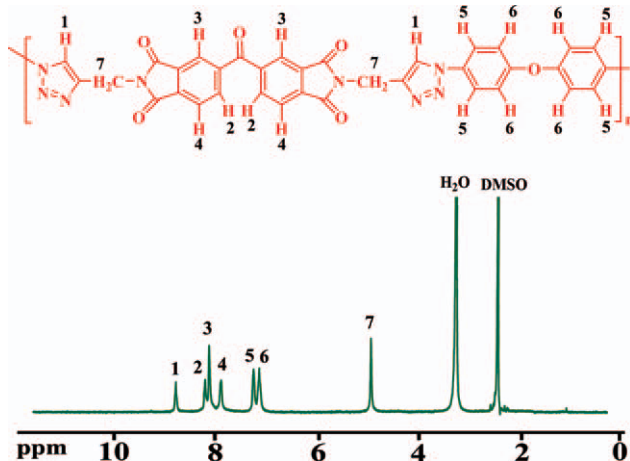


Figure 2 <sup>1</sup>H-NMR (500 MHz) spectrum of polyimide **6ad** in DMSO-*d*<sub>6</sub> at R.T [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com].

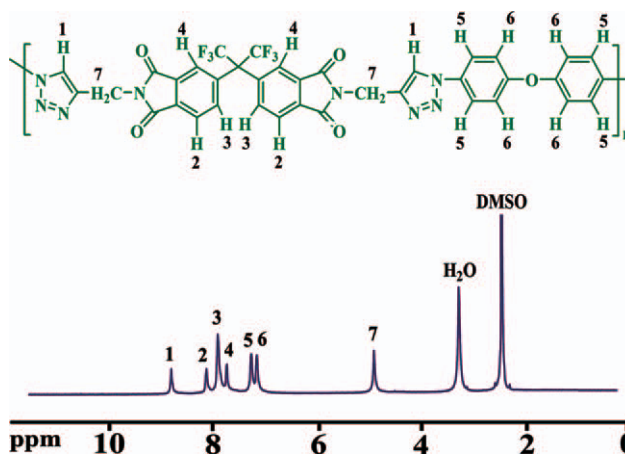


Figure 3 <sup>1</sup>H-NMR (500 MHz) spectrum of polyimide **6bd** in DMSO-*d*<sub>6</sub> at R.T [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com].

Figures 2 and 3 show the <sup>1</sup>H-NMR (500 MHz) spectra of **6ad** and **6bd**. In the <sup>1</sup>H-NMR spectrum of these two polymers, the appearance of the triazole rings protons at 8.75 ppm as a single peak indicates the formation of triazole rings in polymer's chains. The absorption of aromatic protons appeared in the range of 8.19–7.13 ppm. The absorption of CH<sub>2</sub> of diimide appeared at 4.96 and 4.92 ppm, respectively. The resonance observed for the end-group ethynyl protons (≡CH) in monomer **3a** at 3.27 ppm disappears in the polymer.

Under these conditions, yields and inherent viscosities of the resulting polymers ranged between 76.2–87.6% and 0.37–0.53 dL/g, respectively. Some of the physical properties of these polymers are listed in Table II.

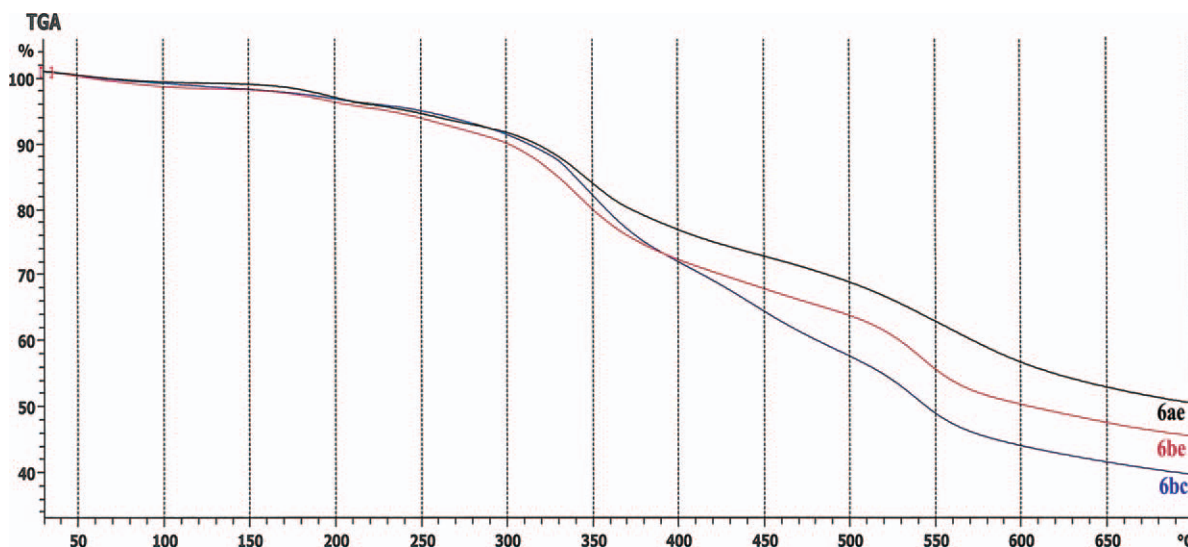
Because of the flexible groups in the polymer backbone, these polymers are expected to have higher solubility. All of them are soluble in organic polar aprotic solvents such as DMF, DMAc, DMSO, and NMP at room temperature and insoluble in water, chloroform, methanol, and acetone.

TABLE II  
Some Physical Properties of the Eight Polymers

| Monomers              | Polymer    | Yield (%) | $\eta_{inh}$ (dL g <sup>-1</sup> ) <sup>a</sup> | Color <sup>b</sup> |
|-----------------------|------------|-----------|---|--------------------|
| <b>3a</b> + <b>5c</b> | <b>6ac</b> | 80.9      | 0.37  | PY                 |
| <b>3a</b> + <b>5d</b> | <b>6ad</b> | 82.0      | 0.40  | PY                 |
| <b>3a</b> + <b>5e</b> | <b>6ae</b> | 87.6      | 0.39  | PY                 |
| <b>3a</b> + <b>5f</b> | <b>6af</b> | 81.2      | 0.43  | PY                 |
| <b>3b</b> + <b>5c</b> | <b>6bc</b> | 79.1      | 0.42  | PY                 |
| <b>3b</b> + <b>5d</b> | <b>6bd</b> | 76.2      | 0.53  | PY                 |
| <b>3b</b> + <b>5e</b> | <b>6be</b> | 78.5      | 0.41  | PY                 |
| <b>3b</b> + <b>5f</b> | <b>6bf</b> | 81.8      | 0.48  | PY                 |

<sup>a</sup> Measured at a concentration of 0.5 g dL<sup>-1</sup> in DMF at 25°C.

<sup>b</sup> PY, pale yellow.



**Figure 4** TGA thermograms of polyimides **6ae**, **6be**, and **6bc** under nitrogen atmosphere and a heating rate of 5°C/min [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com)].

### Thermal properties

The thermal properties of polymers **6ae**, **6bc**, and **6be** were characterized by TGA under nitrogen atmosphere at a heating rate of 5°C/min. All these polymers showed good resistance to thermal decomposition up to 220°C and began to decompose gradually above this temperature. The temperature of 5 and 10% weight loss together with char yield at 650°C has been calculated by thermograms. Figure 4 shows the TGA thermograms of these polymers.

The DSC analyses for polyimides **6ae**, **6bc**, and **6be** show glass transition temperature ( $T_g$ ) around 310, 325, and 320°C, respectively. The thermoanalysis data of these polyimides are summarized in Table III.

**TABLE III**  
Thermal Properties of Some Synthesized Polymers

| Sample     | Decomposition temperature (°C) $T_5^a$ | Decomposition temperature (°C) $T_{10}^b$ | Char yield (%) <sup>c</sup> |
|------------|--|---|-----------------------------|
| <b>6ae</b> | 220                                    | 300                                       | 55                          |
| <b>6bc</b> | 250                                    | 315                                       | 42                          |
| <b>6be</b> | 237                                    | 318                                       | 47                          |

<sup>a</sup> Temperature at which 5% weight loss was recorded by TGA at heating rate of 5°C/min in nitrogen atmosphere.

<sup>b</sup> Temperature at which 10% weight loss was recorded by TGA at heating rate of 5°C/min in nitrogen atmosphere.

<sup>c</sup> Percentage weight of material left undecomposed after TGA analysis at maximum temperature of 650°C in nitrogen atmosphere.

### CONCLUSIONS

In summary, we have successfully used several diazides **5(c-f)** and dimides **3(a,b)** to synthesize eight new heterocyclic polyimides containing triazoles units in the main chain with admirable inherent viscosities (0.37–0.53 dL/g). All of the polymers were characterized using FT-IR, <sup>1</sup>H-NMR, and elemental analysis which confirmed the structure of these novel polymeric materials. These aromatic polyimides are soluble in various organic solvents and have good thermal stability.

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