

## Research Article

# Ionic Liquid-Catalyzed Preparation of Aromatic Polyamides Containing Phthalazinone Moiety

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An unsymmetrical heterocyclic dicarboxylic acid monomer, 4-[4-(4-carboxy phenoxy)-naphthyl]-2-(4-carboxyphenyl) phthalazin-1-one (**1**) was successfully prepared. A series of polyamides containing phthalazinone were prepared from the prepared dicarboxylic acid with various aromatic diamines in an ionic liquid (**IL**) as a green, safe, and eco-friendly medium and also reactions catalysis agent. Evaluation of data shows that **IL** is the better polyamidation medium than the reported method and the catalysis stands on the higher inherent viscosities and yields of the obtained PAs and the rate of polymerizations beyond the greener reaction conditions and deletion of some essential reagents in conventional manner. Characterization was performed by means of <sup>1</sup>H-NMR and FT-IR spectroscopy, elemental analysis, thermogravimetric analysis, and differential scanning calorimetric techniques. Molecular weights of the obtained polyamides were evaluated viscometrically, and the measured inherent viscosities were in the range of 0.46–0.71 dL/g. These polyamides were readily soluble in many organic solvents. These polymers still keep good thermal stability with high glass transition temperatures in the range of 310–345°C and the decomposition temperature under the nitrogen atmosphere for 10% weight-loss temperatures in excess of 488°C.

## 1. Introduction

Due to the increasing demands for high-performance polymers as a replacement for ceramics or metals in the micro-electronic, aerospace and automotive industries, and thermally stable polymers have received much interest over the past decade. Polyimides and their copolymers are certainly one of the most useful classes of high-performance polymers, which have found many applications in industries [1–3]. Aromatic polyimides are an important class of heterocyclic polymers with remarkable heat resistance and superior mechanical and electrical properties, and also durability [4–6]. Poor thermoplastic fluidity and solubility are the major problems in wide application of polyimides. This makes it impossible for most polyimides to be directly processed in their imidized forms; thus, their applications have been restricted in some fields. Processable engineering plastics possessing moderately high softening temperatures and/or solubility in some organic solvents are required for practical use. Therefore, various efforts have been focused on the preparation of soluble and/or thermoplastic

polyimides, while still maintaining the excellent thermal and mechanical properties. Typical approaches have been employed to improve the processability of these polyimides including the incorporation of flexible links [7], bulky pendant or cardo groups [8], kinked or unsymmetrical structures [9], and spiro-skeletons [10] into the polymer chain. These modifications lower the melting temperature and lead to soluble and amorphous polymers. In general, amorphous polymers have a lower softening temperature ( $T_g$ ) and improved solubility with respect to their crystalline analogues. Some of the block copolymers composed of polyethers and polyamides have already been commercialized as thermoplastic elastomers [11]. A number of synthetic routes for polyether-polyimide block copolymers have been known [12]. Ether linkages inserted in the main chains provide them with significantly lower energy of internal rotation. 1,2-dihydro-4-(4-hydroxyphenyl) (2H) phthalazinones as bisphenol-like monomers were introduced by Berard and Hay [13], and then a number of polymers such as poly(phthalazinone ether)s, polyamides, polyimides, and polyesters have been developed from the series of monomer

[14–18]. The incorporation of the twisted, noncoplanar structure into the polymer backbone would reduce the chain packing efficiency and crystallinity which could increase solubility. On the other hand, polycondensation reactions are traditionally performed in polar high-boiling-point organic solvents such as N-methyl-2-pyrrolidone (NMP), N,N-dimethylacetamide (DMAc), and N,N-dimethylformamide (DMF), which generally cause several ecological disadvantages further than the imposition of the overcost on synthetic methods. So development of the effective techniques for replacing or removing hazardous materials is one of the recent environmental issues [19, 20]. Recently, ionic liquids (ILs) have been of interest in many fields of chemistry because of their exclusive properties such as solubility, nonvolatility, high reactivity, low flammability, and possibility of recycling [21–24]. Nonvolatile nature and stability at high temperature make the ILs excellent candidates for the use as polycondensation reactions medium [25–27]. Furthermore, it has been shown that ILs have catalytic effect on the polymerization progresses, resulting from the removal of some of the additives which are essential in conventional polymerization methods [28, 29].

In the present paper the prepared dicarboxylic acid containing phthalazinone (DHPZ) moiety in the main chain [1] was directly polycondensed with various aromatic diamines in an ionic liquid medium to produce a series of organosoluble polyamides. The properties of these polymers, such as solubility and thermal stability of these polymers are also discussed herein.

## 2. Experimental

**2.1. Materials.** 4-[4-(4-Carboxy phenoxy)-naphthyl]-2-(4-carboxyphenyl) phthalazin-1-one (**1**) was prepared as reported [1]. 1,4-phenylenediamine (**2a**), 1,3-phenylenediamine (**2b**), 2,6-pyridinediamine (**2c**), 3,5-pyridinediamine (**2d**), 2,5-diaminotoluene (**2e**), and 4,4'-diaminodiphenylether (**2f**) were purified by sublimation, 4,4'-diaminodiphenylmethane (**2g**) and 4,4'-diaminobiphenyl (**2h**) were purified by recrystallization from water. DMAc was purified by distillation under reduced pressure over barium oxide. Triphenyl phosphite (TPP) was purified by distillation under reduced pressure. All other materials were reagent-grade and used without further.

**2.2. Equipments.** Proton nuclear magnetic resonance  $^1\text{H}$ NMR (400 MHz) spectra were recorded on a Bruker AM instrument (Bruker, Rheinstetten, Germany) using DMSO- $d_6$  as solvent. The FT-IR adsorption spectra were recorded on a Shimadzu FT-IR-680 spectrophotometer with KBr pellets. Elemental analyses were made on a Heraeus CHNS-RAPID instrument. Ultraviolet-visible (UV-vis) spectra of the dilute N,N-dimethylformamide (DMF) solution were recorded on a JASCO V-570 spectrophotometer. Inherent viscosities of polymer solution (0.5% w/v) in DMF were determined at 25°C by a standard procedure using a Cannon Fenske Routine viscometer (Cannon, Mainz, Germany). Melting points were measured in open capillaries with a gallenkamp

instrument. Thermal gravimetric analysis (TGA) data for polymers were taken on a TGA-Perkin Elmer (Pyris 1) at a heating rate of 10°C/min under  $\text{N}_2$  atmosphere. Differential scanning calorimetric analysis (DSC) data were recorded on a DSC-PL-1200 instrument under  $\text{N}_2$  atmosphere.

### 2.2.1. Monomer Preparation

4-[4-(4-Carboxy phenoxy)-naphthyl]-2-(4-carboxyphenyl) phthalazin-1-one (**1**) Was Prepared as Reported [1]. Yield: 90%; white powder; mp. 165–167°C; IR (KBr,  $\text{cm}^{-1}$ ): 3070 (broad, C(O)O–H), 1692 (C=O), 1,239 (C–O–C);  $^1\text{H}$  NMR (DMSO- $d_6$ , ppm):  $\delta$  12.93 (s, 2H), 8.51 (d, 1H), 8.14 (d, 1H), 8.07 (d, 2H), 8.00 (d, 2H), 7.96 (t, 1H), 7.88 (m, 4H), 7.77 (d, 1H), 7.60 (t, 1H), 7.56 (t, 1H), 7.34 (d, 1H), 7.31 (d, 1H), 7.21 (d, 2H);  $^{13}\text{C}$  NMR (DMSO- $d_6$ , ppm):  $\delta$  166.68, 166.64, 161.05, 158.25, 151.82, 146.26, 145.13, 133.97, 133.38, 132.33, 131.71, 129.89, 129.59, 129.54, 128.56, 128.47, 127.99, 127.69, 126.95, 126.86, 126.78, 126.18, 125.91, 125.73, 125.71, 121.69, 117.30, 114.75. Calculated for  $\text{C}_{32}\text{H}_{18}\text{N}_2\text{O}_6$ : C (72.72%), H (3.81%), N (5.30%). Found: C (72.49%), H (3.77%), N (5.45%).

### 2.3. Polymer Preparation

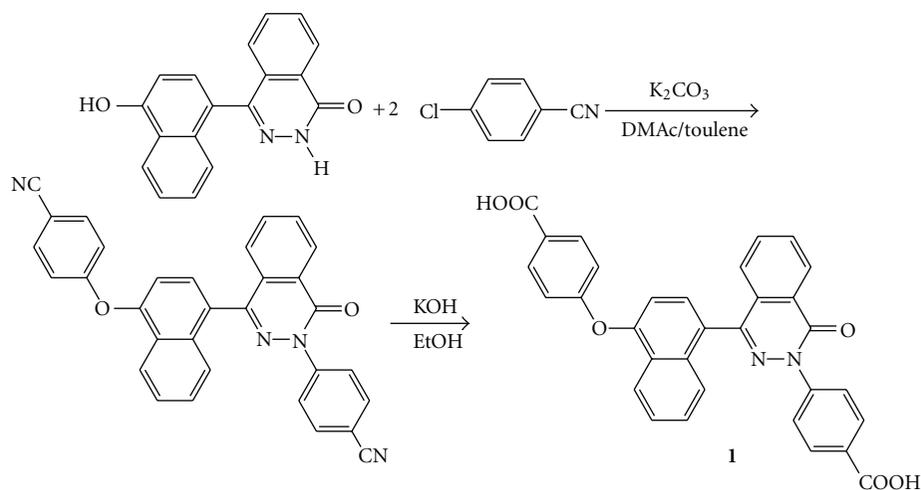
**Preparation of PAs via IL Catalysis Polycondensation.** The PAs were prepared by the following general procedure: as an example for the preparation of PA<sub>1</sub>, 0.1108 g (0.226 mmol) of diacid **1** and 0.0244 g (0.226 mmol) of diamine **2a** were dissolved in 0.15 g of [1-methyl-3-propyl imidazolium]Br, then 0.15 mL (0.565 mmol) of triphenyl phosphite (TPP) was added. The whole solution was heated at 110°C for 2.5 h. The solution becomes more viscous by progress of the polymerization reaction. The resulting product was dissolved in 1 mL of DMF and was poured into 25 mL of stirring methanol, filtered, and dried under vacuum to give 0.1311 g (97%) of PA<sub>1</sub>.

(PA<sub>1</sub>): white solid; FT-IR (KBr,  $\text{cm}^{-1}$ ): 3341 (C(O)N–H, stretching), 1683 (C=O, stretching), 1611 (N–H, bending). Calculated for  $\text{C}_{38}\text{H}_{22}\text{N}_4\text{O}_4$ : C (76.24%), H (3.70%), N (9.36%). Found: C (76.17%), H (3.65%), N (9.31%).

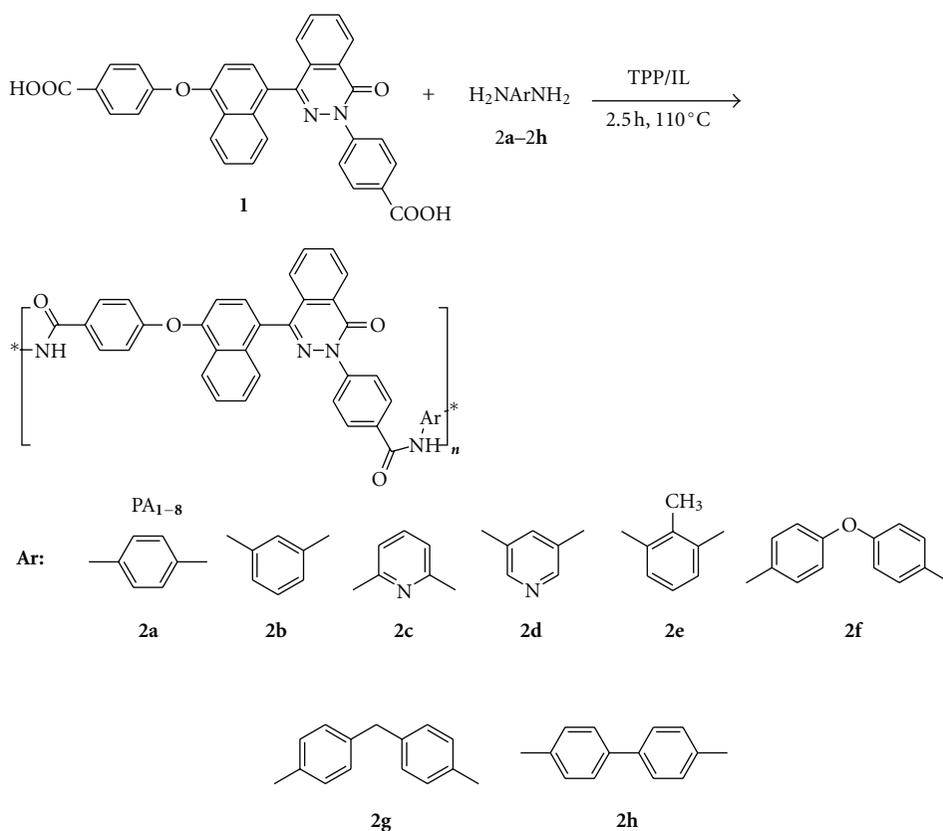
(PA<sub>2</sub>): white solid; FT-IR (KBr,  $\text{cm}^{-1}$ ): 3339 (C(O)N–H, stretching), 1680 (C=O, stretching), 1618 (N–H, bending). Calculated for  $\text{C}_{38}\text{H}_{22}\text{N}_4\text{O}_4$ : C (76.24%), H (3.70%), N (9.36%). Found: C (76.19%), H (3.67%), N (9.30%).

(PA<sub>3</sub>): white solid; FT-IR (KBr,  $\text{cm}^{-1}$ ): 3351 (C(O)N–H, stretching), 1674 (C=O, stretching), 1600 (N–H, bending). Calculated for  $\text{C}_{37}\text{H}_{21}\text{N}_5\text{O}_4$ : C (74.12%), H (3.53%), N (11.68%). Found: C (74.04%), H (3.48%), N (11.59%).

(PA<sub>4</sub>): white solid; FT-IR (KBr,  $\text{cm}^{-1}$ ): 3350 (C(O)N–H, stretching), 1677 (C=O, stretching), 1598 (N–H, bending). Calculated for  $\text{C}_{37}\text{H}_{21}\text{N}_5\text{O}_4$ : C (74.12%), H (3.53%), N (11.68%). Found: C (74.10%), H (3.44%), N (11.53%).



SCHEME 1: Preparation of the unsymmetrical diacid containing phthalazinone moiety.



SCHEME 2: Ionic liquid-catalyzed preparation of aromatic polyamides.

(PA<sub>5</sub>): white solid; FT-IR (KBr, cm<sup>-1</sup>): 3335 (C(O)N–H, stretching), 2867 (CH<sub>3</sub>, stretching), 1680 (C=O, stretching), 1615 (N–H, bending). Calculated for C<sub>39</sub>H<sub>24</sub>N<sub>4</sub>O<sub>4</sub>: C (76.46%), H (3.95%), N (9.14%). Found: C (76.41%), H (3.89%), N (9.11%).

(PA<sub>6</sub>): white solid; FT-IR (KBr, cm<sup>-1</sup>): 3337 (C(O)N–H, stretching), 1635 (C=O, stretching), 1598 (N–H, bending), 1227 and 1124 (C–O–C, stretching).

<sup>1</sup>H NMR (DMSO-d<sub>6</sub>, ppm): δ 10.66 (N–H, 1H), 10.52 (N–H, 1H), 8.52 (1H), 8.32 (2H), 8.21 (1H), 8.01–8.18 (4H), 7.78–8.01 (6H), 7.51–7.66 (2H), 7.32 (4H), 7.12 (4H). Calculated for C<sub>44</sub>H<sub>26</sub>N<sub>4</sub>O<sub>5</sub>: C (76.51%), H (3.79%), N (8.11%). Found: C (76.48%), H (3.76%), N (8.09%).

(PA<sub>7</sub>): white solid; FT-IR (KBr, cm<sup>-1</sup>): 3344 (C(O)N–H, stretching), 2858 (CH<sub>2</sub>, stretching), 1677 (C=O,

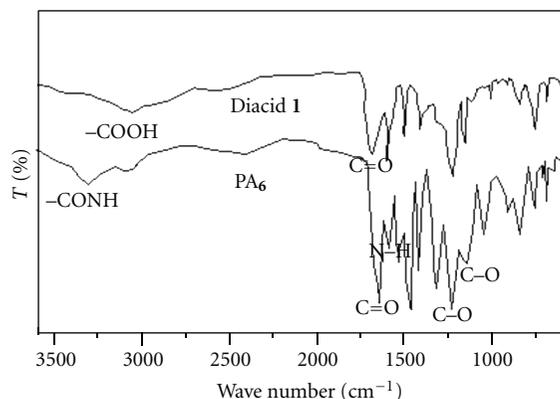


FIGURE 1: FT-IR spectra of diacid (1) and polymer PA<sub>6</sub>.

stretching), 1602 (N–H, bending). Calculated for C<sub>45</sub>H<sub>28</sub>N<sub>4</sub>O<sub>4</sub>: C (78.47%), H (4.10%), N (8.13%). Found: C (78.41%), H (4.07%), N (8.10%).

(PA<sub>8</sub>): white solid; FT-IR (KBr, cm<sup>-1</sup>): 3356 (C(O)N–H, stretching), 1663 (C=O, stretching), 1587 (N–H, bending). Calculated for C<sub>44</sub>H<sub>26</sub>N<sub>4</sub>O<sub>4</sub>: C (78.33%), H (3.88%), N (8.30%). Found: C (78.24%), H (3.77%), N (8.28%).

### 3. Results and Discussion

**3.1. Monomer Preparation.** As shown in Scheme 1, the phthalazinone-containing aromatic dicarboxylic acid was prepared by alkaline hydrolysis of the corresponding dinitrile compound resulting from the nucleophilic substitution reaction of 4-(4-hydroxynaphthalenyl) phthalazin-1-one with p-chlorobenzonitrile.

All prepared compounds were characterized by elemental analysis, FT-IR and <sup>1</sup>H NMR spectroscopy with satisfied results. A broad O–H absorption of diacid (1) appeared in the region of 2600–3500 cm<sup>-1</sup> and that of C=O stretching at 1689 cm<sup>-1</sup> (Figure 1). In <sup>1</sup>H NMR (DMSO-d<sub>6</sub>, ppm), the corresponding peak to CO(O–H) appeared at δ 12.93 (s, 2H).

**3.2. Polymer Preparation.** Polymerization of diacid (1) with stoichiometric amount of different aromatic diamines (2a–2h) was carried out in [1-methyl-3-propyl imidazolium]Br both as solvent and catalyst outlined in Scheme 2.

In addition to provide a safe and green reaction medium, **IL** has also catalytic effect on polymerization reaction because polyamidations did not progress in the absence of either **IL** or TPP and the existence of both **IL** and TPP at the same time was essential for the polycondensation. A comparison between this method and the conventional method proves that the PAs prepared via **IL** method encompass higher inherent viscosities and yields. Furthermore, the use of **IL** causes reduced overall cost of preparation and cleaner reaction. Utilization of **ILs** is often motivated by safety and environmental considerations, where the eligible volatility and nonflammability of **ILs** make them promising green

replacements for more hazardous molecular solvents and overcomes the trouble of solvent evaporation that exists with the long-term use of volatile solvents in organic synthesis. The high polarity and viscosity of **ILs** greatly increases the propagation rate constant (*k<sub>p</sub>*) and decreases the termination rate constant (*k<sub>t</sub>*) in polymerization; consequently, polymers with higher molecular weight and narrower polydispersity are achieved in **ILs**. All polyamidations proceeded in a homogeneous system throughout the reaction, and the polyamides were isolated in quantitative yields. The yields and inherent viscosities of the resulting polyamides are summarized in Table 1.

Inherent viscosities of the polyamides ranged from 0.46–0.71 dL/g, indicative of the formation of high molecular weights. The structures of these polyamides were confirmed by IR spectroscopy. For example, polymer PA<sub>6</sub> (Figure 1) showed characteristic absorptions of amide group occurred at 3337 (C(O)N–H, stretching), 1635 (C=O, stretching), and 1598 (N–H, bending) cm<sup>-1</sup>. The <sup>1</sup>H NMR spectrum of PA<sub>6</sub> was showed in Figure 2.

The absorption peaks at 10.5 and 10.6 ppm was assigned to the N–H protons. Thermal properties and glass transitions temperature (*T<sub>g</sub>*) of the polymers were evaluated with TGA and DSC, respectively (Figures 3 and 4), and the characteristic data are summarized in Table 2. The *T<sub>g</sub>* value of PA<sub>1–8</sub> were in the range of 310–345 °C. In addition more than half of the polymers weight remained at 600 °C (char yields). The excellent thermal stabilities may be due to the presence of very rigid aromatic heterocyclic backbone.

**3.3. Organosolubility.** The solubility behavior of the new aromatic polyamides was determined at concentration of 5% (W/V) in a number of solvents. Almost all prepared polyamides exhibited excellent solubility in polar aprotic solvents such as NMP, DMF, DMAc, dimethyl sulfoxide (DMSO), and even in less polar solvents like Py and m-cresol. High tendency to pack the polymer chains is the predominant origin of insolubility of common PAs because of ability to create high density of interchain H-bonds. In the case of our prepared PAs, the high solubility is attributed to the presence of twist and noncoplanar aromatic heterocyclic moiety in the polymer backbone, which interrupt the regular packing of polymer chains. In addition, the more flexible ether group was introduced in the polymer backbone are especially effective for the high solubility.

### 4. Conclusion

Replacement of the toxic volatile organic solvents with several ecological disadvantages by a green, safe, and eco-friendly reaction medium is the main aspect of this investigation. Therefore, direct polycondensations of diacid monomer (1) and several aromatic diamines have been investigated in **IL** medium, which acts as a green solvent and a reaction catalyst. Comparison with the reported direct method [1] indicates that **IL** in combination with TPP is the superior polyamidation agent. On the other hand, in the case of **IL**-catalyzed polyamidation, removal of some chemicals such

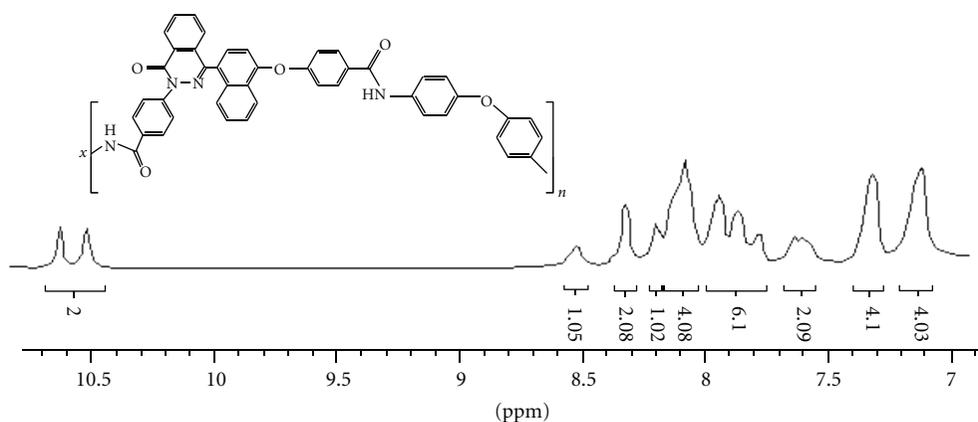
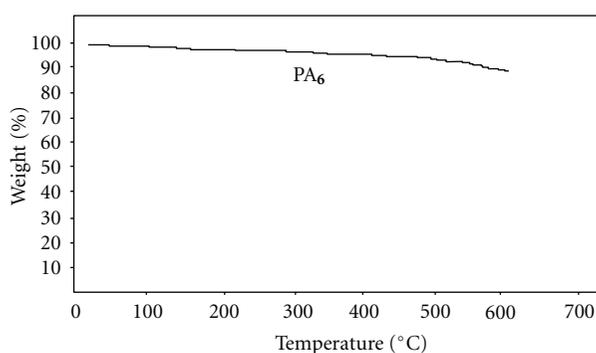
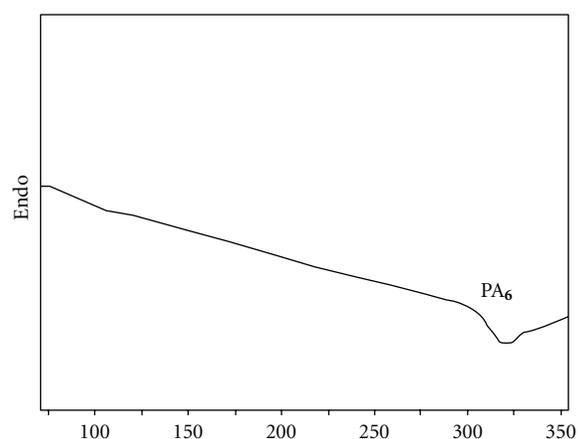
FIGURE 2:  $^1\text{H}$  NMR spectrum of polymer  $\text{PA}_6$  in  $\text{DMSO-d}_6$ .

TABLE 1: Inherent viscosity and yield of polyamids.

Polymer	IL catalysis polycondensation		Conventional polycondensation [1]	
	Yield (%)	$\eta_{\text{inh}}$ ( $\text{dl g}^{-1}$ ) <sup>a</sup>	Yield (%)	$\eta_{\text{inh}}$ ( $\text{dl g}^{-1}$ ) <sup>a</sup>
$\text{PA}_1$	97	0.69	99.1	0.63
$\text{PA}_2$	95	0.56	—	—
$\text{PA}_3$	93	0.54	—	—
$\text{PA}_4$	90	0.48	—	—
$\text{PA}_5$	92	0.46	—	—
$\text{PA}_6$	98.8	0.64	98.4	0.58
$\text{PA}_7$	95	0.49	—	—
$\text{PA}_8$	97	0.71	—	—

<sup>a</sup>Measured at a concentration of 0.5 g/dL in DMAc at 30°C.

FIGURE 3: TGA curve of  $\text{PA}_6$ .FIGURE 4: DSC curve of  $\text{PA}_6$ .

as NMP,  $\text{CaCl}_2$ , and Py, which are essential in conventional methodologies, decreases the cost of polymerization as well as the environmental pollutions, considerably. Here a series of polyamides containing phthalazone were prepared from diacid (**1**) with various aromatic diamines by ionic liquid catalyzed method. The results indicated that adding asymmetrical and noncoplanar heterocyclic unit into polymer

backbone improved the solubility of polyamides with the retention of the thermostability. The properties of these polyamides make them attractive for practical applications such as processable high performance polymeric materials.

TABLE 2: Thermal properties of PA<sub>1-8</sub>.

Polymer	IL catalysis polycondensation			Conventional polycondensation [1]		
	<sup>a</sup> T <sub>10%</sub>	<sup>b</sup> T <sub>g</sub> (°C)	<sup>c</sup> Char yield	T <sub>10%</sub>	T <sub>g</sub> (°C)	Char yield
PA <sub>1</sub>	497	323	61	509	338	—
PA <sub>2</sub>	458	310	62	—	—	—
PA <sub>3</sub>	511	321	56	—	—	—
PA <sub>4</sub>	521	317	53	—	—	—
PA <sub>5</sub>	498	312	42	—	—	—
PA <sub>6</sub>	565	324	87	511	311	—
PA <sub>7</sub>	488	332	50	—	—	—
PA <sub>8</sub>	578	345	68	—	—	—

<sup>a</sup>Temperature at which 10% weight loss was recorded by TGA at a heating rate of 10°C/min under nitrogen atmosphere. <sup>b</sup>Glass transition temperature recorded by DSC at a heating rate of 10°C/min under nitrogen atmosphere. <sup>c</sup>Percentage weight of material left after TGA analysis at maximum temperature 600°C under nitrogen atmosphere.

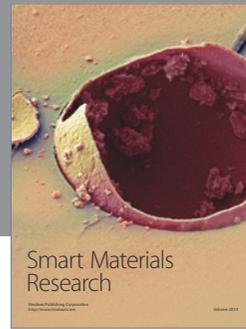
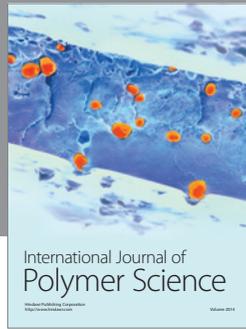
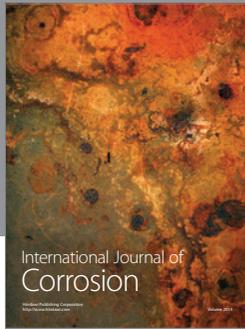
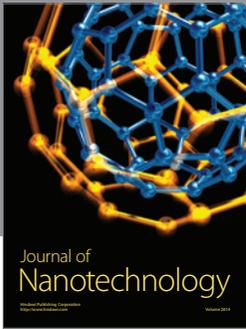
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