# Fluorene-Ring-Containing Diamine and Resultant Soluble Thermally Stable Polyamides

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**ABSTRACT:** A new fluorene-ring-containing diamine, $N'$-[7-(acetyl-4-aminoanilino)-9,9-dioctylflouren-2-yl]-$N'$-4-aminophenyl)acetamide, was prepared through the reaction of 2,7-dibromo-9,9-dioctylfluorene with 4-aminoacetanilide in the presence of 10 mol % CuI, 20 mol % $N,N'$-dimethyl ethylene diamine as a catalyst, and $K_2CO_3$ as a base. A series of novel polyamides were prepared by the direct polycondensation of the new diamine with various commercially available aliphatic and aromatic dicarboxylic acids. The novel diamine and the obtained polyamides were characterized with conventional methods (Fourier transform infrared and NMR spectroscopy) and elemental analysis. All the synthesized polyamides showed good solubility in various solvents such as dimethyl sulfoxide, $N$-methyl-2-pyrrolidone, $N,N$-dimethylacetamide, $N,N$-dimethylformamide, hexamethylene phosphoramide, and pyridine and exhibited inherent viscosities in the range of 0.44–0.55 dL/g. According to the differential scanning calorimetry analysis, the glass-transition temperatures of the polyamides were in the range of 85–196°C. Thermogravimetric analysis of the polyamides indicated a 10% weight loss in the temperature range of 344–417°C in an $N_2$ atmosphere. © 2008 Wiley Periodicals, Inc. J Appl Polym Sci 110: 1730–1738, 2008

**Key words:** polyamides; thermal properties

## INTRODUCTION

Polyamides have attracted much attention because they are synthesized, characterized, and applied as commercial synthetic fibers known commonly as nylons. Aromatic polyamides (aramides) are a class of high-temperature-resistance polymers with good chemical resistance and thermal stability, low flammability, and very good mechanical properties.[1–3] However, because of their limited solubility in organic solvents and high melting and glass-transition temperatures, they are difficult to process. Some of these problems are caused by strong intermolecular hydrogen bonds and donor–acceptor interactions of the amide functions, which cause chain stiffness. The introduction of a flexible bond into the rigid polymer backbone is known to be an effective method for enhancing the solubility and also lowering the glass-transition and melting temperatures of these rigid polymers while minimizing the deterioration of their physical properties. Modified polyamides and polyimides have been prepared with diamine monomers containing flexible groups.[4–11] Another conventional approach to increasing the solubility is the introduction of bulky pendant groups into the polymer backbone.[12–18] This modification lowers the melting temperature and produces amorphous and soluble polymers. However, some efforts in using monomers with bulky flexible side groups have led to polymers with poor solubility in organic solvents.[19–21] This work describes the successful synthesis of a novel diamine monomer, $N'$-[7-(acetyl-4-aminoanilino)-9,9-dioctylflouren-2-yl]-$N'$-4-aminophenyl)acetamide (ADOAc), with a new amidation method based on Cu(I) as a catalyst.[22] The new diamine contains both amide and fluorene with flexible aliphatic side groups. The polyamides are prepared by the direct polycondensation reaction of this diamine with different dicarboxylic acids. The best conditions for polymerization have been obtained through the synthesis of a model compound. The monomer, model compound, and polyamides have been characterized, and properties such as the inherent viscosity, solubility behavior, thermal properties, and thermal stability have been studied.

## EXPERIMENTAL

### Materials

All chemicals were purchased from either Merck (Germany) or Fluka Co. (Germany) 2,7-Dibromo-9,9-dioctylfluorene was synthesized according to the procedure reported in the literature.[23,24] Toluene was purified by distillation over sodium. $N,N$-Dimethylformamide was purified by distillation over

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**TABLE I**
**Characterization Data of the Monomer and Model Compound**

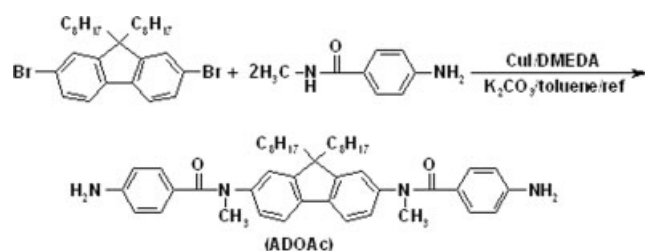| Component | $^1$H-NMR [δ (ppm)] | $^{13}$C-NMR [δ (ppm)] | FTIR [ν (cm$^{-1}$)] |
|---|---|---|---|
| ADOAc | 7.63 (b, 2H), 7.26–7.20 (m, 4H), 7.09 (d, 4H), 6.70 (b, 4H), 3.78 (s, 4H), 2.11 (s, 6H), 1.91 (b, 4H), 1.26–1.04 (m, 20H), 0.88 (t, 6H), 0.66 (b, 4H) | 171.39, 145.06, 143.13, 129.77, 127.73, 127.30, 124.97, 123.30, 121.11, 119.98, 116.01, 55.71, 40.38, 32.20, 30.31, 30.13, 29.64, 29.50, 24.30, 23.04, 14.55 | 3459–3357–3225 (—NH$_2$), 3033 (Ar—H), 2926–2860 (aliphatic stretching C—H), 1656 (C=O), 1583,1466 (aromatic C=C), 1405 (stretching C—N), 725 (—C=O, out-of-plane deformation) |
| Model compound | 10.31 (s, 2H), 7.93 (d, 4H), 7.78 (b, 6H), 7.60 (t, 4H), 7.52 (t, 4H), 7.29 (b, 6H), 2.0 (b, 10H), 1.12 (q, 4H), 1.08 (m, 4H), 1.03–0.93 (m, 12H), 0.73 (t, 6H), 0.50 (b, 4H) | — | 3342 (—NH—), 3050 (Ar—CH), 2919–2867 (aliphatic stretching C—H), 1659 and 1601 (C=O), 1505,1466 (aromatic C=C), 1407 (stretching C—N), 706 (—C=O, out-of-plane deformation) |

calcium hydride under reduced pressure. All other compounds such as terephthalic acid, isophthalic acid, pyridine-2,6-dicarboxylic acid, adipic acid, and sebacic acid and various solvents were used without further purification.

## Instruments

For Fourier transform infrared (FTIR), a Bruker Vector 22 (Germany) (with a KBr plate) was used. For $^1$H- and $^{13}$C-NMR, a Bruker DRX 500-MHz Advance instrument (with CDCl$_3$ and dimethyl sulfoxide-$d_6$ as solvents) was used. For elemental analysis, a Leco CHN-600 was used. For the determination of the melting point, a Branstead Electrothermal Engineering, Ltd. (Germany), model 9200 was used. For the measurement of the inherent viscosities, an Ubbelohde suspended-level viscometer (with a 0.5 g/dL solution in N-methyl pyrrolidone at 25°C) was used. For thermogravimetric analysis, a DuPont Instruments TGA 951 (at 10°C/min under an N$_2$ atmosphere at 20 cm$^3$/min) was used. For differential scanning calorimetry (DSC), a PerkinElmer Pyris 6 differential scanning calorimeter (at 10°C/min under an N$_2$ atmosphere at 20 cm$^3$/min) was used.

## Synthesis of ADOAc

A 100-mL, two-necked flask equipped with a magnetic stirrer, a condenser, and an argon inlet/outlet tube was charged with CuI (0.192 g, 1 mmol, 10 mol %), 9,9-dioctyl-2,7-dibromoflurene (2.74 g, 5 mmol), 4-aminoacetanilide (1.88 g, 12.5 mmol), and K$_2$CO$_3$ (2.8 g, 20.3 mmol), briefly evacuated, and backfilled with argon. N,N′-Dimethyl ethylene diamine (DMEDA; 0.2152 mL, 2 mmol) and toluene (20 mL) were added under argon. The reaction mixture was stirred at 110–115°C for 36 h. During this time, the progress of the reaction was monitored by thin-layer chromatography. The resulting pale-brown suspension was allowed to reach room temperature and then filtered through a 0.5 × 1 cm$^2$ pad of silica gel eluting with 1 : 1 ethyl acetate/dichloromethane (100 mL). The filtrate was evaporated and then purified with column chromatography (SiO$_2$, 1 : 4 ethyl acetate/dichloromethane). A pale brown crystalline product was obtained with an overall yield of 89.6% (3.8 g) and a melting point of 119–121°C.

ANAL. Calcd for the diamine (C$_{45}$H$_{58}$N$_4$O$_2$): C, 78.71%; H, 8.45%; N, 8.16%. Found: C, 78.58%; H, 8.56%; N, —.

The characterization data from $^1$H-NMR, $^{13}$C-NMR, and FTIR spectra are collected in Table I. The reaction for the synthesis of the monomer is presented in Scheme 1.



**Scheme 1** Preparation of ADOAc.
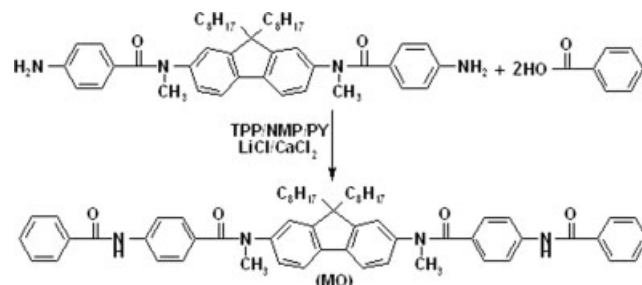


**Scheme 2** Preparation of the model compound (NMP = N-methyl-2-pyrrolidone; PY = pyridine; TPP = triphenyl phosphite).

TABLE II
Yields, Spectroscopic Data, and Elemental Analysis of the Polyamides

| Polyamide code | Yield (%) | $^1$H-NMR [$\delta$ (ppm)] | FTIR [$\nu$ (cm$^{-1}$)] | Elemental analysis | | | | | |
| | | | | Calculated (%) | | | Found (%) | | |
| | | | | C | H | N | C | H | N |
|---|---|---|---|---|---|---|---|---|---|
| **I** ($C_{52}H_{59}N_5O_4$) | 95 | 10.95 (s, 2H), 8.36–8.25 (b, 3H), 7.88–7.77 (d, d, 4H), 7.31–7.01 (m, 10H), 1.96–1.91 (b, 10H), 1.22–0.96 (m, b, 20H), 0.63–0.47 (m, b, 10H) | 3306, 3051, 2926 and 2853, 1670 and 1601, 1521, 1462, 1375, 1376, 817, 750 | 76.38 | 7.22 | 8.57 | 76.28 | 7.02 | — |
| **II** ($C_{53}H_{60}N_4O_4$) | 94 | 10.44 (s, 2H), 8.07 (s, 4H), 7.78 (b, 4H), 7.67 (b, 2H), 7.50–7.32 (m, 6H), 7.08 (m, 2H), 1.97 (s, 6H), 1.91 (s, 4H), 1.11–0.85 (m, 20H), 0.73 (t, 6H), 0.49 (b, 4H) | 3320, 3051, 2926 and 2846, 1656 and 1601, 1517, 1469, 1378, 1316, 830, 721 | 77.94 | 7.35 | 6.86 | 77.72 | 7.48 | — |
| **III** ($C_{53}H_{60}N_4O_4$) | 94 | 10.49 (b, 2H), 8.51 (s, 1H), 8.12 (d, 2H), 7.78 (b, 4H), 6.67–7.61 (b, 3H), 7.29 (b, 6H), 7.06 (m, 2H), 1.96 (s, 6H), 1.91 (s, 4H), 1.18–0.92 (m, 20H), 0.72 (b, 6H), 0.49 (b, 4H) | 3328, 3050, 2933 and 2860, 1659 and 1605, 1513, 1462, 1375, 1312, 1247, 823, 717 | 77.94 | 7.35 | 6.86 | 78.14 | 7.06 | — |
| **IV** ($C_{51}H_{64}N_4O_4$) | 92 | 9.92 (b, 2H), 7.55 (b, 4H), 7.46 (m, 2H), 7.31–7.20 (m, 4H), 7.03–6.95 (m, 4H), 2.29 (m, 4H), 2.01–1.93 (m, 10H), 1.60 (m, 4H), 1.14–0.92 (m, 20H), 0.78 (t, 6H), 0.47 (b, 4H) | 3320, 3057, 2928 and 2860, 1663 and 1605, 1517, 1469, 1375, 1301, 823, 721 | 76.88 | 8.04 | 7.03 | 76.58 | 8.30 | — |
| **V** ($C_{55}H_{72}N_4O_4$) | 90 | 9.91 (b, 2H), 7.55 (b, 4H), 7.45 (m, 2H), 7.31–7.20 (m, 4H), 7.01 (m, 4H), 2.26–2.17 (m, 4H), 2.02–1.82 (m, 10H), 1.56 (b, 2H), 1.26 (b, 8H), 1.14–0.92 (m, 20H), 0.77 (b, 6H), 0.50 (b, 4H) | 3320, 3055, 2928 and 2860, 1663 and 1608, 1553, 1517, 1466, 1305, 823, 725 | 77.76 | 8.45 | 6.57 | 77.72 | 8.21 | — |

## Model compound synthesis

To a 50-mL, two-necked flask equipped with a magnetic stirrer, a condenser, and a nitrogen inlet/outlet tube, a mixture consisting of N-methyl pyrrolidone (10 mL), pyridine (2 mL), LiCl (0.2 g), and CaCl$_2$ (0.6 g) was added. The synthesized diamine (ADOAc; 0.46 g, 0.7 mmol), benzoic acid (0.18 g, 1.5 mmol), and triphenyl phosphite (0.45 g, 1.4 mmol) were added to the flask. The mixture was heated at 110°C for 8 h under a stream of N$_2$. During this time, the progress of the reaction was monitored by thin-layer chromatography. The solution was then poured into water, and the precipitate was filtered off and vacuum-dried. The overall yield of this reaction was 97% (0.61 g), and the melting point of the product was 170–172°C.

ANAL. Calcd for the model compound ($C_{59}H_{66}N_4O_4$): C, 79.19%; H, 7.38%; N, 6.26%. Found: C, 79.08%; H, 7.72%; N, —.

The characterization data from $^1$H-NMR and FTIR spectra are collected in Table I. The reaction for the synthesis of the model compound is shown in Scheme 2.

## Polyamide synthesis

In a two-necked, round-bottom flask equipped with a magnetic stirrer, a condenser, and a nitrogen inlet/outlet tube, the diamine (ADOAc; 0.662 g, 1 mmol) and a dicarboxylic acid (terephthalic acid, isophthalic acid, pyridine-2,6-dicarboxylic acid, adipic acid, or sebacic acid; 1 mmol) was mixed with triphenyl phosphite (0.65 g, 2.1 mmol) in a solvent mixture of N-methyl pyrrolidone (50 mL) and pyridine (3 mL) containing LiCl (0.3 g) and CaCl$_2$ (0.9 g). The reaction mixture was heated at 110°C for 8 h under N$_2$. The reaction solution was poured into water, and the precipitate was filtered and washed with hot water. For purification, the polyamides obtained from pyridine-2,6-dicarboxylic acid (**I**), terephthalic acid (**II**), and isophthalic acid (**III**) were refluxed for 1 h in methanol, and the polyamides
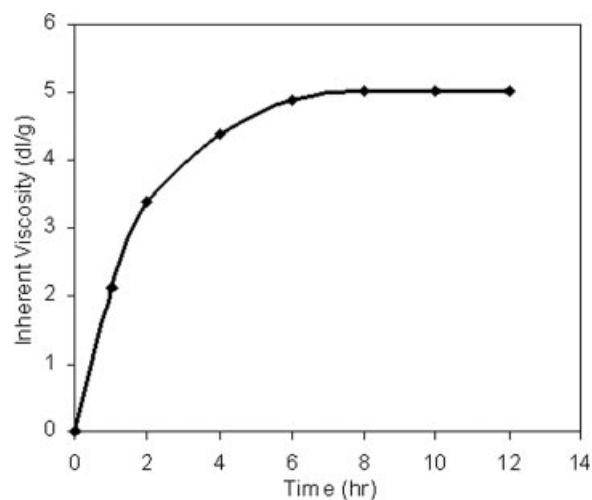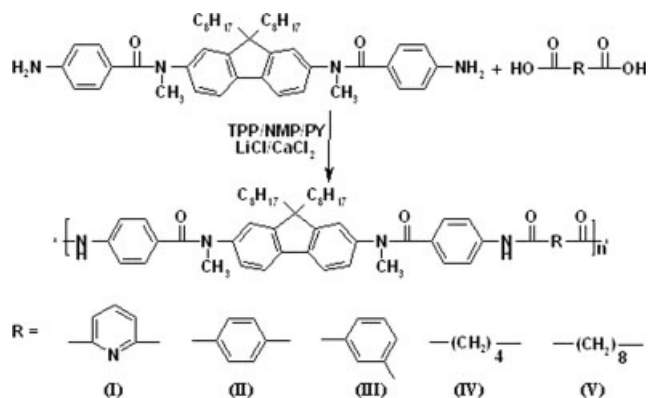
**Figure 1** Changes in the viscosity during the preparation of polyamide **II**.



**Scheme 3** Preparation of the polyamides (NMP = N-methyl-2-pyrrolidone; PY = pyridine; TPP = triphenyl phosphite).

obtained from adipic acid (**IV**) and sebacic acid (**V**) were refluxed for 1 h in water. The extracted polyamides were then filtered and vacuum-dried at 100°C. The yields, elemental analysis data, and spectroscopy data are shown in Table II. The inherent viscosity of polyamide **II** during polyamidation was measured, and the results are shown in Figure 1. The reaction for the synthesis of polyamides is shown in Scheme 3.

## RESULTS AND DISCUSSION

The diamine ADOAc was synthesized in one step through the CuI/DMEDA catalytic C(aryl)–N coupling reaction (amidation) of 2,7-dibromo-9,9-dioctyl-fluorene with 4-aminoacetanilide in the presence of $K_2CO_3$ as a base (Scheme 1). FTIR, $^1$H-NMR, $^{13}$C-NMR, and elemental analysis were used to confirm the structure of the diamine monomer. FTIR spectra of ADOAc showed characteristic bands at 3459–3357 (N—H stretching), 3033 (Ar—CH), 2926–2860 (aliphatic stretching C—H), 1656 (C=O), and 1583 cm$^{-1}$ (C=C). The $^1$H-NMR spectra showed characteristic signals at 3.78 ppm related to $NH_2$ protons. The $^{13}$C-NMR spectra also showed characteristic signals at 171.39 ppm related to the carbon of the carbonyl group. $^1$H-NMR and $^{13}$C-NMR spectra of the synthesized diamine are shown in Figures 2 and 3. The complete characterization data in Table I confirm the formation of the diamine containing a bulky fluorene unit with flexible aliphatic side-chain groups.
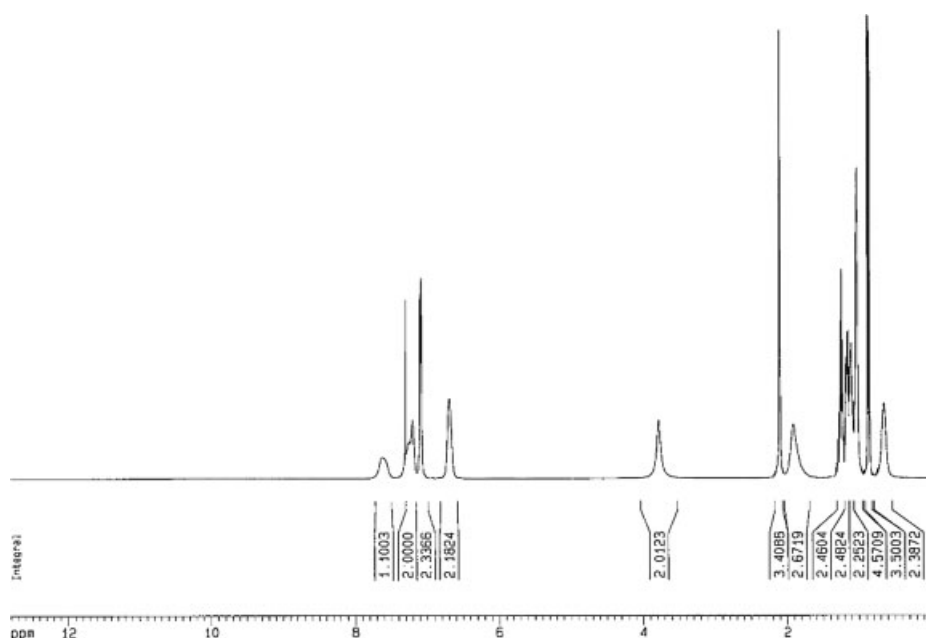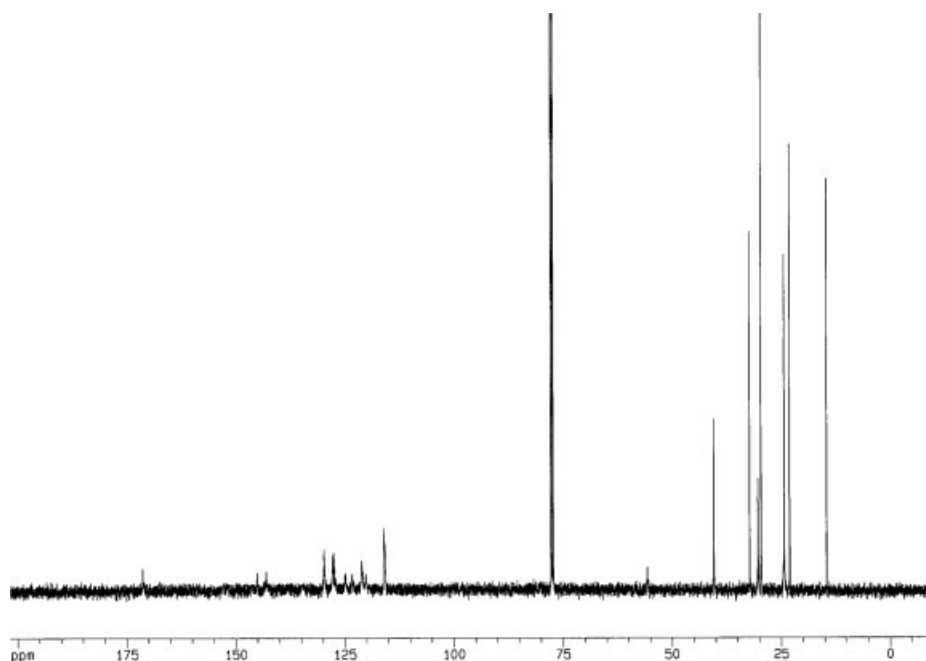


**Figure 2** $^1$H-NMR spectrum of the diamine ADOAc.

**Figure 3** ^{13}C-NMR spectrum of the diamine ADOAc.

Typical condensation reactions of the obtained diamine with benzoic acid resulted in the preparation of the model compound (Scheme 2). The FTIR spectrum of the model compound showed amide bands at 3342 (N—H stretching), 1659 and 1601 (related to two C=O stretchings), and 1407 cm$^{-1}$ (C—N stretching). ^{1}H-NMR spectra showed the characteristic signal of the amidic protons at 10.31 ppm. ^{1}H-NMR and FTIR spectra are shown in Figures 4 and 5. The complete characterization data are listed in Table I.

The optimized conditions obtained from the study of the model compound were applied to the preparation of polyamides. The inherent viscosity of polyamide **II** was measured at a concentration of 0.5 g/dL in N,N-dimethylformamide at 25°C for different reaction times. The viscosity increased with the time of polymerization, as shown in Figure 1, to 0.501 dL/g after 8 h and changed to 0.502 dL/g when the reaction time increased up to 10 h.

The polycondensation reaction of the synthesized diamine with aromatic dicarboxylic acids (pyridine-
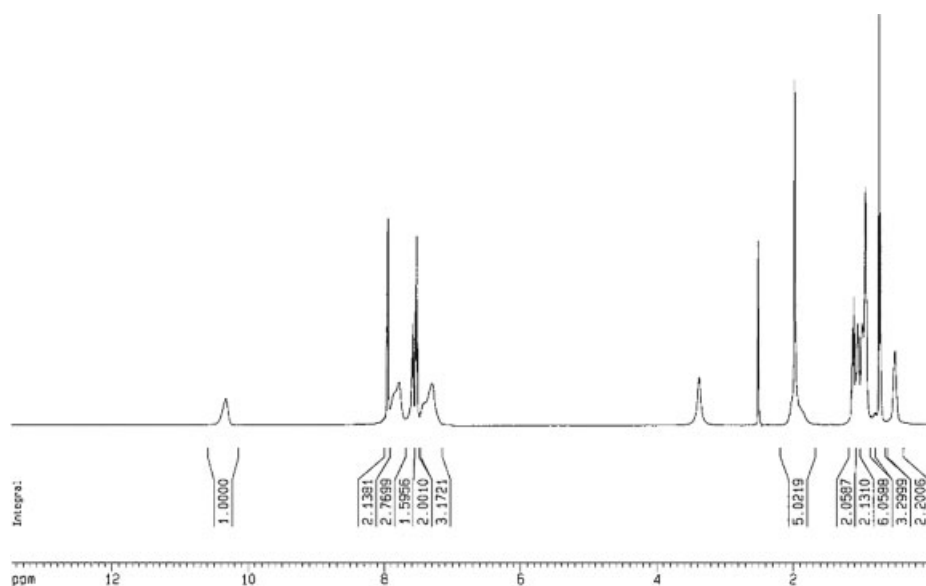


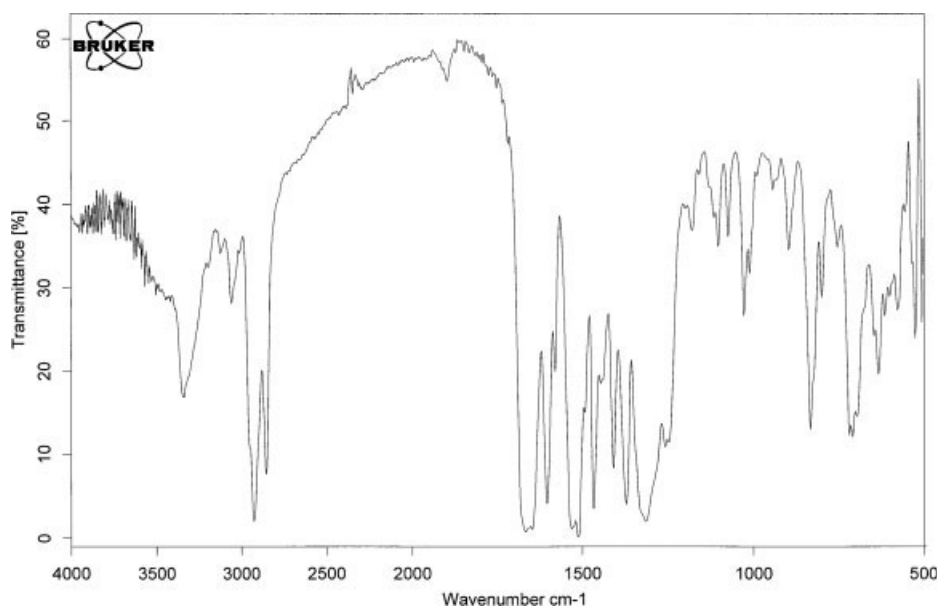**Figure 4** ^{1}H-NMR spectrum of the model compound.

**Figure 5** FTIR spectrum of the model compound.

2,6-dicarboxylic, terephthalic acid, and isophthalic acid) resulted in the preparation of aromatic polyamides. Also, semiaromatic polyamides were prepared from the polycondensation of aliphatic dicarboxylic acids (a dipic acid and sebacic acid) with the diamine (Scheme 3). The chemical structures of the polyamides were characterized with FTIR, ^{1}H-NMR, and elemental analysis. The presence of amidic bands at about 3320 (N—H stretching), 1660 and 1605 (related to two C=O stretchings), and 1520 cm$^{-1}$ (N—H bending and

C—N stretching) in the FTIR spectra (Fig. 6) and the amidic proton at about 10.5 ppm for aromatic polyamides and at 9.90 for semiaromatic polyamides in the ^{1}H-NMR spectra (Fig. 7) confirmed the amidic structure of the polymers. The yields, spectroscopic data, and elemental analysis data of all the polyamides are collected in Table II.

The major objectives of this study were to synthesize a new diamine with a particular method and to produce modified polyamides with improved solubility. This was achieved, in addition to the presence
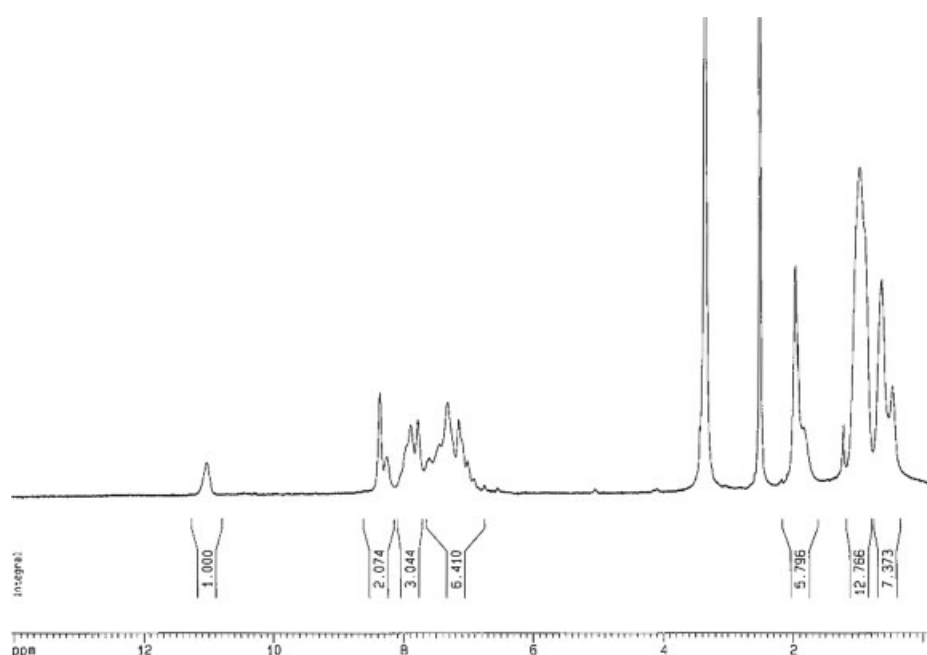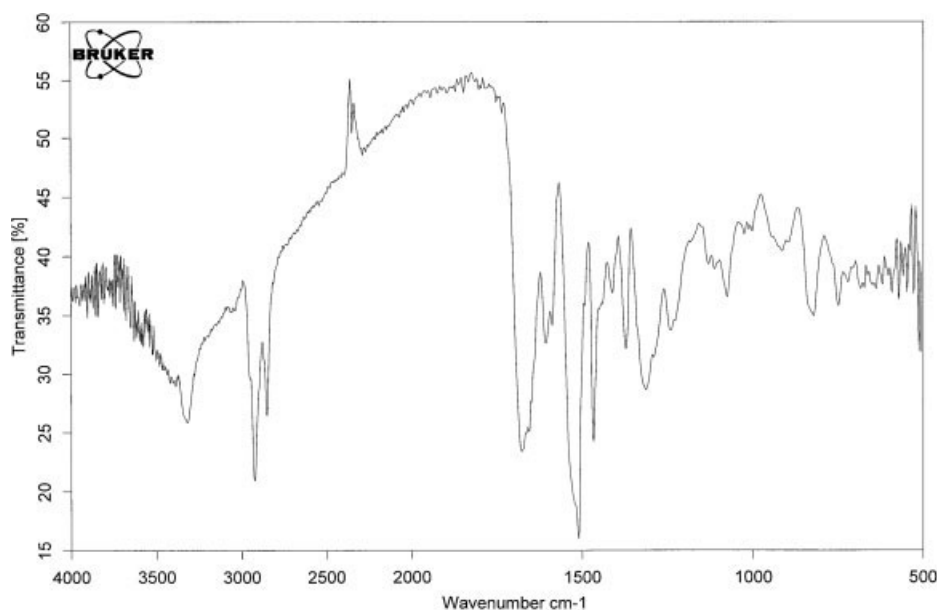


**Figure 6** ^{1}H-NMR spectrum of polyamide **I**.

**Figure 7**  FTIR spectrum of polyamide **I**.

of dioctyl groups at the 9-position, by the introduction of a polar tertiary amide group [—N(CH₃)—CO—] into the repeating unit of the polymer chain, which increased the distance between amide groups (—NH—CO—) capable of the formation of intramolecular hydrogen bonding. As a result of these characteristics in the chains, the polyamides (as shown in Table III) were readily soluble in common polar aprotic solvents. Among these polyamides, **I** was soluble in common solvents with heating at about 60°C, and **IV** and **V** were more soluble and less time was needed for their complete dissolution in the same solvents. For the semiaromatic polyamides **IV** and **V**, the presence of methylene groups in the backbone increased flexibility and also disturbed the planarity of aromatic units, resulting in a reduction of the close packing and the crystallinity. The aromatic polyamides **I**, **II**, and **III** had lower solubility because of the presence of rigid phenylene groups, which reduced the flexibility of polymer chains and caused them to have closer packing. The inherent viscosity of the polymers, as a suitable criterion for the evaluation of the molecular weight, was measured at a concentration of 0.5 g/dL in N-methyl pyrrolidone at 25°C. The inherent viscosities were in the range of 0.44–0.55 dL/g and indicated a moderate molecular weight (Table III).

The thermal behavior of the polyamides was studied with DSC. All the polyamides showed glass-transition temperatures in the range of 85–196°C. The decreasing trend of the glass-transition temperature was comparable to the decreasing order of stiffness and polarity of the polymer backbone. The lower glass-transition temperatures of polymers **IV** and **V** were mainly due to the incorporation of aliphatic groups into the backbone of dicarboxylic acids. None

**TABLE III**
**Inherent Viscosity and Solubility of the Polyamides**

| Polyamide code | Inherent viscosity (dL/g)[a] | Solvent | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | DMAc | DMF | NMP | DMSO | HMPA | DO | PY | THF | CCl₄ |
| **I** | 0.51 | +h | +h | ++ | +h | +h | S | ++ | ± | — |
| **II** | 0.50 | ++ | ++ | ++ | ++ | +h | − | ++ | ± | — |
| **III** | 0.44 | ++ | ++ | ++ | ++ | +h | S | ++ | S | S |
| **IV** | 0.45 | ++ | ++ | ++ | ++ | ++ | ++ | ++ | ++ | +h |
| **V** | 0.55 | ++ | ++ | ++ | ++ | ++ | ++ | ++ | ++ | ++ |

The solubility was measured at a polymer concentration of 0.05 g/mL. ++ = soluble at room temperature; +h = soluble after heating (60°C); ± = partially soluble; − = insoluble; S = swelling; DMAc = N,N-dimethyl acetamide; DMF = N,N′-dimethyl formamide; DMSO = dimethyl sulfoxide; DO = dioxane; HMPA = hexamethyl phosphor amide; NMP = N-methyl pyrrolidone; PY = pyridine; THF = tetrahydrofuran.
[a] Measured at a polymer concentration of 0.5 g/dL in NMP as a solvent at 25°C.

**TABLE IV**
**Thermal Data**

| Polyamide code | $T_g$ (°C) | $T_0$ (°C) | $T_5$ (°C) | $T_{10}$ (°C) | $T_{max}$ (°C) | Char yield at 700°C (%)[a] |
|---|---|---|---|---|---|---|
| I | 196 | 205 | 373 | 417 | 461 | 41.00 |
| II | 184 | 202 | 351 | 399 | 456 | 38.49 |
| III | 166 | 227 | 325 | 398 | 459 | 36.27 |
| IV | 103 | 168 | 320 | 361 | 439 | 34.18 |
| V | 85 | 163 | 306 | 344 | 436 | 32.78 |

$T_0$ = temperature for 0% weight loss; $T_5$ = temperature for 5% weight loss; $T_{10}$ = temperature for 10% weight loss; $T_g$ = glass-transition temperature; $T_{max}$ = maximum decomposition temperature.

[a] Weight of the polymer remaining at 700°C.

of the polyamides showed clear melting endotherms in the DSC thermograms. This could be attributed to the amorphous nature of the polyamides. The obtained results are collected in Table IV, and the representative DSC curves are shown in Figure 8.

The thermal stability of the polyamides was evaluated by thermogravimetric analysis in an $N_2$ atmosphere at the heating rate of 10°C/min. The 5% decomposition temperatures of the polyamides were about 306–373°C, and the temperatures for 10% weight loss, important criteria for the evaluation of the thermal stability, were in the range of 344–417°C. The char yields of the polymers at 700°C were about 32–41%, which indicated good thermal stability. The obtained results are collected in Table IV, and the representative thermogravimetric analysis curves are shown in Figure 9. According to the obtained results, the polyamides showed suitable thermal stability, and the fully aromatic ones were more stable than the semiaromatic polyamides.

## CONCLUSIONS

The design and synthesis of a novel diamine for the preparation of thermally stable polyamides with improved solubility were our main aim. Therefore, a novel diamine, ADOAc, was successfully prepared in a high yield through the amidation reaction of 4-aminoacetanilide with 2,7-dibromo-9,9-dioctylfluorene in the presence of 10 mol % CuI, 20 mol % DMEDA as a catalyst, and $K_2CO_3$ as base. Polycondensation reactions of this diamine with aromatic and aliphatic diacids resulted in polyamides. The overall particularity of the chemical structure of the polyamide chains enhanced the solubility and caused a nice balance between properties such as thermal stability and solubility. The presence of phenyl with mixed amide groups in the backbone of the polyamides and the symmetry in the structure of the monomer were the main structural factors for the suitable properties.
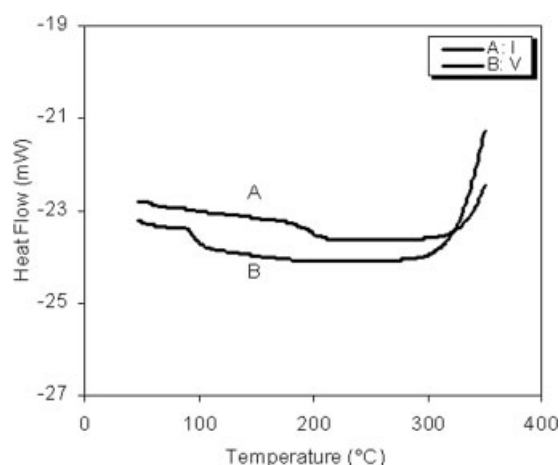


**Figure 8** DSC curves of polyamides **I** and **V**.
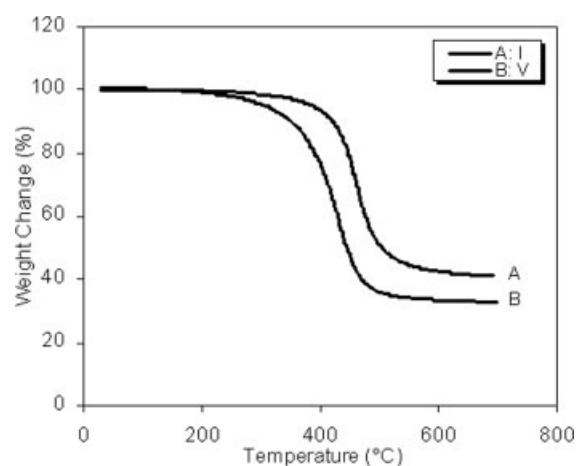


**Figure 9** Thermogravimetric analysis curves of polyamides **I** and **V**.

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