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ORIGINAL PAPER

Synthesis and characterization of novel polyhydrazides containing pendant amide-imide-pyridine moiety

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Abstract Eight novel polyhydrazides (**PH$_{\mathbf{a4-h3}}$**) were prepared from the reaction of two novel diacids, 5-(2-(6-methyl pyridine-2-yl)-1,3-dioxo isoindoline-6-carboxamido) isophthalic acid (**3**) and 5-(2-(4-methyl pyridine-2-yl)-1,3-dioxo isoindoline-6-carboxamido) isophthalic acid (**4**), with four dihydrazides by interfacial polycondensation. These polymers have moderate inherent viscosities (0.12–0.17 dL/g) and are readily soluble in polar aprotic solvents. They start to decompose ($T_{10\%}$) above 187 °C and display glass transition temperatures in the range of 130.3–156.3 °C. All polymers could be thermally converted into the corresponding polyoxadiazole approximately in the region of 250 °C, as evidenced by the DSC thermograms. All of the above polymers were fully characterized by FT-IR and ^{1}HNMR spectroscopy, TGA, DSC, and inherent viscosity measurement.

Keywords Polyhydrazide · Interfacial polymerization · Chelation

Introduction

Polyhydrazides are high performance materials and have been extensively studied as they increase dye ability of synthetic fibers and improve elasticity over other polymer types. These polymers can be spun to fibers of high modulus of elasticity (high modulus fibers) and are used to reinforce plastics as a tire cord. They also

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can be used in protective and decorative coating, strengthening agents, textile finishing agents, high temperature fibers, etc. [1]. The high molecular weight polymers have been obtained by reacting diacid chloride with dihydrazide at low temperature in a dimethylacetamide solvent [2]. They also provide a synthetic base for the chelate polymers [3], as the hydrazide group (–CO–NH–NH–CO–) can react with metal ions to form complexes. They have been cyclized to give polyoxadiazoles and polytriazoles. Pyridine, polyamides and polyhydrazides react with transition metal cations to form stable complexes [4, 5]. Consequently, combination of polyhydrazide with pyridine ring can be extremely effective chelation agent.

The solubility and thermal characteristics of the polymers can be enhanced by having cyclic side groups, such as fluorene, phthalide, and phthalimidine groups on the polymer backbone [6–8]. Thus, the introduction of phthalimide groups into the macromolecular chains of polyhydrazides improves the properties of the resulting materials. So, recently polyamides containing pyridine rings have been synthesized [9–11]. It has been shown that the introduction of pyridine rings in the aromatic polyamide usually enhances their thermal stability, solubility, and adhesion to metals [12, 13].

In this study, aforementioned novel diacids introduce bulky amide-imide pendent groups including pyridine rings. This structure improves properties of the synthesized polyhydrazides as shown in this article. Furthermore, these polymers can potentially uptake heavy metals. Diacids (**3**) and (**4**) were transformed to their corresponding diacyl chlorides, and then the interfacial polymerization was applied to prepare eight new polymers. These two novel diacids and all polymers were characterized by FT-IR and ^{1}HNMR spectroscopy. The outstanding characteristics of these polymers include thermal stability and desired solubility, chelation ability which will be study in the next part of our research.

Experimental

Materials and instruments

Trimellitic anhydride (Merck) was recrystallized from acetic anhydride. 2-Amino 6-methyl pyridine was recrystallized from H_2O/EtOH (2/3) mixture. *N*,*N*-Dimethyl acetamide (DMAc) (Merck) was dried over BaO, followed by fractional distillation. 5-Aminoisophthalic acid (5AIPA) was recrystallized from H_2O/DMF (5/2) mixture. The other chemicals (Merck or Fluka) were used as received. ^{1}HNMR spectra were recorded on 250 or 300 MHz using DMSO-d_6 as solvent. FT-IR spectra were recorded on a Tenssor 27 instrument, using KBr pellets. Thermogravimetric analyses (TGA) were recorded on a TGA-PL 1500 with heating rate of 10 °C min^{-1} under air atmosphere. DSC analyses were preformed on a PL-DSC with heating rate of 5 °C min^{-1} under nitrogen atmosphere. Wide-angle X-ray diffraction measurements were performed at room temperature on a Bruker-D8 X-ray diffractometer (operating at 40 kV and 30 mA) with graphite monochromatized Cu Kα radiation ($\lambda = 1.54118$ Å). The scanning rate was

2°/min. Inherent viscosities of polymers were measured by a standard procedure using a Cannon–Fenske Routine Viscometer (Germany) at 25 °C using DMF as solvent. Melting points were measured in open capillaries with a IA9000 series digital Melting Point Apparatus.

Monomer synthesis

*Synthesis of 2-(6-methyl pyridine-2-yl)-1,3-dioxo isoindoline-5-carboxylic acid (**1**)*

Into a 50-mL-round bottomed flask, 0.500 g (4.58×10^{-3} mol) 2-amino 6-methyl pyridine, 0.880 g (4.58×10^{-3} mol) trimellitic anhydride, 25 mL glacial acetic acid and a stirring bar were placed. The mixture was stirred at r.t. for 3 h and then refluxed for 12 h. The solution was poured into a mixture of H_2O/MeOH (5/2).

A white precipitate was formed, filtered off, and washed with water. The solid was dried to leave 1.03 g (80%) of acid **1** m.p. = 190 °C; IR (KBr): 3544–2400, 1780, 1732, 1607, 1578, 1458, 1371, 1269, 1099, 1014, 792, 730 cm^{-1}. ^{1}HNMR (DMSO-d_6) δ: 2.36 (s, 3H), 6.97 (d, 1H), 7.65 (t, 1H), 7.88 (m, 4H), 10.98 (s, 1H).

*Synthesis of 2-(4-methyl pyridine-2-yl)-1,3-dioxo isoindoline-5-carboxylic acid (**2**)*

Using 2-amino, 4-methyl pyridine and following the above-mentioned procedure, the carboxylic acid of **2** was prepared. Yield: 71.3%; $d = 184$ °C; FT-IR (KBr): 3600–2400, 1789, 1720, 1614, 1424, 1358, 1284, 1234, 1187, 1093, 1015, 849, 804, 727, 699, 649 cm^{-1}. ^{1}HNMR (DMSO-d_6) δ: 2.50 (s, 3H), 7.39 (s, 2H), 8.08 (d, 1H), 8.32 (s, 2H), 8.49 (s, 1H), 13.79 (s, 1H).

*Synthesis of 5-(2-(6-methyl pyridine-2-yl)-1,3-dioxo isoindoline-6-carboxamido) isophthalic acid (**3**)*

Acid **1** was reacted with thionyl chloride to yield the corresponding acyl chloride according to the reported procedure [14]. Into a 25-mL-round-bottomed flask equipped with a magnetic stirrer a solution of 5-amino isophthalic acid (0.642 g, 3.54×10^{-3} mol) in dry DMAc (5 mL) was placed. The reaction mixture was heated in an oil bath at 80 °C for 0.5 h and then cooled to room temperature. The mixture of 1.06 g (3.54×10^{-3} mol) acid chloride in 5 mL of dry DMAc in the presence of 5 mL triethylamine was added to this solution and stirred for 40 h at room temperature. The solution was poured into 100 mL of HCl (5%). The white precipitate was collected by filtration and washed thoroughly with water and dried at 70 °C for 12 h to give 1.10 g of diacid (**3**). Yield: 70%; $d = 314$ °C; IR-(KBr): 3600–2400, 1727, 1551, 1451, 1378, 1208, 904, 757, 719, 669, 641 cm^{-1}. ^{1}H-NMR (DMSO-d_6) δ: 2.39 (s, 3H), 7.35 (t, 2H), 7.89 (t, 1H), 8.12 (d, 1H), 8.23 (s, 1H), 8.47 (d, 1H), 8.60 (s, 1H), 8.69 (s, 2H), 10.94 (s, 1H), 13.33 (s, 2H). ^{13}C-NMR (DMSO-d_6) δ: 14.17, 18.94, 24.09, 120.54, 122.94, 124.13, 124.28, 125.26, 125.84, 132.11, 134.25, 135.14, 139.31, 139.85, 140.21, 145.43, 158.84, 164.16, 166.32, 166.45, 166.89, 181.69 ppm.

*Synthesis of 5-(2-(4-methyl pyridine-2-yl)-1,3-dioxo isoindoline-6-carboxamido) isophthalic acid (**4**)*

Following the above-mentioned procedure, the dicarboxylic acid of **4** was synthesized, Yield: 65%; m.p. = 217 °C; FT-IR (KBr): 3600–2400, 1720, 1609, 1555, 1419, 1239, 906, 760, 670 cm^{-1}. ^{1}HNMR (DMSO-d_6) δ: 2.40 (s, 3H), 6.97 (s, 1H), 7.38 (s, 1H), 8.02 (s, 1H), 8.17 (d, 2H), 8.49(s, 1H), 8.67 (s, 3H), 10.83 (s, 1H), 10.89 (s, 2H).

*Dihydrazide synthesis (**5**–**8**)*

General procedure In a 25-mL-round-bottomed flask equipped with a reflux condenser, a mixture of corresponding dimethyl ester (1.00 mol), hydrazine mono hydrate (2.50 mol), and ethanol (10 mL) were placed and refluxed for 5 h. Upon cooling, crystals separated, which were filtered and washed with ethanol.

*Dihydrazide **5*** White; yield (%) = 95; m.p. = 220 °C; IR (cm^{-1}): 3324, 3033, 1623, 1605, 1540, 1489, 1340, 1291, 1103, 1016, 927, 886, 736, 713, 638; ^{1}HNMR (DMSO-d_6) δ: (ppm): 4.51 (s, 4H), 7.90 (s, 4H), 9.90 (s, 2H)

*Dihydrazide **6*** White; yield (%) = 87; m.p. (°C) = 176–177; IR (cm^{-1}): 3315, 3180, 3046, 2965, 2927, 1636, 1532, 1379, 1330, 1157, 1016, 1001, 954, 711, 618; ^{1}HNMR (DMSO-d_6) δ: (ppm): 1.75 (quin, 2H), 2.00 (t, 4H), 3.51 (s, 4H), 8.90 (s, 2H)

*Dihydrazide **7*** Yellow; yield (%) = 80; m.p. (°C) = 148–149; IR (cm^{-1}): 3304, 3201, 3131, 3034, 2873, 1667, 1647, 1532, 1363, 1248, 1203, 1052, 955, 788, 694; ^{1}HNMR (DMSO-d_6) δ: (ppm): 2.90 (s, 2H), 4.20 (s, 4H), 9.11 (s, 2H)

*Dihydrazide **8*** White-gray; yield (%) = 75; m.p. (°C) = 240–241; IR (cm^{-1}): 3282, 3181, 3007, 1679, 1611, 1534, 1360, 1269, 1097, 998, 793, 537; ^{1}HNMR (300 MHz) δ: (ppm): 4.11 (s, 4H), 9.60 (s, 2H)

Polyhydrazides synthesis, general procedure

Into a 25-mL-round-bottomed flask in an ice bath, 1 mmol diacyl chloride, according to the reported procedure [14], 7 mL of dichloromethane and a stirring bar were placed. A solution of dihydrazide (1 mmol) in water (10 mL), sodium hydroxide (6.3 mL, 0.1 N) and catalytic amount of tetraethylammonium bromide were prepared. This solution was added dropwise to the flask during 10 min with stirring the mixture vigorously. After stirring for 3 h, the polymer was filtered off, washed with methanol, and vacuum dried at 90 °C to a constant weight.

PH_{a4}: Pale pink; yield (%) = 49; η_{inh} (dL g^{-1}) = 0.12; IR (cm^{-1}): 3422, 1783, 1727, 1651, 1559, 1455, 1373, 1244, 1058, 746; ^{1}H NMR (DMSO-d_6) δ: (ppm): 2.45 (3H, s), 7.01–8.69 (13H, m), 10.76 (5H, br).

PH_{b4}: Cream; yield (%) = 46; η_{inh} (dL g^{-1}) = 0.15; IR (cm^{-1}): 3251, 1791, 1721, 1557, 1452, 1374, 1245, 799, 748; ^{1}H NMR (DMSO-d_6) δ: (ppm): 1.86 (2H, m), 2.38 (3H, s), 3.35 (2H, br), 3.77 (2H, br), 6.99–8.48 (9H, m), 9.99 (1H, br), 10.44 (2H, br), 10.90 (2H, br).

PH_{c4}: Cream; yield (%) = 50; η_{inh} (dL g^{-1}) = 0.13; IR (cm^{-1}): 3422, 1742, 1642, 1557, 1451, 1244, 801; ^{1}H NMR (DMSO-d_6) δ: (ppm): 2.24 (3H, s), 3.41 (2H, br), 7.00–8.48 (9H, m), 10.25 (1H, br), 10.91 (4H, br).

PH_{d4}: Cream; yield (%) = 54; η_{inh} (dL g^{-1}) = 0.17; IR (cm^{-1}): 3252, 1791, 1720, 1557, 1452, 1374, 1244, 1066, 799, 748; ^{1}H NMR (DMSO-d_6) δ: (ppm): 2.48 (3H, s), 7.01–8.51 (9H, m), 10.74 (1H, br), 10.96 (4H, br).

PH_{e3}: Pale yellow; yield (%) = 50; η_{inh} (dL g^{-1}) = 0.14; IR (cm^{-1}): 3650–2250, 1721, 1610, 1417, 1389, 1237, 747, 678; ^{1}H NMR (DMSO-d_6) δ: (ppm): 2.41 (3H, s), 6.73–9.19 (13H, m), 10.48 (1H, br), 10.77 (2H, br), 11.17 (2H, br).

PH_{f3}: Pale yellow; yield (%) = 41; η_{inh} (dL g^{-1}) = 0.12; IR (cm^{-1}): 3620–2400, 1722, 1611, 1418, 1369, 1241, 748, 680; ^{1}H NMR (DMSO-d_6) δ: (ppm): 1.87 (2H, m), 2.41 (3H, s), 3.39 (2H, br), 3.79 (2H, br), 7.08–8.93 (9H, m), 10.00 (1H, br), 10.45 (2H, br), 10.57 (2H, br).

PH_{g3}: Pale yellow; yield (%) = 43; η_{inh} (dL g^{-1}) = 0.14; IR (cm^{-1}): 3600–2420, 1723, 1611, 1419, 1369, 1239, 762, 680; ^{1}H NMR (DMSO-d_6) δ: (ppm): 2.41 (3H, s), 3.90 (2H, br), 7.07–8.69 (9H, m), 10.25 (1H, br), 10.96 (4H, br).

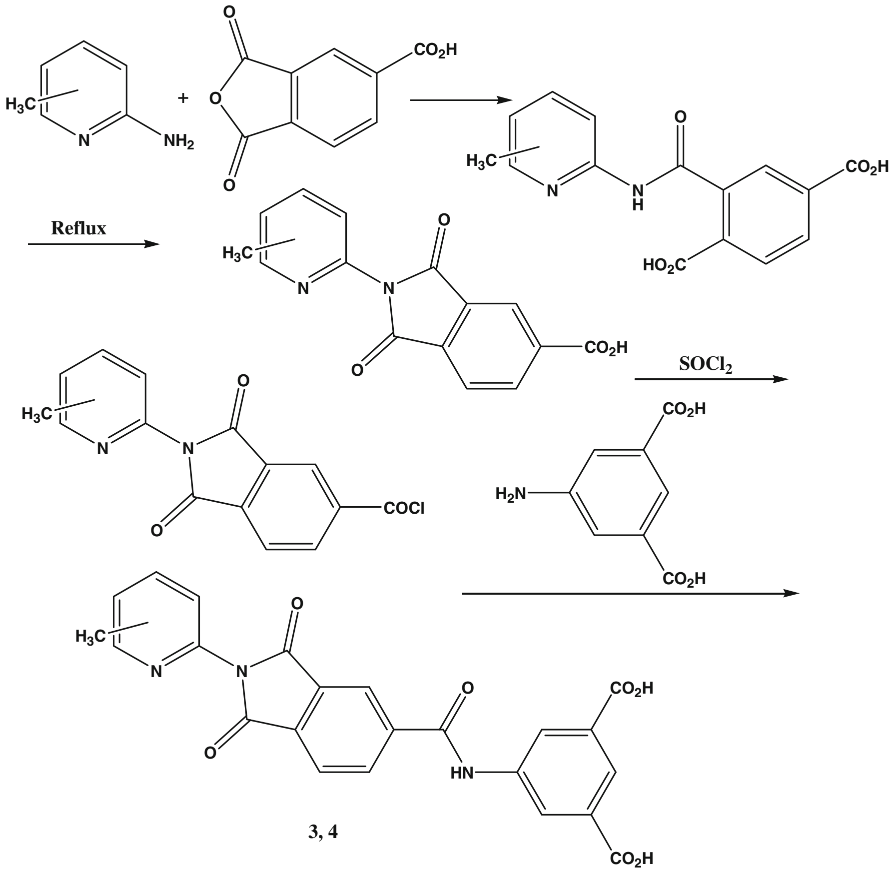
PH_{h3}: Pale yellow; yield (%) = 52; η_{inh} (dL g^{-1}) = 0.16; IR (cm^{-1}): 3610–2400, 1721, 1611, 1583, 1446, 1368, 1241, 679; ^{1}H NMR (DMSO-d_6) δ: (ppm): 2.41 (3H, s), 7.11–8.70 (9H, m), 10.75 (1H, br), 10.96 (4H, br).

Results and discussion

Monomer synthesis

We synthesized the diacids, 5-(2-(6-methyl pyridine-2-yl)-1,3-dioxo isoindoline-6-carboxamido) isophthalic acid (**3**) and 5-(2-(4-methyl pyridine-2-yl)-1,3-dioxo isoindoline-6-carboxamido) isophthalic acid (**4**), by the condensation reaction in refluxing acetic acid in two steps. Washing the residue with concentrated HCl turned them into white solids (Scheme 1). The step II was completed within 2 h, when, the mixtures were completely dissolved in refluxing thionyl chloride. The ^{1}HNMR spectra of diacid (**3**) showed peaks that confirm its chemical structure (Fig. 1).

In FT-IR spectra of these diacids some characteristic peaks could be seen, including a strong and broad peak at 3,600–2,400 cm^{-1} corresponding to the COOH stretching and N–H stretching, C=O asymmetric stretching of imide group at 1,780 cm^{-1}, the C=O symmetric stretching of imide and amide groups at



3, 4

3: Synthesis of 5-(2-(6-methyl pyridine-2-yl)-1,3-dioxo isoindoline-6-carboxamido) isophtalic acid

4: Synthesis of 5-(2-(4-methyl pyridine-2-yl)-1,3-dioxo isoindoline-6-carboxamido) isophtalic acid

Scheme 1 Diacids (**3**), (**4**) synthesis

1,727–1,710 cm^{-1}, C–N stretching at around 1,551 cm^{-1} and two peaks at 1,378 and 757 cm^{-1} due to the cyclic imide groups.

^{1}HNMR spectroscopy shows the corresponding peaks, such as 2.39 (s, 3H, CH_3), 7.35 (t, 2H, Ar), 7.89 (t, 1H, Ar), 8.12 (d, 1H, Ar), 8.23 (s, 1H, Ar), 8.47 (d, 1H, Ar), 8.60 (s, 1H, Ar), 8.69 (s, 2H, Ar), 10.94 (s, 1H, N–H), 13.33 (s, 2H, COOH).

^{13}CNMR ($CDCl_3$, ppm) spectroscopy agrees well with the proposed structures of these two diacids. As pyridine moiety and their orientations, these monomers can potentially act as good complexing agent to trap the heavy metal cations [15]. Therefore, these monomers can be used to synthesize polyhydrazides to achieve this goal.

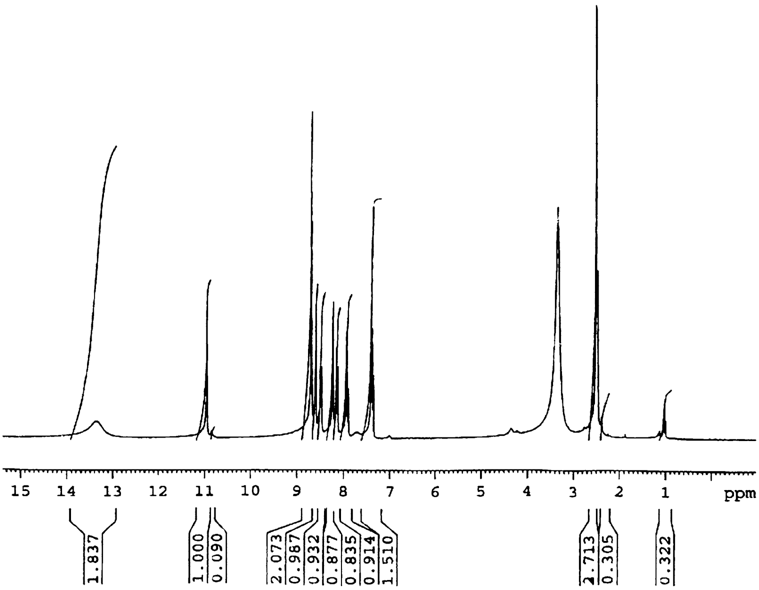


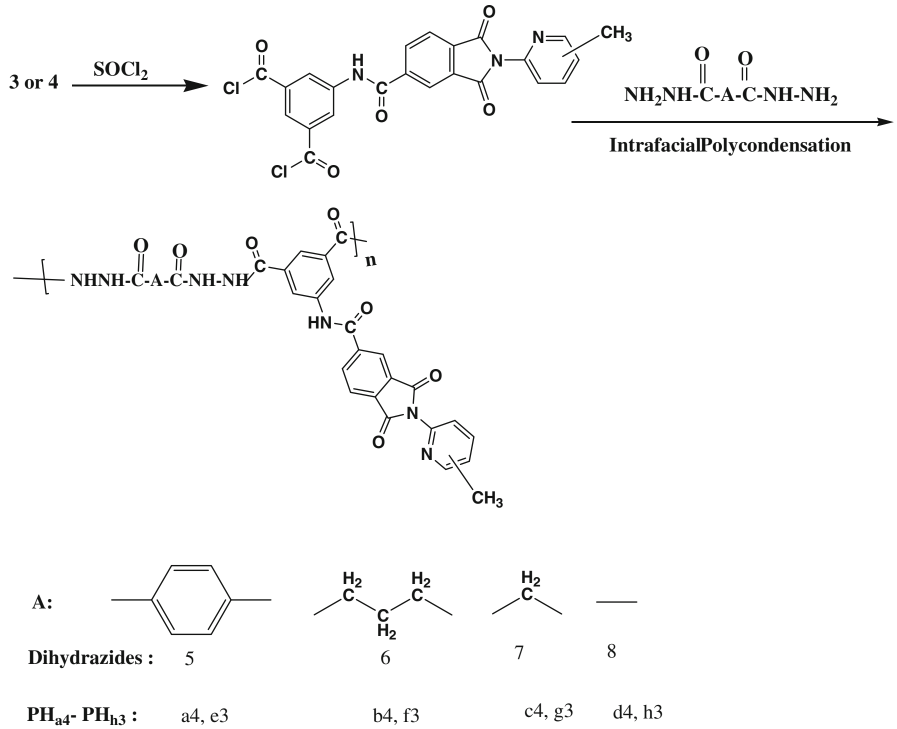
Fig. 1 ^{1}H NMR spectrum of diacid **3**

Polymer synthesis

Interfacial polymerization was applied to prepare the polyhydrazides in the presence of catalytic amount of tetraethylammonium bromide (Scheme 2).

Theses polyhydrazides bear polar pendent groups which may increase crystallinity and restrict the solubility in organic solvents. Introducing hydrazide groups into the backbone of polymers increases backbone flexibility to enhance solubility of theses type of polymers. The **PH**s are obtained with desired inherent viscosities, although the yields of polymerization are not high. The formation of **PH**s was confirmed by FT-IR spectroscopy and ^{1}HNMR spectroscopy. The colors of these polymers are pale yellow to pale pink. The FT-IR spectrum of $\mathbf{PH}_{a4}$ shows the characteristic absorptions at 3422 (N–H, amidic and hydrazide stretchings), 1,780 cm^{-1} (C=O, asymmetric, imidic), 1,727 cm^{-1} (C=O, symmetric, imidic), 1,630 cm^{-1} (C=O stretching of hydrazide group), 1,559 cm^{-1} (C–N stretching of imide), 1,373 and 746 cm^{-1} (imidic ring). ^{1}HNMR spectrum of $\mathbf{PH}_{e3}$ (Fig. 2) exhibits 2.45 (3H, s, CH_3), 7.01–8.69 (13H, m, Ar), 10.76 (5H, br, N–H).

One of the major objectives of this study is to study the solubility and the versatility of these polymers by incorporating the soft segment in the polymer backbone. The solubility of **PH**s was tested qualitatively in various organic solvents and the results are summarized in Table 1. All of the **PH**s are soluble in organic solvents, such as DMF, DMSO, NMP, and H_2SO_4 at room temperature.



Scheme 2 Interfacial polymerization to synthesize **PH_{a4}–PH_{h3}**

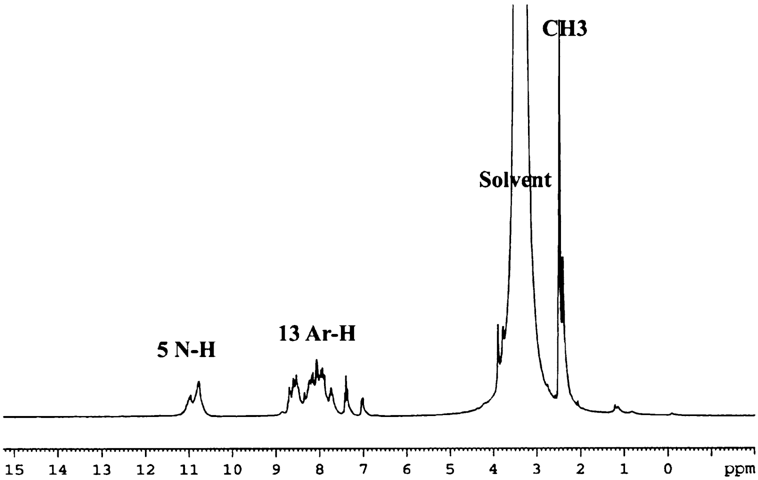


Fig. 2 ^{1}H NMR spectrum of **PH_{e3}**

Table 1 Solubility of polyhydrazides

Solvents	$\mathbf{PH_{a4}}$	$\mathbf{PH_{b4}}$	$\mathbf{PH_{c4}}$	$\mathbf{PH_{d4}}$	$\mathbf{PH_{e3}}$	$\mathbf{PH_{f3}}$	$\mathbf{PH_{g3}}$	$\mathbf{PH_{h3}}$
NMP	+	+	+	+	+	+	+	+
DMSO	+	+	+	+	+	+	+	+
DMAc	+	+	+	+	+	+	+	+
DMF	+	+	+	+	+	+	+	+
H_2SO_4	+	+	+	+	+	+	+	+
CH_2Cl_2	−	−	−	−	−	−	−	−

Concentration: 5 mg mL^{-1}, + soluble at room temperature; − insoluble at room temperature

Table 2 Thermal behavior of polyhydrazides

Polymer	Decomposition temperature (°C) $T^{a}_{10\%}$	T_g	Char yield (%)[b]	LOI
$\mathbf{PH_{a4}}$	234.87	145.6	13.50	22.9
$\mathbf{PH_{b4}}$	196.41	131.5	43.30	34.8
$\mathbf{PH_{c4}}$	278.60	140.3	24.50	27.3
$\mathbf{PH_{d4}}$	239.08	146.4	39.78	33.4
$\mathbf{PH_{e3}}$	187.96	130.3	48.80	37.0
$\mathbf{PH_{f3}}$	210.40	131.7	36.59	32.1
$\mathbf{PH_{g3}}$	215.39	156.3	44.65	35.4
$\mathbf{PH_{h3}}$	237.33	134.1	15.40	23.7

T_g glass transition temperature, *LOI* limiting oxygen index

[a] Temperature at which 10% weight loss was recorded by TGA at a heating rate of 10 °C/min under air atmosphere

[b] Percentage weight of material left after TGA analysis at maximum temperature 600 °C under air atmosphere

The thermal properties of **PH**s were evaluated by means of TGA under air atmosphere and DSC under nitrogen atmosphere. They show two different decomposition maxima and a good resistance to thermal decomposition.

The temperature of 10% weight loss, the residual weight at 600 °C and the T_gs are presented in Table 2. The initial weight loss in TGA can be due to dehydration of hydrazide moieties in polymer backbones to oxadiazols. TGA/DTG and DSC thermograms of $\mathbf{PH_{e3}}$ are presented in Figs. 3 and 4. The DSC diagram exhibits no T_m but a sign of crystalline formation around 240 °C.

The high char yields of the synthetic polymers could be due to their high aromatic content. Char yield can be used as a factor for estimated limiting oxygen index (LOI) of the polymers based on Van Krevelen and Hoftyzer equation. LOI = 17.5 + 0.4 CR, where CR = char yield. On the basis of LOI values, these polymers can be classified as self-extinguishing polymers.

To evaluate the amount of crystallinity in the polymers, the wide-angle X-ray diffraction pattern of the $\mathbf{PH_{a4}}$ in the region of $2\theta = 5°–100°$ at room temperature was studied. The spectrum of XRD of $\mathbf{PH_{a4}}$ (Fig. 5) shows two main peaks at $2\theta = 13°$ and 23°. The crystallinity of the polymers was about 29% (according to the data obtained from the calculation of area of the peaks) due to highly polar pendent groups therefore, it is almost semicrystalline polymer.

As pyridine moiety and their orientations these polymers can potentially act as good chelation agent to trap the heavy metal cations. Indeed the pendant methyl

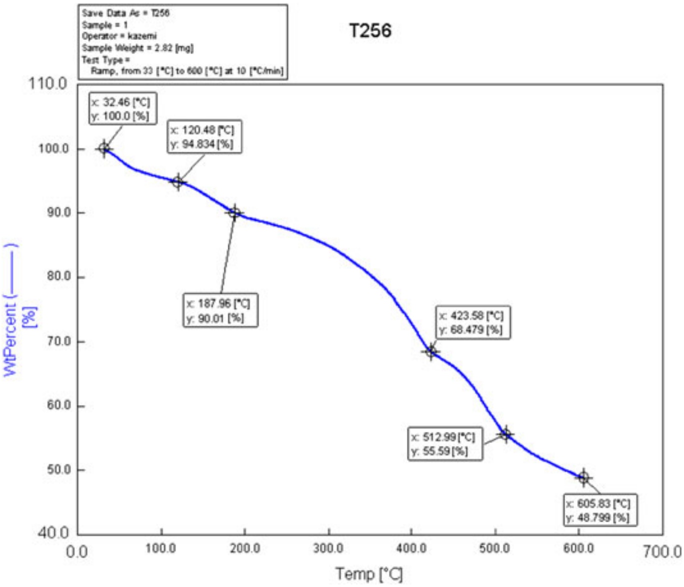


Fig. 3 TGA spectrum of $\mathbf{PH_{e3}}$

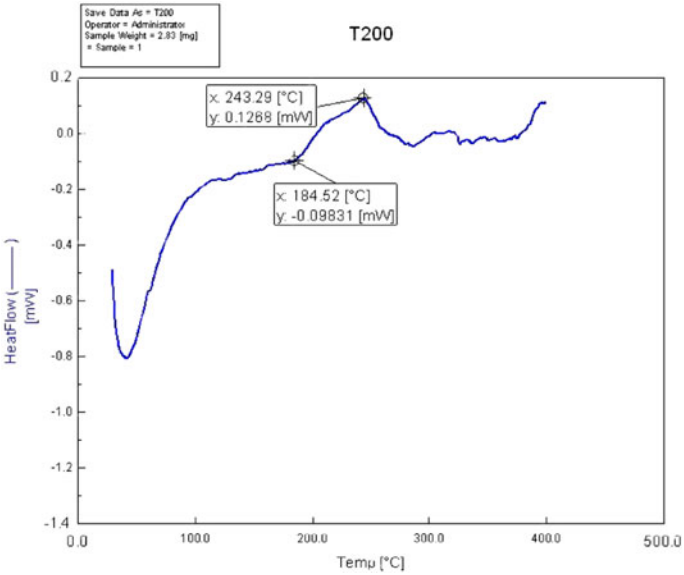


Fig. 4 DSC spectrum of $\mathbf{PH_{e3}}$

group on the pyridine rings can be potentially oxidized to give acidic sites suitable in ion-exchangeable resin design (we are going to do on these abilities in the next part of our research).

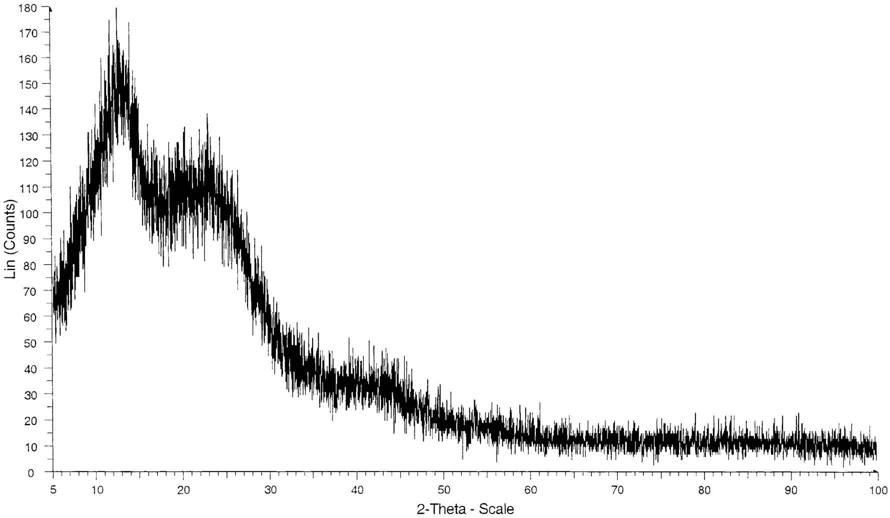


Fig. 5 XRD spectrum of $\mathbf{PH_{a4}}$

Conclusions

A fast and effective procedure to prepare a series of pyridine containing polyhydrazides has been introduced. These polymers are soluble, thermally stable, and are potentially good complexing agents. The resulting polymers are completely identified and characterized.

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