



Synthesis and characterization of novel heat resistant poly(amide imide)s

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Abstract

A series of new poly(amide imide)s was prepared from new diacid containing sulfone, ether, amide and imide groups with various aromatic diamines. The diacid was synthesized via four steps, starting from reaction of 4-aminophenol with 4-nitrobenzoyl chloride in the presence of propylene oxide afforded *N*-(4-hydroxy phenyl)-4-nitrobenzamide. In the second step, reduction of nitro group resulted in preparation of 4-amino-*N*-(4-hydroxy phenyl) benzamide. In the next step for the preparation of diamine, the reaction of 4-amino-*N*-(4-hydroxy phenyl) benzamide with bis-(4-chlorophenyl) sulfone in the presence of K_2CO_3 was achieved. The prepared sulfone ether amide diamine was reacted with two moles of trimellitic anhydride to synthesize related sulfone ether amide imide diacid. The precursors and final monomer were characterized by FT-IR, H-NMR and elemental analysis. Direct polycondensation reaction of the sulfone ether amide imide diacid with different diamines in the presence of triphenyl phosphite afforded five different poly(sulfone ether amide imide amide)s. The obtained polymers were fully characterized and their physical properties including thermal behavior, thermal stability, solubility, and inherent viscosity were studied.

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1. Introduction

Aromatic polyimides are thermally stable polymers, which exhibit excellent mechanical and electrical properties [1,2]. However these polymers are difficult to process because of their high softening temperatures and poor solubility in organic solvents. In order to improve the solubility and processability, several approaches have

been proposed, based on the incorporation of flexible segments bearing sulfone, amide, and ether moieties in the polymer backbone, without sacrificing the heat resistance [3]. Thus, several copolymers have been developed, such as poly(amide imide)s (PAIs), which are known to have high thermal stability and good solubility in polar amide type solvents. Poly(amide imide)s provide a favorable balance between processability and performance and are useful in numerous applications in electrical wire enamel, adhesives, and injection-molding and extrusion products [4,5]. Various approaches have been thus far devised to prepare PAIs [6–19]. Among these methods, direct polycondensation is the most convenient approach for synthesizing high molecular weight PAIs

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on a laboratory scale. Yamazaki et al. reported triphenyl phosphite-promoted polycondensation for synthesis of polyamides in 1975 [20]. The main strategy in the field of thermally stable polymers is to design and synthesize polymers with high thermal stability and improved solubility. It was reported that the polymer chain having a cyclic cardo (bulky pendant) group, for example, a fluorene, a phthalide, or a phthalimidine group, can cause a significant increase in both glass transition temperature and thermal oxidative stability while providing good solubility [21,22]. On the other hand incorporation of flexible units such as $-\text{NHCO}-$, $-\text{O}-$, and $-\text{SO}_2-$ and incorporation of bulky substituents or bulky pendant groups into rigid polymer backbones is an important way to improve the solubility of the polymers [23,24].

The main aim of this study was to prepare novel poly-(amide imide)s with improved solubility without sacrificing their thermal stability. Generally, poly(amide imide)s can be synthesized by different methods including: (1) from trimellitic anhydride with diamines, (2) from monoimide dicarboxylic acids with diamines, (3) from diimide dicarboxylic acids with diamines, (4) from diimide diamines with dicarboxylic acids, (5) from dianhydrides containing amide functions and diisocyanates, and (6) by the Pd catalyzed carbonylation of aromatic dihalides containing an imide skeleton with diamines [25].

So, it can be seen that diamine is the basic component for the preparation of poly(amide imide)s, therefore design and synthesis of diamine is very important. Accordingly, synthesis of a diamine (SEAD) with special functional groups was considered. The diamine with built-in sulfone, ether, and amide units was prepared to induce thermal stability and also solubility character to the final polymers.

2. Experimental

2.1. Materials

All chemicals were purchased either from Merck or Aldrich chemical Co. *N*-Methyl-2-pyrrolidone (NMP), *N,N*-dimethylacetamide (DMAc), *N,N*-dimethylformamide (DMF) and toluene were distilled over calcium hydride under reduced pressure. Trimellitic anhydride (TMA), 4,4'-diamino diphenyl methane, *p*-phenylene diamine, 1,5-diaminonaphthalene, and 2,6-diaminopyridine were recrystallized from tetrahydrofuran (THF), water, ethanol, water and benzene, respectively.

2.2. Instruments

Infrared measurements were performed on a Bruker-IFS 48 FT-IR spectrometer (Ettlingen, Germany). The H-NMR spectra were recorded in DMSO- d_6 solution using a Bruker Avance DPX 250 MHz (GmbH, Ger-

many). Elemental analyses were performed by a CHN-O-Rapid Heraeus elemental analyzer (Wellesley, MA). Differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) were recorded on a Stanton Redcraft STA-780 (London, UK). The dynamic mechanical measurements were recorded on a Polymer Laboratories Dynamic Mechanical Thermal Analyzer (DMTA) in the bending mode at 1 Hz and a heating rate of 5 °C/min (Surrey, UK). Inherent viscosities were measured by using an Ubbelohde viscometer.

2.3. Monomer synthesis

The sulfone ether amide diamine (SEAD) was obtained as described in our previous report [26]. To a 0.01 mol solution of SEAD dissolved in 25 ml of NMP in a 50 ml flask, 0.021 mol of TMA was added. The stirred reaction mixture was heated to 80 °C for 2 h under N_2 . Then, 8 ml of dry toluene was added to the mixture and it was heated to reflux for about 12 h until water was distilled off azeotropically via Dean-Stark trap.

After cooling the reaction mixture to room temperature, the precipitate was collected by filtration, washed several times with methanol and dried in a vacuum oven at 100 °C. (yield 81%). [IR (KBr): ν 3540–3387, 1782, 1722, 1660, 1605, 1587, 1508, 1406, 1375, 1319, 1241, 1180, 1105, 1015, and 727 cm^{-1} , H-NMR (DMSO- d_6): δ 13.65(s, 2H, COOH), 10.45(s, 2H, NH), 8.42(d, 2H, phenyl), 8.32 (s, 2H, phenyl), 8.12(d, 2H, phenyl), 8.08 (d, 4H, phenyl), 7.93 (d, 4H, phenyl), 7.86 (d, 4H, phenyl), 7.64 (d, 4H, phenyl), 7.15 (d, 4H, phenyl), 7.09 (d, 4H, phenyl); elemental analysis: Calculated for $\text{C}_{56}\text{H}_{34}\text{N}_4\text{O}_{14}\text{S}$: C, 66.01%; H, 3.36%; N, 5.50%; Found: C, 65.82%; H, 3.13%; N, 5.78%].

2.4. Poly(amide imide) synthesis

A typical procedure was as follows. A mixture of 1.6 mmol of diamine, 1.6 mmol of sulfone ether amide imide diacid (SEAD), 0.72 g of calcium chloride, 1.6 ml of triphenyl phosphite, 1.6 ml of pyridine, and 8 ml of NMP was refluxed for 3 h. After cooling, the reaction mixture was poured into a large amount of methanol with stirring. The obtained precipitate was filtered and washed with methanol and hot water. Then it was dried at 110 °C under vacuum overnight.

3. Results and discussion

We previously reported the preparation of a novel sulfone ether amide diamine (SEAD) and resulting polyimides [26]. Reaction of the obtained diamine (SEAD) with 2 moles of trimellitic anhydride resulted in preparation of a diimide-dicarboxylic acid (SEAD) (Scheme 1). Structure of the diimide dicarboxylic acid

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