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Note Studies on Heat-Resistant Polymers. Part 2. Synthesis and Characterization of two new Polyamideimides from Bis-1, 1'-(3-Carboxyphthalimidyl)-4,4'-Biphenyl and Aromatic Amines

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NOTE
STUDIES ON HEAT-RESISTANT POLYMERS. PART 2. SYNTHESIS AND CHARACTERIZATION OF TWO NEW POLYAMIDEIMIDES FROM BIS-1,1′-(3-CARBOXYPHTHALIMIDYL)-4,4′-BIPHENYL AND AROMATIC AMINES*

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INTRODUCTION

The significance of polyamideimides as important heat-resistant materials has been well established [2]. In our earlier publications we reported the synthesis of several new polyamideimides with excellent thermooxidative properties [1, 3–5]. This paper reports the convenient preparation of new monomers from trimellitic anhydride and their use in the synthesis of two new polymers by polycondensation with benzidine and/or 1,4-diaminobenzene under appropriate conditions.

EXPERIMENTAL

The starting materials were purchased from Aldrich Chemical Company and were purified before use.

Preparation of Diimidodicarboxylic Acid 3 and Diacid Chloride 4

Condensation of trimellitic anhydride (3.84 g, 0.02 mol) and benzidine (1.84 g, 0.01 mol) in DMF (250 mL) with azeotropic removal of water gave the diacid **3** in

*For Part 1, see Ref. 1.

81% yield. This acid, on reaction with an excess of thionyl chloride, gave the diacid chloride **4** in 89% yield.

Polymerization Procedure

Method A

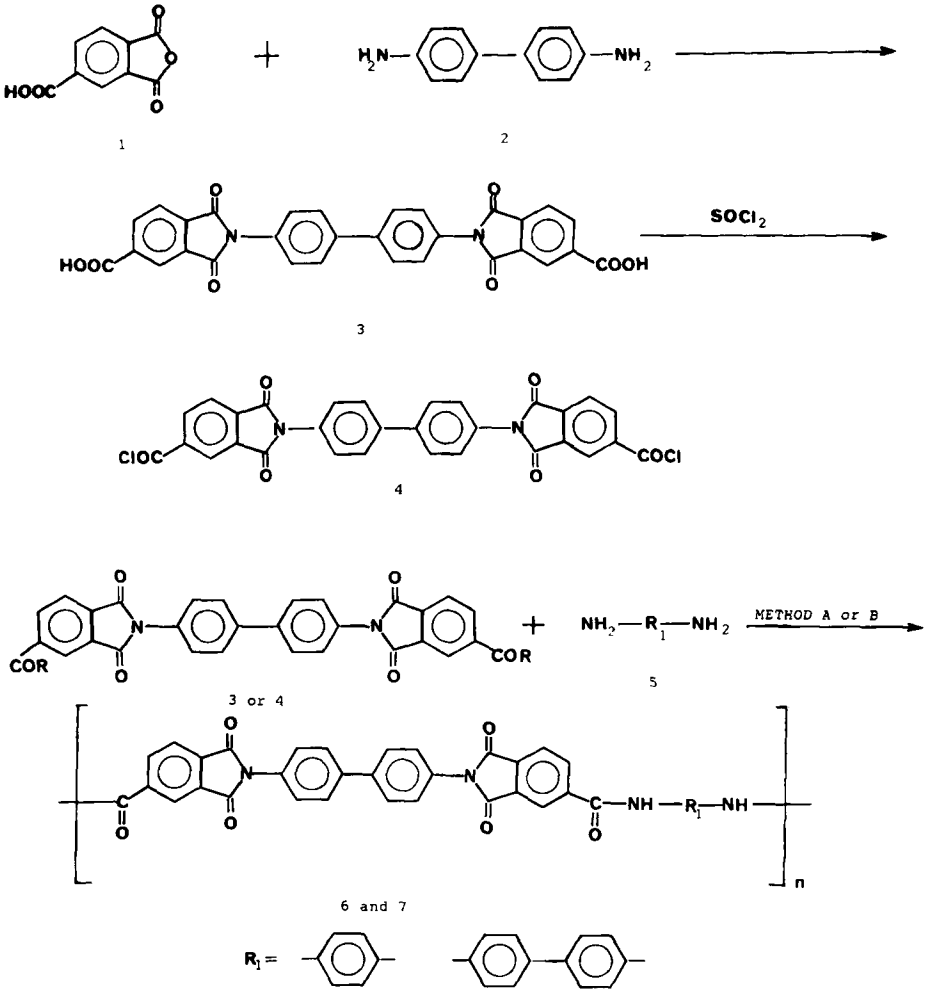
Diacid chloride **4** (2.84 g, 5 mmol) and 150 mL of DMF or NMP were placed in a two-neck round-bottom flask fitted with a stirring unit, a dropping funnel, and a nitrogen inlet system. The reaction mixture was cooled to −45°C, and a solution of diamine **5** (5 mmol) and 1.19 mL (0.01 mol) triethylamine in DMF or NMP (100 mL) was added to it slowly with the reaction mixture kept at −40 to −30°C. The addition of amine was accompanied by a heavy precipitate of polymer. The reaction mixture was well stirred at that temperature for another 4 h, after which the temperature was allowed to rise slowly to room temperature. The polymer was precipitated by addition of cold water. The solid was filtered out and dried at room temperature.

Method B

To a precooled (−20°C) suspension of diacid **3** (2.12 g, 4 mmol) in 150 mL DMF or NMP, thionyl chloride (0.95 g, 8 mmol) was added slowly, and the mixture was stirred for 1 h. A solution of the diamine (4 mmol) and triethylamine (0.88 g, 8 mmol) in 50 mL DMF or NMP was added slowly. After stirring the reaction mixture at −20°C for 3 h, the cooling bath was removed, and the reaction mixture was stirred for another 3 h. Finally, addition of water completed the precipitation of polyamideimides, which were filtered and dried at room temperature to give brown-colored solid material.

RESULTS AND DISCUSSION

The monomers and the polymers were prepared as shown in Scheme 1. The yield of the polymerization reaction was excellent. Interestingly, Monomer **4** and the diamines failed to produce any polymer under the influence of triphenylphosphine, alkyl halide, and a base [4, 5]. Characterization of the monomers and the polymers was done by elemental analysis and spectroscopic data. The polymers are insoluble in common organic polar solvents, partly soluble in formic acid, and completely soluble in concentrated sulfuric acid. X-ray diffraction data show the presence of crystallinity in both polymers. The polymers show moisture absorption below 0.7%.



METHOD A : When R = Cl; i) addition of diamine solution, ii) addition of acid acceptor solution

METHOD B : When R= OH; i) $SOCl_2$ ii) diamine solution, iii) acid acceptor solution

SCHEME 1. Synthetic pathway to prepare monomers and polymers.

Thermal study revealed that the degradation of both the polymers is a single-step process and the maximum weight loss takes place between 625 and 675°C. The glass-transition temperature of Polymers **6** and **7** are 355 and 360°C, respectively. The performance of **6** and **7** at 350°C is excellent. The cumulative weight loss at 350°C after 100 h was found to be 5%.

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