

Synthesis and Characterization of Poly(ether imide)s Containing *m*-Methyl and Phthalazinone Moieties

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Abstract: Three novel poly(ether imide)s were synthesized by one-step solution polymerization from 2-(3, 4-dicarboxyl-*N*-phenyl)-4-(3', 4'-dicarboxyl-phenoxy)-4-(2-methyl)-phenyl)-2, 3-phthalazin-1-one dianhydride and three amines, and characterized. The polymers show good solubility and thermal properties.

Keywords: Synthesis, poly(ether imide)s, phthalazinone, dianhydride.

Aromatic polyimides are distinguished for their excellent mechanical and thermal properties in many engineering fields¹. However, aromatic polyimides are normally insoluble in common organic solvents, which restricts their applications in some fields. Many efforts have been taken to improve their solubility. Introducing bulky side groups and non-coplanar structure into polymer's bone chains is a good way to obtain the polymers with excellent mechanical and thermal properties². In this paper, *m*-methyl and phthalazinone groups have been successfully introduced into the dianhydride monomer and corresponding polymers. Non-coplanar conformation of phthalazinone and *m*-methyl moieties contribute to the good solubility, and still the polymers retain their good thermal properties.

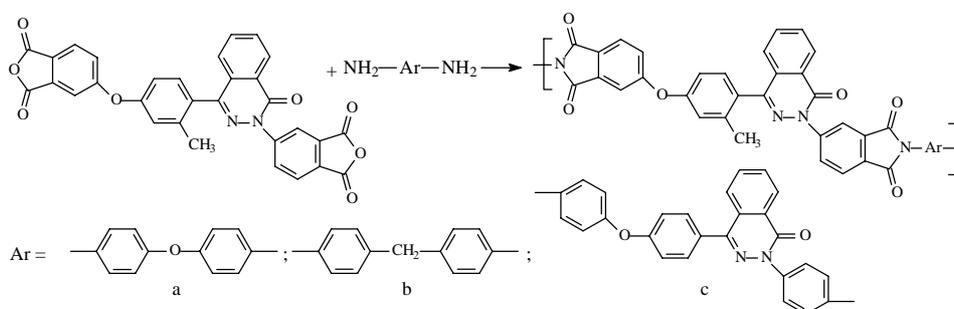
2-(3, 4-Dicarboxyl-*N*-phenyl)-4-(3', 4'-dicarboxyl-phenoxy)-4-(2-methyl)-phenyl)-2, 3-phthalazin-1-one dianhydride was prepared by the method of reference 3. One-step solution polymerization of the dianhydride monomer with **a** 4, 4'-diamino-diphenyl ether, **b** 4, 4'-diaminodiphenylmethane, or **c** 2-(4-amino-*N*-phenyl)-4-(4'-aminophenoxy)-4-phenyl)-2, 3-phthalazin-1-one was carried out in *m*-cresol in the presence of a catalytic amount of isoquinoline at 200°C (**Scheme 1**). High molecular weight poly(ether imide)s were readily formed over 18 h. Then the polymers were refined in *N*-methyl-2-pyrrolidone and dried in vacuum oven at 120°C for 48 h and afterwards 250°C for 4 h.

In FT-IR spectrum of poly(ether imide) from diamine monomer **b**, absorption at 1777 cm⁻¹ and 1721 cm⁻¹ is symmetric and asymmetric absorption of imido acyl group, and absorption signal at 1362 cm⁻¹ is due to stretching absorption of imido C-N bond. The absorption at 2969 cm⁻¹ and 1478 cm⁻¹ belongs to the asymmetric and symmetric

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stretching vibration of methyl group. There is no stretching absorption of N-H bond at 3440 cm^{-1} in FT-IR spectrum, which indicates that imide ring structure was fully formed after drying in the vacuum oven at 120°C and 250°C . The $^1\text{H-NMR}$ spectrum shows good conformity in hydrogen atoms to the polymer's structure. The sharp peaks at 2.25 ppm and 4.07 ppm are peculiar to the protons of methyl and methylene groups respectively, 8.59 ppm to the proton near *endo*-amide bond in phthalazinone, 7.18-8.42 ppm to the phenyl groups. The ratio of their intensity is 3:2:22, which also shows perfect agreement with the calculated amount of hydrogen atoms in the anticipated structure.

Scheme 1



The poly(ether imide)s from **a**, **b** and **c** have inherent viscosity of 1.39, 1.24, 0.97 dl/g respectively, and all show good solubility in 1, 1, 2, 2-tetrachloroethane, *m*-cresol and *N*-methyl-2-pyrrolidone. The glass transition temperatures are 289°C , 282°C , 309°C respectively, and degradation temperatures for 5% weight loss appear at 465°C , 483°C , 478°C respectively.

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References

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