

Synthesis and Characterization of Poly (ether imide)s Containing Phthalazinone and Isopropyl Moieties

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Abstract: A novel poly(ether imide)s containing phthalazinone and isopropyl moieties derived from 2-(4-aminophenyl)-4-[4-(4-aminophenoxy)phenyl]-phthalazin-1-one and bisphenol-A diphthalic anhydride was synthesized by one-step solution condensation polymerization in *m*-cresol. The polymer was characterized by FTIR, NMR, molecular weights, glass transition temperature, thermal degradation temperature and WAXD.

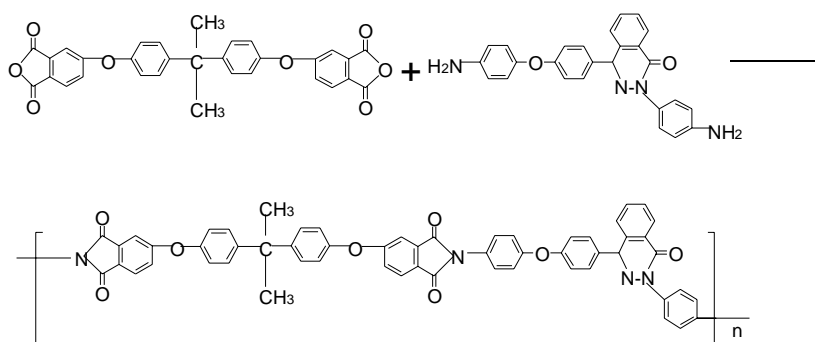
Keywords: Poly (ether imide) s, phthalazinone, one-step solution condensation polymerization.

Uses for aromatic polyimides have developed rapidly in recent years due to their outstanding properties^{1,2}. However, their applications have been limited in many fields because aromatic polyimides are normally insoluble and infusible. The poly (ether imide)s (PEI) prepared by the polymerization of bisphenol-A diphthalic anhydride (BPA dianhydride) and *m*-phenylenediamine (*m*-PDA), developed by General Electric Plastics and marketed under the trade name Ultem 1000, has excellent processability, and has enjoyed much commercial success³. Unfortunately, introducing flexible linkages led to lower its glass transition temperature ($T_g=217^\circ\text{C}$) than general polyimides'. Earlier work of ours demonstrated that the polyamides based on 2-(4-aminophenyl)-4-[4-(4-aminophenoxy) phenyl]-phthalazin-1-one (DHPZ-DA) have excellent properties⁴. The objective of this work is to synthesize a new poly (ether imide) by the polymerization of DHPZ-DA and BPA dianhydride in order to improve thermal property of PEI and maintain its excellent processability.

We were able to successfully polymerize DHPZ-DA with BPA dianhydride in *m*-cresol in the presence of a catalytic amount of isoquinoline at 200°C for 20 h (**Scheme 1**). The polymer had inherent viscosity of 1.05 dL/g in N-methyl-2-pyrrolidone (NMP) at 30°C .

The structure of the polymer was confirmed by spectroscopic means. The FTIR spectrum exhibited the characteristic absorption for imide ring at 1777 and 1723 cm^{-1} , indicative of the asymmetrical and symmetrical C=O stretching vibration, 1362, 1110 and 720 cm^{-1} due to ring deformation. The absorption at 2969 and 1478 cm^{-1} belonged to the asymmetrical and symmetrical methyl stretching vibration. The $^1\text{H-NMR}$ of the polymer also confirmed the formation of poly (ether imide). The signal at 5.00 and 5.26 ppm of the DHPZ-DA⁴ disappeared. The sharp peak at 1.75 ppm was peculiar to

Scheme 1



Reagents and conditions: *m*-cresol, isoquinilone, 200°C, 20 h

the protons of methyl group, 8.57 ppm to the proton of naphthalene which was near to C=O of endo-amide bond, 6.35-7.98 ppm to the phenyl groups. Their intensity ratio was 6:1:29, which show agreement with the calculated amount of the proposed structure. The T_g was 252°C, which was 35°C higher than the T_g of Ultem 1000. The onset temperature taken at 5% weight loss in nitrogen was 487°C. WAXD analysis showed that the polymer was amorphous. Furthermore, the polymer was readily soluble in chloroform, 1,1'2,2'-tetrachloroethane, N,N-dimethylformamide, N, N-dimethylacetamide, NMP and *m*-cresol.

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