

Synthesis and Characterization of a Novel Copoly(aryl ether ketone) Containing 4, 4'-Biphenyl-bis[4-phthalazin-1(2H)-one] Moiety

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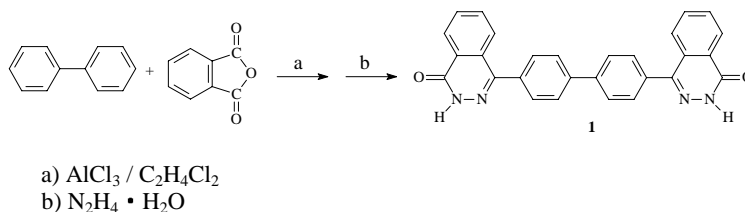
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Abstract: A new monomer of 4, 4'-biphenyl-bis[4-phthalazin-1(2H)-one] was synthesized from biphenyl and phthalic anhydride, and a novel copoly(aryl ether ketone) (PPEK) was synthesized from 2, 2-bis(4-hydroxyphenyl)-propane (BPA), 4, 4'-biphenyl-bis-[4-phthalazin-1(2H)-one], 4, 4'-difluorodiphenylketone (DFK). The monomer and copolymer were characterized by FT-IR and ¹H-NMR. DSC and TGA were used to the novel polymer.

Keywords: Poly(aryl ether ketone), 4,4'-biphenyl-bis[4-phthalazin-1(2H)-one], 2,2-bis(4-hydroxyphenyl)-propane(BPA).

Poly(aryl ether ketone)s are a category of high performance engineering thermoplastics characterized by high glass transition temperature and excellent thermooxidative stability. And they have important applications in electronic, electric, aircraft and aerospace industries¹⁻³. Considerable efforts have been made towards the improvement of solubility or processability of poly(aryl ether ketone)s^{4,5}. In our work, a novel bis(phthalazinone) monomer **1** 4, 4'-biphenyl-bis[4-phthalazin-1(2H)-one] and copoly(aryl ether ketone) containing bis(phthalazinone) moiety (PPEK) were successfully synthesized. Thermal property and solubility of the copolymer were studied. The monomer was synthesized by the reference method² as showed in **Scheme 1**.

Scheme 1

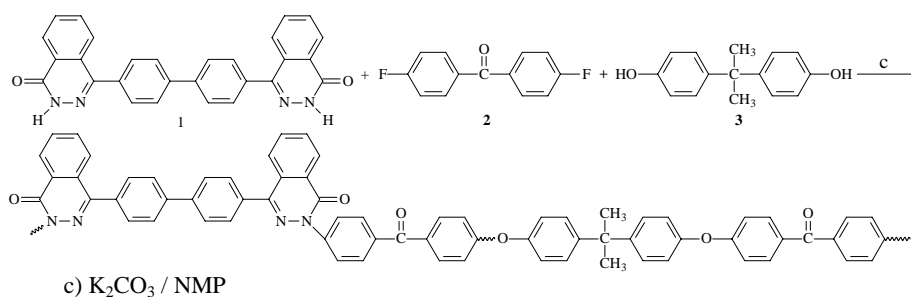


The PPEK containing bis(phthalazinone) was prepared by the reaction of monomer **1** 4, 4'-biphenyl-bis[4-phthalazin-1(2H)-one] and **3** 2, 2-bis(4-hydroxyphenyl)-propane (BPA) with 4, 4'-difluorodiphenylketone (DFK) in N-methyl-2-pyrrolidone (NMP) at

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190°C for 8h in the presence of potassium carbonate after refluxing in toluene for 2-3 h. When the molar feed ratio of **1**, **2** and **3** was 1:2:1, the obtained polymer had intrinsic viscosity 0.54 dL/g in NMP at 25°C. The copoly(aryl ether ketone) was characterized with FT-IR, DSC and TGA.

Scheme 2



The FT-IR⁶ spectrum showed the presence of the strong absorption of carbonyl group at 1654 cm^{-1} , and N-H stretching absorption peak at 3160 cm^{-1} of monomer **1** disappeared. The ¹H-NMR of the product showed the disappearance of peak at 12.74 ppm. DSC analysis showed a unique glass transition temperature at 215°C. The data above confirmed that the product was a copolymer but not a mixture of two homopolymers. The 5% weight loss temperature was above 400°C in nitrogen. Furthermore, the copolymer could not dissolve in DMAc, sulfolane and chlorobenzene, but it was well soluble in CHCl_3 and NMP.

Acknowledgment

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References and Note

1. S. Yoshida and A. S. Hay, *Macromolecules*, **1997**, *30*, 2254.
2. Y. Gao, X. G. Jian, Z. Y. Xu, *et al*, *Chin. Chem. Lett.*, **2001**, *4*, 325.
3. J. F. Wolfe, J. K. Still, *Macromolecules*, **1976**, *9*, 489.
4. Y. Gao, X. G. Jian, and J. Y. Wang, *Chin. Chem. Lett.*, **2000**, *9*, 777.
5. Y. Z. Liu, X. G. Jian, *Acta Polymerica Sinica*, **1999**, (1), 37.
6. Selected data of monomer **1**: FT-IR (KBr, cm^{-1}): 1654(C=O), 3160(N-H), 1559(Ar-H); ¹H-NMR (400MHz, DMSO- d_6 , δ ppm): 7.6-8.2(m, 14H), 8.32(m, 2H), 12.74(-s, 2H); Anal. Calcd for $\text{C}_{28}\text{H}_{18}\text{N}_4\text{O}_2$: C, 76.00; H, 4.1; N, 12.7; Found: C, 76.29; H, 4.50; N, 12.29.

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