

## Synthesis and Characterization of Reactive Poly(Aryl Ether Ketone Ketone) Containing Cyclohexene Ring

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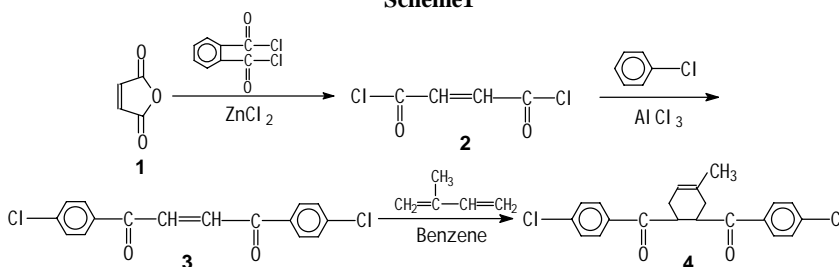
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**Abstract:** A novel heterocyclic poly (aryl ether ketone ketone) (PPEKK) containing cyclohexene ring was prepared from 1-methyl-cyclohex-1-ene-4,5-diyl-bis(4-chlorophenyl methanone) and 4-(4-hydroxyphenyl)(2H) phthalazin-1-one. The polymer was characterized by FTIR, <sup>1</sup>HNMR, DSC, TG and X-ray analysis.

**Keywords:** Engineering thermoplastics, poly(aryl ether ketone ketone), synthesis.

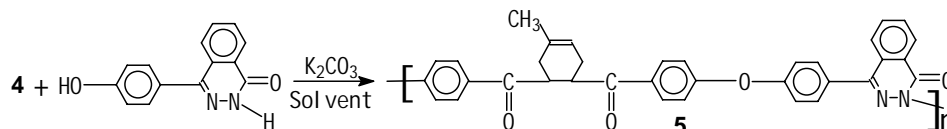
Poly(aryl ether ketone)s are one of the most important high performance engineering thermoplastics, widely used in electronic, electric, aircraft, and aerospace industries. Initially, considerable effort was made to modify their chemical structure, essentially to improve their thermal properties<sup>1</sup>. In the last few years, more attention was paid to their functionalization<sup>2</sup>. This can be achieved either by chemical modification of the polymer or by direct synthesis using functionalized monomers.

Scheme 1



In this letter, we synthesized a novel monomer **4** by introducing cyclohexene ring and poly (ether ketone ketone) based on this novel monomer (**Scheme 1**). Monomer **4**

Scheme 2



was readily synthesized from the maleic anhydride in high yield. The approach involved the chloroformylation with phthalic chloride, Friedel-Crafts reaction with chlorobenzene and Diels-Alder reaction with 2-methyl-1,3-butadiene. The high pure monomer was recrystallized from acetone and alcohol. Its structure was confirmed by FTIR, NMR and MS. The poly(ether ketone ketone) was synthesized from **4** and 4-(4-hydroxyphenyl)(2H) phthalazin-1-one (**Scheme 2**). The polymer was obtained in quantitative yield with inherent viscosity of 0.35 dL/g in CHCl<sub>3</sub> at 25 °C. Spectroscopy confirmed the formation of poly(ether ketone ketone) **5**. The FT-IR spectrum showed the presence of the strong absorption peak of carbonyl group at 1670 cm<sup>-1</sup>. The peak at 1245 cm<sup>-1</sup> was due to the C-O-C bond absorption. Comparing the spectrum of the polymer with that of the monomer, we found that the broad peak at 3200~3100 cm<sup>-1</sup> due to the NH group absorption of the monomer disappeared in the spectrum of the polymer. From <sup>1</sup>H-NMR of the polymer the strong signal of the proton attached to the nitrogen atom could no longer be found in the spectrum of the monomer at 12.74 ppm. The *T<sub>g</sub>* was 252 °C. The temperature for 5% weight loss was above 400 °C in air. X-ray analysis showed that the polymer was amorphous. Furthermore, the polymer was readily soluble in CHCl<sub>3</sub>, DMAc, DMF, DMSO and NMP.

### Acknowledgments

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### References and Notes

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2. F. Wang, J. Roovers, *Macromolecules*, 1993, 26, 5295.
3. Selected data of monomer **4**: m.p.: 178~179 °C. FTIR (KBr, cm<sup>-1</sup>) 1666 (C=O), 1594 (C=C), 1109 (C-Cl). <sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>, ppm): δ = 1.70 (s, 3H), 2.10-2.40 (m, 4H), 4.90-5.10 (m, 2H), 5.40-5.60 (m, 1H), 7.30-8.00 (m, 8H). <sup>13</sup>C-NMR (400 MHz, CDCl<sub>3</sub>, ppm): δ = 22.97 (CH<sub>3</sub>), 29.79, 34.24 (CH<sub>2</sub>), 43.88, 44.31 (CH), 119.62 (-CH=), 128.94, 129.91 (-C<sub>6</sub>H<sub>5</sub>), 132.84, 134.79, 139.50 (-C-), 202.07, 202.18 (C=O). MS (M<sup>+</sup>: 372).

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