# POLY (2,6-DIMETHYL-1,4-PHENYLENE OXIDE): SYNTHESIS, CHARACTERIZATION, MODIFICATION AND APPLICATIONS

by

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*DEDICATED TO MY PARENTS*

## CERTIFICATE

This is to certify that the thesis entitled "POLY (2,6-DIMETHYL-1,4-PHENYLENE OXIDE): SYNTHESIS, CHARACTERIZATION, MODIFICATION AND APPLICATIONS" submitted by Kunal Chander to the Indian Institute of Technology, Delhi for the award of the degree of Doctor of Philosophy is a record of bonafide research work carried out by him. Kunal Chander has worked under our guidance and supervision and has fulfilled the requirements for the submission of the thesis, which to our knowledge has reached the requisite standard.

The results contained in this thesis have not been submitted, in part or full, to any other University or Institute for the award of any degree or diploma.

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## ABSTRACT

The present thesis includes results of investigations of the chemical modification of poly(2,6-dimethyl-1,4-phenylene oxide)(PPO). The modified PPO's thus synthesized, have been evaluated as photostablizers for polyolefins viz. polyethylene and polypropylene.

PPO was synthesized in the laboratory by oxidative coupling of 2,6-dimethylphenol using Cu-pyridine complex as a catalyst. A number of samples of PPO were prepared by varying reaction parameters. PPO samples with high intrinsic vicosity $[\eta] > 1$ dL/g were also prepared but these samples developed yellow colour after drying.

In order to investigate the origin of yellow colour these samples were studied through atomic absorption, UV, ir and NMR spectroscopy.

The modifications of PPO were accomplished through a common intermediate; poly(2-bromomethyl-6-methyl-1,4-phenylene oxide). This bromo PPO (PPOBr) was reacted with various organic compounds to incorporate phosphorous, silicon and benzotriazole moieties in PPO. The resultant products were characterized through their intrinsic viscosities, elemental analysis and various spectroscopic techniques.

PPOBr was prepared by a free radical bromination of PPO

using N-bromosuccinimide (NBS) and benzoyl peroxide. Only one methyl group per repeating unit of PPO was brominated by using 1:1.1 mole ratio of PPO and NBS in the reaction mixture.

Phosphorylation of PPOBr was carried out through Arbuzov's reaction. This reaction is normally accompanied by a gel formation. During the present investigations, the reaction conditions were optimized to eliminate the gel formation.

Incorporation of benzotriazole group in PPO was accomplished by reacting PPOBr with 2-(2,6-dihydroxyphenyl)-2H-benzotriazole in the presence of sodium hydride.

Silylation of PPO was carried out by esterification of p-trimethylsilyl benzoic acid with PPOBr using triethylamine as the base. Silyl benzoic acid in turn was prepared by oxidation of trimethyl-p-tolyl silane with chromium trioxide.

The effect of molecular weight on the thermal behaviour of PPO was investigated by dynamic thermogravimetry. PPO samples were stable in air and nitrogen atmosphere upto 350 °C.

The effect of substitution of hydrogen of the methyl group by bromine, $-\overset{O}{\overset{\|}{P}}(OEt)_2$ and benzotriazole groups on thermal behaviour of PPO was evaluated in nitrogen

atmosphere. PPOBr and PPOP showed a two step decomposition behaviour. The char yield of PPOP and PPOB was higher compared to PPO,

In the DSC traces of PPOP and PPOSi, endothermic transitions corresponding to their melting were observed at 180 °C and 174 °C respectively.

Different weight percent of PPOP (0, 1 and 5) were melt blended with PE or PP and these were studied for their photostability, thermal aging and processing characteristics.

On irradiation blended sample of PE, the intensity of vinylic absorption increased with time but in sample PE/PPOP (5%) no significant change was observed. Mechanical properties measurement (tensile strength, % elongation, yield stress) indicated a better retention of properties in the irradiated PE samples containing PPOP.

Addition of 5% PPOP in PP had a significant effect on fractional retention of 1% secant modulus. Melt flow index studies of extruded material showed that PPOP significantly reduced the MFI of PP.

Effects of combination of commercially available Tinuvin or Irganox with PPOP were also studied on the properties of PP. These results showed that PPOP and Tinuvin have a synergistic effect on the fractional retention yield stress of PP.

Silicon containing bismaleimide was also synthesized and characterized in the laboratory. Thermal behaviour of bismaleimide and chain extended bismaleimide was evaluated. These studies are included in the appendix.

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