# STUDIES ON AROMATIC : ALIPHATIC AND WHOLLY AROMATIC POLYAMIDES

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SUBMITTED IN
FULFILMENT OF THE REQUIREMENT OF
THE DEGREE OF DOCTOR OF PHILOSOPHY

TO THE
INDIAN INSTITUTE OF TECHNOLOGY, DELHI
AUGUST, 1978

DEDICATED TO

MY

BELOVED PARENTS

## CERTIFICATE

This is to certify that the thesis entitled "Studies on aromatic;aliphatic and wholly aromatic polyamides" being submitted by Mrs. V.S. Sundari to the Indian Institute of Technology,Delhi, for the award of the degree of Doctor of Philosophy in Textile Technology, is a record of bonafide research work carried out by her. She has worked under our guidance and supervision and has fulfilled the requirement for the submission of this 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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## ACKNOWLEDGEMENTS

I wish to express with my utmost sincerity the gratitude for the painstaking guidance, constant encouragement offered by my research supervisors Professor D.S. Varma, Professor of Fibre Science, Department of Textile Technology and Professor (Mrs.) I.K. Varma, Professor of Polymer Science and Co-ordinator, School of Materials Science & Technology, Department of Chemistry, Indian Institute of Technology, Delhi. They have been a source of inspiration and moral support in the hour of need.

I am thankful to the teaching and technical staff of the Textile Technology Department for their kind co-operation.

I wish to extend my thanks with pleasure to all my colleagues and friends for their help.

Council of Scientific and Industrial Research, New Delhi, India is also gratefully acknowledged for providing the financial assistance to carry out this work.

Finally thanks are due to neat and accurate typing by Mr. Rajesh Arora and the tracing of figures by Mr. K.G. Padam.

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## ABSTRACT

The synthesis of poly(m-/ p-phenylene sebacamides) by interfacial polycondensation and poly(arylene terephthalamides) by low temperature solution condensation method is reported in the present thesis. Poly(p-phenylene terephthalamide) (PPTA), poly(m-phenylene terephthalamide)(MPTA) and terephthalamide polymers (DATA) of 4,4'-diaminodiphenylmethane were prepared by reacting terephthaloyl chloride and the corresponding diamine in dimethyl acetamide/LiCl system at low temperature. Several PPTA samples with different intrinsic viscosities were prepared by changing the concentration of LiCl, solvent to monomer ratios, and addition of triethylamine. Aromatic polyamides containing both p-phenylene (P) and m-phenylene group (M) in the backbone were synthesized by taking different molar ratios of m- and p-phenylene diamine in the initial monomer feed. These have been named as $M_1P_1$, $M_1P_2$ according to the molar ratios of the diamine taken.

The polyamides/copolyamides were characterised by nitrogen estimation, density determination and powder X-ray diffraction. The intrinsic viscosity of the various polyamides was determined in conc. $H_2SO_4$ at 30°C. The temperature dependence of viscosity of MP copolyamides in conc. $H_2SO_4$ from 30-45°C was determined.

The effect of backbone structure and intrinsic viscosity of the polyamides, the thermal and thermo-oxidative stability was studied by differential thermal analysis in nitrogen and thermogravimetric analysis in air and nitrogen atmosphere. The stability order based on IPDT data in air was found to be DATA > PPTA > MPTA and in nitrogen atmosphere PPTA > MPTA > DATA. The higher stability of DATA in air has been explained by the oxidation of $-CH_2-$ group to -CO- thus stabilising the polymer. Introduction of m-phenylene groups reduced the stability. PPTA samples of higher $\eta_{inh}$ values had good thermal stability with a decomposition temperature above 600°C.

The wet spinning of PPTA, DATA, $M_1P_2$ and $D_1P_3$ were also carried out. The mechanical properties of the as-spun fibres from $D_3$ were inferior to PPTA. The heat-ageing for 16 hrs at 100° and 200°C improved the tenacity and initial modulus of PPTA fibres whereas in hot drawn $D_3$ fibres the mechanical properties showed a decline. X-ray diffraction of PPTA fibres indicated crystalline structure. The hot drawn $D_3$ fibres were essentially amorphous with slight orientation. Studies of the solution viscosity and fractured surface of PPTA fibres indicate a fully extended rod-like structure of polymer molecules.

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