

Reactive Processing and Characterization of PBT/Epoxy Blends

By
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*THESIS SUBMITTED IN FULFILMENT OF THE
REQUIREMENTS FOR THE DEGREE OF*

DOCTOR OF PHILOSOPHY



**CENTRE FOR POLYMER SCIENCE AND ENGINEERING
INDIAN INSTITUTE OF TECHNOLOGY, DELHI
JULY, 2002**

epoxy resin

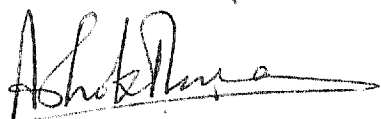
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CERTIFICATE

This is to certify that the thesis entitle 'Reactive Processing and Characterization of PBT/Epoxy Blends' being submitted by Ms. Bhawna Kulshreshtha to the Indian Institute of Technology, Delhi for the award of degree of Doctor of Philosophy, is a record of bonafide research work carried by her. Ms. Bhawna Kulshreshtha has worked under our guidance and supervision and has fulfilled the requirements 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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Dedicated to

My Parents

&

Parents-in-Law

ACKNOWLEDGEMENTS

If it is true that the wealth of a man can best be measured by his generosity, then my supervisors Prof. Ashok Misra and Dr. Anup K. Ghosh own a vast fortune of creativity. Their unstinting donation of advice, ideas, and time has been the foundation of this research. I also wish to place on record their patience and understanding, their encouraging attitude, constant motivation and immense co-operation to accomplish this challenging task.

I am grateful to Prof. S. N. Maiti, Prof. (Mrs.) I. K. Varma, Prof. B. L. Deopura, Prof. A. K. Gupta, Dr. Harpal Singh and Prof. Alok Ray for their encouraging and helpful spirit.

I would like to acknowledge Prof. H.E.H. Meijer, Prof. P.J. Lemstra, Dr. S. Rastogi and Dr. J.G.P. Goossens, Prof. A.L. (Ton) German, TU/e, Eindhoven, The Netherlands for providing me the experimental facilities and valuable suggestions during this research work. Special thanks to Sanjeev, Ankur, Sreepad, Martijn, Mathijs, Ellen, Ronald and Marteen for their kind help during my stay at TU/e.

I am highly obliged to Dr. C. Ramesh, N.C.L. Pune for extending his cooperation by providing the experimental facilities for the X-ray analysis of the blends. I would like to express my sincere respect, recognition and appreciation to Dr. U. K. Saroop, Reliance Industries Ltd. for his special concern, constant moral support and encouragement during the entire period of my research work and in the development of this thesis.

I would like to thank Dr. Arup Ranjan Bhattacharyya, Dr. (Mrs.) Purnima Jain, Mr. Shounak Dey Roy, Mr. Vishal Anand, Mr. Anubhav Saxena, Mrs. Roopali Rai, Ms. Shivani Agarwal and Mr. Sandeep Gupta for their constant help and support throughout the entire period of my work. I am also thankful to Mr. Joy Kishore Pal, Mr. Bandeep Singh, Mr. G.S. Anantha Padmanabha, Mr. Milind Mhalgi, Dr. Bharti Gaur for their help and support.

Beyond words some people make a world of difference by their mere presence, my father for his principles, my mother for her patience, sister and brother-in-law (Mrs. Surekha Kulshreshtha and Mr. Sampann Kulshreshtha), my niece- Prachi and Sanskriti for their help and constant moral support. I offer a deep sense of gratitude to my grandmother, Uncles, Aunts, cousins and all my relatives for their love and encouragement.

Over these last four years, I have been the happy and grateful recipient of much love, advice, and support. Here I would like to mention those who have been most directly involved with my efforts to complete this research. Thanks to my Mother-in-law, Father-in-law for everything. It's a joy to be their daughter-in-law. Thanks to my sister-in-law, Rashmi for her kind help, support and patience. Words would not be sufficient to mention her help and assistance at every stage of this task.

I can not forget to mention the name of my little daughter, 'Tisha' who had been a constant source of enthusiasm and ecstasy by her sweet smile.

Lastly I am left with lack of words to acknowledge, my strength, motivation and stable force, who has an deep influence on my ability to see and understand auras, my path of self-awareness, for all the pains he bore to make me smile, helped me to take the pressure for the completion of this thesis, always stood by me with a smile during the storm of momentary depression, my better half, 'Sandeep'.

Finally, thanks to God for the joy and opportunity to worship Him through learning more about this small part of His creation.


(Bhawna Kulshreshtha)

Abstract

Poly(butylene terephthalate) (PBT) has developed considerable importance as an engineering thermoplastic due to its good dimensional stability, strength, stiffness and toughness. However, at high processing temperature, thermal degradation of PBT occurs through the formation of some cyclic oligomers. Therefore, lowering of its processing temperature and enhancing its processing characteristics are required for a wide range of applications where easy mouldability is desired. In view of these, epoxy resin has been used as a reactive solvent for PBT in the present work. After the processing, in-situ curing of the epoxy is carried out so that the solvent becomes a part of the system and does not require to be removed. The blends of different compositions of PBT and epoxy (epoxy being 10-50 wt.%) were prepared in a Brabender Kneader at 220 °C and cured at 175 °C for 4 hours using a bi-functional amine curing agent.

In the present work, the melting behaviour, crystallization behaviour, mechanical properties and morphology of PBT in the presence of epoxy has been investigated. Differential scanning calorimetry (DSC) studies showed that melting temperature, equilibrium melting temperature and crystallization temperature decreased in the PBT/epoxy uncured blends while they remained constant and the values are close to that of PBT. Multiple melting behaviour shown by PBT, isothermally crystallized at different temperatures, is affected in the presence of epoxy monomer and cured epoxy resin. The nature and shape of melting endotherms show that the presence of the epoxy resin leads to the formation of less stable type of crystals. The half time of crystallization and induction time

decreased in uncured blends while these remained constant in the cured blends with increase in the amount of epoxy resin. The value of Avrami exponent varies between 1 to 2 in pure PBT as well as PBT/epoxy uncured and cured blends, indicating mixed type of spherulitic growth. The interaction parameter between PBT and epoxy monomer was found to be negative, indicating that PBT forms a thermodynamically stable system with epoxy. Scanning electron microscopy shows a morphology of PBT domains in the epoxy solvent in the uncured blends while in the cured blends, a morphology of dispersed cured epoxy resin in the PBT matrix is observed. A small increase in the tensile, flexural strength as well as modulus and a small decrease in the impact strength were observed in the PBT/epoxy cured blends with increase in the amount of epoxy resin. WAXD showed no change in the position of diffraction maxima and d-spacing of pure PBT in the presence of epoxy monomer as well as cured epoxy resin, and showed an increase in crystallinity in uncured blends and decrease in crystallinity in the cured blends with an increase in the level of epoxy. SAXS studies show an increase in the long period with increase in the amount of epoxy resin in the uncured blends, indicating that epoxy is present in the interlamellar region of the PBT phase.

Change in the spherulitic morphology of the PBT in the presence of epoxy monomer and cured epoxy resin for different blend compositions at various crystallization temperatures was studied by polarizing microscopy (PLM) and small angle light scattering (SALS). These show that typical unusual type of spherulites in pure PBT change into the usual type of spherulites in the presence of epoxy

monomer as well as cured epoxy resin when isothermally crystallized at 50⁰C and 215⁰C. For uncured blends, crystallized at both these temperatures, spherulitic radius of PBT increased upto 30% epoxy level and then decreased. In the cured blends, an increase in the radius of the spherulites is observed up to the 20% epoxy after which there is no change, while in blends with higher epoxy levels, the H_v patterns were too diffused for any analysis. The SALS observations correlate well with PLM observations. Pure PBT, PBT/epoxy 80/20 uncured and cured blends isothermally crystallized at different temperatures varying between 50⁰C to 215⁰C showed an increase in the spherulitic radius with increase in the crystallization temperature levelling off at about 175⁰C.

In conclusion, this study gives an insight about the use of epoxy as reactive solvent to enhance the processing characteristics of PBT. Furthermore, it provides an overall picture about the changes in crystallization behaviour, melting behaviour, morphology and mechanical properties of PBT in the presence of epoxy monomer as well as cured epoxy resin. Overall, it may be observed that epoxy monomer acts as a diluent and affects the crystallization behaviour to a great extent as compared to that of the cured epoxy resin. Spherulitic morphology is affected by both epoxy monomer as well as cured epoxy resin, while the effect on mechanical properties is marginal. A small change in the mechanical properties is observed in PBT/epoxy cured blends and the properties are mainly dominated by the PBT phase. Thus this route can be successfully used for enhancing the processing characteristics of PBT while retaining majority of properties of PBT.

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