

**DEVELOPMENT OF MODIFIED BAMBOO FIBER
REINFORCED PLASTIC NANOCOMPOSITES AND
STUDY OF THEIR DIELECTRIC, MECHANICAL
AND THERMAL PROPERTIES**

by

VIVEK KUMAR

INSTRUMENT DESIGN DEVELOPMENT CENTRE

Submitted

in fulfillment of the requirements for the degree of

DOCTOR OF PHILOSOPHY

to the



INDIAN INSTITUTE OF TECHNOLOGY DELHI

OCTOBER 2012

I. I. T. DELHI.
LIBRARY
Acc. No. T.H. 4335.....

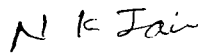
DEDICATED TO MY PARENTS

I am so happy and lucky to be your son. Thank you for your endless love, moral, spiritual and financial support throughout my life. I really do not know what my life would be without all sacrifices you made for the life and education of your child. My sincere gratitude and love forever.

CERTIFICATE

This is to certify that the thesis entitled “**DEVELOPMENT OF MODIFIED BAMBOO FIBER REINFORCED PLASTIC NANOCOMPOSITES AND STUDY OF THEIR DIELECTRIC, MECHANICAL AND THERMAL PROPERTIES**” being submitted by **Vivek Kumar** to the Indian Institute of Technology, Delhi for the award of the degree of “**DOCTOR OF PHILOSOPHY**”, is a record of the authentic research work carried out by him. Mr. Vivek Kumar has worked under our supervision and guidance. He has fulfilled all the requirements for submission of the thesis, which to the best of our knowledge has reached the required standard.

The results contained in this thesis are original and have not been submitted, in part or full, to any other university or institute for the award of any degree or diploma.



Dr. N.K. Jain

Instrument design development centre
Indian institute of technology Delhi
New Delhi -110016



Dr. Rakesh Kumar

Instrument design development centre
Indian institute of technology Delhi
New Delhi -110016

ACKNOWLEDGEMENTS

First and foremost, I would like to express my deepest gratitude to my supervisor, Dr. Rakesh Kumar for his painstaking guidance, motivation, invaluable suggestions and never-ending patience. His communication skills, leadership qualities, enthusiasm and integral view on research have left a deep impression on my mind. I wish to sincerely express my grateful appreciation to another supervisor Dr. N.K. Jain for his continued support, invaluable suggestions and constant encouragement throughout my study, especially at my most difficult times. His patience and personality will not be forgotten.

I would like to thank SRC chairman and all SRC members for evaluating my performance from time to time and suggesting improvements. The suggestions received from Head and faculty members of IDDC are gratefully acknowledged. I express my deepest sense of reverence and heartfelt gratitude to Prof R. K. Shevgaonkar, Director IIT Delhi for providing academic ambience that has inspired me a lot. I am thankful to Dr. A. M. Biradar, Scientist G, National Physical Laboratory, for helping me in this research work.

I am also thankful to all the technical staff members of Mechanical Fabrication facility, SEM Lab, TEM Lab, XRD Lab, Materials testing Lab and all those who helped me in the preparation cutting, and testing of samples during this program. I would like to thank in particular Mr. Satish Bansal and Mr. Surinder kumar for extending their valuable help at all times.

In addition to these people, I was lucky enough to have the support of my friends Rajendra Malik, Udit, J.P. Rana, Ajay, Anu malik, H.S. Jaggi and Abhishek Gandhi. I do not know how adequately to thank my parents Sri Ashok Kumar and Smt. Pushpa Devi who formed part of my vision. I am grateful to my younger brothers, Mr. Pradeep Kumar and

Mahendra Singh for rendering me the sense and value of brotherhood and for their helpful attitude. Last but not the least, my heartfelt appreciation to Garima Pal, my wife, and my son Shubh, not necessarily for coming along the right time but for bearing my long absence due to my busy academic life.

Finally, I thank all those who have helped me directly or indirectly in the successful completion of this thesis. The chain of gratitude would be definitely incomplete if I forget to thank the first cause of this chain, The Prime Mover, to whom I offer my deepest and sincere gratitude for inspiring and guiding this humble being.

Vivek Kumar

(Vivek Kumar)

ABSTRACT

Environmental awareness and government regulations are leading to preserve the world's existing forests and human health because of pollution. These facts are major driving forces for the growing need of biofiber polymer composites in place of synthetic fiber composites and wood composites due to the positive environmental benefits such as biodegradability, renewable character, availability in a variety of forms throughout the world, raw material utilization and free from health hazard.

Modified bamboo fiber mat and polymer (epoxy, polylactic acid) resin incorporated with cloisite 30B nanoclay are used to prepare the hybrid composite laminates employed for dielectric and structural applications. A critical overview of the simultaneous effect of nanoclay filler and chemical treatment on the various aspects of bamboo fiber reinforced plastic (BFRP) composite is rationalized in this research work. An analysis of diverse relations among dielectric properties (dielectric constant, loss factor etc.), mechanical properties (tensile, flexural and impact), thermal properties and moisture absorption properties, as well as methods of preparation and product qualities of BFRP nanocomposites is demonstrated. The main thrust of current research work is to obtain relatively low dielectric constant BFRP nanodielectrics with improved mechanical, thermal and water resistance properties. Bamboo-epoxy composites of two fiber orientation parallel (0°) and perpendicular (90°) to electric field were prepared. Dielectric constant, conductivity and dissipation factor of 0° oriented bamboo-epoxy composites were higher than that of 90° oriented bamboo-epoxy composites. Dielectric constant and dissipation factor of bamboo fiber reinforced plastic composites decreased with increase in frequency at all the temperatures under study.

Bamboo–epoxy composites with varying content of nanoclay ranging from 0 to 10 wt% were also developed. Ultrasound sonication and twin screw extruder was used for dispersion of nanoclay in epoxy resin and polylactic acid resin respectively. Dispersion of nanoclay in BFRP composite was confirmed by SEM (scanning electron microscopy), TEM (Transmission electron microscope) and XRD (X-ray diffraction). The results show that there exists an optimum limit in which the mechanical properties, dielectric properties, thermal properties and water resistance properties of composites improved by continuously increasing the nanoclay content. The highest value of impact strength was obtained for 1 wt% nanoclay content bamboo-epoxy nanocomposites.

The tensile and flexural strength of bamboo-epoxy nanocomposites with 3wt% nanoclay increased by 40% and 27%, respectively as compared to pure composites. Also the least value of dielectric constant and water absorption was observed for 3 wt% nanoclay content bamboo-epoxy composites. Further reduction in dielectric constant and water absorption was observed for silane and alkali treated bamboo-epoxy nanocomposites. The minimum value of dielectric constant was observed for 3 wt% nanoclay content mercerized aminopropyltriethoxy silane treated bamboo-epoxy nanocomposites. The water absorption of 3 wt% nanoclay content mercerized aminopropyltriethoxy silane treated and alkali treated bamboo-epoxy nanocomposites decreased from 46.44% (for pure BFRE composite) to 22.35% and 24.55% respectively. Tensile strength and flexural strength of 3 wt% nanoclay content alkali treated bamboo-epoxy nanocomposites increased by 60% and 42% respectively as compared to pure composite. Thermal degradation temperature of alkali treated bamboo-epoxy nanocomposites was increased by 21.1°C. SEM was used to study the interfacial adhesion of polymer layer on the surface of alkali and silane treated bamboo fiber composites. FTIR (Fourier

transform Infrared spectroscopy) was used to analyze the change in structure of bamboo fiber due to alkali and silane treatment. Significant improvement in impact strength of bamboo-poly(lactic acid) bionanocomposites was observed as compared to virgin composites.

Alkali and silane treatment of bamboo fiber improved interfacial adhesion between fiber and matrix. Alkali treatment allows better fiber wetting by fibrillation (breakdown of the fiber bundle into smaller fibers) which increase the effective surface area available for contact with the wet matrix. Further, nanoclay particles improve the mechanical interlocking inside the bamboo-epoxy nanocomposite by taking the space into the rough surface of alkali treated bamboo fiber.

TABLE OF CONTENTS

| | |
|--|-------------|
| <i>CERTIFICATE</i> | <i>i</i> |
| <i>ACKNOWLEDGEMENTS</i> | <i>ii</i> |
| <i>ABSTRACT</i> | <i>iv</i> |
| <i>CONTENTS</i> | <i>vii</i> |
| <i>LIST OF FIGURES</i> | <i>xi</i> |
| <i>LIST OF TABLES</i> | <i>xvi</i> |
| <i>SYMBOLS AND ABBREVIATIONS</i> | <i>xvii</i> |

CHAPTER 1: GENERAL INTRODUCTION

| | |
|--|----|
| 1.1 Introduction..... | 1 |
| 1.2 Current Status and Future Prospects of NFRP nanocomposites | 2 |
| 1.3 Natural Fibers..... | 3 |
| 1.4 Bamboo Fiber - A Brief Analysis | 5 |
| 1.4.1 Characteristics of Bamboo Fiber | 6 |
| 1.4.2 Chemical Composition of Bamboo Fiber | 6 |
| 1.4.3 Structure of Bamboo Fiber | 7 |
| 1.4.4 Applications of Bamboo Fiber | 9 |
| 1.5 Drawbacks of Natural Fiber Composites | 11 |
| 1.6 Chemical Modification of Natural Fibers..... | 12 |
| 1.7 Nanoclay Fillers | 13 |
| 1.8 Polymer Matrix | 15 |
| 1.8.1 Epoxy Resin | 16 |
| 1.8.2 Poly Lactic Acid (PLA) Resin | 18 |
| 1.9 Nanocomposites | 19 |
| 1.9.1 Advantages of Nanocomposites | 19 |

| | |
|--------------------------------------|----|
| 1.9.2 Areas of Applications | 20 |
| 1.10 Literature Review | 22 |
| 1.11 Objective of Present Work | 29 |
| 1.12 Plan of Thesis | 30 |

CHAPTER 2: EXPERIMENTAL

| | |
|---|----|
| 2.1 Raw Materials Used in this Research Work | 34 |
| 2.1.1 Bamboo Fiber Mat | 34 |
| 2.1.2 Polymer Resins | 36 |
| 2.1.3 Nanoclay Filler and Chemicals | 36 |
| 2.2 The Process Used to Mix the Polymers and Nanoclay | 37 |
| 2.3 The Techniques Used in Making Composite Laminates | 37 |
| 2.4 Fabrication Process of Composites | 39 |
| 2.4.1 Bamboo-Fiber Reinforced Epoxy Composites | 39 |
| 2.4.2 Bamboo-PLA Composites | 40 |
| 2.5 Fiber Volume Fraction | 41 |
| 2.6 Characterization Techniques | 41 |
| 2.6.1 Electric Testing | 41 |
| 2.6.2 Mechanical Testing | 42 |
| 2.6.3 Water Absorption Test | 43 |
| 2.6.4 Thermal Properties | 44 |
| 2.6.5 Morphology Characterization | 44 |

CHAPTER 3: EFFECT OF FIBER ORIENTATION ON DIELECTRIC BEHAVIOR OF BAMBOO-EPOXY COMPOSITES

| | |
|--|----|
| 3.1 Introduction..... | 47 |
| 3.2 Results and Discussion | 49 |
| 3.2.1 Dielectric Constant and Dissipation Factor | 49 |
| 3.2.2 Volume Resistivity..... | 54 |
| 3.2.3 Conductivity..... | 56 |
| 3.2.4 Electric Modulus | 59 |
| 3.3 Conclusions | 62 |

**CHAPTER 4: EFFECT OF NANOCLAY ON DIELECTRIC,
MECHANICAL AND THERMAL PROPERTIES OF
BFRE COMPOSITES**

4.1 Introduction64
4.2 Results and Discussion65
 4.2.1 Mechanical Properties65
 4.2.1.1 Tensile and Flexural Properties65
 4.2.1.2 Impact Strength68
 4.2.2 Dielectric Properties70
 4.2.3 Water Absorption Study72
 4.2.4 Thermal Degradation of Bamboo–Epoxy Composites76
 4.2.5 Structure and Morphology of Bamboo–Epoxy Nanocomposites80
4.3 Conclusions87

CHAPTER 5: CHEMICALLY MODIFIED BFRE NANOCOMPOSITES

5.1 Introduction88
5.2 Results and Discussion99
 5.2.1 Dielectric Properties99
 5.2.1.1 Dielectric Constant99
 5.2.1.2 Dissipation Factor98
 5.2.1.3 Volume Resistivity100
 5.2.2 Mechanical Properties101
 5.2.2.1 Tensile and Flexural Properties101
 5.2.2.2 Impact Strength105
 5.2.3 Water Absorption Study106
 5.2.4 Thermal Degradation of Bamboo-Epoxy Composites110
 5.2.5 Structure and Morphology of Bamboo-Epoxy Nanocomposites114
5.3 Conclusions118

CHAPTER 6: BAMBOO-PLA BIONANOCOMPOSITES

| | |
|---|-----|
| 6.1 Introduction | 120 |
| 6.2 Results and Discussions | 123 |
| 6.2.1 Structure and Morphology of Bamboo-PLA Nanocomposites | 123 |
| 6.2.2 Dielectric Properties | 127 |
| 6.2.3 Mechanical Properties | 130 |
| 6.2.4 Thermal Degradation of Bamboo Composites | 131 |
| 6.3 Conclusions | 135 |

CHAPTER 7: CONCLUSIONS

136

FUTURE SCOPE OF WORK

139

REFERENCES

140

LIST OF PUBLICATIONS

159