PLASTINATION FEASIBILITY STUDY OF NATURAL FIBRES: TOWARDS A NEW BAMBOO-Glass FIBRE HYBRID COMPOSITE

by

Daanvir Karan Dhir


A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF
THE REQUIREMENTS FOR THE DEGREE OF

MASTER OF APPLIED SCIENCE

in

THE COLLEGE OF GRADUATE STUDIES

(Mechanical Engineering)

THE UNIVERSITY OF BRITISH COLUMBIA

(Okanagan)

September 2019

© Daanvir Dhir, 2019
The following individuals certify that they have read, and recommend to the Faculty of Graduate and Postdoctoral Studies for acceptance, the dissertation entitled:

Plastination feasibility study of natural fibres: Towards a new bamboo-glass fibre hybrid composite

submitted by Daanvir Karan Dhir in partial fulfillment of the requirements of

the degree of Master of Applied Science

Dr. Abbas S. Milani, School of Engineering, UBCO

Supervisor

Dr. Kasun Hewage, School of Engineering, UBCO

Supervisory Committee Member

Dr. Kevin Golovin, School of Engineering, UBCO

Supervisory Committee Member

Dr. Solomon Tesfamariam, School of Engineering, UBCO

University Examiner
Abstract

Natural fibers are gaining wide attention by manufacturers due to a great emphasis by government regulators on the use of sustainable materials, especially the natural fiber reinforced polymer composites (NFPCs), which can replace most metals and synthetic fiber composites. However, one of the biggest challenges in working with natural fibers is known to be their moisture absorption ability, leading to fast degradation rates.

This research presents an experimental, feasibility study towards enhancing the performance of lignocellulosic fibers (here bamboo) and their structural application as a filler material in a typical glass fiber composite. The first part of the study involves the adaptation of a unique preservation technique called plastination, which is originally used for the preservation of human and animal body tissues. Plastination was applied to the bamboo natural fibers for the very first time, with the hope to hinder their bio-degradability. It was observed that plastination tends to decrease the residual moisture in bamboo and also enhances its mechanical properties. Secondly, an experimental study on a hybrid composite (glass fiber composite with plastinated bamboo as filler) revealed that plastinated bamboo could in fact alter the failure response of the glass fibre composite, without significantly affecting its elastic properties, while increasing its impact energy-absorbing ability and making the material less dense. Next, a material durability testing, involving the exposure of samples to moisture at elevated temperatures, proved that the plastination decreases the composite degradation rate. Moreover, it was observed that the flexural stiffness, which lowers for the regular glass fiber composite after moisture conditioning, slightly increased in the case of hybrid composite. Various parametric and non-parametric hypotheses tests were
conducted throughout the work to statistically analyze the significance of process variables involved.
Lay Summary

Natural fibres (cotton, wool, bamboo, etc.) are being increasingly preferred by many industries today, for structural and non-structural applications over human-made glass/carbon fibres due to their much lower carbon footprint and economic factors. However, a significant problem with natural fibres is their degradability due to moisture absorption. Plastination is a technique initially developed to replace water from dead animal and human tissues with a curable polymer to hinder their bio-degradation. This research presents an investigation towards adapting the same Plastination technique on bamboo natural fibres, with the hope to enhance their durability. The plastinated bamboo has been further characterized individually and as a filler material in a synthetic fibreglass composite (hybrid), under various physical and mechanical properties testing techniques. The effect of plastination on durability of these materials is also evaluated as part of the study.
Preface

Parts of this thesis has been published as a poster presentation as follows:


The figures shown in this thesis without any reference have been taken and/or prepared by the author, without referring to any sources. For the figures containing a reference, an acknowledgement/reference to the corresponding source has been provided, as suggested by the publisher(s).
# Table of Contents

Abstract .................................................................................................................................................. iii
Lay Summary .......................................................................................................................................... v
Preface .................................................................................................................................................... vi
Table of Contents ................................................................................................................................... vii
List of Tables .......................................................................................................................................... xi
List of Figures ......................................................................................................................................... xii
List of Abbreviations ............................................................................................................................ xxi
List of Symbols ......................................................................................................................................... xxii
Acknowledgements ............................................................................................................................... xxiv
Dedication ................................................................................................................................................ xxv

## Chapter 1: Introduction ......................................................................................................................... 1

1.1 Background overview ...................................................................................................................... 1
1.2 Motivation and objectives ................................................................................................................. 2
1.3 Thesis framework ............................................................................................................................. 3

## Chapter 2: Literature Review .................................................................................................................. 5

2.1 Natural fibres ..................................................................................................................................... 5
2.1.1 Processing of natural fibres ........................................................................................................... 8
2.1.2 Bamboo natural fibres .................................................................................................................. 9
2.1.3 Characteristics of bamboo ......................................................................................................... 9
2.2 Fiber-reinforced composites .......................................................................................................... 12
2.2.1 Natural fibre polymer composites ............................................................................................. 13
2.3 Plastination ....................................................................................................................................... 15
Chapter 3: Materials and Methods ................................................................. 26

3.1  Plastination and testing of bamboo ......................................................... 26

3.1.1  Overview ......................................................................................... 26

3.1.2  Plastination of bamboo ..................................................................... 26

3.1.2.1  Material ..................................................................................... 26

3.1.2.2  Plastination procedure ................................................................. 27

3.1.3  Characterization of plastinated bamboo ........................................... 31

3.1.3.1  Density measurement ................................................................. 31

3.1.3.2  Tensile test setup ...................................................................... 31

3.1.3.3  Environmental conditioning test .................................................. 34

3.2  Design and testing of hybrid composite ............................................. 35

3.2.1  Overview ......................................................................................... 35

3.2.2  Laminate design ............................................................................. 35

3.2.3  Characterization of composite samples ......................................... 38
3.2.3.1 Tensile test ................................................................. 39
3.2.3.2 Three-point bending test ............................................. 41
3.2.3.3 Fracture test ............................................................... 43
3.2.3.4 Impact test ................................................................. 46

3.3 Environmental conditioning .................................................. 47
3.3.1 Conditioning of bamboo .................................................. 47
3.3.1.1 Overview and setup ................................................... 47
3.3.1.2 Characterization of conditioned bamboo .......................... 48
3.3.2 Conditioning of composite ................................................ 49
3.3.2.1 Overview and setup ................................................... 49
3.3.2.2 Characterization of conditioned composites ....................... 49

3.4 Statistical hypothesis testing ............................................... 50

Chapter 4: Results and Discussions ........................................... 52

4.1 Characterization of bamboo ................................................. 52
4.1.1 Density ............................................................................. 52
4.1.2 Tensile tests ................................................................. 56
4.1.3 Moisture tests ................................................................. 61

4.2 Hybrid composite ................................................................. 64
4.2.1 Physical properties ......................................................... 64
4.2.2 Tensile test results ........................................................ 65
4.2.3 Flexural test results ......................................................... 68
4.2.4 Fracture tests ................................................................. 72
4.2.5 Impact tests ................................................................. 79
Chapter 4: Environmental conditioning

4.3 Environmental conditioning................................................................. 89

4.3.1 Characterization of conditioned bamboo ............................................ 89

4.3.1.1 Density results................................................................................. 89

4.3.1.2 Moisture test results ................................................................. 90

4.3.1.3 Tensile tests................................................................................ 91

4.3.2 Characterization of conditioned Composites ....................................... 97

4.3.2.1 Density .......................................................................................... 97

4.3.2.2 Tensile tests................................................................................ 99

4.3.2.3 Flexural tests .............................................................................. 106

Chapter 5: Conclusion and Future Work Recommendations.......................... 109

5.1 Summary.............................................................................................. 109

5.2 Main contributions to knowledge ......................................................... 112

5.3 Limitations to work............................................................................. 112

5.4 Future work ....................................................................................... 113

Bibliography .............................................................................................. 115

Appendices................................................................................................. 123

Appendix A.............................................................................................. 123

Appendix B .............................................................................................. 124
List of Tables

Table 2.1. Advantages and disadvantages of natural fibres over synthetic fibres [3, 98] .......... 7
Table 3.1. Specifications for the tested TWINTEX TPP60N22P-060 [115] ......................... 36
Table 3.2. Sample dimensions for three-point bending ...................................................... 41
Table 3.3. DCB sample dimensions for mode I fracture test ............................................. 44
Table 4.1. Chemical composition of plastinated bamboo derived using EDS ...................... 53
Table 4.2. Tensile properties of bamboo fibre bundles ......................................................... 56
Table 4.3. Physical properties of GFRP and BGFRP .......................................................... 64
Table 4.4. Failure modes for tensile test samples ................................................................. 68
Table 4.5. Interlaminar fracture toughness values for GFRP sample 5 ............................... 77
Table 4.6. Tensile strength comparison of different bamboo specimens tested ...................... 91
Table 4.7. Tensile modulus comparison of different bamboo specimens tested .................... 95
List of Figures

Figure 1.1. Organization of the thesis ................................................................. 4

Figure 2.1. Applications of natural fibers [9] ......................................................... 6

Figure 2.2. Moso bamboo cross-section under microscope. The density of vascular bundles
decreases from outer to inner portion of the culm [24] .................................. 10

Figure 2.3. Specific tensile strength plot for natural and E-glass fibres (processed from [3]) .... 11

Figure 2.4. Specific Young’s modulus plot for natural and E-glass fibres (processed from [3]) 11

Figure 2.5. Classification of composites [35] ....................................................... 13

Figure 2.6. Modes of Fracture: (a) Mode I (Opening mode), (b) Mode II (Sliding mode), (c)
Mode III (Tearing mode) [60] ................................................................. 20

Figure 2.7. Classical Fickian sorption behavior [90] .......................................... 25

Figure 3.1. Inversa Bambusoide used in the study. ............................................. 27

Figure 3.2. (a) Acetone dehydration illustrative (ISP). The acetone in the dehydration bath was
stirred every day and replaced if the concentration of acetone fell below 99%. (b).
Acetone concentration check using an acetonometer. ........................................ 28

Figure 3.3. Silicone impregnation apparatus. The impregnation chamber (with orange lid cover
was connected to the vacuum chamber via a catch-pot so that the acetone
evaporating for the chamber doesn’t reach the pump........................................ 29

Figure 3.4. (a) Bubble formation in silicone chamber due to pressure reduction. (b) S3 sprayed
bamboo samples covered in a plastic wrap so that the S3 stays in contact with the
impregnated bamboo.................................................................................. 30

Figure 3.5. Paper frame test: (a) Paper frame tensile testing of bamboo fibres [92]. (b) Paper
clip sample prepared in the current study..................................................... 32
Figure 3.6. Square cross-section strips extracted from bamboo culm. Multiple strips of same length were machined out of the same culm................................................................. 33
Figure 3.7. Customized tensile test bamboo strip in the test fixture............................................ 33
Figure 3.8. Hybrid composite layup .......................................................................................... 37
Figure 3.9. Randomly oriented chopped bamboo layer over Twintex .......................................... 37
Figure 3.10. Hybrid composite fabrication flow chart................................................................. 38
Figure 3.11. Tensile test samples of BGFRP ........................................................................... 39
Figure 3.12. Three-point bending-loading diagram [99]. ............................................................42
Figure 3.13. BGFRP sample setup on a three-point bending fixture ........................................... 43
Figure 3.14. Double Cantilever Beam illustrative [64]..............................................................44
Figure 3.15. Use of piano hinges for loading DCB sample ......................................................... 45
Figure 3.16. Illustrative of piano hinge used for loading DCB specimen [64] .............................. 46
Figure 3.17. Impact specimen in drop tower. (a) GFRP sample fixed in the drop tower test chamber. (b) Hemispherical impactor falling on the composite laminate ........... 47
Figure 3.18. Composite samples immersed in water at 70⁰C for 96 hours for moisture conditioning .................................................................................................................. 49
Figure 4.1. Density bar graph for virgin and plastinated bamboo. The increased density of plastinated bamboo is due to the large volume of silicone being impregnated into the bamboo................................................................. 52
Figure 4.2. EDS analysis of plastinated bamboo denoting Si content. The variation of silicon denotes a concentration gradient with lower concentration at the middle of the cross-section ........................................................................ 54
Figure 4.3. X-ray micro-CT images of virgin bamboo. (a) XY plane (b) YZ plane (c) XZ plane. The white vertical bundles denote the Xylem while the cellular voids around denote Phloem................................................................. 55

Figure 4.4. X-ray microCT images of virgin bamboo. (a) XY plane (b) YZ plane (c) XZ plane. Bright white contrast in vertical bundles denotes impregnated silicone mixture. ..... 56

Figure 4.5. QQ plot for virgin bamboo tensile strength data points. All data points along the line denote normal distribution and no outliers. ............................................ 57

Figure 4.6. QQ plot for plastinated bamboo tensile strength data points. All data points along the line denote normal distribution and no outliers............................................. 58

Figure 4.7. Tensile strength bar graph for virgin and plastinated bamboo. *Significance level, p-value<0.05 (One-way ANOVA). Results show that plastination increases the tensile strength of virgin bamboo by 71% and this effect is statistically significant at 95% confidence level. .................................................................................. 58

Figure 4.8. Tensile modulus bar graph for virgin and plastinated bamboo. *Significance level, p-value<0.05 (One-way ANOVA). Results show that plastination increases the tensile modulus of virgin bamboo by 23% and this effect is statistically significant at 95% confidence level. .................................................................................. 59

Figure 4.9. Tensile stress vs. longitudinal strain curves for plastinated and virgin bamboo showing increased tensile strength, modulus and decreased strain to failure of plastinated bamboo. .................................................................................................. 60

Figure 4.10. Fractured tensile test specimen of plastinated bamboo. Lower vascular bundles on the inner side of bamboo result in dislocation/deformation of more fibres on the inner side during tensile test................................................................. 61
Figure 4.11. Moisture desorption curves for virgin and plastinated bamboo. Lower residual moisture in plastinated bamboo with reduced moisture diffusivity constant as compared to virgin bamboo.

Figure 4.12. Residual moisture content in plastinated bamboo using different dehydration techniques showing reduced residual moisture after repetitive acetone soaking.

Figure 4.13. Residual moisture content among different bamboo species, before and after plastination showing that a higher moisture content before plastination results in a lower residual moisture after plastination.

Figure 4.14. Tensile strength bar graph for GFRP and BGFRP. *Significance level, p-value<0.05 (One-way non-parametric ANOVA). Results show that adding plastinated bamboo fillers to GFRP decreases the tensile strength of GFRP and this effect is statistically significant at 95% confidence level.

Figure 4.15. Chord modulus elasticity bar graph for GFRP and BGFRP. Comparison shows no statistical significance, p-value>0.05 (One-way non-parametric ANOVA). In other words, the results show that adding plastinated bamboo fillers to GFRP can only randomly decrease the tensile modulus of GFRP and the effect is not statistically significant at 95% confidence level.

Figure 4.16. Tensile stress vs. longitudinal strain curves for GFRP and BGFRP showing reduced tensile strength for BGFRP. Toe region denotes the fibre straightening/uncrimping region.

Figure 4.17. Flexural strength bar graph for GFRP and BGFRP. *Significance level, p-value<0.05 (One-way non-parametric ANOVA). Results show that adding
plastinated bamboo fillers decreases the flexural strength of GFRP and this effect is statistically significant at 95% confidence interval...

Figure 4.18. Flexural chord modulus of elasticity bar graph for GFRP and BGFRP. No statistical significance is observed between the two sample groups; p-value>0.05 (One-way non-parametric ANOVA). In other words, the results show that adding plastinated bamboo fillers to GFRP can only randomly decrease the flexural modulus of GFRP and the effect is not statistically significant at 95% confidence interval.

Figure 4.19. Flexural stress-strain curves for GFRP and BGFRP showing adding plastinated bamboo fillers to GFRP reduces the flexural properties and adds randomness to its response.

Figure 4.20. Microscopic image of brittle fractured fibres of GFRP during flexural testing.

Figure 4.21. Microscopic image showing interlaminar delamination (matrix cracking) in BGFRP due to flexural loading.

Figure 4.22. Monotonous trend observed in load-displacement curve for a typical unidirectional composite I DCB specimen [62].

Figure 4.23. Load-displacement curve for GFRP DCB specimen 5 showing INST (point of crack growth) and ARREST (point where the crack propagation stops) points during mode I fracture characterization of woven GFRP.

Figure 4.24. Flexural failure of a 4-layer GFRP DCB specimen during mode I fracture testing.

Figure 4.25. Flexural failure of 12-layer GFRP DCB specimen during mode I fracture testing.
Figure 4.26. Post fracture SEM fractography of the GFRP DCB specimen; (a) Fibre imprints in matrix denoting fibre pull out, (b) Matrix de-bonded split out fibres due to fibre bridging. ................................................................. 78

Figure 4.27. Post fracture SEM fractography of the GFRP DCB specimen; (a) Stand-alone fractured fibre, denoting typical fibre and matrix fractography, (b) Fractured split out fibres because of tensile failure. ................................................................. 79

Figure 4.28. Impact response data observed for GFRP and BGFRP (a) Specific impact energy-time, (b) Specific contact force-displacement, (c) Specific contact force-time. ..... 81

Figure 4.29. Impact response for BGFRP denoting various damage modes during low velocity impact testing................................................................. 82

Figure 4.30. Post impact specimens; (a) Singular micro crack in GFRP, (b) Multiples macro cracks in BGFRP seem to spread from point of impact in various directions........ 83

Figure 4.31. SEM microstructure images of post impact BGFRP specimen; (a) Numerous voids, fractured glass fibres and bamboo denote notable damage, (b) Crack propagation (red dotted curve) along bamboo-glass fibre interface, (c) Glass fibre tow farthest from the impact surface with least damage......................... 85

Figure 4.32. SEM microstructure image of post impact GFRP specimen with smaller voids and less fractured glass fibres denoting lesser damage than BGFRP..................... 86

Figure 4.33. (a) Contact force-time curves for BGFRP samples, (b) Contact force-time curves for GFRP samples.................................................................................. 87

Figure 4.34. Impact energy per unit density bar graph for GFRP and BGFRP. * Significance level, p-value<0.05 (One-way non-parametric ANOVA). Results confirms that
adding plastinated bamboo fillers increases the energy absorption ability of
GFRP at 95% confidence interval. ................................................................. 88

Figure 4.35. Peak-force per unit density bar graph for GFRP and BGFRP. *Significance level, p-value<0.05 (One-way non-parametric ANOVA). Results confirms that adding plastinated bamboo fillers increases the peak-force during impact in GFRP at 95% confidence interval. ................................................................. 88

Figure 4.36. Density of virgin and plastinated bamboo, before and after moisture conditioning showing higher increase in density of virgin bamboo than plastinated bamboo..... 89

Figure 4.37. Weight fraction of water desorbed by virgin and plastinated bamboo vs. drying time in the oven showing lower moisture absorbed by plastinated bamboo. ........... 90

Figure 4.38. QQ plot for moist virgin bamboo tensile strength data points. All data points along the line denote normal distribution and no outliers. ......................... 92

Figure 4.39. QQ plot for moist plastinated bamboo tensile strength data points. All data points along the line denote normal distribution and no outliers. ......................... 92

Figure 4.40. Tensile strength of virgin and plastinated bamboo, before and after moisture conditioning. *Significance level, P<0.05 (Two-sided t-test). Results denote that both study factors i.e. bamboo type and conditioning are statistically significant as well as their interaction. Tukey’s test further reported that individual factors make all pairwise comparisons of samples to be significantly different, while the interaction factor only made the mean values of moist virgin bamboo and dry plastinated bamboo sample to be significantly different at 95% confidence interval. ........................................................................................................... 94
Figure 4.41. Tensile modulus of virgin and plastinated bamboo, before and after moisture conditioning. *Significance level, p<0.05. Results denote that both the factors i.e. bamboo type and conditioning are statistically significant as well as their interaction at 95% confidence interval. ................................................................. 96

Figure 4.42. Tensile stress vs. longitudinal strain curves for plastinated bamboo, before and conditioning showing increased ductility after moisture conditioning. ................. 97

Figure 4.43. Density bargraph for different composites, before and after moisture conditioning. ........................................................................................................ 98

Figure 4.44. Weight gain bar-graph for different composites, due to moisture conditioning. ..... 99

Figure 4.45. Tensile strength variation for different tested composites, before and after moisture conditioning. *Significance level, p<0.05. Results show that fibre type and conditioning are overall statistically significant factors, but their interaction is not at 95% confidence interval. ................................................................. 101

Figure 4.46. Tensile modulus variation for different tested composites, before and after moisture conditioning. *Significance level, p<0.05. Results show that only fibre type is a statistically significant factor at 95% confidence level. ................. 102

Figure 4.47. Post tensile test SEM images; (a) Unconditioned GFRP sample with well consolidated matrix, (b) Conditioned GFRP matrix with micro-cracks and voids that lead to Non-Fickian behaviour of matrix material, (c) Cracked and flaked matrix in conditioned GFRP sample. ................................................................. 104

Figure 4.48. Post tensile test SEM images of unconditioned GFRP, (a) Fractured glass fibres coated with polypropylene matrix, (b) fibre bundles adhered together with the matrix, signifying high interfacial strength. ................................................................. 105
Figure 4.49. Post tensile test SEM images of conditioned GFRP; (a) Fractured glass fibres with minimal/no polypropylene matrix on surface, (b) fibre bundles without any matrix, signifying weak interfacial strength. .......................................................... 105

Figure 4.50. Flexural strength variation for different composite samples, before and after moisture conditioning. *Significance level, p<0.05. Results show that fibre type and conditioning are statistically significant factors, but their interaction is not at 95% confidence interval. .......................................................... 106

Figure 4.51. Flexural modulus variation for different composite sample, before and after moisture conditioning. Results show that there is no factor that significantly affects (at 95% confidence interval) the flexural modulus of GFRP and BGFRP, neither fibre type, nor the conditioning................................................. 107

Figure 4.52. Illustrative reasoning for increase in bending modulus of BGFRP below 5% moisture absorption (present case). .......................................................... 108

Figure 4.53. Illustrative reasoning for decrease in bending stiffness of BGFRP above 5% moisture absorption, as observed in [83].......................... 108
List of Abbreviations

FRC: Fibre reinforced composite
NFC: Natural Fibre Composite
BFRP: Bamboo Fibre Reinforced Polymer composite
TWINTEX, GFRP: Commercial name for comingled E-glass/Polypropylene fabric tested
BGFRP: GFRP reinforced with plastinated bamboo fillers
BGFRP_V: GFRP reinforced with virgin bamboo fillers
PP: Polypropylene
CT: Computed Tomography
MC: Moisture Content
DCB: Double Cantilever Beam
SEM/EDS: Scanning Electron Microscopy/Energy-dispersive X-Ray Spectroscopy
FSP: Fibre Saturation Point
List of Symbols

$\sigma_{max}$  ultimate tensile strength of bamboo

$P_{max}$  maximum force before failure

$A$  average cross-sectional area

$E$  tensile modulus of elasticity

$\Delta \sigma$  difference in applied tensile stress between the two strain points

$\Delta \varepsilon$  difference between the two strain points

$A^*$  original mass of the bamboo

$B$  the oven-dry mass of bamboo

$P_{tu}$  ultimate tensile strength of composite

$\varepsilon_i$  tensile strain in the composite at $i$th data point

$\delta_i$  Instron head displacement at $i$th data point

$L_g$  gauge length of the composite tensile test specimen

$E^{chord}$  tensile chord modulus of elasticity

$\sigma$  flexural stress at the outer surface in the load-span region

$P$  applied load

$L$  support span

$b$  width of the beam

$h$  beam thickness

$\varepsilon$  maximum strain at the outer surface

$\delta$  mid-span deflection

$E_f^{chord}$  flexural chord modulus of elasticity
δ* load point displacement
A delamination length
Δ determined experimentally by generating a least-squares plot of the cube root of compliance, \( C^{1/3} \)
\( G_1 \) strain energy release rate
\( E \) potential energy of the impactor prior to drop
\( C_E \) specified ratio of impact energy to specimen thickness
\( F \) contact force
\( x \) displacement of the impactor
Acknowledgements

I would like to start by acknowledging my supervisor, Dr. Abbas S. Milani for his unwavering support and motivation throughout my Master’s. Dr. Milani is a distinguished teacher and an amazing mentor. His willingness to help students so altruistically makes him even a better person.

I would also like to thank Dr. Grant Bogyo and Ron Ryde from Ryde Holdings Ltd. and Dr. Robert Henry from The University of Tennessee, for sharing their ideas and deep insights into my Master’s project. My sincere gratitude to my committee members, Dr. Kasun Hewage and Dr. Kevin Golovin for their valuable inputs to this project and the thesis. I am obliged to my course professors at UBC and colleagues at Tesla for imparting knowledge to me.

Not to forget all my colleagues at CRN Okanagan lab for their valuable help, especially Armin Rashidi and Bryn Crawford, who answered my endless questions, thanks a lot. I must appreciate the efforts made by the technicians in the machine shop towards this project.

I would like to thank my friends in Canada and India for their constant encouragement and support. Lastly, I am grateful to my family for always believing in me and supporting me in every possible way. My graduate studies would not have been likely without them.
Dedication

...In dedication to my parents and the almighty...
Chapter 1: Introduction

1.1 Background overview

Due to growing concerns for environmental issues such as increasing manufacturing pollution, increasing carbon footprint, and raising awareness about the environmental hazards caused by human-made materials like synthetic fibre composites, great demand and interest for sustainable natural fibre composites have developed over recent years. There is a progression in industry to use natural fibres as reinforcing fibres and/or fillers, e.g. within polymer/biopolymer matrices, in place of synthetic fibres. Owing to their low density, low cost, high stiffness, and strength-to-weight ratios and improved sustainability, natural fibres are gaining a wide demand, including in structural applications [1-3].

Among various natural fibres, bamboo is one of the most desirable options due to its low density and high strength properties. Furthermore, bamboo has over 1,450 species, and the worldwide bamboo production is approximately 30 million tons/year, the highest among all-natural fibres [1-3]. Despite the aforementioned desirable attributes of natural fibres, however, they are prone to degradation due to the effect of moisture and weathering.

In anatomical sciences, the long-term preservation of animal and human tissues has been carried out for decades and among the various preservation techniques, “plastination” is a well-established technique. First applied in 1977 by Dr. Gunther Von Hagens, plastination, also called forced polymerization, has been extensively employed for preserving different bodies and body parts of living organisms [4-6]. In essence, plastination replaces the water and lipids in the tissue with curable polymers. It involves preserving perishable biological specimens using a series of procedures that replaces tissue water and part of tissue fat with curable polymers, mostly silicone,
and epoxy that hinders the decay of body tissue. Besides the wide range of applications of plastination techniques among human, animal and yeast tissues, there is no report on whether plastination could be possibly applied to preserve natural fibres, as a way to enhance the strength and durability of these fibres and their composites like bamboo fibre reinforced plastics.

1.2 Motivation and objectives

A major drawback of using natural fibres is their durability. Lignocellulosic fibers tend to have hydrophilic groups attached, due to which they absorb a lot of moisture. This moisture weakens the cellulose structure and the cell wall of these natural fibers, which results in the degradation of their properties. Lately, a large amount of interest has been developed by researchers to develop techniques/methods that can add to the durability of natural fibers (detailed literature review to follow in Chapter 2).

Plastination is a technique developed in 1977 by Dr. V. Hagens as a sophisticated technique to preserve body tissues of humans and animals. Over the past couple of years, optimizations to this technique have been suggested to make the preservation process less complicated and cheaper. However, the scope of this technique has never been extended outside human and animal tissues. Further, no study has been ever conducted to quantitatively analyze the effect of plastination on physical and mechanical properties and durability of the specimen involved.

A fundamental question arising in this context would be, ‘can plastination make natural fibers durable too?’ In response to this, the initial step would be to test the feasibility of applying the same plastination technique to natural fibers (from manufacturing perspective), and accordingly to investigate how this plastination would affect the properties of the fibers and their composites (from design/application perspective).
With a relatively high strength to weight ratio and a large number of existing structural/non-structural applications, bamboo has been selected for this research, with potentially enhanced durability that can then replace synthetic, e.g. glass fibers in structural applications.

Specifically, the primary objective of this study is to investigate the feasibility of plastinating bamboo natural fibres through room-temperature S-10 plastination method. The sub-objectives defined for this research are as follows:

1. Characterize changes in the material properties of the bamboo due to plastination; including density, moisture content, and tensile strength.

2. Design a plastinated bamboo-glass fiber (hybrid) polymer composite and characterize its mechanical properties.

3. Perform a durability/environmental testing to examine the effect of moisture conditioning on plastinated specimens and the hybrid composite.

1.3 Thesis framework

This thesis is comprised of five chapters. Chapter 2 provides an insight into the background of natural fibres and bamboo, plastination, and a recent history of natural-synthetic fiber (hybrid) composites. Chapter 3 presents the methodology used for this research. Namely, it includes a detailed procedure developed for plastination of bamboo natural fibres, techniques for characterizing the mechanical, physical and chemical properties of plastinated and virgin bamboo. Furthermore, the chapter outlined the various test methods adopted to conduct tensile, flexural, impact and bending tests on the hybrid composite. The remaining part of the chapter discusses moisture conditioning (durability study) experiments of bamboo and the hybrid composite.
Following the same trend as chapter 3, the fourth chapter presents and discusses the findings of the research, focusing on three key themes: successfully applying plastination to bamboo and characterizing it (fulfilling the primary objective and sub-objective 1), analyzing various mechanical properties of the hybrid composite (achieving sub-objective 2), and assessing the environmental durability of plastinated bamboo and the hybrid composite (fulfilling sub-objective 3). Lastly, Chapter 5 outlines the main findings and conclusions of this thesis and suggests pertinent future work.

Figure 1.1. Organization of the thesis
Chapter 2: Literature Review

2.1 Natural fibres

Recently, with the concept of sustainability becoming ubiquitous in engineering design, an abrupt increase in research efforts regarding the applications of green materials and green composites has been observed [1]. It is noticed that natural fibres make up a major portion of these materials as they are extracted from plants and animals. The examples of such natural fibres include bamboo, linen, cotton and wool [1]. The global market for natural fiber-based materials is expected to reach $10.89 billion by 2024 [7]. Moreover, the natural fiber composites market grew, e.g. at an annual compound growth rate of 8.2% between 2015 and 2020 [7]. The growing demand for lightweight and environmentally friendly composites in various industry sectors including automotive, building and construction, is the main driver for the growth of these ecologically friendly fibres [8]. Figure 2.1 summarizes some of the main areas where natural fibres are used today.
Lignocellulose based natural fibers are composed of cellulose microfibrils with lignin and hemicellulose amorphous matrix. Several fibrils constitute a fibre and are arranged along the length of the fiber: comprising thin primary wall surrounding a thicker secondary layer, resembling a single wood fiber [28]. Table 2.1 shows various advantages and disadvantages of natural fibres over synthetic (man-made) fibres.
Table 2.1. Advantages and disadvantages of natural fibres over synthetic fibres [3, 98]

<table>
<thead>
<tr>
<th>Advantages</th>
<th>Disadvantages</th>
</tr>
</thead>
<tbody>
<tr>
<td>• Low density</td>
<td>• Lower durability</td>
</tr>
<tr>
<td>• Renewable/low embodied energy/CO₂ absorption</td>
<td>• Higher moisture absorbing ability</td>
</tr>
<tr>
<td>• Lower cost</td>
<td>• Lower impact strength</td>
</tr>
<tr>
<td>• Less hazardous manufacturing</td>
<td>• Higher variation in properties</td>
</tr>
<tr>
<td>• Less emission of toxins at the end of life</td>
<td>• Lower processing temperature limiting matrix options</td>
</tr>
<tr>
<td>• Less abrasion damage to the processing equipment</td>
<td>• Prone to microbial attacks</td>
</tr>
</tbody>
</table>
2.1.1 Processing of natural fibres

As noticed from Table 2.1, a significant disadvantage of using natural fibres is their lower durability and high moisture absorption. Existing research efforts recognize various physical and chemical processes to enhance the durability and strength of these fibres [3]. Sinha and Panigrahi [10] found that plasma-treated fibres have superior surface hydrophobicity and improved shear strength. Moreover, recent research on the treatment of natural fibers involved heating the fibers to temperatures close to those that can cause their degradation and affect their properties [11]. Huber et al. [12] found that electron radiation can improve the interfacial bond between natural fibres and PP from 21% to 53% due to the generation of free radicals that promote fibre/matrix crosslinking. Further, beating of the fibres results in a 10% increase in kraft fibre/Polypropylene strength due to fiber defibrillation and the corresponding increase in contact area and mechanical interlocking [13].

A vast bulk of natural fibre processing literature has also focused on chemical treatment of natural fibres, that have shown improved physical strengths and interfacial strengths of these fibres [3, 11]. Chemically treating often involves the use of alkali, acetyl, silane, benzyl, acrylic, permanganate, peroxide, isocyanate, titanate, zirconate and acrylonitrile treatments, along with a maleic anhydride graft interfacial agent [14]. The alkaline treatment removes fibrous constituents such as lignin, hemicellulose, wax and pectin, thereby exposing the cellulose and increasing the roughness/surface area, thus improving interfacial bonding [15].

**Remark 1.** However, much of the research involving processing/pre-treatment of natural fibres has been carried out to enhance the mechanical and interfacial properties of natural fibres. These techniques do not address the problem of the hydrophobic nature of these fibres, which requires adequate attention for their durable long-term usage.
2.1.2 Bamboo natural fibres

The subsequent sections in this study consider bamboo as the primary material used for characterization and as filler in typical structural composites.

2.1.3 Characteristics of bamboo

Bamboo can be classified as a group of perennial evergreen plants belonging to the true family of grasses “Poaceae”, from the subfamily “Bambusoideae”, to the tribe Bambuseae [16]. More than 1450 species of bamboo coming from 70 different genera can be found in different climatic zones - from cold mountains to warm tropical regions [16]. Bamboo has a high growth rate and CO$_2$ fixation rate, and some bamboo species grow more than 90 cm/day [17]. Moreover, where wood takes about ten years to reach its maturity, bamboo matures in just 6-8 months [18]. Bamboo also has a small harvesting cycle due to its very high growth rate [19]. Given its rapid growth rate and low carbon footprint during life cycle, it is also referred to as “CO$_2$ better” i.e., CO$_2$ negative [20].

In raw form, bamboo is usually a cylindrical grass with mostly hollow culm and its fibres aligned in the direction of the lignin matrix [19]. Bamboo fiber for a long time has been widely used in construction, pulp, and paper industries [16]. Due to its superior strength and fast growth rate, the use of bamboo fibers in composites as reinforcement of concrete offers an enhanced to technique to increase the dependence of the construction industry on renewable resources [21].

Vascular bundles constitute the fundamental part of the bamboo culm and are responsible for providing the structural support and transport water nutrients in bamboo. The abundance of vascular bundles near the outer wall of the culm accounts for high bending stiffness and structural strength of bamboo culm [22]. Bamboo fibres can even exceed the strength of e.g. glass fibres during bending [2]. Further, data from studies suggest that the lignin content in bamboo is highest in comparison to other lignocellulosic fibres (about 32%) and has a small microfibrillar angle (2°-
These properties also contribute to the high tensile and flexural strength, and high rigidity of bamboo fibres [16]. A study conducted by Sharma et al. [19] used standard test specifications to compare the engineered bamboo and commercial timber, and the results revealed that engineered bamboo products (laminated and scrimber bamboo) have properties either close to or exceed that of timber.

**Figure 2.2. Moso bamboo cross-section under microscope. The density of vascular bundles decreases from outer to inner portion of the culm [24].**

Figure 2.3 shows specific tensile strength comparison for different natural fibres and glass fibre (E glass). The box plots denote the range of data sets obtained due to the variation of properties among different species of same fibre type [3]. It is worth mentioning that the peak values for flex and bamboo are comparable to that of glass fibre. Figure 2.4 shows a plot for stiffness to weight ratio between various fibres and the peak value for natural fibres exceed that of glass fibres [3].
Bamboo fibre has chlorophyll and sodium copper chlorophyllin that are natural antibiotics and impart bamboo a strong bacterial resistance. In just 24 h, bamboo fabric inhibits upto 71% of bacteria population. [25, 26]. The non-abrasive nature of bamboo fibres doesn't lead to significant machine wear, as compared to synthetic glass and ceramic fibres. Their non-abrasive nature also allows for high-volume fraction for filling thus, resulting in good mechanical properties [27, 28].
However, bamboo has some inherent disadvantages such as hydrophilic properties and low fungi resistance, which significantly shortens its service life [29-31].

**Remark 2.** Despite a superior structural performance, the use of bamboo and other natural fibres like Flex in outdoor structural applications appears to be avoided in the current applications due to its biodegradation, which in essence abbreviates its durability [16, 32-34].

### 2.2 Fiber-reinforced composites

Fiber-reinforced composite (FRC) can overcome several of the limitations associated with bamboo as discussed in the previous section [16, 31-34]. FRC consists of a combination of two or more dissimilar constituents with distinct properties, i.e., fibre/fabric reinforced with the matrix (often polymer) material. While designing FRCs, fibre orientation significantly affects the mechanical properties of the composite. The matrix material could be ceramics, polymers, or metals. Figure 2.5 shows classification of composites based upon the type of reinforcement.
2.2.1 Natural fibre polymer composites

Most of the literature on natural fibres is devoted to the design and characterization of polymer composites reinforced with natural fibres. Various natural fibres such as hemp, flax, bamboo and jute are reinforced with polymers; usually thermoplastics and epoxy resins, to form natural fibre-reinforced polymers replacing synthetic fibre composites in various industries [3, 25, 34, 36]. The literature suggests that significant work is being conducted on bamboo reinforced with thermoplastic polymers, with leading thermoplastics being Polypropylene (PP) and Polyvinyl chloride (PVC) [16]. It is believed that these bamboo/polypropylene composites contribute towards sustainability and in appeasing environmental issues because of its abundance, recyclability, low price and decent mechanical properties as a matrix material and bamboo’s cheap
price, easy availability in abundance and eco-friendly nature [16]. The plastic matrix constitutes 30-70% of the composite material with short and randomly distributed bamboo fibres. With the length of these fibres varying between 1-6 mm, these laminas can be considered as quasi-isotropic [16, 25, 34]. Prasad and Rao [37] compared the tensile and flexural strength of polyester resin reinforced bamboo, sisal and jowal, extracted under similar lab conditions and found that bamboo polymer composite possessed good flexural strength and the highest tensile strength among the three composites.

Moreover, Chen et al. [38] examined the mechanical properties of different bamboo shapes and sizes including single bamboo fibre, fibre bundles and strips obtained using chemical and mechanical retting. The study revealed that the outer portion of the culm has higher tensile modulus and strength as compared to the inner part. Further, the tensile strength of single bamboo fibre was least in comparison to the fibre strips and bundles.

Apart from the use of a single kind of fibres in FRCs, these composites can be designed as hybrids. A hybrid composite contains two or more distinct fibres, in such a manner that the desired characteristics of one type of fibre complement the lacking properties of another fibre. Along with providing balanced performance, it also reduces the cost through proper material designing [39, 40].

In the past, it has been observed that the moisture absorption and mechanical properties of natural fibres can be enhanced by their hybridization [41, 42]. Aji et. al. [41] studied the variation in mechanical properties of hybridized kenaf and pineapple leaf fibre reinforced high-density polyethylene (HDPE) composites. The outcomes demonstrated that pineapple leaf fiber expanded the tensile and flexural quality while kenaf improved impact strength and decreased moisture uptake. Moreover, Thomas et al. [42] reported that tensile properties of banana/sisal fibre
reinforced polyester composite were improved by adding short banana fibres. Although hybridization leads to strength enhancement and reduced moisture content, such expositions are not an entirely satisfactory solution because the remainder moisture uptake value is still 78% of the actual.

More recently, researchers have started focusing on the design of natural-synthetic (hybrid) fibre polymer reinforced composites. Thwe and Liao prepared a Bamboo/glass fibre reinforced PP hybrid to study influence of short bamboo/glass fibre on the thermal, mechanical and rheological properties. The study showed change in strength and thermal properties of hybrid composite with change in length of bamboo fibre and amount of interfacial agent [43]. Moreover, it was also concluded that hybridization with synthetic fibres could be a viable approach to improve the mechanical and environmental properties of natural fibre composites [43-45].

**Remark 3.** Based on the above-reviewed works, the author believes that the use of natural fibres in structural applications mostly involves their use with a natural/synthetic polymer, which may provide desired strength and enhanced durability. Despite the fact that BFRPs offer a low cost, there are several limitations such as lower modulus, and relatively poor moisture resistance and interfacial strength, relative to synthetic fibres, due to the hydrophilic nature of these lignocellulosic fibres. Chemically altering the biological structure of these fibres could be one of the solutions to overcome the above limitations.

### 2.3 Plastination

Students, instructors and technicians in anatomy, histology and embalming labs are often exposed to concentrated levels of formaldehyde during dissection of specimens, which usually results in irritation of eyes and the respiratory tract. Formaldehyde is known to cause acute toxic effects on humans [46]. Realizing the dangers associated in dealing with formaldehyde, studies have been
conducted to reduce the exposure levels and contact time with formaldehyde, thus minimizing its adverse health hazards [46]. Plastination is a recently developed method, excellent for long term preservation of biological tissues.

Plastination involves preserving perishable biological specimens using a series of processes that replaces tissue water and part of tissue fat with curable polymers, mostly polyester, silicone and epoxy that hinders the decay of body tissue. First applied in 1977 by Dr. Gunther von Hagens, plastination, also called forced polymerization had been extensively used since then in multiple disciplines for preserving bodies and body parts of different living organisms [5, 6, 47]. For example, in anatomy, plastination is used for preserving brain, heart and body slices; in zoology, plastination is employed for preserving small animal like chicken, spiders and rats; and in botany, investigations reveal the successful application of plastination to a variety of fungi including mushrooms [6, 48]. Specimens treated with plastination usually tend to retain their original texture and color and result in samples that are odorless, dry and have higher durability [6, 49]. In comparison to frozen, dehydrated or paraffin-treated specimens, plastinated ones are more stable, i.e. they are resistant against mechanical stresses. The latter also retains their texture and cellular identity at microscopic level and requires minimal aftercare than the former [49-50].

Plastination comprises of four major steps [6, 48-50]. Depending upon the subject to be plastinated, these steps may be omitted/modified to fulfill the requirement:

I. Fixation and Staining

Fixation is performed using standard fixation techniques like formaldehyde fixation. Colouring is done by injecting epoxy resin into the vessels of the organ. It can also be achieved by short fixation.

II. Dehydration
Dehydration is an important step in plastination before the impregnation. The easiest and most common method for dehydration is freeze-substitution. It involves placing the specimen in acetone bath at -25°C, until the concentration of acetone in the bath is greater than 99%.

III. Forced Impregnation

This step is the most important part of the plastination process since it involves impregnating the specimen with the curable polymers. The polymer mixture is impregnated into the sample by creating a pressure difference built using a vacuum pump.

IV. Curing

The impregnated specimen is cured using a cross-linking polymer that hardens the internal polymer.

Based upon the polymer to be used in the forced impregnation, the three types of plastination techniques, i.e. silicone, epoxy, and polyester can be further categorized into S-10, E 12, PEM 27 and so on [6].

2.3.1 Silicone S-10 plastination technique

Silicone plastination is one of the simplest and most versatile types of plastination involving silicone polymers as impregnation mixtures and hardeners. Among different silicone polymers used, the S10 (polydimethylsiloxane) is the most popular and widely used polymer and results in opaque specimens, and more natural-looking [50]. The S-10 technique has the same plastination steps as described earlier, namely, fixation, dehydration, forced impregnation and curing.

The standard S-10 plastination, also called cold temperature plastination uses a cold impregnation polymer mixture at -15°C to -25°C. The polymer mixture comprises 99% S10 and 1% dibutyltindilaurate (S3) as a catalyst. The viscosity of this polymer mixture increases over time if
stored at room temperature. To prolong the viscosity change, the mixture is stored at -20°C and the impregnation is carried out in a deep freezer [51-52].

In 1998, Glover et al. [53] developed S-10 room temperature technique that involved S10 (polydimethylsiloxane) as the major impregnating polymer and cross-linker tetraethoxysilane (S6) in the mixture. This mixture is stable at room temperature without much change in viscosity over a period of the next 3-4 months, thus eliminating the need for a freezer. Today, about 20% of the laboratories worldwide use room temperature plastination [54].

Starchik and Henry [52] evaluated specimen properties involving the use of cold and room temperature plastination technique. The study demonstrated that cold temperature plastination results in more shrinkage, has a higher duration and specimens produced are more flexible and elastic. It was also suggested that cold temperature plastination should be used for specimens used for exhibition purposes and the room temperature technique for samples in the domains of archelogy and fossils. However, the latter is more economical and reduces surface deformation.

Another investigation by Looney and Henry [48] involved preservation of mushrooms using cold and room temperature plastination techniques revealed that room temperature plastination could also be used to alter the aesthetics and morphological features of plastinated specimen.

**Remark 4.** Besides the wide range of applications of plastination techniques to human, animal and yeast tissues, the author believes that there is no data related to use of plastination technique in the preservation of natural fibres. In addition, no research has been found that surveyed the residual moisture content in a plastinated sample to verify the fundamental idea of complete moisture removal during plastination. As outlined in Chapter 1, the main motivation of the present work is to investigate the feasibility of plastinating natural fibres (here bamboo) using the S-10
room temperature technique to possibly hinder their moisture absorption ability, and thus degradation.

2.4 Material Characterization

2.4.1 Characterization of bamboo

The variation in the mechanical properties of bamboo in different directions, i.e. longitudinal, and transverse radial makes it an anisotropic material. In the raw form, bamboo is usually a cylindrical grass with mostly hollow culm and its fibres aligned in the direction of lignin matrix [19]. The extraction of undamaged long fibers for testing and application has been a major impediment to the use of bamboo fibers for many years [17]. Due to complex geometry of bamboo, there is a lack of appropriate test codes for measuring the mechanical properties of bamboo, unlike wood and timber [55]. As a consequence, majority of the research involving characterization of bamboo includes test methods developed for wood and timber [16-17].

2.4.2 Composite characterization

In recent years, there has been increasing interest in improving the properties and applications of the natural fibre composites (NFCs) by designing hybrid composites where natural fibers become a filler. However, the performance of these hybrid composites is less clear regarding when to use lignocellulosic fibers as a filler [45]. Studies remain narrow in dealing with the hybrid composites, especially their dynamic properties and microstructure. The present thesis attempts to investigate the manufacturability and mechanical properties of a hybrid Twintex (GFRP) composite reinforced with bamboo filler, and its feasibility to be used for structural applications. In the sub-sections to follow general characterization techniques used for composites, as pertinent to this thesis, are reviewed.
2.4.2.1 Characterization of fracture

Currently, fiberglass and carbon fibers are widely used as reinforcing materials in synthesis composites, typically with thermoset and thermoplastic matrices for various structural applications. In particular, as a low-cost option, fabric-reinforced epoxy composites exhibit high ratios of strength/weight and stiffness/weight. For this reason, GFRP composites have found numerous applications in the areas where high performance and light structures are required [56-58].

The interlaminar fracture of fiber-reinforced composites must be understood to make their structures and parts more efficient and reliable during service. Three independent modes are typically used to define different fracture deformations of a given composite structure [59-61]:

I. Opening mode (mode I), i.e., when the faces of the crack are pulled away in the crack’s plane.

II. Sliding (shearing) mode (mode II), i.e., when the faces of the crack are sliding over each other in the crack’s plane.

III. Tearing mode (mode III), i.e., when two faces of the crack are taken apart out of the crack’s plane.

Figure 2.6. Modes of Fracture: (a) Mode I (Opening mode), (b) Mode II (Sliding mode), (c) Mode III (Tearing mode) [60]
Considerable attention has been devoted to Mode 1 (opening mode) fracture with the development of standardized test procedures for testing of unidirectional composites [62-63]. Motamedi [59] proposed an extended finite element method that simulated fracture in unidirectional composites and showed a good agreement to the experimental results. Czabaj and Ratcliffe [61] studied the mode I fracture toughness of unidirectional glass/epoxy composite using Compression testing and Double Cantilever Beam methods. They observed that during crack propagation, the CT specimens exhibited more extensive fibre bridging as compared to the DCB specimens.

**Remark 5.** Most of the Mode 1 fracture studies have focused on the testing of unidirectional fibre composites (UDs) without much work to discover the fracture phenomenon in woven composites due to their non-planar interplay geometry [64]. Since it is equally important to characterize the mode I delamination behavior of woven composites from a design perspective, this study opted to focus on the investigation of delamination (mode I) resistance of a typical glass fibre/PP woven composite, with and without the plastinated bamboo.

### 2.4.2.2 Impact damage characterization

As composites play a crucial role in our lives today, a comprehensive understanding of the impact properties of these materials in the advanced composites industry is one of the keys to designing optimal and safe structures [65]. The Impact behavior of glass-fiber reinforced composites in low-velocity tests has been a subject of experimental research, especially, the damage tolerance of glass fibre/polypropylene composites [66-69]. Alemiardakani [65] analyzed glass/polypropylene laminates with different fiber architectures and used X-ray microtomography to inspect the damage. The study revealed that fibre architecture has a significant effect on the damage induced by the impact. Moreover, Leonard et al. [70] mapped interplay damage in impact tested composite
laminates by visualizing and quantifying delamination in 3D from X-ray CT datasets. However, there has not been much study conducted on the impact characterization of natural-synthetic fibre reinforced (hybrid) composites [71].

Interestingly, researchers are now focusing on the effects of natural fabrics on impact properties in Polypropylene based matrices [72]. Some studies have reported the behavior of NFCs in low-velocity impact tests. However, with hybrid composites, consisting of natural and fiberglass in PP matrices, no such research has been conducted. A thermoplastic resin such as polyester has been used in most of the works on hybrid composites based on natural fibres [73-75]. For example, Ahmed et al. [76] hybridized a woven jute-glass fabric composites and studied it under low-velocity impact. Their results showed that jute composites have a better impact energy absorption capacity than glass-jute hybrid composites, but have a lower damage resistance.

Pandita et al. [77] performed low-velocity impact tests on glass-polyester jute composites and concluded that woven glass on the outer surface of a composite could act as a solid skin. Furthermore, microanalysis is an essential element in the characterization of materials [65]. Scanning Electron Microscopy (SEM) is largely used for fractographic analysis. The fractographic analysis can be used to study the micro-mechanisms of fractures, analyze the failure of laboratory structures and to study the components in post mortem service [78-79]. Similar to the above studies, in the present study, SEM will be used as a tool to analyze the fractography of impact and the fracture of tested specimens.
2.5 Weathering and environmental conditioning

2.5.1 Conditioning of bamboo

A number of studies have recognized the influence of moisture content and analyzed the moisture-dependent properties of unconditioned/untreated bamboo [80-81]. After examining the effect of moisture on various mechanical properties of bamboo, it was observed that the shear strength of bamboo is most affected due to moisture conditioning [80]. However, the influence of moisture content on mechanical properties decreases with increasing target density of bamboo [80]. Xu et al. [33] tested the effect of moisture content on the compressive, tensile, shearing and transverse splitting properties of untreated bamboo lumber and found that the Fibre Saturation Point of untreated bamboo lumber is approximately 30%.

Remark 6. However, no information is available in the current literature on the effect of moisture content on the mechanical properties of plastinated bamboo.

2.5.2 Conditioning of FRCs

When considering a fiberglass/polypropylene composite for automotive, offshore and marine structures, it is necessary to characterize the effects of the environment on mechanical properties. In particular, water can penetrate into the fiber-reinforced polymer and then significantly attack the resin/fiber interface, thereby creating a low-stress transfer efficiency and thus reducing strength and rigidity. Therefore, many studies have been conducted to observe the influence of moist environment on the mechanical and physical properties of fiber-reinforced polymer composites [82-83]. Shiota et al. [84] studied the effect of hot water immersion on mechanical properties of glass fibre/PP composite. The study reported a decrease in tensile and fatigue properties due to degradation of the PP matrix and leaching of the interfacial agent. Morii et al. [85] identified the
effect of moisture uptake on bending properties of glass fibre/PP composite. A linear decrease in bending properties with an increase in immersion time and moisture uptake was observed.

The penetration of moisture into composites occurs through three different mechanisms. Firstly, water penetrates the micro-gaps between polymer chains through diffusion. Secondly, water travels into the voids through capillary transportation and flows at the fibre-polymer interface. It also transports through the micro-cracks in the polymer initiated during the manufacturing phase.

The third process involves the transportation of micro-cracks that are formed as a result of fibre-swelling (especially in the case of NFPCs). In general, based upon these mechanisms, the moisture mobility in the polymer can be classified to Fickian, Non-Fickian or an intermediate behaviour between Fickian and Non-Fickian. [86-89]. Figure 2.7 shows typical Fickian diffusion behavior where $M_o$ is the sorbed penetrant content and $t$ is the sorption time.

**Remark 7.** However, little is known about the moisture absorption of polymer composites, in particular, the effect of moisture on the mechanical properties of hybrid natural-synthetic fiber composites, which is again one of the main objectives of the present work as outlined in Chapter 1.
Figure 2.7. Classical Fickian sorption behavior [90]
Chapter 3: Materials and Methods

3.1 Plastination and testing of bamboo

3.1.1 Overview

This section provides an insight into the experimental setup designed in the current work to plastinate the bamboo. Further, it discusses the detailed methodology to define and optimize the plastination of natural fibres, due to lack of any similar previous work as referred to in section 2.3.1. The section also presents the test methods employed to characterize the physical, mechanical and chemical properties of plastinated and virgin bamboos; the characterization of the hybrid natural-synthesis fiber composite will be presented in Section 3.2. Select material parameters, namely density, tensile strength, and moisture are tested for bamboo with an analysis to measure the change in chemical composition due to plastination, using Energy Dispersive X-ray Spectroscopy and the 3-dimensional morphology of plastinated bamboo is recorded and studied using Micro-tomography.

3.1.2 Plastination of bamboo

3.1.2.1 Material

The specific bamboo species that was primarily used for all of the following experiments was Inversa Bambusoide, also called ‘Yellow Strip Timber’ (Figure 3.1). It is a medium-sized Asian timber bamboo that often has a culm diameter of 3 inches and a height of around 35-45 feet [91]. It establishes quickly, forming a large grove in only four to five years [91]. Another bamboo species used only for a few moisture experiments was Phyllostachys Edulis, which is also commonly called ‘Moso bamboo’. Phyllostachys Edulis is the most important species covering 30% of bamboo forests and a total area of over 7.2 million hectares in China [26]. The two-bamboo species were derived from a partnering supplier, in the form of raw bamboo culms and
fibre bundles. Single bamboo fibres were technically made up of multiple elementary fibres [92]. The initial attempts for plastination of bamboo were made using the raw bamboo culms and the fibre bundles using room temperature S-10 procedure. As the feasibility of the process proved to be successful, some experiments were conducted to optimize the time duration of this natural fibres’ plastination process.

![Image of Inversa Bambusoide used in the study.](image)

**Figure 3.1. Inversa Bambusoide used in the study.**

### 3.1.2.2 Plastination procedure

The first step of the standard S-10 plastination procedure involves degreasing to remove lipids. However, in the present case, natural fibres did not possess much lipids and hence this step was skipped, and the process was started with the second step; i.e., acetone dehydration. As mentioned in chapter 2, the idea behind acetone dehydration is to diffuse acetone into the cells and replace water. For this, the raw bamboo culms and fibre bundles were completely immersed in a 100% concentrated solution of acetone. The acetone volume in the container was enough to keep the bamboo samples completely immersed. The acetone container was placed in a deep freezer to
maintain a temperature of about \(-25^\circ\text{C}\) to prevent evaporation of volatile acetone, as shown in Figure 3.2 (a).

As the bamboo specimens were immersed in fresh acetone \(-25^\circ\text{C}\), the bamboo was observed to float on the surface of acetone due to a higher density of cold acetone than the density of bamboo at room temperature. The acetone in the container was stirred once daily and the first acetone concentration check was done after three days of bamboo immersion. Unlike the large and complex human body parts that contain much higher aqueous content, the acetone concentration in the container after 3 days was greater than 99%. The concentration was measured using an acetonometer as shown in Figure 3.2 (b), at \(20^\circ\text{C}\), i.e. the calibration point of the acetonometer.

**Figure 3.2.** (a) Acetone dehydration illustrative (ISP). The acetone in the dehydration bath was stirred every day and replaced if the concentration of acetone fell below 99%. (b). Acetone concentration check using an acetonometer.

After ensuring the acetone concentration to be higher than 99%, the bamboo samples were taken towards the next crucial step of plastination, silicon impregnation. Figure 3.3 shows the apparatus
for silicon impregnation. The room temperature impregnation process was followed through all the plastination experiments in this thesis.

Figure 3.3. Silicone impregnation apparatus. The impregnation chamber (with orange lid cover) was connected to the vacuum chamber via a catch-pot so that the acetone evaporating for the chamber doesn’t reach the pump.

As mentioned in chapter 2, the high boiling point and low vapor pressure mixture is impregnated into the cells of the specimen by vaporizing the low boiling point and high vapor pressure acetone. For this, the dehydrated samples from the acetone bath were quickly immersed in the silicone mixture. These samples were kept immersed in the silicone mixture overnight to achieve an equilibrium between the concentrations of acetone and silicone mixture.

On the second day, the pressure chamber was sealed and pressure reduction inside the chamber was achieved using a vacuum pump through a catch pot. Initially, the pressure was decreased to 17.5” Hg. The formation of bubbles was observed as the pressure decreased, as shown in Figure
3.4 (a). The mixture was allowed to stand this pressure until no bubbles were observed on the surface.

The vacuum was then further increased in a similar manner in increments of 4” Hg. The disappearance of bubbles assured that acetone within the bamboo has been removed and has been replaced by silicone.

At around 28” Hg (close to perfect vacuum) the disappearance of bubbles was considered as the point of complete impregnation. Theoretically, total vacuum is the point of complete impregnation, but due to limitations of the experimental setup, 28” was the final point.

After impregnation, the samples were cured using a spraying technique. The curing mixture was sprayed on the samples which initiated a cross-linking reaction between the S3 and S10 & S6 mixture. Figure 3.4 (b) shows samples that were wrapped in a plastic wrap to make sure S3 was sealed and stayed in contact with the impregnated bamboo specimens.

![Figure 3.4. (a) Bubble formation in the silicone chamber due to pressure reduction. (b) S3 sprayed bamboo samples covered in a plastic wrap so that the S3 stays in contact with the impregnated bamboo.](image-url)
3.1.3 Characterization of plastinated bamboo

After the plastination process, the plastinated bamboo samples were characterized to analyze the change in their physical, chemical and mechanical properties caused by the plastination process.

3.1.3.1 Density measurement

Density of an object is a fundamental parameter in design, used commonly in the industry. The effect on physical properties was measured by comparing the density of virgin and plastinated bamboo. To best of the author’s knowledge, no prior studies investigated the content of silicone that flows into the plastinated sample. The Energy Dispersive X-Ray Spectroscopy was conducted on different cross-sections of a plastinated bamboo culm to calculate the amount of silicone flowing into the bamboo. The cross-sections of the bamboo samples were coated with a platinum layer to enable conduction of electrical current [93]. The platinum sputtering thickness was about 6000-8000 nm. A Tescan Mira 3 XMU Scanning Electron Microscope was used for the investigation. For the x-ray micro-CT, a Zeiss/Xradia X-400 was used with 40 kV and 250 µA current. The 360-degree rotation scan had a step size of 0.144 degrees.

3.1.3.2 Tensile test setup

An Instron 5969 with a 50 KN load cell was used for all the tensile tests. For calculating the tensile strength of bamboo, different methods and bamboo geometries were used. To determine the tensile strength of fibre bundles, Defoirdt et al. [92] worked on single fibre tensile testing of bamboo fibres using paper clip method. This method involved gluing the ends of bamboo fibre to two paper clips which were held in the tensile testing machine grips, as seen in Figure 3.5. For simplicity, the cross-section and diameter of the bamboo fibre bundle was assumed to be uniform throughout its length. The clips were pulled apart with the fibre loaded at 1mm/min [92]. Six fibre bundle
replicates were used for this test. However, a problem with this method was the inaccuracy to calculate the exact cross-sectional area of the fibre bundles and detachment of fibres from paper clips. Due to lack of test codes for tensile testing of bamboo, a customized sample geometry was developed, with the rest of test guidelines derived from ASTM D4761 [94]. This involved $4 \times 4$ mm$^2$ sections of bamboo being extracted from the culm (Figure 3.6), with a height equal to the maximum length of culm about 110 mm (mostly uniform in all samples). This method provided results much more consistently. The tensile strength parameters were then derived using the load-displacement curve generated during the tests. For this test, six replicates each of the plastinated and virgin bamboo strips was tested.

Figure 3.5. Paper frame test: (a) Paper frame tensile testing of bamboo fibres [92]. (b) Paper clip sample prepared in the current study.
Figure 3.6. Square cross-section strips extracted from the bamboo culm. Multiple strips of same length were machined out of the same culm.

Figure 3.7. Customized tensile test bamboo strip in the test fixture.

The ultimate tensile strength of bamboo was calculated using:

$$\sigma_{max} = \frac{P_{max}}{A}$$  \hspace{1cm} (3.1)

where, $\sigma_{max}$ is the ultimate tensile strength, $P_{max}$ is the maximum force before failure, and $A$ is the average cross-sectional area.
The tensile modulus of elasticity, in the direction parallel to the bamboo fibres (direction of applied uniaxial load) was calculated using:

\[
E = \frac{\Delta \sigma}{\Delta \varepsilon}
\]  

(3.2)

where, \( E \), the tensile modulus of elasticity, \( \Delta \sigma \) is the difference in applied tensile stress between the two strain points, and \( \Delta \varepsilon \) is the difference between the two strain points.

For simplicity and relative comparison purposes, the toe region (fibre uncrimping/straightening) observed in the stress-strain curves for the tested samples was resolved and removed for calculating the tensile test parameters consistently.

### 3.1.3.3 Environmental conditioning test

The theory of plastination states that water and fat present in the cells are replaced by a polymer, silicone in this case [4]. To verify this reasoning, the plastinated bamboo samples were checked for residual moisture levels. Virgin bamboo was tested along for a relative analysis. To understand the effect of variation in existing moisture levels of bamboo, another bamboo specie namely Moso bamboo was also tested. As mentioned before, due to a lack of test specifications for bamboo, the moisture tests were conducted following ASTM D4442, which mentions the procedure of moisture testing of wood using oven dry method [95]. The samples were weighed for mass (accurate to 10 mg) and kept in an oven maintained at 103±2°C with regular mass readings observations after three hours until the mass change between two readings was less than equal to twice the least count of the weighing scale used, 20 mg in this case. The moisture content was defined using:
where, $A^*$ is the original mass of the sample and B is the oven dry mass.

Further, to reduce the residual moisture content in plastinated bamboo, an altered plastination procedure was also studied. In this ‘Three-soak method’, the initial process of acetone dehydration as mentioned in section 3.1.2. was repeated thrice consecutively, following which the bamboo culms were taken for the forced impregnation process. The residual moisture in these samples was then calculated following the same aforementioned equation.

3.2 Design and testing of hybrid composite

3.2.1 Overview

In this sub-section, the design and characterization of a hybrid and a woven glass fibre composite has been outlined. Section 3.2.2 discusses the design and fabrication of glass fibre and hybrid composites. Also, the hybrid composite constituting short bamboo fibres (both plastinated and virgin) as filler material in a glass fibre/PP composite was characterized for density, tensile properties, flexural properties, fracture toughness; and impact strength was also calculated relative to the regular glass fibre composite to understand the effect of adding these bamboo fillers. Standardized test procedures were followed for the most part to conduct the aforementioned tests, along with an optimized tensile test method for the hybrid composite.

3.2.2 Laminate design

The base composite laminate used in the experiments constituted plies of comingled glass fibre and homopolymer polypropylene (PP) filaments, called Twintex commercially. This material is a plain weave composite that has about 60% glass fibres by weight and approximately 40%
polypropylene. Table 3.1 shows material specifications for glass fibres and PP used in the Twintex. The author would like to make a note here that Twintex material also consists of an interfacial agent, the name/properties of which have not been mentioned in the datasheets, being a trade secret.

Four plies of Twintex were hand laid-up to constitute the Glass fibre Reinforced Polymer (GFRP) composite and the Bamboo Glass Fibre (hybrid) Reinforced Polymer (BGFRP) composite. For GFRP, the stacked layup was compression-molded on steel plates at 190°C with a subsequent consolidation within the press following a custom recipe. 300 × 300 mm² plies were stacked for fabrication in the press and samples of required size and geometry were later cut from these consolidated sheets using waterjet cutting.

<table>
<thead>
<tr>
<th>Property</th>
<th>Glass</th>
<th>Polypropylene</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tensile Modulus, GPa</td>
<td>73.1</td>
<td>1-1.4</td>
</tr>
<tr>
<td>Volume Fraction, %</td>
<td>35</td>
<td>65</td>
</tr>
<tr>
<td>Poisson’s ratio</td>
<td>0.22</td>
<td>0.3</td>
</tr>
<tr>
<td>Density, kg/m³</td>
<td>2610</td>
<td>910</td>
</tr>
<tr>
<td>Average fibre diameter, µm</td>
<td>17</td>
<td>25</td>
</tr>
<tr>
<td>Number of fibres in a tow</td>
<td>879</td>
<td>3642</td>
</tr>
</tbody>
</table>

For BGFRP_P, plastinated bamboo fibre bundles were chopped to 5 mm length with an average diameter of about 0.5 mm, and were randomly distributed to sandwich between the middle layers (2nd and 3rd) of the four-layered GFRP as shown in Figures 3.8 and 3.9. In BGFRP, plastinated
bamboo constituted 10% of the total weight. In case of BGFRP_V, chopped virgin bamboo was sandwiched between the Twintex layers with the same weight percentage as plastinated bamboo. A similar compression molding recipe was followed for BGFRP and BGFRP_V as for GFRP, as outlined in Figure 3.10.

<table>
<thead>
<tr>
<th>Layer 1: Twintex</th>
</tr>
</thead>
<tbody>
<tr>
<td>Layer 2: Twintex</td>
</tr>
<tr>
<td>Layer 3: Chopped bamboo layer</td>
</tr>
<tr>
<td>Layer 4: Twintex</td>
</tr>
<tr>
<td>Layer 5: Twintex</td>
</tr>
</tbody>
</table>

Figure 3.8. Hybrid composite layup

Figure 3.9. Randomly oriented chopped bamboo layer over Twintex
3.2.3 Characterization of composite samples

The density of the various composites mentioned above was calculated using a densimeter to analyze the physical effect(s) of adding virgin/plastinated bamboo to a synthetic fibre composite. Apart from analyzing the effect on physical properties by adding bamboo, the tensile, flexural, fracture and impact properties of the hybrid composite and the synthetic fibre composite were compared to examine the difference in mechanical strength and properties. To observe detailed fractography and the damaged microstructure and the interface post-testing, the tested samples were further characterized by optical microscopy and SEM.
3.2.3.1 Tensile test

The tensile test was conducted on an Instron 5969 and samples were prepared and tested in accordance with ASTM D3039 [96] which is originally meant for unidirectional composites, due to lack of any tensile test specifications for woven fabric composites. Due to the material availability limitations, the sample size for various mechanical stress tests of all the composite samples was set to two. Based upon the fibre-orientation, the dimensions for each sample for tensile testing was $250 \times 25 \text{ mm}^2$ (Figure 3.11) and the cross-head speed was set to 2 mm/min. As per ASTM D3039 [96], metallic tabs should be attached to the composite tensile specimens on the area to be gripped in the fixture clamps, to avoid any stress concentration in the gripping zone. However, due to polypropylene’s (matrix material) poor adhesion properties, tabs could not be used for any tensile test experiments in this study. Instead, a test set-up optimization study was conducted to increase the likelihood of the specimen failure in the gauge length (desired failure zone). As per this finding, if a pressure of less than 3.5 MPa is exerted on the gripped zone of the GF/PP composite, metallic tabs may be avoided as the sample is highly likely to fail in the gauge length [97].

![Figure 3.11. Tensile test samples of BGFRP](image)

**Figure 3.11. Tensile test samples of BGFRP**
The load-extension data obtained from the test was post-processed to evaluate the maximum tensile strength, tensile modulus and strain to failure. Failure identification codes for each sample were derived per the criteria mentioned in Appendix A, following ASTM D3039 [96].

The ultimate tensile strength was calculated using:

\[ F_{tu} = \frac{P_{max}}{A} \]  \hspace{1cm} (3.4)

where, \( F_{tu} \) is the ultimate tensile strength of the composite, \( P_{max} \) is the maximum force before failure, and \( A \) is the average cross-sectional area.

The tensile strain at each data point was calculated from the indicated displacement using:

\[ \varepsilon_i = \frac{\delta_i}{L_g} \]  \hspace{1cm} (3.5)

where, \( \varepsilon_i \) is the tensile strain at \( i \)th data point, \( \delta_i \) is the head displacement at \( i \)th data point and \( L_g \) is the gauge length. The strain range for modulus calculation is contained in the lower half of the stress/strain curve, between 25% to 50% of the ultimate strain.

The tensile chord modulus of elasticity is the ratio of stress range and corresponding strain range. In this case, it was calculated using:

\[ E_{chord} = \frac{\Delta \sigma}{\Delta \varepsilon} \]  \hspace{1cm} (3.6)
Where, \( E_{chord} \) is the tensile chord modulus of elasticity, \( \Delta \sigma \) is the difference in applied tensile stress between the two strain points and \( \Delta \varepsilon \) is the difference between the two strain points (nominally 0.002).

### 3.2.3.2 Three-point bending test

All the flexural testing was done using the three-point bending mode per ASTM D7264, since the effect of shear can also be accounted for in three-point bending as compared to only tension-compression in four-point bending [99]. The three-point bend fixture was set up on an Instron 5969. The span to width ratio selected was 32:1, with a span length of 60.8 mm and 73.6 mm for GFRP and BGFRP composites, respectively. The samples were loaded at 2 mm/min and the sample dimensions being shown in Table 3.2. Each sample was tested until a sharp fall in the load-deflection curve was observed, signifying final failure i.e. fibre fracture on tensile side. The flexural strength, flexural strain and flexural chord modulus of elasticity were calculated from the load-extension data [99].

<table>
<thead>
<tr>
<th>Sample</th>
<th>Span length (mm)</th>
<th>Total length (mm)</th>
<th>Width (mm)</th>
<th>Thickness (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>BGFRP</td>
<td>60.80</td>
<td>65</td>
<td>13</td>
<td>1.90</td>
</tr>
<tr>
<td>GFRP</td>
<td>73.60</td>
<td>80</td>
<td>13</td>
<td>2.30</td>
</tr>
</tbody>
</table>

The maximum flexural stress in the beam was calculated using:

\[
\sigma = \frac{3PL}{2bh^2}
\] (3.7)
where $\sigma$ is the stress at the outer surface in the load-span region, $P$ is the applied load, $L$ is the support span, $b$ is the width of the beam and $h$ is the thickness.

The maximum strain at the outer surface occurring at mid-span was calculated using:

$$\varepsilon = \frac{6\delta h}{L^2} \quad (3.8)$$

where $\varepsilon$ is the maximum strain at the outer surface, $\delta$ is the mid-span deflection, $L$ is the support span and $h$ is the beam thickness.

The flexural chord modulus of elasticity is the ratio of stress range and corresponding strain range. In this case, it was calculated using:

$$E_{f}^{\text{chord}} = \frac{\Delta \sigma}{\Delta \varepsilon} \quad (3.9)$$

where $E_{f}^{\text{chord}}$ is the flexural chord modulus of elasticity, $\Delta \sigma$ is the difference in flexural stress between the two selected strain points and $\Delta \varepsilon$ is the difference between the two selected strain points. For the calculation of the flexural chord modulus, the recommended strain range is 0.002 with a start point of 0.001 and an endpoint 0.003 [99].

**Figure 3.12.** Three-point bending-loading diagram [99].
3.2.3.3 Fracture test

For fracture toughness property, the composites were tested under Mode-I fracture (opening mode) using the Double Cantilever Beam (DCB) technique, namely for calculating $G_I$. ASTM D5528 was primarily followed to set up the Mode I fracture experiment at room temperature, due to the absence of any existing test specification for fracture testing of woven composites [64]. The DCB samples were prepared using a vacuum bagging (50 $\mu$m thick) as an insert material placed at mid-thickness during the lay up to generate a pre-crack of desired length in the consolidated specimen. Piano hinges were screwed into the two beams of the DCB samples. Hence, holes were drilled and tapped for screws to mechanically fasten the hinges used, owing to the inability of glue/adhesive agents to bond with PP matrix due low surface energy of PP matrix. DCB samples of different sizes were tested during this experiment. Table 3.3 shows the different width, initial crack length and thickness used for the samples.
Table 3.3. DCB sample dimensions for mode I fracture test

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Twintex layers</th>
<th>Thickness (mm)</th>
<th>Pre-crack length (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>4</td>
<td>1.9</td>
<td>50</td>
</tr>
<tr>
<td>2</td>
<td>4</td>
<td>1.9</td>
<td>22.5</td>
</tr>
<tr>
<td>3</td>
<td>6</td>
<td>37</td>
<td>50</td>
</tr>
<tr>
<td>4</td>
<td>6</td>
<td>37</td>
<td>22.5</td>
</tr>
<tr>
<td>5</td>
<td>12</td>
<td>54</td>
<td>50</td>
</tr>
<tr>
<td>6</td>
<td>12</td>
<td>54</td>
<td>22.5</td>
</tr>
</tbody>
</table>

The piano hinges were gripped and a monotonic loading displacement rate of 10 mm/min was applied (Figure 3.14). The direction of crack propagation in the woven lamina was in the warp direction. One side of each sample was coated with white correction fluid and marked with equidistant points to trace the position of crack tip, as shown in Figure 3.15. The position of the growing crack tip was simultaneously recorded on the real-time load-displacement curve derived from the Bluehill software. A $3 \times 3$ mm$^2$ sample from the fractured face was analyzed to reveal the microstructure of the cracked PP matrix and fractured fibres. It is also worth mentioning that for fabricating the 12-layer Twintex DCB sample, the standard composite thermoforming recipe was altered and thermocouples were used to cater and monitor the flow of heat through the thickness.

Figure 3.14. Double Cantilever Beam illustrative [64]
For calculating the interlaminar fracture toughness, Modified Beam theory was used as the Beam theory overestimates the value of interlaminar fracture toughness. As a correction, the rotation occurring at the delamination front is accounted for by using slightly longer delamination, $a + |\Delta|$. Thus, the strain energy release rate was calculated $G_I$ per the equation [64]:

$$G_I = \frac{3P\delta^*}{2b(a + |\Delta|)} \quad (3.10)$$

where $P$ is the load, $\delta^*$ is the load point displacement, $b$ is the specimen width, $a$ is the delamination length and $\Delta$ determined experimentally by generating a least-squares plot of the cube root of compliance, $C^{1/3}$, as a function of delamination length.

Further, due to excessive bending and large displacement, $G_I$ was multiplied by a correction factor $F$, calculated using:

$$F = 1 - \frac{3}{10} \left( \frac{\delta^*}{a} \right)^2 - \frac{3}{2} \left( \frac{\delta^* t}{a^2} \right) \quad (3.11)$$

where $t$ is shown in Figure 3.15.
3.2.3.4 Impact test

To examine the damage resistance of the developed composites, low-velocity impact testing was conducted on an Instron Ceast 9340 impact machine. Two samples each of GFRP and BGRFP were impact tested per ASTM D7136 [100]. 150 x 100 mm$^2$ sample was loaded in the drop tower, as shown in Figure 3.17, and impacted with 3.9 kg striker with a hemispherical tip having a 1-inch diameter. The potential energy of the impactor prior to drop was based upon the thickness of the lamina being tested and was calculated using:

\[ E = C_E h \]  

(3.12)

where, $E$ is the potential energy of the impactor prior to drop, $C_E$ is the specified ratio of impact energy to specimen thickness i.e. 6.7 J/mm and $h$ is the nominal specimen thickness. The energy $E$ is sufficient enough to damage the sample causing a noticeable change in its stiffness properties [100]. This impact energy was calculated to be 12.73 and 15.17 J for GFRP and BGFRP samples, respectively.
The impact contact force (reaction force) time-series data were collected using a quartz piezoelectric load cell placed on the nose of the hemispherical tup. The striker displacement was reported by the strain gauges and the impact energy was calculated by integrating the contact force and time as:

\[ E(t) = \int F(t) \, dx \]  \hspace{1cm} (3.13)

where \( F \) is the contact force and \( x \) is the impactor displacement.

3.3 Environmental conditioning

3.3.1 Conditioning of bamboo

3.3.1.1 Overview and setup

One of the main objectives of this study was to analyze how plastination affects the durability of bamboo. Since bamboo fibres are hydrophilic, they tend to absorb moisture notably, which leads to material degradation and loss of strength [32-34]. Plastination removes water from the tissues.
to make them more durable, however there has been no study so far to understand the effect of moisture on plastinated specimens. Hence, this section outlines the method adopted to understand the effect of moisture conditioning on plastinated bamboo and to how it behaves in a moist environment as compared to virgin bamboo.

For this, six plastinated and virgin bamboo specimens were submerged in tap-water at room-temperature i.e. 20±2°C for 4 days. The duration of conditioning and the water temperature were determined such that these parameters were consistent with the other standardized moisture experiments in this thesis. The sample dimensions were also kept uniform for the tensile test (section 3.13.2) and moisture test specimens to eliminate any undesired noise related to sample geometry.

3.3.1.2 Characterization of conditioned bamboo

Post conditioning, the bamboo samples were tested for moisture content, density and tensile strength. For all these tests, the samples were taken out of the water container and wiped off the excess water from the surface with a dry cloth [82]. These were then quickly tested per the required test procedures as discussed in section 3.1.3.

The determination of the moisture content of the wetted samples was done in accordance with ASTM D4442 [95]. This moisture content value gave an idea about the nature of hydrophilic behaviour of plastinated samples as compared to virgin samples and their moisture desorption behaviour. The density of the moistened plastinated and virgin bamboo samples was calculated using densimeter to understand how plastination affected the moisture absorption (mass change) and swelling (volume change) for bamboo fibres.
3.3.2 Conditioning of composite

3.3.2.1 Overview and setup

This section outlines the method used for moisture conditioning of the hybrid and glass fibre composites; which was conducted to analyze the moisture effect on glass fibres, PP matrix, plastinated and virgin bamboo fibres as they act as filler materials. All the conditioned samples were tested post conditioning for moisture, density, tensile and flexural properties.

3.3.2.2 Characterization of conditioned composites

The conditioning test parameters were calculated in compliance with ASTM D5229, ‘Moisture absorption of Polymer Matrix Composite Material’ [101]. A non-equilibrium moisture profile was taken into account, as specified by this test standard. This resulted in a specific moisture content value and established a documented procedure that is reported in sufficient detail to allow duplication of the conditioning result by the independent part. In this case, the composite samples were immersed in water for four days at 70±2°C, as can be seen in Figure 3.18. The role of temperature was to accelerate the water ageing and 70°C immersion temperature is considered a moderate temperature in accelerating ageing of polymer composites [101, 102].

![Figure 3.18. Composite samples immersed in water at 70°C for 96 hours for moisture conditioning.](image-url)
The moist samples were subjected to tensile testing to understand the effect of moisture on mechanical properties due to changed physical and chemical properties [82, 88]. Since water absorption in fibrous composites also depends upon exposed surface area [102], the uniform specimen geometries and test procedure used for the tensile and bending tests complied to those mentioned in ASTM D3039 and ASTM D7264. The immersed samples were taken out of the water and wiped with a dry cloth to remove surface water just before testing.

For tensile and flexural testing, the sample geometry and procedure followed was the same as in section 3.2.3. It should be noted that glass fibre composite with virgin bamboo fibres as filler was only used in tensile testing under destructive testing due to limited material availability. SEM was used for observing the topography of the conditioned composites and the fractography of the fibres in untested and tensile tested samples.

### 3.4 Statistical hypothesis testing

Analysis of Variance (ANOVA) was initially used for hypothesis testing to determine the effect of various factors and their levels on the response variables. For experiments involving a relatively larger sample size (six), parametric one way and two ANOVA were conducted. Since the model errors in a parametric ANOVA are assumed to be normally and independently distributed, QQ plots were used as a measure to check the validity of this assumption. However, since most of the experiments in this study have a sample size of two, the assumption of normal distribution could not be always validated. Hence, for such cases, a non-parametric ANOVA was conducted; namely the Aligned Rank Transform (ART) method [103]. A fixed significance level testing i.e. a p-value approach (equal to 0.05) with a 95% Confidence level was used as a measure to determine rejection/acceptance of null hypothesis.
Wobbrock et al. [103] presented the Aligned Rank Transform (ART) method to overcome the complexity and paucity of nonparametric factorial analysis. In this work, the ARTool program was used for alignment and ranking of the raw datasets. The methodology employed for the alignment and ranking technique has been discussed in detail in Appendix B. After this step, a parametric ANOVA was conducted on the aligned and ranked data to analyze the significance of study factors. For both parametric and non-parametric ANOVA, Tukey’s Post-hoc analysis was also run to determine the effect of individual levels in each factor [104].

The results of these analyses have been incorporated into the upcoming results in chapter 4. To ease the visual representation of comparisons between multiple data sets tests, a ‘*’ sign will be used to represent rejection of the null hypothesis cases (i.e. when p<0.05).
Chapter 4: Results and Discussions

4.1 Characterization of bamboo

As discussed in section 3.1.3, the plastinated bamboo samples were tested for density, tensile properties and moisture.

4.1.1 Density

Figure 4.1 shows plastinated bamboo has about 19% higher density than virgin bamboo. Interestingly, the relative densities of the S10 and S6 solutions i.e. the impregnation mixture, are less than water. The increased density of plastinated samples suggest that the impregnation solution not only replaces some water, but it also occupies the voids that were earlier empty in bamboo.

![Density bar graph for virgin and plastinated bamboo. The increased density of plastinated bamboo is due to the large volume of silicone being impregnated into the bamboo.](image-url)
Table 4.1 shows that, on average, about 23.94% Si is present in a cross-section of plastinated bamboo vs. 7.5% moisture pre-present before impregnation. It is also worth mentioning that from the trend seen in the EDS analysis in Figure 4.2, the Si content in the middle of the cross-section was usually lower than the content on the outer regions. It can thus be suggested that silicone solution may not be able to impregnate completely to the innermost regions of the bamboo cross-section using the present technique.

Table 4.1. Chemical composition of plastinated bamboo-derived using EDS

<table>
<thead>
<tr>
<th>Statistics</th>
<th>C</th>
<th>O</th>
<th>Si</th>
</tr>
</thead>
<tbody>
<tr>
<td>Max, %</td>
<td>49.51</td>
<td>38.30</td>
<td>30.41</td>
</tr>
<tr>
<td>Min, %</td>
<td>36.03</td>
<td>23.06</td>
<td>11.38</td>
</tr>
<tr>
<td>Average, %</td>
<td>39.35</td>
<td>33.24</td>
<td>23.94</td>
</tr>
<tr>
<td>Standard Deviation, %</td>
<td>4.13</td>
<td>4.32</td>
<td>6.35</td>
</tr>
</tbody>
</table>
Figure 4.2. EDS analysis of plastinated bamboo denoting Si content. The variation of silicon denotes a concentration gradient with lower concentration at the middle of the cross-section.

The micro-CT results could well distinguish the long fibre sap (Xylem) from the soft cellular tissues (Phloem) in bamboo, as shown in Figure 4.3. The white contrast in the plastinated bamboo micro-CT images (Figure 4.4) inside the Xylem tissue denotes an impregnated silicone mixture. This relates well to the idea of liquid uptake in bamboo since the Xylem is responsible for transport of water across the plant vertically. However, the non-uniform presence of these white spots in Xylem signifies improper impregnation. Comparing Figures 4.3 and 4.4., it is also interesting to note that dehydration (moisture removal) has assisted to obtain higher resolution (non-fuzzy) images.
Figure 4.3. X-ray micro-CT images of virgin bamboo. (a) XY plane (b) YZ plane (c) XZ plane. The white vertical bundles denote the Xylem while the cellular voids around denote Phloem.
Figure 4.4. X-ray micro-CT images of plastinated bamboo. (a) XY plane (b) YZ plane (c) XZ plane. Bright white contrast in vertical bundles denotes an impregnated silicone mixture.

4.1.2 Tensile tests

Table 4.2 shows the tensile properties of bamboo fibre bundles. The relatively large standard deviations recorded can be indicative of e.g. the natural variation of the cross-sectional area of the fibre culms (more formal hypothesis testing to follow below).

Table 4.2. Tensile properties of bamboo fibre bundles

<table>
<thead>
<tr>
<th>Bamboo Fibre bundles</th>
<th>Tensile Strength, MPa</th>
<th>Tensile Modulus, GPa</th>
<th>Strain</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean</td>
<td>60</td>
<td>6</td>
<td>0.058</td>
</tr>
<tr>
<td>Standard deviation</td>
<td>17</td>
<td>1.3</td>
<td>0.007</td>
</tr>
</tbody>
</table>
Tensile properties of plastinated and virgin bamboo are visually compared in Figures 4.7 and 4.8. From the data, it is apparent that with a 70% increase in tensile strength and a 23% increase in tensile modulus, plastination tends to add to the mechanical strength of bamboo. The data sets obtained for strength and modulus for various bamboo samples followed a normal distribution with all the data points lying over/along the line without any outliers, as can be seen in Figures 4.5 and 4.6. With the validation of the normal distribution assumption of the data sets, one-way ANOVA was conducted, which rejects the null hypothesis in the case of both tensile strength and modulus. Thus, it can be concluded that the effect of plastination on tensile strength and modulus of bamboo has been statistically significant at 5% significance level.

Figure 4.5. QQ plot for virgin bamboo tensile strength data points. All data points along the line denote normal distribution and no outliers.
Figure 4.6. QQ plot for plastinated bamboo tensile strength data points. All data points along the line denote normal distribution and no outliers.

Figure 4.7. Tensile strength bar graph for virgin and plastinated bamboo. *Significance level, p-value<0.05 (One-way ANOVA). Results show that plastination increases the tensile strength of virgin bamboo by 71% and this effect is statistically significant at 95% confidence level.
Figure 4.8. Tensile modulus bar graph for virgin and plastinated bamboo. *Significance level, p-value<0.05 (One-way ANOVA). Results show that plastination increases the tensile modulus of virgin bamboo by 23% and this effect is statistically significant at a 95% confidence level.

However, observing the decreased strain to failure shown in the stress-strain curves of plastinated and virgin bamboo in Figure 4.9, it is possible to hypothesize that after plastination, the impregnated silicone acts as a matrix between the bamboo fibres and occupies the voids in the hollow cellular structure, thus, sharing and distributing the load more uniformly. As the load approaches the failure limit, this binding matrix fails causing a sudden failure of the brittle bamboo fibres. With the increased strength and tensile modulus, the strain to failure drops by 47% in plastinated bamboo.
Figure 4.9. Tensile stress vs. longitudinal strain curves for plastinated and virgin bamboo showing increased tensile strength, modulus and decreased strain to failure of plastinated bamboo.

Further, slow-motion video analysis and the failed bamboo strips revealed that the outer side of the bamboo culm is mechanically stronger than the inner side. It may be further deduced from Figure 4.10, that fracture initiated on the inner side, towards the hollow part of the culm and propagated towards the center, leading to failure. The complete dislocation of the bamboo fibres on the inner side of the culm as compared to relatively lower damage on the outside further supports this finding. Similar results were also reported by Chen et. al. [38]. The major factors affecting the strength of bamboo are the density of vascular fibre bundles and fibre tissues. The number of vascular bundles and fibres constituting these bundles were greater on the outer part of the culm than the inner portion [38] (Figure 2.2).
Figure 4.10. Fractured tensile test specimen of plastinated bamboo. Lower vascular bundles on the inner side of bamboo result in dislocation/deformation of more fibres on the inner side during the tensile test.

4.1.3 Moisture tests

As discussed in section 3.1.3.3, plastinated samples should ideally not have any residual moisture content. Contrary to expectations, plastinated bamboo samples showed moisture levels, that were however lower than what was observed in the virgin bamboo, per set out in Figure 4.11. It is also apparent from this figure that the slope of water desorption i.e. the rate of moisture diffusivity constant is lower for plastinated bamboo. Overall, plastinated bamboo has about 13% lower
moisture content than virgin bamboo with a lower moisture diffusivity constant, denoted by the slope of water desorption during the initial 3 hours of testing.

![Figure 4.11](image.png)

**Figure 4.11.** Moisture desorption curves for virgin and plastinated bamboo. Lower residual moisture in plastinated bamboo with reduced moisture diffusivity constant as compared to virgin bamboo.

Interestingly, the samples from the ‘Three soak method’ did result in a slight decrease of residual moisture as shown in Figure 4.12. It can be thus suggested that not all the moisture has been removed from virgin bamboo during the acetone dehydration and it results in residual moisture content in plastinated samples. Prolonged exposure to acetone (in case of three soak method) dehydrates the bamboo slightly more, resulting in a 17% lower moisture content but does not result in complete dehydration.
To understand this further, Moso bamboo (with lower moisture content as compared to Inversa Bambusoide) was plastinated. Unexpectedly, the difference in moisture levels of virgin and plastinated bamboo was higher for Inversa Bambusoide (specie with higher initial moisture content) than Moso bamboo as shown in Figure 4.13. About 20% fall in moisture was observed for Inversa Bambusioide as compared to only 5.6% for Moso bamboo. It is possible, therefore, that higher initial moisture content has resulted in lower residual moisture content after plastination.
Figure 4.13. Residual moisture content among different bamboo species, before and after plastination showing that a higher moisture content before plastination results in a lower residual moisture after plastination.

4.2 Hybrid composite

4.2.1 Physical properties

Table 4.3 shows the final thickness and density of the regular glass fibre composite (GFRP) and bamboo-glass fibre composite (BGFRP). It is somewhat surprising that with a 21% increase in thickness and a 10% increase in mass, the final density of BGFRP is about 9% lower than GFRP. This result can be explained by the reasoning that adding the less dense bamboo fibres does increase the volume but with a relatively lower increase in mass as compared to glass fibres.

Table 4.3. Physical properties of GFRP and BGFRP

<table>
<thead>
<tr>
<th>Parameter</th>
<th>GFRP</th>
<th>BGFRP</th>
<th>% Relative change</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness, mm</td>
<td>1.90</td>
<td>2.30</td>
<td>21% (increase)</td>
</tr>
<tr>
<td>Density, kg/m³</td>
<td>1510</td>
<td>1370</td>
<td>6% (decrease)</td>
</tr>
</tbody>
</table>
4.2.2 Tensile test results

As can be seen from Figures 4.14 and 4.15, adding plastinated chopped bamboo results in lowering of tensile strength and chord modulus of elasticity. This decrease in strength and modulus by 15.4% and 2.7%, respectively, may be attributed to the decreased interfacial strength between fillers and the matrix. Since silicone acts as a poor interfacial agent [105], its presence inside and around the surface of plastinated bamboo leads to weaker interfacial strength. Also, randomly distributed bamboo fibres can add to the waviness of the fibre strands during thermoforming (discussed in upcoming section, 4.2.3), thus decreasing the strength further. The 10% standard deviation observed in case of GRRP could be due to varying number of yarns in the tensile test sample geometry that led to difference in failure load, with higher number of yarns failing at a higher load and vice versa. In case of BGFRP another factor causing uncertainty is a weakened interface. Most of these samples failed due to interlaminar delamination followed by fibre fracture, as shown in Table 4.4.

A non-parametric one-way ANOVA was conducted and it showed a significant difference between tensile strengths of GFRP and BGFRP sample, whereas no statistical difference was seen between tensile moduli of GFRP and BGFRP at 5% significance level, as depicted in Figures 4.14 and 4.15, respectively. This could be explained by a relatively larger change in the strength than in the modulus of BGFRP. Since tensile modulus is calculated within the small elastic deformation zone (i.e. the sample with no damage induced) and tensile strength is derived from the non-linear deformation zone of the behavior up to the catastrophic failure, it is likely that adding plastinated bamboo fillers can more affect the damage initiation of GFRP. This has been further discussed and visualized in the upcoming sections of this chapter.
Figure 4.14. Tensile strength bar graph for GFRP and BGFRP. *Significance level, p-value<0.05 (One-way non-parametric ANOVA). Results show that adding plastinated bamboo fillers to GFRP decreases the tensile strength of GFRP and this effect is statistically significant at 95% confidence level.
Figure 4.15. Chord modulus elasticity bar graph for GFRP and BGFRP. Comparison shows no statistical significance, p-value>0.05 (One-way non-parametric ANOVA). In other words, the results show that adding plastinated bamboo fillers to GFRP can only randomly decrease the tensile modulus of GFRP and the effect is not statistically significant at 95% confidence level.

It can be clearly seen from Figure 4.16 that BGFRP fails at a lower strength and strain due to decreased interfacial strength. A closer inspection of these curves also suggests that the matrix cracking in BGFRP starts at an earlier stage as compared to GFRP, as can be seen by the early shift in linear behaviour of BGFRP towards lower stiffness due to weaker interface and hence a poor load transfer compared to the GFRP. The slight fall in modulus observed in the stress-strain curve before the final fibre fracture marks the onset of matrix cracking [106].
Figure 4.16. Tensile stress vs. longitudinal strain curves for GFRP and BGFRP showing reduced tensile strength for BGFRP. Toe region denotes the fibre straightening/uncrimping region.

The test set-up optimization study, described in section 3.2.3.1, has somewhat resulted in deriving the desired failure modes for the tensile samples. Table 4.4 shows the failure modes of different tensile samples. The delamination failure in the gauge length of BGFRP further supports the reasoning of weakening interfacial strength.

Table 4.4. Failure modes observed in tensile test samples

<table>
<thead>
<tr>
<th>Material</th>
<th>Failure Mode</th>
<th>No. of sample(s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>GFRP</td>
<td>LGM</td>
<td>2</td>
</tr>
<tr>
<td>BGFRP</td>
<td>DGM</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>SGM</td>
<td>1</td>
</tr>
</tbody>
</table>

4.2.3 Flexural test results

As aforementioned, adding plastinated bamboo leads to a decreased interfacial strength in BGFRP, thus reducing the inter-layer shear strength of the laminate. Previous studies have shown that bending is a combination of tension, compression and shear [65]. The loss in shear strength of
BGFRP could be explained by weak interfacial strength. Adding plastinated bamboo results in lower bending strength and modulus as shown in Figure 4.17 and 4.18, respectively. However, non-parametric one-way ANOVA suggests that plastinated bamboo fillers significantly affect the flexural strength of GFRP, while modulus for these two materials is not statistically significant at 5% significance level. These results are similar to what was observed in the tensile tests results in section 4.2.2 and further reinforce the claim stated above, i.e. chopped plastinated bamboo fillers can significantly affect only the non-elastic behaviour of GFRP.

![Figure 4.17. Flexural strength bar graph for GFRP and BGFRP. * Significance level, p-value<0.05 (One-way non-parametric ANOVA). Results show that adding plastinated bamboo fillers decreases the flexural strength of GFRP and this effect is statistically significant at a 95% confidence interval.](image-url)
Figure 4.18. Flexural chord modulus of elasticity bar graph for GFRP and BGFRP. No statistical significance is observed between the two sample groups; p-value > 0.05 (One-way non-parametric ANOVA). In other words, the results show that adding plastinated bamboo fillers to GFRP can only randomly decrease the flexural modulus of GFRP and the effect is not statistically significant at 95% confidence interval.

Similar to tensile tests, the stress-strain curve for BGFRP bending samples show an accelerated matrix cracking, thus an early failure. The random orientation of bamboo fibres may contribute to the formation of voids and porosity, which would act as stress concentration spots. These also result in lower strength of BGFRP. The curves in Figure 4.19 also suggest that adding bamboo filler clearly adds to the randomness in the response of the material.
Figure 4.19. Flexural stress-strain curves for GFRP and BGFRP showing adding plastinated bamboo fillers to GFRP reduces the flexural properties and adds randomness to its response.

The micro-cracks on the surface of the GFRP sample can be observed in Figure 4.20. These cracks are due to brittle fractured fibres. Further, it is observed that these fractured fibres lie on the compressive side of the bending failed sample, thus failing in compression. In the case of BGFRP, a crack along the cross-section can be observed in Figure 4.21. This crack shows an interlaminar-delamination due to weak interface. Moreover, the compressive force on the BGFRP sample during bending is not as high as GFRP (due to lower failure load), hence significantly fractured fibres are not prominent and only micro-cracks are seen on the surface. However, fibre compression in BGFRP led to the formation of small kink bands on the surface as highlighted in Figure 4.21.
4.2.4 Fracture tests

Unlike unidirectional composites that normally show a monotonous trend during mode I fracture opening as depicted in Figure 4.22, woven composites seem to show jumps in the delamination front as shown in Figure 4.23. Further, the number of jumps observed depends on the number of
tows the crack has to propagate through, that are oriented perpendicular to the direction of crack propagation due to the 0/90 interface formed at the surface of the weave [107].

Figure 4.22. Monotonous trend observed in load-displacement curve for a typical unidirectional composite I DCB specimen [62].

Figure 4.23. Load-displacement curve for GFRP DCB specimen 5 showing INST (point of crack growth) and ARREST (point where the crack propagation stops) points during mode I fracture characterization of woven GFRP.
Previous research also suggests that since delamination surface is not straight, the obtained fracture toughness may not be pure mode-I. [107]. Few studies have defined this fracture behaviour exhibited by woven composites into 3 different points: INST where the sudden crack growth starts, ARREST where the crack propagation is stopped after load reduction and STABLE where a stable crack growth is observed [107-109]. In a study by Olave et. al. [107], the fracture toughness value was calculated at the INST position; i.e. the maximum load position so that the fracture toughness value is not underestimated. Moreover, for calculating the fracture toughness value, the second INST value was used for the calculation because the crack at the beginning of the delamination might be located anywhere on the surface. Figure 4.23 shows the load-extension curve obtained during mode I crack propagation, for one of the GFRP samples tested.

An interesting new observation in this study was failing of all the DCB woven composite test samples under bending and their excessive displacement as observed in Figure 4.24 and 4.25. To control this effect and perform a meaningful test, after the flexural failure was observed initially in the first DCB sample with 4 Twintex layers, the thickness of the sample i.e. number of laminate plies were increased to increment the flexural strength of the material system and prohibit the excessive bending. Further, the pre-crack length in the sample was also reduced to avoid large deflection and the undesired failures [62]. A six-layer GFRP DCB sample failed in the same fashion as the four layers, after which the pre-crack length was reduced to half to further reduce the bending moment on the cantilevers. Failure of this sample suggested a further increase in the bending stiffens to withstand the high bending moment during crack propagation. For this, 12 layers of Twintex were used to prepare a DCB sample. Surprisingly, this sample failed in the same manner as others due to bending resulting in excessive displacement. To decrease the bending
moment, the pre-crack length was again reduced from 50 to 22.5 mm. However, no difference was observed this time and the sample failed again due to bending, as seen in Figure 4.25.

Figure 4.24. Flexural failure of a 4-layer GFRP DCB specimen during mode I fracture testing.
Per the trend observed above, we can thus infer that the energy required to propagate the crack through transverse tow (fracture toughness at transverse tows) exceeds the energy required to cause flexural failure of one or both specimen arms (beams). Thus, we see a flexural failure of the cantilevers during crack propagation.

A high tow size increases the peak load i.e. the INST value due to the increased size of the unit cell. Further, it was observed by Olave et al. [107] that significant fibre birding is observed during delamination in woven composites with high tow sizes. In the present research, even with a tow size of 3642, the large diameter of glass fibres increased the unit cell size, therefore an enhanced interlocking at the interface. These factors may have led to failure of very thick test samples. Samples with a further increase in Twintex layers could not be developed and tested due to sample manufacturing and preparation limitations.
Hence, for calculating fracture toughness, sample 5 was selected since it has the toughness value relatively closest to the existing literature \([107]\). To compensate for the excessive displacement and since the observed ratio of Instron head displacement to crack length was greater than 0.4 in all cases, a correction factor (equation 3.11) was used. Table 4.5 shows fracture toughness for sample 5, before and after the correction factor was applied.

<table>
<thead>
<tr>
<th></th>
<th>Calculated experimental value</th>
<th>Fracture toughness (KJ/m(^2))</th>
</tr>
</thead>
<tbody>
<tr>
<td>Before correction</td>
<td>12.96</td>
<td>10.5</td>
</tr>
<tr>
<td>After correction</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Remark:** Due to these undesired failures in GFRP (woven composite) DCB specimens under mode I fracture testing, the BGFRP specimen could not be successfully tested for this mode. A tapered DCB geometry can be used to resolve the issue, where the flexural failure of the beam would not precede crack propagation. Such geometry can potentially enhance the flexural stiffness that allows crack propagation without excessive bending \([110]\). In the present study, however, such a geometry modification could not be adopted due to limitations in the thermoforming process of woven composites at our laboratory.

Figure 4.26 shows SEM images of the fractured cross-section to analyze fibre and matrix fractography. In this case, the warp fibre tows lie parallel to the crack propagation direction. Figure 4.26 (a) shows fibre imprints in the matrix and fibre bundle pull outs in warp direction illustrating tensile failure of consolidated fibres. Further, Figure 4.26 (b) illustrates weft fibre roving debonded from matrix with some matrix debris scattered. These selectively shown fibre/matrix rich
regions at the interface would account for high variation in the peak values (INST positions). Also, fibre splitting is evident and fibre bridging can be observed from Figure 4.26 (b).

![Figure 4.26. Post fracture SEM fractography of the GFRP DCB specimen; (a) Fibre imprints in matrix denoting fibre pull out, (b) Matrix de-bonded split out fibres due to fibre bridging.](image)

The split glass fibre in Figure 4.27 (a) still has the matrix coating, which signifies good fibre-matrix adhesion. The cross-section of this fibre has a serrated fracture indicative of a brittle fracture and the riverlines observed on the matrix surface denote isotropic matrix failure. The decrease in cross-section of the split fibre can be accounted to localised fibre necking due to tension. As can be seen in Figure 4.27 (b), the resin pockets and agglomerates observed on the cracked morphology denote unstable crack growth [62], which along with split out fibres (fibre bridging) contribute towards the non-linearity and periodic peaks in loading (stiffness) during delamination.
4.2.5 Impact tests

As discussed in section 3.2.3.4, the GFRP and BGFRP impact specimens were subjected to different impact energies due to variations in their thickness. In industries like aeronautical and automotive, the high-performance structural materials are required to have high specific properties (density-based). High structural efficiency has numerous advantages including pay-load maximization, fuel-saving, and extended fleet autonomy [111]. Tarpani et al. conducted a similar impact toughness comparison study for different carbon-reinforced polymer fibre materials and divided the impact energy with the respective density of each material to obtain specific impact toughness [111]. Therefore, the output impact parameters in this research were normalized with the density of each material to compare their impact properties.

Figure 4.27. Post fracture SEM fractography of the GFRP DCB specimen; (a) Stand-alone fractured fibre, denoting typical fibre and matrix fractography, (b) Fractured split out fibres because of tensile failure.
Figure 4.28 (a) illustrates the impact energy per unit density (specific impact energy) of impactor for the two samples. The energy dissipated by each material is shown by the non-zero curve ending for energy in each case. This absorbed energy per unit density is 0.429 Jcm$^3$/g for BGFRP and 0.115 Jcm$^3$/g for GFRP, as denoted by the higher curve ending for BGFRP than GFRP. Thus, the dissipated energy caused more damage in BGFRP than GFRP because the more the energy absorbed by the material, more severe is the damage [65]. Hence, it may be concluded that adding bamboo increases the energy absorbance in GFRP. It may also be noted from Figure 4.28 (b) and (c) that BGFRP has shown a higher contact force; this could be due to the added stiffness in BGFRP caused by thicker sample size. Further normalizing the contact force by density of BGFRP (less than density of GFRP) increased the overall contact force values, contrary to GFRP which decreased (due to a higher density than BGFRP). A higher contact duration is representative of higher structural stiffness [112]. In this case, GFRP has higher contact duration, even with a thinner laminate denoting much higher stiffness.

Moving average method is extensively used for data filtering and extrapolation of the impact test data points obtained, in order to smooth the excessive oscillations [113]. In this study, the running average technique was used, as shown in Figure 4.29 to characterize various stages of the impact on the BGFRP sample. As depicted in Figure 4.29, until the initial kink in the force-time curve, the elastic zone is observed. This zone corresponds to an approximate linear behaviour after which the damage is initiated i.e. the matrix cracking. The force recorded at this point is called Critical Force. Matrix cracking is responsible for the numerous small oscillations seen on this time series curve. As we move towards the maximum load, oscillations of similar amplitude can be observed, denoting brittle fibre fracture as a result of bending. The peak load represents maximum load the laminate can tolerate before undergoing major deformations. Beyond the peak load, the crack
propagates as the laminas fracture and delaminate, and the impact tup perforates into the sample [112-113].

Figure 4.28. Impact response data observed for GFRP and BGFRP (a) Specific impact energy-time, (b) Specific contact force-displacement, (c) Specific contact force-time.
Figure 4.29. Impact response for BGFRP denoting various damage modes during low velocity impact testing.

Furthermore, as seen in section 4.2.3, adding plastinated bamboo fillers to GFRP reduces the flexural elastic modulus. The present impact test results reinforce this finding, as the increased energy absorption of BGFRP signifies reduced stiffness and increased displacement. Whereas, in the case of GFRP, a higher bending modulus obtained during quasi-static bending (section 4.2.3) means stiffer flexural behaviour and thus, lesser damage-induced energy absorption. In general, it seems that adding plastinated bamboo to GFRP can result in a less dense material with increased energy absorption but decreased damage resistance (i.e. showing extended damage zones).

An interesting trend observed in this impact study was an open loop for force-displacement curve, as seen in Figure 4.28 (b). The loop is a representative of spring-back in the impact specimen after impact. A possible explanation of this could be that the deformation levels recorded for present samples are high due to a lower level of stiffness contrary to thick and stiffer samples with lower displacement and no perforation. The contact time of the tup is majorly utilized in measuring the displacement as the sample undergoes excessive bending while displacing downwards and the tup
perforates into the sample. By the time the sample rebounds, the tup has left contact without measuring the upward displacement. A side study was conducted with thicker samples (8 and 12-layer laminates) of Twintex to confirm that this ‘spring back’ is typical of stiffer samples since there is no perforation, thus reinforcing the above hypothesis.

From a macroscopic/visual inspection of post impact samples of GFRP and BGFRP (Figure 4.30), it is apparent that multiple cracks have propagated radially in different directions from the point of impact in BGFRP due to higher energy absorbed with respect to a single macro crack seen in GFRP due to lower energy absorbed.

![Figure 4.30. Post impact specimens; (a) Singular micro crack in GFRP, (b) Multiples macro cracks in BGFRP seem to spread from point of impact in various directions.](image)

SEM images in Figure 4.31 show significant cavities formed in the impacted cross-section of the BGFRP sample. Fractured and bent fibres, in the direction of impact, can also be seen. A closer analysis of the cross-section reveals interlaminar delamination and fractured bamboo. Multiple
spots of intralaminar decohesion in the glass fibre strands can be observed in Figure 4.31 (b). The highlighted cracks (red dotted curves) show micro-crack propagation along the bamboo-glass fibre interface. The cracks propagate along the bamboo-glass fibre interface because energy dissipation increases across layers of dissimilar materials [105]. And the more compliant randomly distributed bamboo decreases the global stiffness, thus, increasing the maximum displacement and leading to higher energy absorption. However, the laminas further down the bamboo layer do not seem to be much damaged because tensile failure is not much prominent in the lower layers.
Figure 4. 31. SEM microstructure images of post impact BGFRP specimen; (a) Numerous voids, fractured glass fibres and bamboo denote notable damage, (b) Crack propagation (red dotted curve) along bamboo-glass fibre interface, (c) Glass fibre tow farthest from the impact surface with least damage.
However, the microstructure of GFRP shows signs of lesser damage. In Figure 4.32, we can see localised fracture of fibre bundles located in the plane of impact. Cavities can be observed on the cross-section but not as extensive as in BGFRP. Both inter and intra-laminar de-cohesion spots can be seen within a smaller region, and the reduced number of micro-cracks point out the reasoning that lower energy absorption by GFRP leads to lower damage. Thus, the microstructure images of BGFRP and GFRP samples reinforce the hypothesis of higher structural damage in BGFRP as compared to GFRP, due to higher energy absorption capacity.

![Figure 4.32. SEM microstructure image of post impact GFRP specimen with smaller voids and less fractured glass fibres denoting lesser damage than BGFRP.](image)

Two replicates of GFRP and BGFRP were tested under the same test conditions. It can be noticed in the force-time series curves for the BGFRP samples that after reaching an identical value of the
critical load, one of the replicates has a higher peak load followed by a consistent difference in the response between the two samples. Similar to BGFRP, the peak load for the two GFRP replicates is slightly different but the curve for the damage propagation stage is similar and coincides. And the standard deviation for the specific contact force between GFRP samples is 0.2% of the mean value, while for BGFRP is 5% of the mean, as shown in Figure 4.33. This difference in the dynamic response behaviour of the BGFRP samples is likely to be related to the randomness caused by adding arbitrarily oriented plastinated bamboo fibres, as was also seen earlier in section 4.2.3.

The results from this impact study showed a statistical significance in a one-way non-parametric ANOVA. Figure 4.34 and 4.35 illustrates that both, specific impact energy and specific peak contact force of GFRP were significantly changed after the addition of plastinated bamboo fillers. Since energy absorbed and peak force in an impact depend upon the plastic behaviour, these results also fortify the hypothesis stated in tensile and bending tests in sections 4.2.2 and 4.2.3 respectively, per which adding plastinated bamboo fibres significantly alters the damage response of GFRP.

![Figure 4.33. (a) Contact force-time curves for BGFRP samples, (b) Contact force-time curves for GFRP samples.](image-url)
Figure 4.34. Impact energy per unit density bar graph for GFRP and BGFRP. * Significance level, p-value<0.05 (One-way non-parametric ANOVA). Results confirm that adding plastinated bamboo fillers increases the energy absorption ability of GFRP at a 95% confidence interval.

Figure 4.35. Peak-force per unit density bar graph for GFRP and BGFRP. *Significance level, p-value<0.05 (One-way non-parametric ANOVA). Results confirm that adding plastinated bamboo fillers increases the peak-force during impact in GFRP at a 95% confidence interval.
4.3 Environmental conditioning

4.3.1 Characterization of conditioned bamboo

4.3.1.1 Density results

Figure 4.36 demonstrates that both virgin and plastinated bamboo have a density increase, post moisture conditioning. This increase in density can be attributed to water absorption owing to the hydrophilic nature of bamboo. Lesser increase in density of plastinated bamboo in comparison to virgin denotes the reduced moisture up taking capability of plastinated bamboo. This, however, also denotes water being present in the internal voids of the cellular structure of plastinated bamboo, besides the presence of silicone at those sites. This discrepancy could be attributed to the water transport through silicone which led to water molecules reach the bamboo cell wall and interact with it.

![Figure 4.36](image.png)

**Figure 4.36.** Density of virgin and plastinated bamboo, before and after moisture conditioning showing a higher increase in density of virgin bamboo than plastinated bamboo.
4.3.1.2 Moisture test results

Figure 4.37 shows the experimental data on the moisture content of plastinated and virgin bamboo with an average moisture content of 109% for virgin bamboo. Surprisingly, plastinated bamboo seems to absorb moisture during the water bath with an average moisture content of 95%. As aforementioned in the density results section, a possible reason for this moisture gain could be due to the transport of water through silicone to reach bamboo cell wall. This led to an increase in the bound water and increased transport of bound and free water in bamboo cells as they absorbed water. Further, a smaller desorption slope for plastinated bamboo denotes a lower permeability rate of water absorbed and trapped within the cellular bamboo and silicone. A closer inspection of Figure 4.37 reveals that moisture desorption of plastinated bamboo may be a two-step process where more than half of the water is lost within the first three hours, followed by a gradual loss until equilibrium.

![Figure 4.37](image)

Figure 4.37. Weight fraction of water desorbed by virgin and plastinated bamboo vs. drying time in the oven showing lower moisture absorbed by plastinated bamboo.
4.3.1.3 Tensile tests

Table 4.6 shows that moisture conditioning led to a 51.3% and 50.7% loss in strength of virgin and plastinated bamboo respectively. These changes denote that both plastinated and virgin bamboo are affected by moisture conditioning. Such reduction in strength of plastinated bamboo could be attributed to water transport through the silicone and degrading the bamboo cell wall as water acts as a plasticizer to cellulose. However, Table 4.6 also shows that not only plastination increases the tensile strength of unconditioned bamboo, even after moisture conditioning the tensile strength of plastinated bamboo is still 73% higher than virgin bamboo. Thus, it can be concluded that although moisture conditioning decreases the tensile strength of plastinated bamboo, plastinated bamboo still seems to be a much durable option overall as it can support greater loads under moisture.

<table>
<thead>
<tr>
<th></th>
<th>Virgin bamboo (MPa)</th>
<th>Plastinated bamboo (MPa)</th>
<th>% Relative change</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unconditioned</td>
<td>128.5</td>
<td>219.5</td>
<td>70.8% (increase)</td>
</tr>
<tr>
<td>bamboo</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Conditioned</td>
<td>62.5</td>
<td>108.3</td>
<td>73.3% (increase)</td>
</tr>
<tr>
<td>bamboo</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>% Relative</td>
<td>51.3% (decrease)</td>
<td>50.7% (decrease)</td>
<td></td>
</tr>
<tr>
<td>change</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Figure 4.38 and 4.39 show the QQ plots for moist virgin and plastinated bamboo tensile strength data sets, respectively, and they validate the normality assumption since the data points lie over/closely by the line without any outliers. For statistical analysis, a two-way ANOVA was conducted on the tensile strength and modulus and data sets with the bamboo type (plastinated or virgin) and conditioning type (dry or moist) as two factors with two levels each. As a part of the
Post-hoc analysis, Tukey’s test was carried out on the statistically significant factors to compare and analyze various level means and their individual significance.

Figure 4.38. QQ plot for moist virgin bamboo tensile strength data points. All data points along the line denote normal distribution and no outliers.

Figure 4.39. QQ plot for moist plastinated bamboo tensile strength data points. All data points along the line denote normal distribution and no outliers.
The results suggest that both these factors along with their interaction are statistically significant at a 5% significance level, as seen in Figure 4.40. Plastination significantly increases the strength of bamboo, and moisture conditioning significantly decreases the strength of plastinated and virgin bamboo. Interestingly, the interaction of these factors suggest that the strength of moist virgin bamboo was significantly lower than dry plastinated bamboo which could be reason to plasticization of cellulose in moist bamboo and no silicone present to uniformly distribute the load. Therefore, following plastination, a significant increase in the tensile strength and moisture durability of bamboo can be observed.
Figure 4.40. Tensile strength of virgin and plastinated bamboo, before and after moisture conditioning. *Significance level, P<0.05 (Two-sided t-test). Results denote that both study factors i.e. bamboo type and conditioning are statistically significant as well as their interaction. Tukey’s test further reported that the individual factors make all pairwise comparisons of samples to be significantly different, while the interaction factor only made the mean values of moist virgin bamboo and dry plastinated bamboo sample to be significantly different at a 95% confidence interval.

Although the decrease in tensile strength of conditioned virgin and plastinated bamboo is similar, a cross-comparison study from Table 4.6 illustrates that the final strength of conditioned plastinated bamboo is about 82% of the strength of virgin unconditioned bamboo. The tensile strength of virgin conditioned bamboo is only 28.4% of the tensile strength of unconditioned plastinated bamboo, which is statistically significant. Thus, even after moisture conditioning at elevated temperature, plastinated bamboo retains 82% of the tensile strength of original unconditioned bamboo.
In terms of modulus, however, the fall in stiffness of conditioned plastinated bamboo is much lower at 38%, as compared to 51% for virgin bamboo as shown in Table 4.7, as a result of plasticization. Moreover, the observation that conditioned plastinated bamboo provides a 55% higher modulus than conditioned virgin, definitely deems plastination suitable for bamboo processing to enhance its strength and modulus in both moisture conditioned and unconditioned environments. The results obtained for a two-way ANOVA on the tensile modulus datasets were very similar to what was observed for tensile strength, as shown in Figure 4.41. It can be seen that moisture conditioning significantly affects the tensile modulus of plastinated and virgin bamboo and that, the modulus of moist plastinated bamboo is significantly different from moist virgin bamboo at 5% significance level.

<table>
<thead>
<tr>
<th>Material type</th>
<th>Virgin bamboo (GPa)</th>
<th>Plastinated bamboo (GPa)</th>
<th>% Relative change</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unconditioned</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>bamboo</td>
<td>9.6</td>
<td>11.8</td>
<td>22.9% (increase)</td>
</tr>
<tr>
<td>Conditioned</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>bamboo</td>
<td>4.7</td>
<td>7.3</td>
<td>55.3% (decrease)</td>
</tr>
<tr>
<td>% Relative</td>
<td>51% (decrease)</td>
<td>38.1% (decrease)</td>
<td></td>
</tr>
<tr>
<td>change</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Figure 4.41. Tensile modulus of virgin and plastinated bamboo, before and after moisture conditioning. *Significance level, p<0.05. Results denote that both the factors i.e. bamboo type and conditioning are statistically significant as well as their interaction at a 95% confidence interval.

Further, a cross-comparison of modulus from Table 4.7 reveals that moistened plastinated bamboo has about 23% lower modulus than unconditioned virgin bamboo while this percentage is 60% in case of virgin conditioned and unconditioned plastinated bamboo. Conditioned plastinated bamboo retains 77% of the stiffness of original bamboo as opposed to only 49% for conditioned virgin bamboo.

The fall in strength and modulus of bamboo after conditioning may be explained by the fact that water presence dramatically softens the cell walls. During the plasticization of cellulose present in bamboo is affected by water, the hydrogen bonds between different polymer chains in cellulose can break. Subsequently, hydrogen bonds form with water instead, as they are small, polar molecules and hence can penetrate into the polymer chains. These water molecules form hydrogen
bonds with cellulose, stronger than those existing between cellulose and cellulose. This softens the cellulose micro-fibrils as they are no longer so strongly bonded to each other, which results in decreased stiffness of bamboo. And as the water expands the cell wall, there are also fewer cellulose micro-fibrils per unit area. Hence the strength of the bamboo decreases [114].

The increased strain to failure of conditioned plastinated bamboo in Figure 4.42 denotes the plasticization of the semi-crystalline cellulose by water which decreases the tensile strength and modulus with an increased elongation while failure due to increased ductility.

![Tensile stress vs. longitudinal strain curves for plastinated bamboo, before and conditioning showing increased ductility after moisture conditioning.](image)

**Figure 4.42.** Tensile stress vs. longitudinal strain curves for plastinated bamboo, before and conditioning showing increased ductility after moisture conditioning.

### 4.3.2 Characterization of conditioned Composites

#### 4.3.2.1 Density

Figure 4.43 illustrates that the maximum density increase was realized for BGFRP_V by 13.5%.

For the unconditioned samples, BGFRP_V has the lowest density due to the presence of virgin bamboo fillers, which tend to have the least density in comparison to glass fibres and plastinated
bamboo. However, after conditioning, BGFRP_V has higher moisture absorption than GFRP and BGFRP, because of the higher content of hydrophilic bamboo fillers. Moreover, the density of virgin bamboo is less than plastinated bamboo, due to which more virgin fibres can be added for the same mass percentage in BGFRP_V.

![Density bar-graph for different composites, before and after moisture conditioning.](image)

**Figure 4.43.** Density bar-graph for different composites, before and after moisture conditioning.

Further, the density increase may be correlated to weight gain due to moisture absorption in these composites. The weight gain in the case of BGFRP_V is maximum, about 5% as shown in Figure 4.44, owing to highest fibre content. These results confirm the association between moisture uptake and natural fibre content, per which the percentage of moisture uptake increases as the fibre volume fraction increases due to the high cellulose content [84].
In the case of GFRP, the 4.4% increase in density could be attributed to the degradation/leaching of the interfacial agent and water filling the interfacial region between glass fibres and polypropylene. The polymer matrix, in this case, was also degraded in terms of cracking and void formation due to water attack at elevated temperature. The glass fibres may also tend to dissolve by a small amount in water at elevated temperatures. These results reflect those of Shiota et al. [84] and Baillie et al. [83] that water at an elevated temperature causes degradation of the hydrophobic polymer matrix, leaching of interfacial agents and some dissolution of glass fibres leading to an increased weight as well. Additionally, these degradation mechanisms also contribute to density and weight increase of BGFRP and BGFRP_V, since they constitute GFRP as well. The highest weight gain in BGFRP_V can be accounted for the highest volume fraction of fibres.

4.3.2.2 Tensile tests

Figure 4.45 shows the comparison of the tensile strength of different conditioned and unconditioned composites with a maximum reduction in the strength for BGFRP by 38% after
conditioning, followed by 28% and 26% for BGFRP_V and GFRP, respectively. The 26% standard deviation observed in the strength of conditioned BGFRP could be due to the varying number of fibre yarns in the test specimens, as discussed before in section 4.2.2. The reason for loss in strength of GFRP and partly in the case of BGFRP and BGFRP_V could be due to the leaching of the interfacial agent and slight degradation of polypropylene matrix. Different swelling and shrinkage rates for fibres and matrix may lead to a weaker interfacial strength as well. The lower tensile strength of BGFRP than BGFRP_V could be due to the weakest interfacial strength of BGFRP as compared to the other composite samples, as a result of moisture and silicone presence at the interface. Thwe and Liao had reported similar results of matrix and natural fibre degradation during moisture conditioning. Additionally, they also mentioned about stress corrosion cracking of glass fibres that was observed during long term exposure (1600 h) to moisture [45].

A two-way non-parametric ANOVA was conducted on the tensile test data sets with filler type having three levels (no-bamboo, plastinated bamboo and virgin bamboo), and the conditioning factor having two levels (dry and moist). The results denoted that both factors are significant while their interaction is not at 5% significance level. Figure 4.45 shows the results from a non-parametric ANOVA, illustrating that adding virgin and plastinated bamboo fillers to GFRP significantly affects the composite tensile strength. However, there is no significant difference in the strengths of BGFRP and BGFRP_V in both dry and moist conditions (derived from Tukey’s pairwise comparison), clearly visible from the bar plots as well. The ANOVA also reported that the moisture conditioning produces a statistically significant difference between all the three composite materials; i.e. GFRP, BGFRP and BGFRP_V.
Figure 4.45. Tensile strength variation for different tested composites, before and after moisture conditioning. *Significance level, \( p<0.05 \). Results show that fibre type and conditioning are overall statistically significant factors, but their interaction is not at a 95% confidence interval.

Figure 4.46 shows that the maximum fall in modulus can be observed in the case of BGFRP, which could be because of the weak interface of fibers and the matrix due to the presence of silicone. The higher modulus observed in the unconditioned BGFRP than BGFRP_V could be due to the presence of stiffer plastinated bamboo in BGFRP. Interestingly, the higher mean modulus value was observed in the case of moist BGFRP_V, compared to the moist BGFRP, which could be due to the large error (standard deviations), or higher volume of fibre content in the BGFRP_V as discussed previously. The large standard deviation observed in the tensile modulus for most of these composite samples could also be due to the straightening/uncrimping effect of the woven fibres that crumple during the consolidation process, adding uncertainty or noise to the modulus (response).

In this case, the filler type was the only overall significant factor seen through ANOVA. No statistical significance was observed between GFRP and BGFRP samples at 5% significance level.
The non-significance in modulus of dry and moist GFRP by adding plastinated bamboo fillers relates to the hypothesis discussed in section 4.3.1.3, that plastinated bamboo does not significantly affect the elastic properties of GFRP in dry and low moisture conditions (moisture absorbed less than 5%). However, the significant difference in the moduli of GFRP and BGFRP_V denotes that bamboo does affect the elastic properties of GFRP significantly. Based on these findings, we may infer that virgin bamboo affects both elastic and damage properties of GFRP, while plastinated bamboo only alters the damage properties. This could be due to the higher modulus of plastinated bamboo fillers, which does not lead to such reduction in the modulus of GFRP as in the case of adding virgin bamboo fillers to GFRP.

Figure 4.46. Tensile modulus variation for different tested composites, before and after moisture conditioning. *Significance level, p<0.05. Results show that only fibre type is a statistically significant factor at a 95% confidence level.

Figure 4.47 (a) shows SEM images of the polymer matrix of a dry GFRP sample post tensile testing where the polymer matrix seems well consolidated, without much damage or voids. However, Figure 4.47 (b), (c) clearly depicts the degradation of polypropylene caused at elevated
temperature. These figures support the idea that exposure to water at high temperature leads to the cracking of matrix and voids. This leads to active water transportation through these cracks, leading to a Non-Fickian behaviour at elevated temperatures.
Figure 4.47. Post tensile test SEM images; (a) Unconditioned GFRP sample with well-consolidated matrix, (b) Conditioned GFRP matrix with micro-cracks and voids that lead to Non-Fickian behaviour of matrix material, (c) Cracked and flaked matrix in conditioned GFRP sample.

These results corroborate the idea of Dhakal et. al [82], who suggested that water at an elevated temperature significantly alters the moisture absorption in polymer matrix composites due to moisture-induced degradation.

Figure 4.48 illustrates polymer matrix adhered to the fractured glass fibres and broke along with the fibres. These matrix coated fibres and fibre bundles show strong interfacial strength in unconditioned composites. However, it can be observed from Figure 4.49, that only certain resin regions adhere to the fractured glass fibres after conditioning. The loss of adherence in this conditioned composite supports the idea of leaching of the interfacial agent during moisture conditioning, which leads to a weaker interface. These results are also in agreement with those obtained by Shakeri and Ghasemian [86].
Figure 4.48. Post tensile test SEM images of unconditioned GFRP; (a) Fractured glass fibres coated with polypropylene matrix, (b) fibre bundles adhered together with the matrix, signifying high interfacial strength.

Figure 4.49. Post tensile test SEM images of conditioned GFRP; (a) Fractured glass fibres with minimal/no polypropylene matrix on surface, (b) fibre bundles without any matrix, signifying weak interfacial strength.
4.3.2.3 Flexural tests

The bar plots in Figure 4.50 illustrate that the flexural strengths of GFRP and BGFRP decrease after conditioning. Both materials have the same interfacial agent which dissolves/leaches by water, leaving behind voids at the fibre-polymer interface, leading to poor stress transfer during loading. The polypropylene matrix absorbs some water at elevated temperature and develops voids and cracks, leading to a Non-Fickian moisture absorption. Glass is not affected by elevated water except for some dissolution while bamboo absorbs significant moisture owing to its hydrophilic nature. This causes moisture swelling in bamboo, thus affecting the interfacial shear strength. This may be the reason that we see a higher fall in the flexural strength of BGFRP than GFRP. Thus, the effect of moisture on the flexural strength of GFRP and BGFRP is statistically significant.

![Figure 4.50. Flexural strength variation for different composite samples, before and after moisture conditioning. *Significance level, p<0.05. Results show that fibre type and conditioning are statistically significant factors, but their interaction is not at a 95% confidence interval.](image-url)
One unexpected finding in this part of the study was a seemingly small increase of average flexural modulus of the BGFRP after moisture conditioning of about 5%, as seen in Figure 4.51; even though the hypothesis testing between the sample groups did not show statistical significance via the non-parametric ANOVA with $\alpha=5\%$ (the statistical test would be significant at higher risk levels $\sim10\%$). Theoretically, the flexural modulus should decrease markedly at sufficiently high levels of moisture absorption [83]. Interestingly, Stamboulis et. al. [83] similar to our case, observed an initial small increase in flexural modulus followed by its decrease with increasing the moisture content. The above observations in the flexural modulus variation as a function of moisture level are further speculated through Figures 4.52 and 4.53. It is worth noting that the flexural test results are also deemed aligned with the inference made from the tensile test results in section 4.3.1.3, where plastinated bamboo did not significantly affect the elastic behaviour of GFRP between the dry and low-moisture conditioned samples.

![Flexural modulus variation](image)

**Figure 4.51.** Flexural modulus variation for different composite sample, before and after moisture conditioning. Results show that there is no factor that significantly affects (at 95% confidence interval) the flexural modulus of GFRP and BGFRP, neither fibre type, nor the conditioning.
Figure 4.52. Illustrative reasoning for the increase in bending modulus of BGFRP below 5% moisture absorption (present case).

Figure 4.53. Illustrative reasoning for the decrease in bending stiffness of BGFRP above 5% moisture absorption, as observed in [83].
Chapter 5: Conclusion and Future Work Recommendations

5.1 Summary

Natural fibres with their high strength to weight ratios and negligible carbon footprint as compared to synthetic fibres continue to provide high potential for various structural and non-structural applications. Cost-effectiveness is another aspect as to why these fibres are gaining much attention in the industry today. A problem with natural fibres, however, is their bio-degradability. The degradation of the bio-fibres is accelerated exponentially if they are subjected to a moist environment. Overcoming the bio-degradation phenomenon and the hydrophilic tendency of these fibres requires an alteration in the fundamental (cellular) structure of the base material, in a way that it increases/does not reduce the required mechanical performance.

The present thesis was designed to study the feasibility of applying a plastination technique to natural fibres and analyzing their mechanical performance, with the motivation to prevent their degradation. A search of the literature revealed the lack of existing work on the application of plastination to any field beyond animal and human body organs. Chapter 3 presented a step-by-step process to plastinate a select natural fiber (here bamboo) along with its characterization to understand the effect of plastination on both physical and mechanical properties as well as environmental durability. The chapter also discussed the design and characterization of a hybrid glass fibre-bamboo polypropylene composite that was explored for static and dynamic properties along with its environmental performance.

Next, chapter 4 proved the effect of plastination on increased tensile properties and enhanced moisture durability of bamboo. Adding plastinated bamboo to a GFRP composite also resulted in specific desirable changes in the behaviour of the ensuing hybrid material system. The micro-level
investigation of the specimens, along with statistical analyses, further reinforced the macro-findings of the aforementioned tests. A summary of the specific conclusions drawn from these chapters is as follows.

*Investigation of plastination feasibility of bamboo and its characterization*

- It was shown that plastination could be successfully applied to bamboo natural fibres, with a much shorter duration than commonly plastinated specimens including animal and human body organs. With a 57% increase in density after plastination, silicone replaces some water present in bamboo and also occupies empty cellular voids in bamboo.

- Using the current technique, comparing the virgin and plastinated bamboo samples for moisture content suggested that bamboo after plastination still retains some moisture, but lower than that of virgin bamboo. The experiments also confirmed that higher moisture content in virgin bamboo before plastination results in lower residual moisture after plastination. Further, it was perceived that increasing the acetone dehydration duration also results in lower residual moisture after plastination.

- A customized tensile test method revealed that plastination tends to add to the tensile strength of bamboo, by almost 70% for the bamboo species used herein. The stiffness of bamboo also increased by 22%.

- Another significant finding was the moisture absorption by plastinated bamboo. Bamboo impregnated with silicone tends to have a lower moisture diffusivity constant. The micro-CT results from plastinated bamboo revealed the non-uniform distribution of silicone inside bamboo.
Design and characterization of hybrid composite:

- Adding the virgin and plastinated bamboo fillers to the glass fibre-polypropylene composite resulted in a lower density hybrid composite. These experiments denote that plastinated and virgin bamboo fillers reduce the tensile and flexural strengths of the GFRP composite due to weaker natural fiber/matrix interface. These experiments confirmed that adding plastinated bamboo fillers does not change the elastic behaviour of GFRP notably, but it alters the non-elastic/damage response in dry and low moisture conditions. Virgin bamboo, on the other hand, decreases both elastic and non-elastic properties of GFRP, which could be attributed to the much lower stiffness of virgin bamboo as compared to BGFRP.

- The investigation of mode I fracture energy for GFRP showed an unusual behaviour (unstable deformation) during the standard DCB test, which could be related to the bidirectional fibre tows. This, as a result, caused unusual non-linearities in the load-displacement curve and premature failure of the test specimens. Accordingly, the laminate thickness of GFRP samples was adjusted to avoid the above unstable deformation; though the method could still not be applied to the BGFRP samples.

- The study identified that adding plastinated bamboo increase the impact absorbance of GFRP. Namely, the reduced stiffness of BGFRP by means of adding plastinated bamboo increases specific impact energy absorbance, while the damage resistance in the samples decreases.
• The results revealed that the composite with a higher volume fraction of natural fibres has a higher water absorption capacity. The tensile and bending properties of the hybrid composites were reduced after moisture conditioning, except for bending stiffness of BGFRP_P, which increased slightly due to possibly swelling of the virgin bamboo fibres.

5.2 Main contributions to knowledge

• The study for the first time showed the feasibility of applying plastination S-10 technique to natural fibres. Plastination proved to enhance the mechanical properties of bamboo while also improving its environmental durability.

• It was interestingly found that the plastinated bamboo specimens, with the S-10 technique, yield a residual moisture content which decreases with the “increase” in the initial moisture content of the virgin bamboo to be plastinated. Also, we found that successive acetone dehydration, i.e. increased exposure to highly concentrated acetone (three-soak method), can decrease the residual moisture content in plastinated samples.

• It was shown for the first time that adding plastinated bamboo fibre as fillers to a typical fibreglass woven composite can improve select properties of the new hybrid (natural fiber-synthesis fiber) material system. In particular, it resulted in a less dense engineering material with an improved impact energy absorption capacity; which can be of high interest e.g. for helmet/shock-sensitive sport gears manufacturing applications.

5.3 Limitations to work

• Being limited to bamboo, this study lacks the application of the plastination technique to other natural fibres.

• A major limitation of this study is the small sample size of the hybrid composites in all the mechanical tests. Thus, the generalisability of these results may be subject to certain limitations.
• The scope of the study is limited to just tensile testing of virgin and plastinated bamboo, due to a lack of standardized test codes to characterize different mechanical properties of bamboo.

• One source of weakness in this study which could have affected the plastination process was the measurement of acetone concentration during the dehydration process using an acetonometer. A more robust technique to verify the dehydration of natural fibres could be developed.

5.4 Future work

The observations during testing and analyses in this research could outline the following future research suggestions:

• A main question left for most beneficial application of plastination, is the residual moisture content observed in plastinated bamboo using the standard S-10 technique. This could be further assessed by exploring the possible use of other polymers than silicone. The material selection criteria could be a polymer with a high hydrophobic tendency, low/minimal water permeability, low specific gravity, low viscosity and low flammability. Polymer solutions that qualify the most of these criteria should be checked for plastination feasibility of natural fibres. A multi-criteria decision making approach could be used to select the best available polymer for such process optimization.

• Although the major polymer used in the plastination study i.e. PDMS is physically and chemically inert, thermally stable and non-toxic, a life cycle study to access the non-environmentally friendly aspect of plastinating natural fibres could be worthwhile. More
broadly, the effect of plastinated natural fibres on sustainability and pertinent human health aspects could be better understood in the future.

- Information on how plastinated natural fibres behave under microbial and termite attack, alkaline and acidic environments could be another area of interest for future work.
- Extending plastination technique to other natural fibres, like hemp and jute, can be worthwhile e.g. for use with thermoplastic matrices like PP/PET.
- Further research might also examine how plastination affects other mechanical properties of bamboo and other natural fibres, like compressive strength, torsional rigidity and creep. Exploring the flammability properties of plastinated bamboo and the effect of extended high-temperature exposure could be another area of interest.
- A further investigation using the design of experiments (DOE) would be helpful to optimize the process parameters of the plastination, e.g. the dehydration time, impregnation pressure, etc.
- The issue of the flexural failure of woven composite (GFRP) under fracture testing was deemed intriguing and could be explored in more detail. Woven GFRP could also be tested for mode II and mixed-mode fracture to analyze if the same flexural failure is observed.
- A study involving unidirectional bamboo fibres in a regular glass fibre composite could be studied to analyze how these long natural fibres affect the mechanical, environmental and chemical properties of the GFRP.
Bibliography


for nonparametric factorial analyses using only anova procedures,” 2011, p. 143.


Appendices

Appendix A
This appendix includes the failure codes assigned to the GFRP and BGFRP tensile test specimens using ASTM D3039 [96]. The failure modes are denoted by three characters as per Figure A.1. (a). The GFRP specimens failed as LGM (Figure A.1. (b)) while failure mode observed in BGFRP was DGM.

![Table of Failure Codes](image1.png)

![Failure Modes](image2.png)

Figure A.1. Tensile test failure codes [96]; (a) Nomenclature to denote various failures. (b) Common failure modes observed in present study.
Appendix B

This appendix includes the method used to conduct the non-parametric statistical tests using the **Aligned Rank Transform method** to align and rank the data sets, following which the parametric ANOVA can be applied [103]. In data alignment, the estimated effects of the independent variables are “stripped” from the response variable so that the effects of all variables are removed except the one being tested for [103].

In the present study, the datasets from experiments were aligned and ranked using the ARTool [103]. As an example, Table B.1. shows data set from the tensile strength test results from section 4.3.2.2, that was prepared in the format pre-requisite and fed to the ARTool. The following data had two factors:

1. Natural Fibre type: No natural fibres (N), Plastinated bamboo (P), and Virgin bamboo (V)
2. Specimen Condition: Dry (D) and Moist (M).
Table B.1. Tensile test raw dataset used for processing in ARTool

<table>
<thead>
<tr>
<th>Material</th>
<th>Fibre</th>
<th>Condition</th>
<th>Tensile strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>GFRP</td>
<td>N</td>
<td>D</td>
<td>216.8</td>
</tr>
<tr>
<td>GFRP</td>
<td>N</td>
<td>D</td>
<td>214</td>
</tr>
<tr>
<td>GFRP_M</td>
<td>N</td>
<td>M</td>
<td>169.8</td>
</tr>
<tr>
<td>GFRP_M</td>
<td>N</td>
<td>M</td>
<td>159.18</td>
</tr>
<tr>
<td>BGFRP</td>
<td>P</td>
<td>D</td>
<td>203.62</td>
</tr>
<tr>
<td>BGFRP</td>
<td>P</td>
<td>D</td>
<td>180</td>
</tr>
<tr>
<td>BGFRP_M</td>
<td>P</td>
<td>M</td>
<td>140.5</td>
</tr>
<tr>
<td>BGFRP_M</td>
<td>P</td>
<td>M</td>
<td>96.9</td>
</tr>
<tr>
<td>BGFRP_V</td>
<td>V</td>
<td>D</td>
<td>192.6</td>
</tr>
<tr>
<td>BGFRP_V</td>
<td>V</td>
<td>D</td>
<td>196</td>
</tr>
<tr>
<td>BGFRP_VM</td>
<td>V</td>
<td>M</td>
<td>145.8</td>
</tr>
<tr>
<td>BGFRP_VM</td>
<td>V</td>
<td>M</td>
<td>137.1</td>
</tr>
</tbody>
</table>

The ARtool aligned and ranked the data sets as follows:

Step 1. Computing residuals for each response.

Step 2. Computing estimated effects for all main and interactions effects

Step 3. Compute aligned response

Step 4. Assign averaged ranks to the aligned response

Lastly, a full-factorial ANOVA on assigned ranks (ART* column in Table B.2.) was performed on each factor and their interaction separately to analyze the significance of the process variables involved.
Table B.2. Data found by the ARTool containing aligned and ranked columns for factor ‘Fibre’

<table>
<thead>
<tr>
<th>Material</th>
<th>Fibre</th>
<th>Condition</th>
<th>Tensile strength (MPa)</th>
<th>Aligned(Tensile strength) for Fibre</th>
<th>ART* (Tensile strength) for Fibre</th>
</tr>
</thead>
<tbody>
<tr>
<td>GFRP</td>
<td>N</td>
<td>D</td>
<td>216.8</td>
<td>20.32</td>
<td>11</td>
</tr>
<tr>
<td>GFRP</td>
<td>N</td>
<td>D</td>
<td>214</td>
<td>17.52</td>
<td>10</td>
</tr>
<tr>
<td>GFRP_M</td>
<td>N</td>
<td>M</td>
<td>169.8</td>
<td>24.23</td>
<td>12</td>
</tr>
<tr>
<td>GFRP_M</td>
<td>N</td>
<td>M</td>
<td>159.18</td>
<td>13.61</td>
<td>9</td>
</tr>
<tr>
<td>BGFRP</td>
<td>P</td>
<td>D</td>
<td>203.62</td>
<td>-3.96</td>
<td>5</td>
</tr>
<tr>
<td>BGFRP</td>
<td>P</td>
<td>D</td>
<td>180</td>
<td>-27.58</td>
<td>2</td>
</tr>
<tr>
<td>BGFRP_M</td>
<td>P</td>
<td>M</td>
<td>140.5</td>
<td>6.03</td>
<td>8</td>
</tr>
<tr>
<td>BGFRP_M</td>
<td>P</td>
<td>M</td>
<td>96.9</td>
<td>-37.57</td>
<td>1</td>
</tr>
<tr>
<td>BGFRP_V</td>
<td>V</td>
<td>D</td>
<td>192.6</td>
<td>-4.85</td>
<td>4</td>
</tr>
<tr>
<td>BGFRP_V</td>
<td>V</td>
<td>D</td>
<td>196</td>
<td>-1.45</td>
<td>6</td>
</tr>
<tr>
<td>BGFRP_VM</td>
<td>V</td>
<td>M</td>
<td>145.8</td>
<td>1.2</td>
<td>7</td>
</tr>
<tr>
<td>BGFRP_VM</td>
<td>V</td>
<td>M</td>
<td>137.1</td>
<td>-7.5</td>
<td>3</td>
</tr>
</tbody>
</table>