

**STUDY ON CREEP BEHAVIOUR OF PLANT  
FIBRE/EPOXY COMPOSITES**

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# **STUDY ON CREEP BEHAVIOUR OF PLANT FIBRE/EPOXY COMPOSITES**

*by*

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*Dedicated to my parents, parent in-laws, sister, nephew,  
husband, who have always been a constant source of support  
and encouragement in my life.*

## Certificate

This is to certify that the thesis entitled “Study on Creep Behaviour of Plant Fibre/Epoxy Composites” being submitted by Debarati Bhattacharyya to the Indian Institute of Technology Delhi for the award of the degree of Doctor of Philosophy is a record of bona fide research work carried out by her. Debarati has worked under my guidance and supervision and has fulfilled the requirements for submitting this thesis, which to our knowledge has reached the requisite standard. The results contained in this thesis are original and have not been submitted, in part or full, to any other university or institute for the award of any other degree or diploma.

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**Debarati Bhattacharyya**

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Debarati Bhattacharyya

# Abstract

Recently, the plant-based textile structural composites gained importance because of their greater strength-to-weight ratio, high corrosion resistance, and lower maintenance. The plant fibres are gradually replacing the synthetic fibres as reinforcements for the development of environment friendly products (door trim panels, engine and transmission enclosures, etc.) However, the plant fibres have few limitations over synthetic fibres especially in textile structural composites (subsea equipment, airplane and the oil gas industry, etc.), where they need to demonstrate superior long term mechanical performance to cyclic (fatigue) as well as long term constant load (creep) situations in harsh environments of varying temperature and humidity levels. This could result in dimensional instability and even structural failure of composites due to poor creep properties. In this thesis, we try to understand the creep behaviour of plant fibre-based textile structural composites during their service lifetime and exposure to aggressive environments conditions.

In the first study, the surface of jute fabrics was modified by alkali, ozone, and plasma treatments to remove the non-cellulosic materials and thereby improve their adhesion with epoxy matrix. The performance of surface treatments was evaluated based on surface morphology, mechanical properties, hydrophilicity, etc. The ozone surface treatment was found to remove lignin and increase hydrophilicity of jute fibres to greater extent as compared to other treatments, however with higher tendency of defibrillation and fibre rupture.

In the second study, the effect of alkali, ozone, and plasma surface treatments on the variability of the tensile strength of jute fibres was characterized by the Weibull analysis. It was found that the shape parameter (i.e. Weibull modulus) of surface treated jute fibres was higher than that of the untreated jute fibres. The plasma and ozone treatments of jute fibres

showed best results in terms of high Weibull modulus, low standard deviation and higher coefficient of correlation ( $R^2$ ).

In the third study, the creep behaviour of jute fabric/epoxy composites at different environment temperatures was studied using several creep models (i.e., Burger's model, Findley's power law model, and Coupling Model). Ozone-treated samples demonstrated higher creep resistance than alkali- and untreated samples over most of the temperature range, whereas alkali-treated fibres exhibited superior behaviour at lower temperatures. Furthermore, the ozone treated composites shown higher elastic deformation occurring instantaneously at elevated temperatures of 70 °C and 100 °C, whereas less amount of deformation occurring due to the viscous regions as compared to alkali-treated jute fabric composites. The various creep models suggested that the improvement in fibre/matrix interface is very important to ensure longer life of natural fibre-based composites.

In the fourth study, the effect of surface treatment and subsequent separation of individual cellulose micro/nano fibrils from jute, corn husk and sisal plant fibres were studied. The ozone pre-treatment followed by pulverization in ball milling was selected for this purpose. The effect of ozone treatment was examined from morphological, physical, and mechanical properties using FTIR, XRD, SEM, TGA, etc techniques. The tensile strength of the fibres weakened gradually as the treatment time of ozonisation increased. The maximum drop in tensile strength was seen in case of corn husk fibres followed by sisal and jute fibres after the ozone treatments. The tensile strength was reduced by 70 %, 60 %, and 47 % in case of corn husk, sisal, and jute fibres respectively after 15 min of ozone treatment. When pulverization of plant fibres was carried out in ball milling, the ozone treatment was observed beneficial for easier defibrillation of cellulose micro/nano fibrils. The pulverization of jute, corn husk and sisal fibres produced the particles with diameter of 610 nm, 380 nm, and 530 nm after 15 min of ozone treatment respectively.

In the fifth study, the creep performance of ozone treated jute fabric/epoxy composites was studied at different environment temperatures after incorporation of 1, 3 and 5 wt. % of ozonized and pulverized cellulose particles. The incorporation of cellulose particles was found to lower the creep deformation of jute fabric/epoxy composites. The creep resistance of 5 wt.% filler loaded composites was found superior, especially at higher temperatures. The higher elastic deformation and slower viscous deformation was found in case of filler loaded composites at elevated temperatures of 70 °C and 100 °C. The various creep models were used to estimate the durability of composites by validating the obtained creep results. The coupling model predicted the creep behaviour of composites more accurately in addition to explanation on the creep mechanism.

## सारांश

हाल ही में पौधों पर आधारित वस्त्र संरचनात्मक सम्मिश्र ने अपने उच्च बल-भार अनुपात, अधिक क्षरण प्रतिरोध तथा न्यूनतम रखरखाव के कारण महत्त्व प्राप्त किया है। कृत्रिम रेशों के स्थान पर पर्यावरण-अनुकूल उत्पादों (जैसे दरवाजे के पैनल, इंजन एवं ट्रांसमिशन आवरण आदि) के विकास हेतु पौधों के रेशों का उपयोग बढ़ रहा है। तथापि, इन रेशों की कुछ सीमाएँ भी हैं, विशेषकर वस्त्र संरचनात्मक सम्मिश्रों में (जैसे सबसागर उपकरण, विमानन, तथा तेल-गैस उद्योग), जहाँ दीर्घकालिक यांत्रिक गुणधर्मों—चक्रीय थकान तथा विरूपण व्यवहार का कठोर परिवेश (भिन्न तापमान व आर्द्रता) में स्थायित्व आवश्यक होता है। इस कारण विरूपण गुणधर्म की कमी आयामी अस्थिरता अथवा संरचनात्मक विफलता उत्पन्न कर सकती है। प्रस्तुत शोध-कार्य में पौध-आधारित वस्त्र संरचनात्मक सम्मिश्रों की विरूपण प्रवृत्ति को उनकी कार्यावधि एवं आक्रामक वातावरणीय स्थितियों के अंतर्गत समझने का प्रयास किया गया है।

प्रथम अध्ययन में, जूट वस्त्र की सतह को क्षार उपचार, ओज़ोन उपचार तथा प्लाज़्मा उपचार द्वारा संशोधित किया गया जिससे गैर-सेलूलोसिक पदार्थ हटाकर एपॉक्सी मैट्रिक्स से आसंजन सुधारा जा सके। सतह संशोधन का आकलन सतही संरचना, यांत्रिक गुणधर्म एवं जलाभिलाषिता के आधार पर किया गया। ओज़ोन उपचार ने लिग्निन हटाने व जलाभिलाषिता बढ़ाने में सर्वाधिक प्रभावी परिणाम दिए, किंतु इसके साथ रेशा-वियोजन तथा रेशा-विच्छेदन की प्रवृत्ति भी अधिक पाई गई।

द्वितीय अध्ययन में, उपरोक्त सतह उपचारों के बाद जूट रेशों की तन्यता शक्ति की परिवर्तनशीलता का मूल्यांकन वेइबुल विश्लेषण से किया गया। सतह-उपचारित रेशों का वेइबुल गुणांक असंशोधित रेशों से अधिक पाया गया। प्लाज़्मा एवं ओज़ोन उपचारित रेशों ने उच्च वेइबुल गुणांक, निम्न मानक विचलन तथा उच्च सहसंबंध गुणांक ( $R^2$ ) प्रदर्शित किया।

तृतीय अध्ययन में, विभिन्न तापमानों पर जूट वस्त्र/एपॉक्सी सम्मिश्रों के सरीजन व्यवहार का परीक्षण बर्गर मॉडल, फाईंडले की घात नियम मॉडल तथा युग्मन मॉडल द्वारा किया गया। ओज़ोन-उपचारित नमूनों ने

प्रायः सम्पूर्ण तापमान सीमा पर सर्वाधिक विरूपण प्रतिरोध दर्शाई, जबकि क्षार-उपचारित रेशों का प्रदर्शन निम्न तापमान पर श्रेष्ठ पाया गया। उच्च तापमान (70 °C तथा 100 °C) पर ओज़ोन-उपचारित सम्मिश्रों में अधिक तात्कालिक लोचदार विकृति तो देखी गई, किंतु श्यान विकृति अपेक्षाकृत कम पाई गई। अध्ययन से स्पष्ट हुआ कि रेशा/मैट्रिक्स अंतरफलक में सुधार प्राकृतिक रेशा-आधारित सम्मिश्रों की दीर्घायु हेतु अनिवार्य है।

चतुर्थ अध्ययन में, सतह उपचार एवं तत्पश्चात् जूट, मक्का भूसी तथा सिसल रेशों से व्यक्तिगत सेल्यूलोज़ सूक्ष्म/नैनो रेशिकाएँ पृथक्करण का अध्ययन किया गया। इसके लिए ओज़ोन प्री-उपचार एवं गेंद पीसाई का प्रयोग किया गया। फ़ोरियर रूपांतर अवरक्त वर्णक्रमिकी, एक्स-रे विवर्तन, स्कैनिंग इलेक्ट्रॉन माइक्रोस्कोपी, तथा थर्मोग्रैविमेट्रिक विश्लेषण, आदि तकनीकों द्वारा सतही एवं यांत्रिक गुणधर्मों का मूल्यांकन किया गया। ओज़ोन उपचार समय बढ़ने पर तन्यता शक्ति क्रमशः घटती गई, जिसमें अधिकतम कमी मक्का भूसी (70%), तत्पश्चात् सिसल (60%) एवं जूट (47%) में 15 मिनट उपचार उपरांत देखी गई। साथ ही, ओज़ोन उपचार ने रेशिकाओं के आसान वियोजन में सहायक भूमिका निभाई।

पंचम अध्ययन में, ओज़ोन-उपचारित जूट वस्त्र/एपॉक्सी सम्मिश्रों में 1, 3 तथा 5 भार प्रतिशत ओज़ोनित एवं गेंद-पीसाई सेल्यूलोज़ कणों को सम्मिलित कर विभिन्न तापमानों पर विरूपण प्रदर्शन का परीक्षण किया गया। कणों की उपस्थिति से सरीजन विकृति में कमी देखी गई। 5 भार प्रतिशत युक्त सम्मिश्रों ने विशेषतः उच्च तापमानों पर श्रेष्ठ सरीजन प्रतिरोध प्रदर्शित किया। इन नमूनों में उच्च लोचदार विकृति तथा धीमी श्यान विकृति पाई गई। युग्मन मॉडल ने सरीजन व्यवहार का सर्वाधिक सटीक पूर्वानुमान प्रस्तुत किया एवं विरूपण तंत्र की व्याख्या भी की।

# Table of Contents

Abstract.....	i
सारांश.....	iv
Table of Contents.....	vi
List of Figures.....	xi
List of Tables.....	xv
Abbreviations.....	xviii
List of Symbols.....	xxi
<b>CHAPTER 1. INTRODUCTION.....</b>	<b>1</b>
<b>CHAPTER 2. LITERATURE REVIEW.....</b>	<b>5</b>
2.1 Plant Fibres.....	6
2.1.1 Structure of plant fibres.....	9
2.1.2 Limitations of plant fibres.....	10
2.2 Surface modification of plant fibres.....	10
2.2.1 Chemical treatments of plant fibre.....	11
2.2.1.1 Peroxide treatment.....	11
2.2.1.2 Alkali treatment.....	11
2.2.1.3 Acetylation.....	13
2.2.1.4 Isocyanate treatment.....	14
2.2.1.5 Benzoylation.....	15
2.2.1.6 Permanganate treatment.....	15
2.2.1.7 Silane treatment.....	16

2.2.2 Physical treatment of plant fibres.....	16
2.2.2.1 Plasma treatment.....	17
2.2.2.2 Steam explosion.....	18
2.2.2.3 Ozone treatment.....	19
2.2.3 Biological treatment.....	19
2.2.3.1 Enzyme treatment.....	20
2.2.3.2 Fungi treatment.....	20
2.3 Creep properties of plant fibre composites.....	22
2.4 Recent Developments in plant fibres.....	27
2.4.1 Acid hydrolysis.....	29
2.4.2 TEMPO oxidation.....	30
2.4.3 Cryocrushing.....	31
2.4.4 Multiple mechanical shearing.....	31
2.4.5 High shear grinding.....	32
2.4.6 High pressure homogenization.....	32
2.4.7 Microwave irradiation.....	33
2.4.8 Ultrasonic treatment.....	33
2.4.9 Enzymatic hydrolysis.....	34
2.4.10 Ball milling.....	34
2.5 Creep behaviour of plant fibre composites with nanocellulose as fillers.....	36

<b>CHAPTER 3. RESEARCH GAPS.....</b>	<b>60</b>
<b>CHAPTER 4. RESEARCH OBJECTIVES.....</b>	<b>63</b>
<b>CHAPTER 5. STUDY ON THE SURFACE MODIFICATION OF JUTE FIBRES BY ALKALI, OZONE, AND PLASMA TREATMENTS.....</b>	<b>67</b>
5.1 Introduction.....	67
5.2 Materials and Methods.....	68
5.2.1 Materials.....	68
5.2.2 Alkali treatment of jute fabric.....	68
5.2.3 Ozone treatment of jute fabric.....	68
5.2.4 Plasma treatment of jute fabric.....	69
5.3 Characterization of properties of jute fabrics after treatments.....	69
5.4 Results and Discussions.....	72
5.4.1 Surface microstructure of jute.....	72
5.4.2 XRD analysis of jute.....	75
5.4.3 FTIR analysis of jute.....	77
5.4.4 Mechanical properties of jute yarns.....	79
5.4.5 Hydrophilic properties of jute fibre.....	80
5.4.6 Thermogravimetric analysis of jute yarns.....	85
5.5 Conclusions.....	87
<b>CHAPTER 6. STUDY ON THE EFFECTIVENESS OF SURFACE TREATMENTS OF JUTE FIBRES ON INTERFACIAL PROPERTIES OF EPOXY COMPOSITES.....</b>	<b>96</b>
6.1 Introduction.....	96
6.2 Materials and Methods.....	97
6.3 Results and Discussions.....	98

6.3.1	Weibull analysis of tensile strength of jute fibres.....	98
6.3.2	Weibull analysis of interfacial shear strength of jute fibre/epoxy composites.....	101
6.4	Conclusions.....	107
<b>CHAPTER 7. STUDY ON THE SHORT-TERM CREEP BEHAVIOUR OF OZONE TREATED JUTE FABRIC/EPOXY COMPOSITES.....</b>		<b>110</b>
7.1	Introduction.....	110
7.2	Materials and Methods.....	111
7.2.1	Fabrication of composites.....	111
7.2.2	Creep testing of composites.....	111
7.3	Results and Discussions.....	114
7.3.1	Creep behaviour of jute fabric/epoxy composites.....	114
7.4	Conclusions.....	120
<b>CHAPTER 8. STUDY ON THE EFFECT OF OZONE TREATMENT ON PULVERIZATION OF DIFFERENT PLANT FIBRES.....</b>		<b>123</b>
8.1	Introduction.....	123
8.2	Materials and Methods.....	123
8.2.1	Materials.....	123
8.2.2	Ball Milling of ozone treated plant fibres.....	124
8.2.3	Characterisation of pulverized fibres.....	124
8.3	Results and Discussions.....	124
8.3.1	Surface microstructure of plant fibres.....	124
8.3.2	XRD analysis of plant fibres.....	128
8.3.3	FTIR analysis of plant fibres.....	130

8.3.4	Mechanical properties of plant fibres.....	132
8.3.5	Hydrophilic properties of plant fibres.....	134
8.3.6	Lightness values of plant fibres.....	135
8.3.7	Pulverization of plant fibres.....	137
8.4	Conclusions.....	147
<b>CHAPTER 9. STUDY ON THE EFFECT OF INCORPORATION OF PULVERIZED CELLULOSE PARTICLES ON THE SHORT-TERM CREEP BEHAVIOUR OF OZONE TREATED JUTE FABRIC/EPOXY COMPOSITES.....</b>		<b>151</b>
9.1	Introduction.....	151
9.2	Results and Discussions.....	151
9.2.1	Creep behaviour of jute fabric/epoxy composites.....	151
9.2.2	Nanoidentation of jute fabric/epoxy composites.....	157
9.2.3	Interfacial shear properties of jute fabric/epoxy composites.....	165
9.3	Conclusions.....	167
<b>CHAPTER 10. CONCLUSIONS AND FUTURE SCOPE OF WORK.....</b>		<b>170</b>
10.1	Conclusions.....	170
10.2	Future Scope of Work.....	172
<b>ANNEXURE.....</b>		<b>173</b>
<b>CURRICULUM VITAE.....</b>		<b>185</b>

## List of Figures

Figure 1.1. Global market demand of natural fibres from the year 2014 to 2024.....	2
Figure 1.2. Natural fibre composite market revenue globally from the year 2010 to 2020.....	2
Figure. 2.1. Classification of natural fibres.....	5
Figure. 2.2. Properties of plant fibres according to their constituents.....	8
Figure. 2.3. Chemical structure of cellulose.....	9
Figure. 2.4. Structure of cellulose fibre cell.....	10
Figure. 2.5. Typical structure of cellulosic fibre.....	12
Figure. 2.6: Mechanism of atmospheric pressure plasma process.....	18
Figure. 2.7. (a) Creep deformation at a constant stress, (b) response to corresponding strain.....	23
Figure. 2.8. Stages occurring during creep deformation.....	23
Figure 2.9: Creep deformation of UD flax fibre composite in fibre direction.....	26
Figure 2.10. Creep behaviour of composites (a) at 30°C (b) at 60°C.....	26
Figure 2.11. Generic flow charts of obtaining nanocellulose through different steps .....	29
Figure 2.12. Schematic representation of nanocellulose extraction by acid hydrolysis.....	30
Figure 2.13. Mechanism of cellulose oxidation to form nanocellulose using different chemicals TEMPO, NaBr and NaClO .....	31
Figure 2.14. (a) Creep behavior of sisal/hemp composites before weathering, (b) Creep behavior of hybrid composites after weathering.....	39

Figure 2.15. Creep curves of various cellulosic and non-cellulosic filler-loaded composites compared with neat epoxy.....	40
Figure 5.1. Surface morphology of untreated and surface treated jute fabric.....	74
Figure 5.2. Crystalline structure of jute fibres before and after treatments.....	77
Figure 5.3. FTIR spectra of jute fibres before and after treatments.....	78
Figure 5.4. Load-elongation curve of the jute yarns before and after treatments.....	80
Figure 5.5. Color of jute fibres before and after treatments.....	85
Figure 5.6. Thermal degradation of jute yarns before and after treatments.....	87
Figure 6.1. Graph showing the Weibull plots for tenacity of untreated and surface treated jute yarns.....	101
Figure 6.2. Measurement of interfacial shear strength of jute.....	102
Figure 6.3. Maximum load required to debond the reinforcement from the matrix.....	103
Figure 6.4. Graph showing the Weibull plots for tenacity of untreated and surface treated jute yarns.....	105
Figure 6.5. Fractured surfaces of composites after microbead pull out test.....	106
Figure 7.1. Creep behaviour of jute fabric/epoxy composites by different models.....	118
Figure 8.1. Surface microstructure of Jute fibres after ozone treatment.....	125
Figure 8.2. Surface microstructure of Cornhusk fibres after ozone treatment.....	126
Figure 8.3. Surface microstructure of Sisal fibres after ozone treatment.....	127

Figure 8.4. Crystalline structure of plant fibres after ozone treatment.....	130
Figure 8.5. Chemical structure of plant fibres after ozone treatment.....	132
Figure 8.6. Single fibre strength of plant fibres after ozone treatment.....	134
Figure 8.7. Change in colour of plant fibres after ozone treatment.....	136
Figure 8.8. Particle size distribution of pulverized plant fibres before and after ozone treatment.....	138
Figure 8.9. Effect of ozone treatment on pulverization of jute fibres.....	140
Figure 8.10. Effect of ozone treatment on pulverization of cornhusk fibres.....	141
Figure 8.11. Effect of ozone treatment on pulverization of sisal fibres.....	142
Figure 8.12. Schematic of extraction of cellulose microfibrils by combined action of ozone treatment and ball milling.....	144
Figure 9.1. Creep behaviour of jute fabric/epoxy composites validated by different models at 40 °C.....	155
Figure 9.2. Creep behaviour of jute fabric/epoxy composites validated by different models at 70 °C.....	156
Figure 9.3. Creep behaviour of jute fabric/epoxy composites validated by different models at 100 °C.....	157
Figure 9.4. Load penetration depth curves of various filler loaded composites.....	159
Figure 9.5. Spread of interfacial properties of cornhusk filler loaded composites at different spacing.....	160

Figure 9.6. Spread of interfacial properties of jute filler loaded composites at different spacing.....	160
Figure 9.7. Spread of interfacial properties of sisal filler loaded composites at different spacing.....	161
Figure 9.8. Indentation marks on cornhusk filler loaded surface of composites.....	162
Figure 9.9. Indentation marks on jute filler loaded surface of composites.....	163
Figure 9.10. Indentation marks on sisal filler loaded surface of composites.....	164
Figure 9.11. Maximum load required to debond the jute yarns from the epoxy matrix.....	166

## List of Tables

Table 2.1. Properties of commonly used fibres in composites.....	6
Table 2.2. Chemical composition of plant fibres.....	7
Table 2.3. Different criteria for various surface treatments.....	21
Table. 2.4. Comparison of mechanical properties of organic and inorganic fillers.....	27
Table 2.5. Principle, advantages and disadvantages of various nanocellulose extraction techniques.....	35
Table 5.1. Chemical contents of jute fibres before and after treatments.....	75
Table 5.2. Crystallinity of jute fibres before and after treatments.....	76
Table 5.3. Tensile properties of jute yarns before and after treatments.....	80
Table 5.4. Contact angle and surface free energy of jute yarns before and after treatments.....	83
Table 5.5. Wicking properties of jute yarns before and after treatments.....	84
Table 6.1. Tensile strength and Weibull distribution for untreated and surface treated jute yarns.....	99
Table 6.2. Forces required to debond the fibres from the matrix.....	103
Table 6.3. Interfacial shear stress and Weibull distribution for untreated and surface-treated jute yarns and epoxy.....	104
Table 7.1. Estimation of creep parameters of untreated jute fabric/epoxy composites.....	118

Table 7.2. Estimation of creep parameters of alkali treated jute fabric/epoxy composites...	119
Table 7.3. Estimation of creep parameters of ozone treated jute fabric/epoxy composites.....	120
Table 8.1. Determined chemical contents of plant fibres before and after ozone treatment.....	128
Table 8.2. Effect of ozone treatment on crystallinity of plant fibres.....	129
Table 8.3. Effect of ozone treatment on mechanical properties of plant fibres.....	134
Table 8.4. Contact angle and surface free energy of plant fibres before and after ozone treatment.....	135
Table 8.5. Effect of ozone treatment on colour strength values of plant fibres.....	137
Table 8.6. Particle size results and required stress for pulverization plant fibres before and after ozone treatment.....	139
Table 8.7. Effect of ozone treatment on required stress levels to fracture plant fibres.....	143
Table 8.8. Summary of all the properties obtained for both untreated and ozone treated plant fibres .....	145
Table 9.1. Estimation of creep parameters of jute fabric/epoxy composites filled with 1 wt% fillers at 40°C.....	173
Table 9.2. Estimation of creep parameters of jute fabric/epoxy composites filled with 3 wt% fillers at 40°C.....	174
Table 9.3. Estimation of creep parameters of jute fabric/epoxy composites filled with 5 wt% at 40°C.....	175

Table 9.4. Estimation of creep parameters of jute fabric/epoxy composites filled with 1 wt% fillers at 70°C.....	176
Table 9.5. Estimation of creep parameters of jute fabric/epoxy composites filled with 3 wt% fillers at 70°C.....	177
Table 9.6. Estimation of creep parameters of jute fabric/epoxy composites filled with 5 wt% fillers at 70°C.....	179
Table 9.7. Estimation of creep parameters of jute fabric/epoxy composites filled with 1 wt% fillers at 100°C.....	180
Table 9.8. Estimation of creep parameters of jute fabric/epoxy composites filled with 3 wt% fillers at 100°C.....	181
Table 9.9. Estimation of creep parameters of jute fabric/epoxy composites filled with 5 wt% fillers at 100°C.....	183
Table 9.10. Nanoindentation characteristics of various composites.....	164
Table 9.11. Interfacial shear strength of jute fabric/epoxy composites with different filler loadings.....	167

## Abbreviations

<b>ASTM</b>	American Society for Testing and Materials
<b>AJF</b>	Alkali treated jute fabric
<b>AJY+E</b>	Alkali treated jute yarn +epoxy
<b>BNC</b>	Bacterial Nanofibrillated Cellulose
<b>CGAR</b>	Compound Annual Growth Rate
<b>CNC</b>	Nanocrystals
<b>CNF</b>	Nanofibre
<b>EPI</b>	Ends per Inch
<b>E<sub>0</sub></b>	Intial Modulus
<b>E<sub>e</sub></b>	Equilibrium Modulus
<b>FTIR</b>	Fourier Transform Infrared Spectroscopy
<b>FESEM</b>	Fourier Scanning Electron Microscope
<b>HDPE</b>	High Density Polyethylene
<b>K/S</b>	Colour Strength
<b>L</b>	Lightness
<b>MCC</b>	Microcrystalline Cellulose
<b>m</b>	Weibull Modulus
<b>NFC</b>	Nanofibrillated Cellulose
<b>NCC</b>	Nanocrystalline Cellulose
<b>NaBr</b>	Sodium Bromide

<b>NaClO</b>	Sodium Hypochlorite
<b>NPP</b>	Neat Polypropylene
<b>O<sub>3</sub></b>	Ozone
<b>OJF</b>	Ozone treated fabric
<b>OJY+E</b>	Ozone treated jute yarn + epoxy
<b>OTJF, OTCHF OTSF</b>	Ozone treated jute fibres, corn husk fibres and sisal fibres
<b>PBAT</b>	Polybutylene adipate terephthalate
<b>PPI</b>	Picks per Inch
<b>PVC</b>	Polyvinyl Chloride
<b>PJF</b>	Plasma treated fabric
<b>PVA</b>	Polyvinyl Alcohol
<b>PJY+E</b>	Plasma treated jute yarn +epoxy
<b>R<sup>2</sup></b>	Coefficient of Determination
<b>RHA</b>	Rice Husk Ash
<b>RPP</b>	Recycled polypropylene
<b>SEM</b>	Scanning Electron Microscopy
<b>TEMPO</b>	2,2,6,6-tetramethylpiperidine-1-oxyl
<b>TGA</b>	Thermogravimetric Analysis
<b>UTM</b>	Universal Testing Machine
<b>UJF</b>	Untreated Jute Fabric

**UJY+E** Untreated jute yarn + epoxy

**XRD** X-ray Diffraction

## List of Symbols

$\%$	Percentage
$\chi_c$	Crystallinity
$\gamma_l^d, \gamma_l^p$	Surface energies of liquid dispersive and polar components
$\gamma_s^d, \gamma_s^p$	Surface energies of solid dispersive and polar components
$\gamma_s$	Surface free energy of solid
$F(x)$	Cumulative distribution function of Weibull distribution
$R(x)$	Probability of survival of the variable $x$ , reliability
$\tau$	Interfacial shear force
$\varepsilon(t)$	Strain at a given time
$\varepsilon_0$	Instantaneous strain.
$\sigma_0$	Applied stress
$E_M, E_K$	Elastic modulus of Maxwell Spring and Kelvin Spring
$\eta$	Viscosity of dashpot
$\varepsilon_M$	Constant value of creep with time,
$\varepsilon_K$	Earliest saturated value of creep in short time
$\varepsilon_\infty$	Long-time value of creep
$\eta_M, \eta_K$	Represents the viscosities of the Maxwell and Kelvin dashpots

$D(t)$	Creep compliance dependent on time
$D_0, D_e$	Initial and Equilibrium compliances
$<, >$	Less than, Greater than