

Influence of Fibre Surface Treatments and Textile Process on the Properties of Natural Composites

By

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Abstract

The objective of this work is to understand the influence of fibre surface treatment as well as yarn twist on the tensile properties of unidirectional (UD) jute polyester composites. Jute and polyester are chosen in this work as both are cheap. As received jute yarn, physical and chemical surface treated jute yarn and raw jute fibre are used to manufacture UD composites. Physical surface treatment includes removing yarn hairs, sizing by hydroxyethyl cellulose (HEC), water treatment, sizing by cellulose nano-particle (CNP) and chemical treatment includes mercerisation with and without applying tension.

Composites manufactured with unsized hairy yarn show an increase in tensile modulus and a decrease in strain at failure compared to composites manufactured with unsized hairless yarn in the longitudinal direction. However yarn hairs do not affect the tensile strength of composites. Application of HEC improves the tensile properties of composites with respect to composites manufactured with unsized yarn. Water treatment of yarn with tension enhances the tensile properties of composites and water treatment of yarn without tension decreases the tensile properties of composite with respect to composites manufactured with untreated yarn. Similar to water treatment, composites manufactured with alkali treated yarn with tension show higher tensile strength with respect to composites manufactured with alkali treated yarn without tension. Non-dried CNP treatment of yarn improves the tensile properties of composites. Composites manufactured with raw fibres have increased tensile properties with respect to composites manufactured with twisted yarn.

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TABLE OF CONTENTS

1 Introduction 1

1.1 Introduction 1

1.2 Objectives 4

1.3 Thesis outline 4

2 Literature review 7

2.1 Summary 7

2.2 A brief history of composite materials 7

2.3 Prospects and challenges of plant fibre composites (PFCs) 9

2.4 Introduction to natural fibres 11

2.4.1 General discussion on the chemical composition of plant fibre cell 13

2.4.2 Morphology of plant fibres 21

2.4.3 Physical properties of plant fibres 23

2.4.3.(I) Tensile strength of plant fibres 25

2.4.3.(II) Young's modulus of plant fibres 25

2.4.3.(III) Fibre elongation at failure 26

2.4.3.(IV) Impact strength of plant fibres 26

2.4.3.(V) Resistance to atmospheric conditions 26

2.5. Introduction to Plant Fibre Composites 27

2.5.1. Selection of fibre 27

2.5.2 Selection of matrix 28

2.5.3 Modification of fibre and matrix to improve the properties of composites 33

2.5.3. (I) Physical modification of fibres 33

2.5.3. (II) Chemical modification of fibres 36

(II).a Alkali/NaOH treatment 36

(II).b Acetylation 45

(II).c Benzoylation 46

(II).d Peroxide treatment 47

(II).e Sodium chlorite treatment 48

(II).f Acrylation and acrylonitrile grafting 48

2.5.3.(III) Use of coupling agent/matrix modification 49

(III).a Silane treatment 49

(III).b Maleated coupling agents 53

2.5.4 Processing of plant fibre reinforced composites 54

2.5.5 Influence of yarn twist on the properties of composites 55

2.5.6 Mechanical properties of plant fibre composites 58
2.6 Conclusions 66

3 Influence of yarn hairiness on the mechanical properties of composites 69
3.1 Summary 69
3.2 Introduction 69
3.3 Materials and Methods 71
3.3.1 Materials 71
3.3.2 Preparation of yarn 72
3.3.3 Binding mechanism between HEC and fibre 75
3.3.4 Manufacturing of composites 78
3.3.5 Preparation of composites specimen for microscopy analysis 79
3.3.6 Preparation of composites for tensile testing 80
3.4 Testing 81
3.4.1 Single yarn tensile test 81
3.4.2 X-ray photoelectron spectroscopy (XPS) 82
3.4.3 Differential scanning calorimetry (DSC) analysis 82
3.4.4 Fourier transform infrared (FTIR) spectroscopy analysis 83
3.4.5 Microscopy 83
3.4.6 Composite tensile test 84
3.4.7 Statistical hypothesis test 84
3.4.8 Fibre and matrix volume fraction in composite 85
3.4.9 Longitudinal modulus 86
3.4.10 Transverse modulus 87
3.4.11 Longitudinal tensile strength 88
3.4.12 Transverse tensile strength 88
3.5. Results and Discussion 89
3.5.1 Single yarn test results 89
3.5.2 XPS analysis for surface chemical of hairy and hairless yarns 92
3.5.3 Thermal analysis 92
3.5.4 Fourier transform infrared (FTIR) analysis 94
3.5.5 Contribution of lumen on void content of composites 95
3.5.6 Composites test results 97
3.5.7.(I) Longitudinal direction of yarn 97

3.5.7.(II) Transverse direction of yarn 103
3.6 Conclusions 109
4 Influence of water and hydroxyethyl cellulose (HEC) treatment on the mechanical properties of jute fibres, and composites 111
4.1 Summary 111
4.2 Introduction 111
4.3 Materials and Methods 113
4.3.1 Materials 113
4.3.2 Preparation of yarn for water treatment, sizing and composites 113
4.3.3 Water treatment and preparation of composites 114
4.3.4 Single fibre preparation for tensile test 115
4.4 Testing 116
4.4.1 Single fibre tensile test 116
4.4.2 Single yarn and composites tensile test 117
4.5 Results and Discussion 118
4.5.1 Fibre test results 118
4.5.2 Yarn test results 127
4.5.3 Composites test results 138
4.6 Conclusions 147

5 Influence of alkali treatment of jute fibre with and without tension on the tensile properties of jute ud composites 150
5.1 Summary 150
5.2 Introduction 150
5.3 Materials and Methods 151
5.3.1 Materials 151
5.3.2 Preparation of yarn for alkali treatment, sizing and composites 151
5.3.3 Alkali Treatment 152
5.3.4 HEC sizing and manufacturing of composites 152
5.3.5 Preparation of materials for testing 152
5.4 Testing 153
5.5 Results and Discussion 153
5.5.1 Fibre test results 153
5.5.2 Composites test results 159
5.6 Conclusions 171

6 Influence of cellulose nano-particles (CNPs) treatment on the mechanical properties of jute composites 173
6.1 Summary 173
6.2 Introduction 173
6.3 Materials and methods 175
6.3.1 Materials 175
6.3.2 Cellulose nano-whiskers (CNWs) preparation 175
6.3.3 Dispersion of CNP and the significance of sulphate ester groups 177
6.3.4 CNP treatment of jute yarn 178
6.3.5 Preparation of composites 179
6.4 Testing 179
6.4.1 Composite test 179
6.5 Results and Discussion 179
6.5.1 Composites test result 179
6.5.1.(I) Composites manufactured from fridge dried (FD) CNPs treated yarn 182
6.5.1.(II) Composites manufactured from RTND CNPs treated yarn 186
6.5.1.(III) Composites manufactured from NRTND CNPs treated yarn 187
6.5.1.(IV) Composites manufactured from CNP mixed in resin 188
6.5.1.(V) Stress-strain curves of composite plaques 189
6.6 Conclusions 194

7 Mechanical properties of UD composites manufactured from raw jute fibre 196
7.1 Summary 196
7.2 Introduction 196
7.3 Materials and Methods 200
7.3.1 Materials 200
7.3.2 Preparation of fibres and composites 201
7.4: Testing 201
7.5 Results and Discussion 202
7.5.1 Fibre test results 202
7.5.1.(I) Diameter of fibres 202

7.5.1.(II) Tensile modulus of fibres 204
7.5.1.(III) Tensile strength of fibres 205
7.5.1.(IV) Strain at failure of fibres 207
7.5.1.(V) Force-displacement curves of single fibres 208
7.5.2 Fibre diameter/cross-section correction factor 214
7.5.3 Composites test results 216
7.6 Conclusions 230

8 **Discussion and conclusions** 232
8.1 Introduction 232
8.2 General Discussion 232
8.3 Major Conclusions 239
8.4 Summary of original contribution 241
8.5 Future Work 242

References 245

9 **Appendices** 266
9.1 Appendix A: Raw tested data of Chapter 3 266
9.2 Appendix B: Raw tested data of Chapter 4 271
9.3 Appendix C: Raw tested data of Chapter 5 287
9.4 Appendix D: Raw tested data of Chapter 6 296
9.5 Appendix E: Raw tested data of Chapter 7 299
9.6 Appendix F: List of publication 312

CHAPTER 1

INTRODUCTION

1.1 Introduction

Due to ongoing environmental awareness composite researchers are shifting their interest from synthetic fibres to plant fibres. This can be observed by the increase in research publications on plant fibre composites (PFCs) over last two decades. Plant fibres are inexpensive, light and have suitable mechanical properties to be used in composites. They are non-toxic, renewable, nonabrasive, non-pollutant to the environment and little emission of greenhouse gas occurs during their production. As a biodegradable [1-3] material, plant fibre is easily disposable [4].

Under these circumstances researchers are paying attention to plant fibre composites nowadays. However some adverse properties of PFCs limit their use in specific purposes such as engineering applications and building constructions. PFC has low tensile strength, high moisture absorption, poor interfacial properties, poor fire resistance, poor durability and inherent faults with respect to glass fibre composites [5] which squeezes their field of use. Researchers are trying to overcome these problems. Considering the demand of the present era and ongoing ecological awareness, plant fibre composite was chosen as the area of research in this study.

Most plant fibre composite research has been performed on bast fibres [6] (fibres collected from inner bark of the plant). Jute is an important bast fibre and possesses various advantages. It is a low density (density 1.45 gm/cc [7]) fibre and has good mechanical properties. Jute is less abrasive to the processing equipment has good dimensional stability [4] and is harmless to the operators. Jute is a cheap fibre [8], eco-friendly and the second most important natural fibre after cotton according to worldwide

annual production [9]. Biodegradable jute fibres rot quickly and merge with the soil after use which provides compost to nourish the soil [10]. Cultivation of jute requires almost no fertilizers and practically no use of pesticides. All these attractive properties inspired the author to select jute fibre as the reinforcement for this work.

The major use of PFC is for non-structural applications in the automotive and building industries [11] due to poor impact and tensile properties. The next challenge of PFC use is structural applications where very high mechanical properties of composites are the principal criteria. Mechanical properties of composites are greatly influenced by the alignment of fibres. Madsen [12] mentioned that axial ultimate stress is reduced 40% due to 10^0 off axis angle of fibres in composites. The primary use of plant fibre is in textiles [13] and various types of textile products are available which are suitable for composite manufacture and researchers are getting excellent results using these textile products in composite applications. In textile processing a wide range of techniques are practised to develop fibre alignment and a highly oriented fibrous finished textile product is yarn [12]. Hence textile yarn occupies a strong position to produce unidirectional (UD) composites. Considering its suitability jute yarn is selected in this work to produce aligned fibre composites.

The cross-section of a natural fibre yarn contains 60-200 [14-16] fibres. Twist is the most common technique to hold these fibres together [17]. Twist provides mechanical strength to the yarn by developing frictional force among fibres [13]. Twist forces fibres to follow a helical path throughout the length of the yarn [18]. The helix angle decreases from yarn surface to centre [19] and the fibre(s) at the longitudinal axis of yarn

should be straight. To understand the effect of fibre misalignment due to twist, raw jute fibre is also used to manufacture composites.

Jute fibre is suitable to infuse with thermoset and thermoplastic matrices [20]. Both the matrices are widely used to manufacture natural composites. Thermoset matrix has some superior characteristics over thermoplastic matrix, such as better mechanical properties particularly strength and stiffness [21]. It shows better thermal stability and lower water absorption. Common thermoset resins such as epoxy, polyester, polyurethane and phenolic are liquid at room temperature which helps to infuse easily via resin transfer moulding (RTM). In RTM method dry reinforced fibres are infused with liquid resin in a closed mould to avoid harmful volatile exposure to workers [22]. Application of vacuum helps to reduce voids in composites [23]. Fibre volume fraction and dimension of the composite plaque can be closely controlled in this process [24]. A wide range of resin and fibres can be processed by this technique. Distribution of resin is better here and less defects are generated [25]. This is a compatible method for infusing staple fibres such as jute and is relatively cheap [26]. Considering the advantages, RTM is chosen for this study.

In the presence of catalyst the raw uncured resin molecules are cross linked through a chemical reaction [27] and resin creates extremely strong bonds giving a stronger composite. This chemical reaction enables resin to change from a liquid to solid state. This is an exothermic reaction and curing can be performed at room temperature [28]. Liquid resin helps to wet reinforcement fibres more efficiently thus strong bonds between fibre and matrix can be achieved. Thus thermoset resin is chosen in this work. However the major disadvantages of thermoset composites are the

inability to remould or reshape once cured hence it is extremely difficult to recycle [29].

1.2 Objectives

The principal objective of this research is to gain better understanding regarding the mechanical properties of jute/polyester UD thermoset composites. Composites will be manufactured using untreated and treated jute yarn as well as untreated jute fibre and the objectives can be summarised as follow:

a) To understand the influence of hairiness (stray, transverse fibres) on the mechanical properties of UD jute/polyester composites.

b) To investigate the effect of water treatment of jute yarn on the tensile properties of UD jute/polyester composites.

c) To study the effect of alkali treatment on the tensile properties of UD jute/ polyester composites.

d) To investigate cellulose nano-particle treatment of yarn on the properties of UD jute/polyester composites.

e) To observe the influence of yarn twist on the mechanical properties of UD jute/polyester composites.

1.3 Thesis outline

This thesis is divided into eight chapters.

Chapter 1 contains a general introduction and objectives of this work.

Chapter 2 gives the literature review related to this work. It provides an understanding of plant fibres and specially jute fibre composites. This chapter covers jute polyester thermoset composites, different surface

treatments on plant fibres including jute and its effect on the mechanical properties of thermoset composites.

Chapter 3 presents the influence of yarn hairiness on the mechanical properties of composites. Hairy and hairless jute yarns are used to manufacture UD composites and the mechanical properties of composites are compared in both longitudinal and transverse directions of yarns with respect to composites manufactured with raw yarn.

Chapter 4 reports the influence of water treatment on jute yarn with and without tension and their effect on the tensile properties of composites. Jute yarn is dipped in water for 20 minutes with and without tension. After drying composites are manufactured using these yarns to understand the influence of water treatment of jute yarn on the mechanical properties of composites. Hydroxyethyl cellulose (HEC) a natural water based material is used to bind the yarns to form UD mat. Composite plaques are manufactured using HEC sized and unsized yarn to understand the effect of HEC on the tensile properties of composites.

Chapter 5 describes the influence of alkali treatment of jute on the tensile properties of UD jute/polyester composites. Jute yarns are treated with 1%, 3% and 5% (by weight) alkali solution at room temperature for 20 minutes with and without tension. These yarns are used to manufacture UD composites to understand the influence of this chemical treatment on the mechanical properties of UD composites.

Chapter 6 reports the influence of cellulose nano-particle (CNP) treatment on the mechanical properties of UD jute/polyester composites. CNP is produced by acid hydrolysis of bleached cotton fibre. These nano-particles are used to size the jute yarn at various concentrations followed by composite manufacturing to examine its effect on the mechanical

properties. CNP is also mixed with resin before infusion to investigate its effect on the tensile properties of composites.

Chapter 7 presents the effect of yarn twist on the mechanical properties of UD jute composites. UD composites are manufactured using twisted jute yarns and raw fibres and their mechanical properties are investigated to understand the effect of yarn twist on the mechanical properties of composites.

Chapter 8 presents the overall discussion, conclusions and a suggestion for future work.

CHAPTER 2

LITERATURE REVIEW

2.1 Summary

This chapter considers a review of the literature related to plant fibres and plant fibre reinforced composites. It has five sections including a brief history of composites, prospects and challenges of plant fibre composites, general discussion on plant fibres covering briefly the mechanical properties and chemical compositions and overview on plant fibre composites incorporating tensile properties of composites and factors that influence the tensile properties of plant fibre composites, and finally conclusions regarding this chapter.

2.2 A Brief History of Composite Materials

It is likely that the first use of man-made fibrous composites started around 4000 BC when Egyptians made paper [30] from the papyrus plant. Straw and mud bricks is also an example of ancient use of fibrous composites in Egypt circa 1300 BC [30] where straw was used to make bricks stronger.

In 1200 AD, the Mongols invented an excellent composite made with wood, bone, and animal glue [31]. They used this new material to make a powerful bow which was also very accurate. Genghis Khan's military success was partly due to this bow. Natural resins were used as binders before the invention of oil based resins. Natural resins were derived from animals or plants [32-34] and hence the sources were limited. Various synthetic plastics such as vinyl, polystyrene, phenolic and polyester were

developed in the early 1900s [34] which could replace natural resins to begin the modern composites era.

From an application point of view, sole plastic materials are too weak for structural components of specific use. This limitation can be overcome by incorporating reinforcement fibres within plastics. Owens Corning was the pioneer to perform this work by using glass fibres in 1938 [35]. Glass fibre combined with a suitable resin provides a composite material that is also lighter with respect to metals.

Phenol formaldehyde resins were the first synthetic plastics invented in 1907 by Baekeland [36]. From around 1942 the United States of America used fibre reinforced plastics (FRPs) commercially to construct aircraft radomes and polyester resin was developed at this time [37]. Scott Bader used unsaturated polyester resin to manufacture composites in 1946 in the UK. These were hot-cure resin and cold curing polyester resin was first introduced in 1947 [37]. Epoxy resin was developed in 1937 however commercial production of this resin was undertaken from around 1950 [37]. At present several thermoset resins are available for FRPs applications such as phenolics, polyamides, vinyl esters, furans, silicones, polyurethanes and others. Various thermoplastic resins are also used in FRPs, however their use is limited due to low thermal resistance, low mechanical properties and processing difficulties.

High quality carbon fibres were developed in 1963 [37] and are used extensively nowadays in composites manufacturing. In 1965 a polyaramid fibre called Kevlar was invented [38] and found suitable for several areas of application.

2.3 Prospects and Challenges of Plant Fibre Composites (PFCs)

Various metals and their alloys are used for engineering purposes [39] due to their suitability and reliability in engineering applications. Nowadays fibre reinforced composites are slowly displacing the position of metals [40].

Among FRPs the majority is glass/polyester [41] composites. This is because low cost glass/polyester provides potentially improved durability under aggressive environments [42]. However the production of glass fibre consumes considerable energy (48.33 MJ/kg during fibre production and 54.7 MJ/kg for mat production [43]) and thus it provides a poor ecological profile, in addition disposal at the end of life is problematic as the materials are not bio-degradable. Incineration of glass fibre consumes 1.7 MJ/kg energy [44]. On the other hand plant fibres are cheap, consume less energy to produce (10 MJ/kg from jute fibre cultivation to fabric production [45]), are renewable, biodegradable and offer low carbon emission. Due to ongoing ecological concerns and to find a cheap reinforcement fibre, researchers are paying attention to plant based natural fibres such as flax, hemp, jute, kenaf, sisal, bamboo, banana and pineapple, since these fibres provide benefits over man-made fibres, especially glass fibre [46].

Plant fibres possess some suitable properties for the composites industry such as low density, high specific strength, high tensile modulus and excellent ecological aspects. All of these features make PFCs attractive to the automotive industry. A good growth tendency in the use of natural fibre composites is observed in German cars from 10,000 tonnes in 2000 to 19,000 tonnes in 2005 [46]. Table 2.1 contains a comparison of typical properties for natural fibre, glass fibre and carbon fibre [47].

Table 2.1: Comparison between natural and synthetic (glass and carbon) fibres [47]

	Natural Fibre	Glass Fibre	Carbon fibre
Annual Production [tonnes]	31,000,000	4,000,000	55,000
Density [$gmcm^{-3}$]	Low (1.35-1.55)	High (2.50-2.70)	Low (1.70-2.2.0)
Cost [£/kg]	Low (0.5-1.5)	Low (1.3-20.0)	High (>12.0)
Diameter [μm]	High (40-5000)	Low (10-20)	Low (5-7)
Elastic Modulus [GPa]	Moderate (30-80)	Moderate (70-85)	High (150-500)
Tensile Strength [GPa]	Low (0.4-1.5)	Moderate (2.0-3.5)	High (4.0-5.5)
Specific Stiffness [$GPa.cm^3g^{-1}$]	Moderate (20-60)	Low (27-34)	High (68-290)
Specific Strength [$GPa.\ cm^3g^{-1}$]	Moderate (0.3-1.1)	Moderate (0.7-1.5)	High (0.6-3.7)
Failure Strain (%)	Low (1.4-3.2)	Low (2.5-5.3)	Low (0.3-2.2)
Renewability	Yes	No	No
Recyclability	Yes	Partly	No
Energy consumption [MJ/Kg]	Low (4-15)	High (30-50)	High (>130)
Abrasion to machines	No	Yes	Yes
Health risk when inhaled	No	Yes	Yes
Disposal	Biodegradable	Non- biodegradable	Non-biodegradable

However some barriers have to be overcome for widespread use of plant fibres for structural applications. Nature defines the properties of plant fibres and thus a wide variability of plant fibre properties is observed depending upon various influencing parameters such as climatic conditions,

method of cultivation and pest control. Therefore consistent quality of plant fibre supply is practically impossible, which is very important for engineering applications. Another drawback of plant fibres is their hydrophilic nature and due to this property plant fibres create a poor bond with common hydrophobic matrices (such as unsaturated polyesters, epoxies and phenols). The result is unsatisfactory interfacial properties which is a major challenge for PFCs. These constraints need to be overcome to use plant fibres for structural applications.

2.4 Introduction to Natural Fibres

Nature offers an abundant supply of fibrous material from various sources. It is the principal source of textile raw materials. Apart from textile applications, natural fibres are the source of human and animal food, agro-fine chemicals, building materials, environmentally friendly cosmetics, sources of biopolymers and energy [45]. The sources of natural fibres include plants, animals and minerals [2]. Plants produce the highest volume of natural fibres. Almost 1000 types of plants [48] produce 180 billion tonnes [49] of fibre every year around the world. Various parts of the plants such as bark, leaf, seed, woody core, grass, straw, and cane [45] produce these fibres. Depending on the fibre sources plant fibres can be categorised according to Table 2.2.

The main constituent of animal fibres are proteins [50, 51]. Various mammals such as sheep, horses, camels, rabbits, goats and yak provide fibres as hair. Different species of birds give fibre as feathers and silkworms or spiders deliver it as secretions [45]. Delicate animal fibres such as wool, cashmere, rabbit hair, camel hair and yak hair are used widely for clothes. Animal fibres are also used for floor coverings, upholstery, furnishing fabrics and bedding [51].

Table 2.2: Classification of plant fibres and their sources [46]

Bast	Leaf	Seed				Core	Grass/ Reeds	Other Fibres
		Pod	Husk	Fruit	Hulls			
Hemp	Pineapple	Cotton				Kenaf	Wheat	Wood
Ramie	Sisal	Kapok				Jute	Oat	Roots
Flax	Agava	Loofah				Hemp	Barley	Galmpi
Kenaf	Henequen	Milk weed				Flax	Rice	
Jute	Curaua		Coir				Bamboo	
Mesta	Banana			Oil palm			Bagasse	
Urena	Abaca				Rice		Corn	
Roselle	Palm				Oat		Rape	
	Cabuja				Wheat		Rye	
	Albardine				Rye		Esparto	
	Raphia						Sabai	
							Canary	
							Grass	

Minerals such as asbestos, glass, carbon, basalt, ceramic, silicate, borate and aluminium are also a source of natural fibres [45]. In the 19th century asbestos was very popular for structural applications, because asbestos has good thermal, electrical and chemical properties. However due to its carcinogenic nature [52] the EU and several other countries banned the uses of asbestos from the end of the 20th century [53].

2.4.1 General discussion on the chemical composition of plant fibre cell

Table 2.3: Chemical composition of common plant fibres [54]

Fibre type	Cellulose %	Hemi-cellulose %	Lignin %	Pectin %	DP*	Wax %
Abaca	61-64	21	12	0.8	1990	-
Bagasse	32-48	21	19.9-24	10	-	-
Banana	60-65	6-19	5-10	3-5	-	-
Bamboo	26-43	15-26	21-31	-	-	-
Coir	46	0.3	45	4	-	-
Cotton	82-96	2-6	0.5-1	5-7	2020	0.6
Flax	60-81	14-19	2-3	0.9	2420	1.7
Hemp	70-92	18-22	3-5	0.9	2300	0.8
Jute	51-84	12-20	5-13	0.2	1920	0.5
Kapok	13.16	-	-	-	-	-
Kenaf	44-57	21	15-19	2	-	-
Phormium	67	30	11	-	-	-
Pineapple	80-81	16-19	4.6-12	2-3	-	-
Ramie	68-76	13-15	0.6-1	1.9-2	2160	0.3
Sisal	43-78	10-13	4-12	0.8-2	2660	0.2
Wood	45-50	23-30	27	2-2.5	-	-

*****Degree of polymerisation

Since the sources of plant fibres are widespread, the chemical compositions of plant fibres vary to a great extent. However the principal element of plant fibre cells is cellulose and it is generally 60-80% [55] of the weight of the fibres. It is the most common organic compound in the world [56]. The second most important component of plant fibre (especially bast fibre), is hemicellulose. Apart from cellulose and

Table 2.4: Dimensions of some typical plant fibres [54]

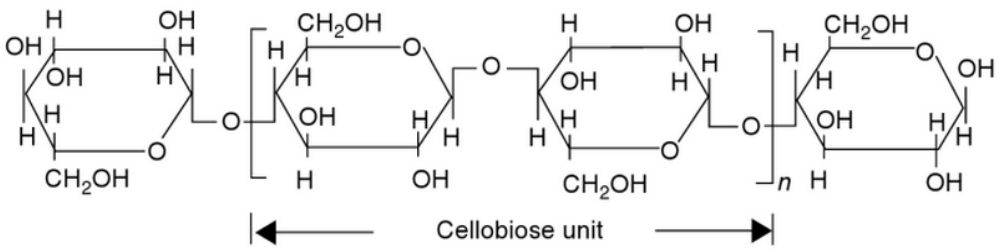
Fibre type	Diameter (μm)	Length (mm)	Aspect Ratio (l/d)	MFA** (θ)	Bulk Density (kg/m^3)	Moisture Regain*** (%)
Abaca	17-21.4	4.6-5.2	257	-	1500	14
Bagasse	20	1.7	-	-	550-1250	-
Banana	-	2-3.8	-	11-12	1300-1350	-
Bamboo	10-40	2.7	-	-	1500	-
Coir	16.2-19.5	0.9-1.2	64	39-49	1250	13
Cotton	11.5-17	20-64	2752	20-30	1550	8.5
Flax	17.8-21.6	27.4-36.1	1258	5	1400-1500	12
Hemp	17-22.8	8.3-14.1	549	6.2	1400-1500	12
Jute	15.9-20.7	1.9-3.2	157	8.1	1300-1500	17
Kapok	15-35	32	724	-	384	10.9
Kenaf	17.7-21.9	2-2.7	119	-	1220-1400	17
Phormium	15.4-16.4	5-5.7	337	-	-	-
Pineapple	20-80	-	-	6-14	1520-1560	-
Ramie	28.1-35	60-250	4639	-	1550	8.5
Sisal	18.3-23.7	1.8-3.1	115	10-22	1300-1500	14

**Micro fibril angle. ***The ratio between the weight of water absorbed by fibre at standard atmospheric condition and the oven dry weight of fibre expressed in percentage. Standard atmospheric condition in textile is (20±2)^{0}C and (65±2)% relative humidity and oven dry weight is the weight of the sample excluding moisture.

Hemicellulose bast fibres and some other plant fibres contain a cementing material known as lignin. The amount of these three polymers varies (Table 2.3) from fibre to fibre. This phenomenon plays a major role for selecting plant fibres to use as reinforcements. Tables 2.3 and 2.4 contain

the chemical composition as well as some dimensions of some common plant fibres.

It is widely accepted that cellulose is a linear macromolecule chain and is a homopolysaccharide polymer consisting of a monomer made with two units of β-D-glucopyranose covalently bonded by a β-1,4-glycosidic linkage [11, 57]. When two β-D-glucopyranose units are bound together, one glucose unit has to move 180° with respect to the other unit (Fig. 2.1) to form a repeating unit comprising two units of glucose known as a cellobiose unit. Due to β configured linkages of glucose units, cellulose possesses a flat ribbon like structure having long chains with a half convolution. Cellulose chains are oriented along the axis of the fibre. Cellulose creates strong hydrogen bonds between intra-molecular chains and inter-molecular chains. The linear molecular chains contribute an anisotropic nature to cellulose [58] chains and hence to fibres also. The orientation of these linear molecular chains is not identical throughout the fibre length. Sometimes they are straight and parallel (crystalline region) and sometimes they are randomly oriented (amorphous region). Crystalline regions provide strength and amorphous regions provide flexibility to the fibres. The ratio of crystalline and amorphous regions varies from fibre to fibre. Valonia which is a highly crystalline cellulose has up to 94% crystallinity however



(n=3,000-10,000)

Fig. 2.1: Chemical structure of cellulose [45]

the primary cell wall of the fibre contains only 20% crystalline region [57]. 5-6 nm diameter crystals are assumed to be nearly square shaped in cross-sections and about 100 chains per crystal [57].

A single polymer chain comprises from hundreds to millions of monomer units [59] and they are called a repeating unit. The number of repeating units contained by a chain is known as degree of polymerisation (DP) [60]. The mechanical properties of the fibre, such as strength and elongation at failure are partially governed by DP. It is also related to the resistance from physical, chemical and radiation damage of fibres. Native cellulose (cellulose I) has a very high DP of up to 10,000 or even 15,000 [11, 57]. However different physical and chemical treatments during extraction and processing reduce the DP of cellulose. Each repeating unit of cellulose (cellobiose unit) contains six hydroxyl groups. Since hydroxyl groups are very prone to create hydrogen bonds so these fibres are hydrophilic. Due to their hydrophilic nature, cellulosic fibres have high moisture content and they hydrolyse in acid easily.

Cellulose has very high stiffness and theoretically it can be up to 134 GPa for native cellulose [61]. Cellulose I, cellulose II and amorphous cellulose have different ultimate stress values and theoretically it is estimated 13-17 GPa, ~9 GPa and 800±100 MPa [46] respectively. However experimental results give a maximum tensile strength of around 2 GPa [46]. This large difference of tensile strength between theoretical estimation and experimental result is due to a number of reasons, such as natural defects in the fibres as well as addition of processing faults. The result indicates that attention needs to be paid regarding fibre extraction in addition to processing techniques. For example mechanical fibre decortication techniques (e.g. ramie) or steam explosion processes (high temperature

and pressure) can add defects to the fibres and may reduce native cellulose content as well as the DP. Since native cellulose has high stiffness, good strength and a very high DP thus it is desirable to have a higher portion of cellulose I for a plant fibre to obtain better mechanical properties.

Hemicellulose is a collective name for an extremely heterogeneous group of polysaccharides [62] (Fig. 2.2). The main component of hemicellulose is β-1,4-linked D-xylopyranol units with different lengths of branch chains comprising L-arabinose, D-glucuronic acid, or its 4-*O*-methylether, D-galactose and possibly D-glucose [63].

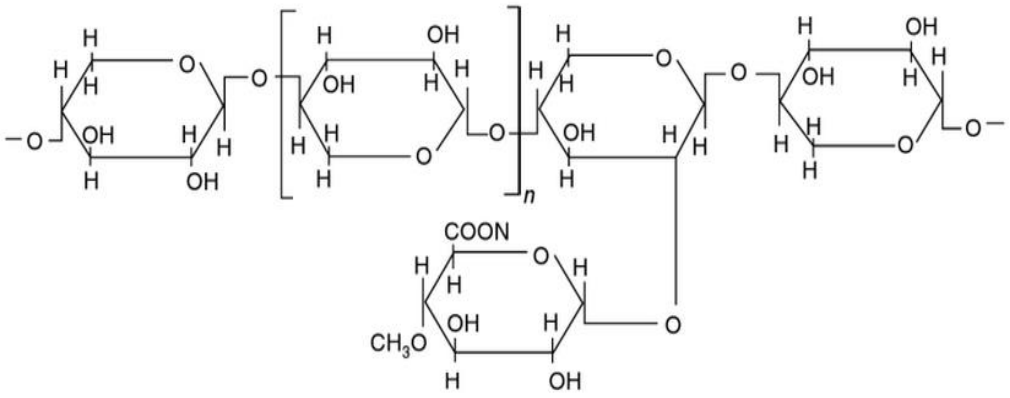


Fig. 2.2 : Chemical structure of hemicellulose (n=up to 200) [45]

Generally hemicelluloses are considered as matrix materials and may be found at the middle lamella of fibres that bind cell walls [62]. In three different aspects hemicellulose is different from cellulose and they are (i) hemicellulose contains many different sugar units, whilst cellulose contains only 1,4-β-D-glucopyranose units (ii) hemicellulose chains are highly branched and (iii) it has very low DP (10-100 times lower than cellulose I) i.e. short polymer chain and lower molecular weight [63]. Hemicelluloses are hydrophilic in nature [64], soluble in an alkali solution and hydrolysable in an acid solution.

The third important constituent of bast fibre is lignin which plays a role as a cementing material. It provides rigidity to plants [65] and also increases the compression strength by cementing the fibres together. Lignin acts as a filler between cellulose, hemicellulose and pectin. It is a very high molecular weight little understood hydrocarbon (see Fig. 2.3) polymer consisting of both aromatic and aliphatic portions [63] and acts as adhesive material around the cellulose chains that holds the natural structure together. From a hygroscopic point of view it is opposite to cellulose or hemicelluloses. It is amorphous [63] and thermoplastic in nature having a glass transition temperature (Tg) of around 90°C and a melting temperature (Tm) close to 170°C. Lignin has a stiffness of 4 GPa [46] and DP of 60. It is an isotropic polymer.

Lignin deposition on flax fibres decreases the contact angle and hence increases wettability by resin and fracture surfaces have shown that delamination happens between fibre and lignin rather than lignin and matrix [66]. Lignin as a compatibilizer with coir and polypropylene increased the flexural strength and decreased water absorption and swelling. No effect was observed on the tensile strength of composites [67]. However a reduction of lignin increased the tensile strength of single wood fibre [68]. Low amounts of lignin removal from jute fibre make the middle lamella more homogeneous due to elimination of micro voids and hence increases the tensile strength and tensile modulus. Reduction of lignin down to 5.7% or lower decreases the tensile strength and tensile modulus of jute fibre. [69].

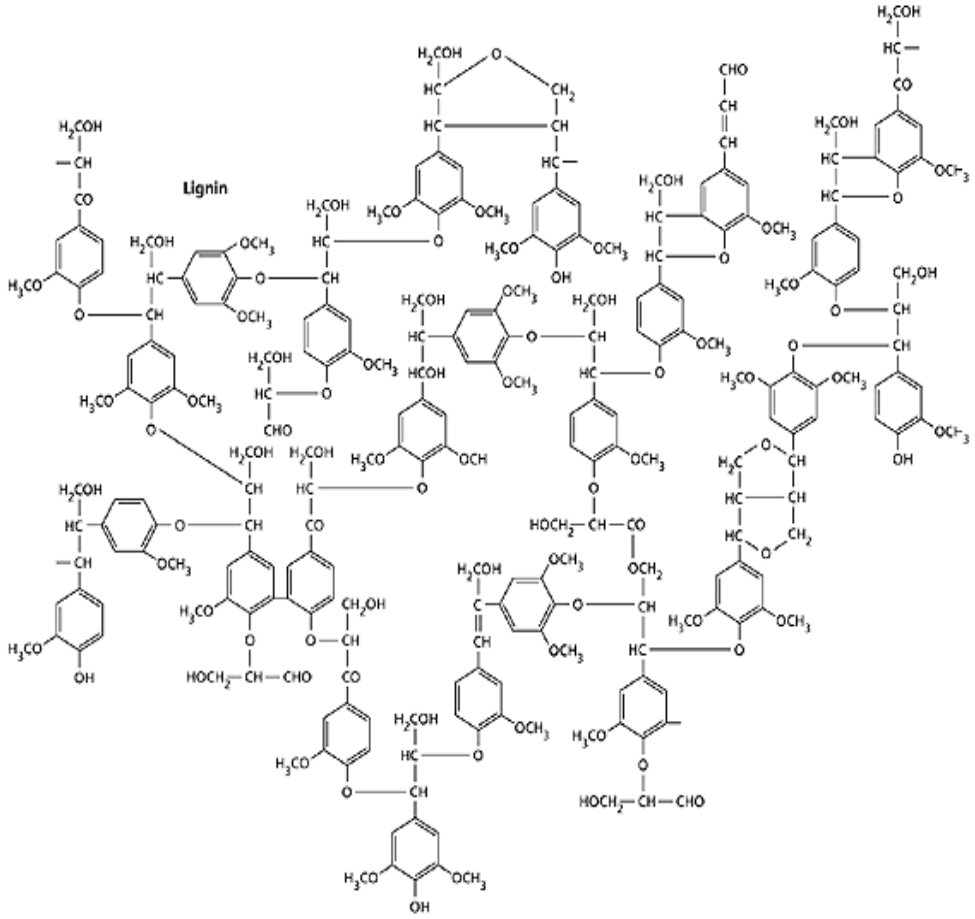


Fig. 2.3: Partial structure of a hypothetical lignin molecule from European beech (*Fagus sylvatica*) [70]

Plant cells also contain pectin (see Fig. 2.4) which is a collective name of heteropolysaccharides [63] that consists of α-(1,4)-D glycosidic linked galacturonic acid units, sugar units of various composition and their respective methyl esters [71]. It is a linear poly saccharide that binds cells together. Monovalent cation (alkali metal) salts of pectinic and pectic acids is generally water soluble however di and trivalent cations are partially water soluble or insoluble. The pectin chain contains a few hundred to thousand saccharide units. The highest amount of pectin is available in the middle lamella of the cell wall [72]. Pectin is highly hydrophilic [73] and thus removing it is helpful to improve the fibre/matrix interfacial properties. Pectin can be removed partially by alkali treatment [74] or by enzyme

(pectinase) treatment [75] and removal of pectin from fibre has improved the tensile (35.8% modulus and 9% strength) and flexural (37.4% modulus and 18.4% strength) properties of hemp/poly propylene composites [75].

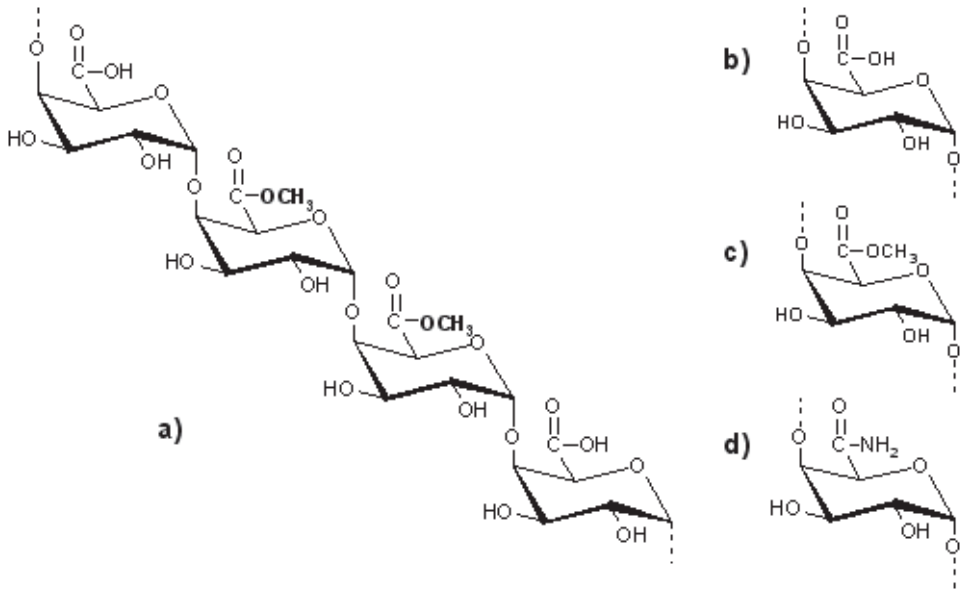


Fig. 2.4 (a) A repeating segment of pectin molecule and functional groups: (b) carboxyl; (c) ester; (d) amide in pectin chain [72].

Waxes are a diverse group of hydrocarbons [62] and represents a small percentage of the fibre. Waxes act as surface coatings and protect fibres from mechanical damage. Wax is also insoluble in water as well as several acids. Waxes hinder fibre matrix adhesion hence high levels of waxes are not desirable.

After discussing the constituents of natural fibres it can be concluded that:

- Higher portion of native cellulose content gives high strength and stiffness to fibres.
- Low hemicellulose content is desirable since it absorbs moisture and hence accelerates fibre degradation.

- Lignin acts as a bonding material among cellulose, hemicelluloses and pectin and it is hydrophobic in nature. Reduction of lignin increases the tensile strength of fibre. However lignin as compatibilizer or deposition of lignin on the fibre surface can improve the properties of composites.
- Pectin is hydrophilic and removal of pectin improves the mechanical properties of composites.
- Waxes stay at the surface of the fibres and prevent wetting and also reduce fibre-matrix adhesion properties, so a minimum level of wax is desirable.

2.4.2 Morphology of plant fibres

Each plant fibre is a single cell and this cell consists of a cell wall and a central cavity called a lumen [63]. At the development stage of cells, lumens are full of cell sap containing nutrients to the cell, and they help to transport water [76]. When the cells become mature all the sap is consumed and a central cavity is formed.

Hair shape plant fibre exhibits a high aspect ratio (ratio of length to diameter) and a large variation of aspect ratio is noticed from fibre to fibre. In case of bast fibre the individual fibres are packed together to form a polygonal shape and oriented in the fibre axis (see Fig. 2.5).

The plant cell wall structure and organisation are inhomogeneous. Naturally plant cell walls are composed of several layers, such as a thin layer of primary cell wall and layers of secondary cell wall. The primary cell wall develops during the period of cell growth encircling the secondary cell wall. The major constituent of a single plant cell is the secondary cell wall

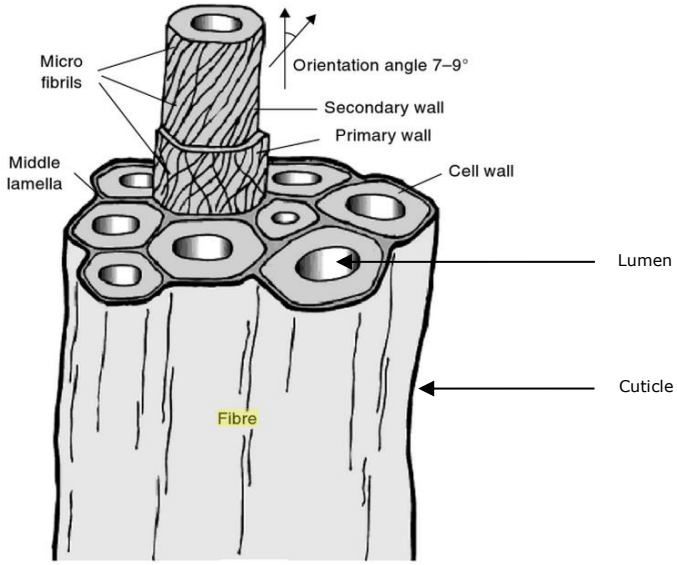


Fig. 2.5: Jute fibre morphology: microstructure [45]

that consists of three layers (S_1, S_2 and S_3) [2]. Cell walls are composed of an oriented form of cellulose micro fibrils together with hemicellulose and lignin. These micro fibrils are not parallel to the fibre axis, rather they are oriented at an angle to the fibre axis, called the micro fibrillar angle (MFA) [2]. Beyond these cell walls, plant fibres have an outer skin called a cuticle which protects the fibre from abrasion or other mechanical damage. The cuticle is also surrounded by a thin layer of wax [63].

Micro fibrils create a different MFA in each layer and this also varies from layer to layer (Fig. 2.5) of the cell wall. For example micro fibrils present in the primary cell wall are not parallel to each other and not even arranged in a single direction. This is because plants follow a 'multi-net growth theory' to grow.[77]. Thus a large variation of MFA in the primary cell wall is observed. Micro fibrils lay almost in the transverse direction toward the fibre axis at S_1 and S_3 layers, so MFA is very high there [78]. Micro fibrils in the S_2 layer stay more parallel to the fibre axis and thus have lower MFA [79] and this layer is the thickest, making up to 80% of the thickness of

the cell wall [80]. This layer essentially determines the mechanical properties of the fibre.

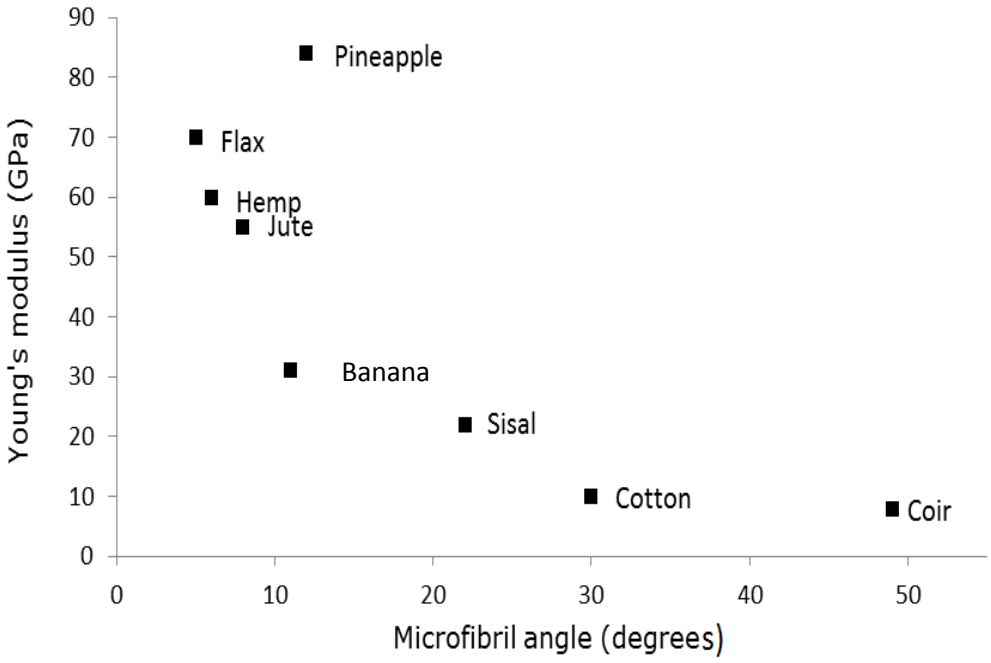


Fig. 2.6: The relationship between Young's Modulus and plant fibre micro fibril angle [54]

Lower MFA results in a rigid fibre with higher stiffness and strength properties. High MFA incorporated micro fibrils provides a more ductile fibre, having higher toughness and impact properties. Higher MFA also reduces the tensile strength and stiffness [81]. Thus to achieve maximum tensile properties a low MFA is preferred. Fig. 2.6 presents the relation between Young's modulus and MFA of some common plant fibres [54].

2.4.3 Physical properties of plant fibres

Several mechanical properties such as tensile strength, tensile modulus, elongation at failure, water absorption, impact strength and fibre swelling are considered for different uses of fibres. All these properties may not have equal importance for every use. For example tensile strength, tensile

modulus and impact strength are very important for composites whilst water absorption and fibre swelling are disadvantageous. On the other hand water absorption is very important for clothing used in tropical climates as they need to absorb perspiration and diffuse it to atmosphere [82].

Table 2.5: Physical properties of glass and some typical plant fibres [83]

Properties	Fibre								
	E-glass	flax	hemp	jute	ramie	coir	sisal	abaca	Cotton
Density g/cm^3	2.55	1.4	1.48	1.46	1.5	1.25	1.33	1.5	1.51
Tensile strength MPa	2400	800 - 1500	550 - 900	400 - 800	500	220	600-700	980	400
E-modulus (GPa)	73	60 - 80	70	10 - 30	44	6	38		12
Specific modulus (E/density)	29	26 - 46	47	7 - 21	29	5	29		8
Elongation at failure (%)	3	1.2 - 1.6	1.6	1.8	2	15 - 25	2 – 3		3 – 10
Moisture absorption (%)	-	7	8	12	12 -17	10	11		8 – 25
Price/Kg ($), raw (mat/fabric)	1.3 (1.7/3.8)	1.5 (2/4)	0.6 - 1.8 (2/4)	0.35 1.5/ 0.9 - 2	1.5 - 2.5	0.25-0.5	0.6 - 0.7	1.5 - 2.5	1.5- 2.2

Physical properties of fibres are affected greatly by chemical composition and some other properties such as fibre length, fineness, maturity, micro fibril angle, aspect ratio, cell geometry and arrangement, DP value, defects and faults incurred [80, 82, 83]. Some important physical properties of plant fibres are presented in Table 2.5 [83]. Research has been carried out since 1930s [84] to identify the mechanical properties of plant fibres (especially wood fibres) affected by these parameters. One interesting observation is that cell shape and arrangement do not play a major role in determining the physical properties of fibres [84].

2.4.3.(I) Tensile strength of plant fibres

Tensile strength of reinforcement fibre is very important, since this parameter determines the tensile strength of composites. McLaughlin and Tait [81] studied different types of plant fibres and mentioned that increasing cellulose content and decreasing micro fibril angle enhanced the values of tensile strength and Young's modulus. Satyanarayana et al. [85] also made similar observations. Alves Fidelis et al. [86] studied the tensile properties of jute, sisal, curaua, coir and piassava fibres and mentioned that less lumen area and higher secondary wall thickness increase the tensile strength and Young's modulus of fibres. Zhang et al. [68] studied the effect of lignin on the properties of single wood fibre tensile behaviour and commented that reduction of lignin increases fibre tensile strength. However the presence of defects can also reduce the fibre strength [87].

2.4.3.(II) Young's modulus of plant fibres

As with tensile strength, Young's modulus is also an important parameter of any engineering or structural material. Young's modulus of composite materials depends primarily on the stiffness of reinforcement fibres. MFA and cellulose content play a vital role to determine the stiffness of fibres. Lower MFA (Fig. 2.6) and higher cellulose content enhance the tensile modulus of fibres [81, 85, 88].

Placet et al. [89] commented that the tensile modulus of plant fibres is dependent on the lumen size and fibre diameter. Larger lumen decreases the modulus for a constant diameter fibre. They reported that this structural property is affected by the cylindrical geometry, the multi-layered organisation of the fibre and the orientation of the cellulose microfibrils only. They also mentioned that the Young's modulus of fibres is

influenced by morphological, dimensional and structural parameters however not by weak link effect.

2.4.3.(III) Fibre elongation at failure

Elongation at failure depends upon the chemical components and geometric properties of the fibre. Increase in cellulose content [90] and decrease lignin content [91] increase the elongation of plant fibres. Generally cellulosic fibres show higher elongation at failure at wet condition than dry. Higher degrees of crystallinity lead to lower elongation at failure [92]. Zhang et al. [68] mentioned that the reduction of lignin increases the elongation at failure of single wood fibres. McLaughlin and Tait [81] reported that uncoiling of micro-fibrils happen due to loading and higher MFA increase the fracture strain of wood fibre.

2.4.3.(IV) Impact strength of plant fibres

Pavitharan et al. [93] quoted that the major influential factor of fibre impact strength is MFA and it shows an opposite to tensile behaviour. This means fibres having high MFA value (e.g. seed and leaf fibres such as coir, pineapple, sisal and banana) will show higher impact strength than those with low MFA (e.g. bast fibres such as flax, jute, hemp). A similar result was also found by Reis [94].

2.4.3.(V) Resistance to atmospheric conditions

Water and thermal resistance are very important properties for plant fibre reinforced composite materials. Plant fibres are hydrophilic in nature and absorb water readily. This is a major constraint to the manufacture of composites with hydrophobic resins and hinders wider use of plant fibres. Plant fibres have poor resistance to heat and at high temperature the fibre cell walls start to degrade or even become char (normally above 200^0C)

[46, 95, 96]. This characteristic sometimes becomes a constraint especially for manufacturing of thermoplastic composites or for thermoset composites with a high exothermic temperature.

2.5 Introduction to Plant Fibre Composites

Different plant fibres such as flax, hemp, jute and sisal reinforced polymer composites have proved to be very successful for engineering applications [97]. Available textile forms of plant fibres such as non-woven fabric, yarn, woven fabric, knitted fabric or even fibre itself are found suitable for composite manufacture. In recent years an increasing number of research papers, reviews and books denote the growing interest in PFCs.

2.5.1 Selection of fibre

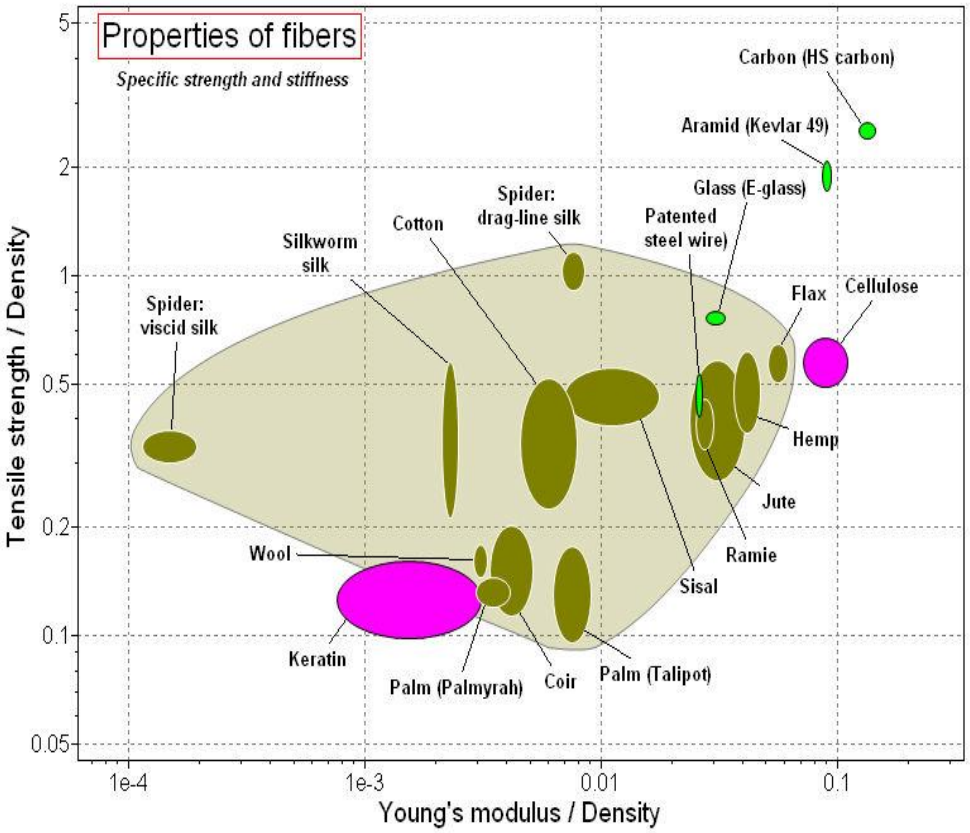


Fig. 2.7: The specific strength and stiffness of natural fibres compared to those of the strongest man-made fibres. [98].

Selection of materials for a specific end use is an important criterion. This parameter is influenced by a set of attributes such as mechanical, electrical, thermal, chemical and optical properties in addition to processing characteristics, cost, density, availability and environmental issues [99]. Among different variables material weight, cost, mechanical properties such as tensile modulus and tensile strength are considered as primary parameters to select a material to be used in engineering purposes. Ashby presented 'material performance indices' based on component function, objectives and constraints as design criteria [99]. On the basis of these criteria he explained a method to select a specific material by comparing relative performance such as specific tensile stiffness (tensile stiffness/density), specific tensile strength (tensile strength/density), specific flexural stiffness (flexural stiffness/density) and specific flexural strength (flexural strength/density). Ashby presented the specific tensile properties of plant fibres against the specific tensile properties of synthetic fibres (Fig. 2.7) and it was noticed that specific tensile stiffness of several plant fibres such as flax, hemp, jute, ramie is comparable to the specific tensile stiffness of glass fibre, however not the specific tensile strength.

Properties of composites not only depend on the properties of reinforcement fibres but also the fibre volume fraction, fibre/matrix interface properties, packing arrangement, orientation of fibres and the quality of matrix [21]. Hence opportunities are there to utilise the characteristics of plant fibres to maximise the properties of composites.

2.5.2 Selection of matrix

A fibre reinforced composite has three distinct parts and they are (i) fibres as discontinuous phase, (ii) matrix as continuous phase and (iii) fibre matrix interface [100]. In a composite, fibres are embedded into a

homogeneous and monolithic matrix material. The matrix acts as a binder, holds fibres together and transfer stresses between them. It protects the fibres from atmospheric damage and provides the composite shape, colour, durability, finish, texture and functionality. There are three types of matrix material available for composites manufacturing: (i) ceramic matrix (ii) metal matrix and (iii) polymer matrix [100].

Polymer matrices are of two types: thermoplastic and thermoset. Sources of polymer matrices are non-renewable petroleum and renewable bio-polymer. Fig. 2.8 indicates the matrices in common use, their sources and biodegradability [101]. Intensive work has been performed on plant fibres with thermoplastic and thermoset matrices, both fossil-based and bio-based. Polyethylene is a common fossil-based thermoplastic resin and frequent use of this resin is observed to manufacture PFCs. For example jute, flax, hemp, sisal, bagasse, rice hull, rape straw flour and wood fibres were successfully fabricated with polyethylene. Polypropylene (PP) is also a commonly studied thermoplastic matrix to manufacture PFCs with jute, hemp, kenaf, sisal, wood fibre. However limited investigations have been undertaken on polyvinyl chloride matrix with plant fibre reinforced composites [102].

In recent years much work has been performed on bio-based thermoplastic resin such as polylactic acid (PLA), polyhydroxybutyrate (PHB), polybutylene succinate (PBS), starch, soy oil based resin, cashew nut shell oil and polycaprolactone with different plant fibres. As bio-based resins are derived from nature thus they are more eco-friendly than mineral oil based resins. However natural resins suffer some drawbacks that impede their widespread applications. Generally they are biodegradable and have poor mechanical performance. PLA has low Tg (60°C [103]) and the processing

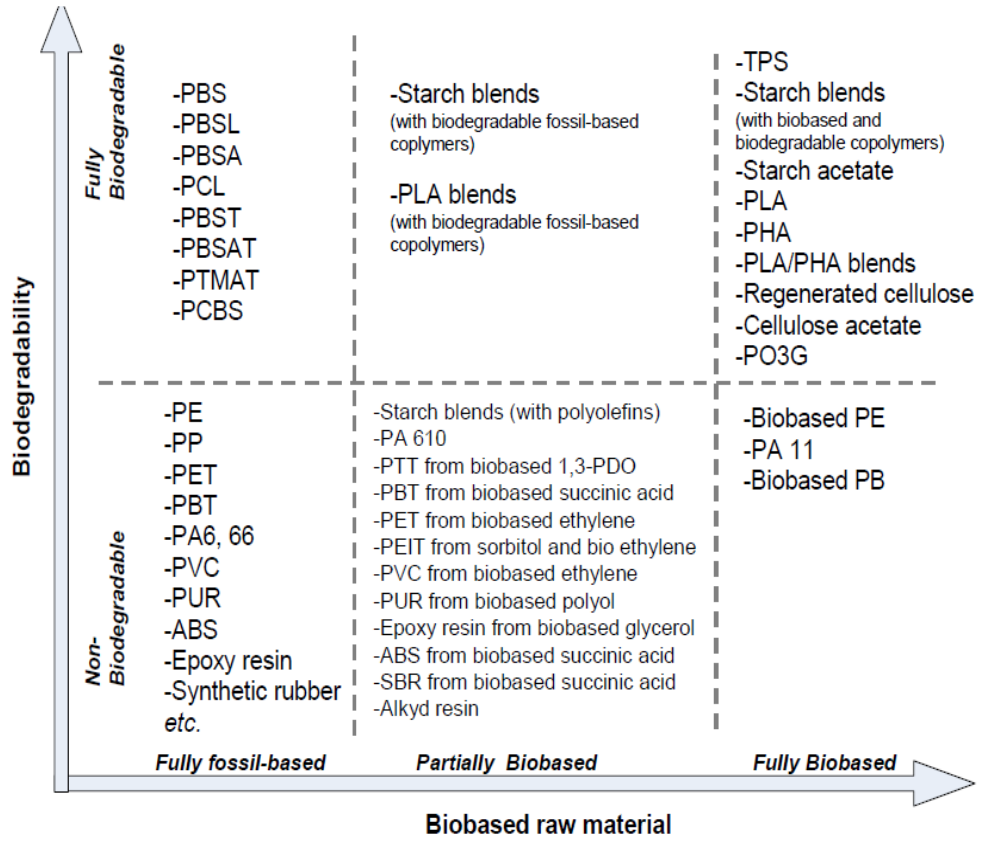


Fig. 2.8 Current and emerging plastics and their biodegradability [101]

temperature is not recommended to be over 200 ^{O}C [46]. It is also brittle in nature. Starch is very sensitive to moisture thus not suitable for several applications [46]. They have high viscosity which restricts the ability to wet the reinforcement fibres properly [104]. Poly(β-hydroxybutyrate) (PHB), a natural thermoplastic resin, is also brittle in nature [46]. The very low Tg (-60°C) and Tm (60°C) of poly (α-hydroxyalkanoate) is the major challenge for widespread use [46]. Disadvantages of soy oil resin are that it is highly hydrophilic, brittle under dry conditions and has low strength [105].

Apart from thermoplastic matrices much work has been performed on thermoset resins. Some well cited thermoset resins are polyester, epoxy and phenolic studied intensively with plant fibres. Polyester is a common

thermoset resin used in polymer composites. Jute, sisal, abaca, bamboo, banana, flax, hemp, kenaf, curaua, pineapple and their blends were fabricated successfully with polyester resin and studied for their various properties. Epoxy is also a very common thermoset resin used frequently with plant fibres such as jute, flax, hemp, kenaf, bamboo, bagasse, banana, pineapple and others. Jute, oil palm, bamboo and date palm leaf fibres were investigated with phenolic (phenol formaldehyde) resin.

Shah [47] presented graphically the absolute (see Fig. 2.9) and specific (see Fig. 2.10) tensile properties of thermoset and thermoplastic PFCs and it is noticed that thermoset PFCs showed better tensile properties than thermoplastic PFCs. This is due to large dimensional cross-linked molecular structure for thermoset PFCs [21] and hence they are more appropriate for load bearing applications with respect to thermoplastic PFCs. Processing temperature of thermoset resins are low (typically below 100^0C) which helps to prevent thermal degradation of plant fibres which may happen during processing of thermoplastic composites. Better wetting and lower void contents are expected for thermoset based PFCs due to low viscosity (0.1-10 Pas) of these matrices. Low viscosity thermoset matrices have enabled the use of common moulding techniques such as hand lay-up and resin transfer moulding (RTM). On the other hand up to 200^0C processing temperature and 100-10000 Pas viscosity of thermoplastic matrices are constraints for plant fibre processing [46].

Thermoset resins are cross-linked in the presence of accelerator and catalyst [106] and form a strong 3-dimensional structure [107]. They have good strength, excellent resistance to heat, have good electrical insulation properties and are dimensionally stable [108]. Thus thermoset resin was chosen in this work. However application of heat cannot untangle the

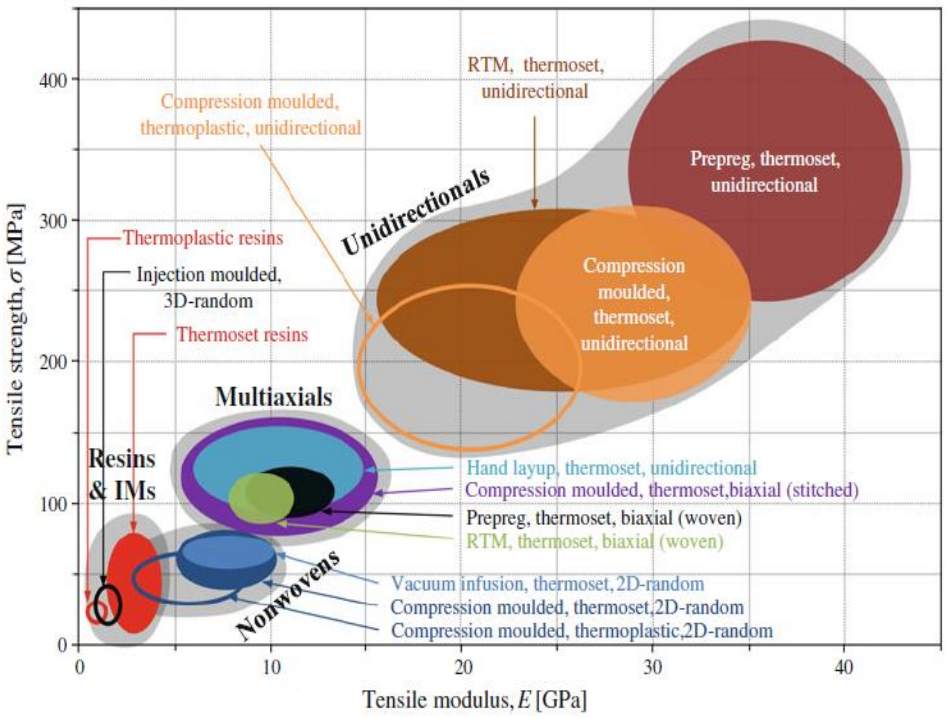


Fig. 2.9: Typical absolute tensile properties for plant fibre reinforced plastics (PFRPs) manufactured with thermoplastic/thermoset resins, short-random/long-aligned fibre reinforcements and various manufacturing routes [47].

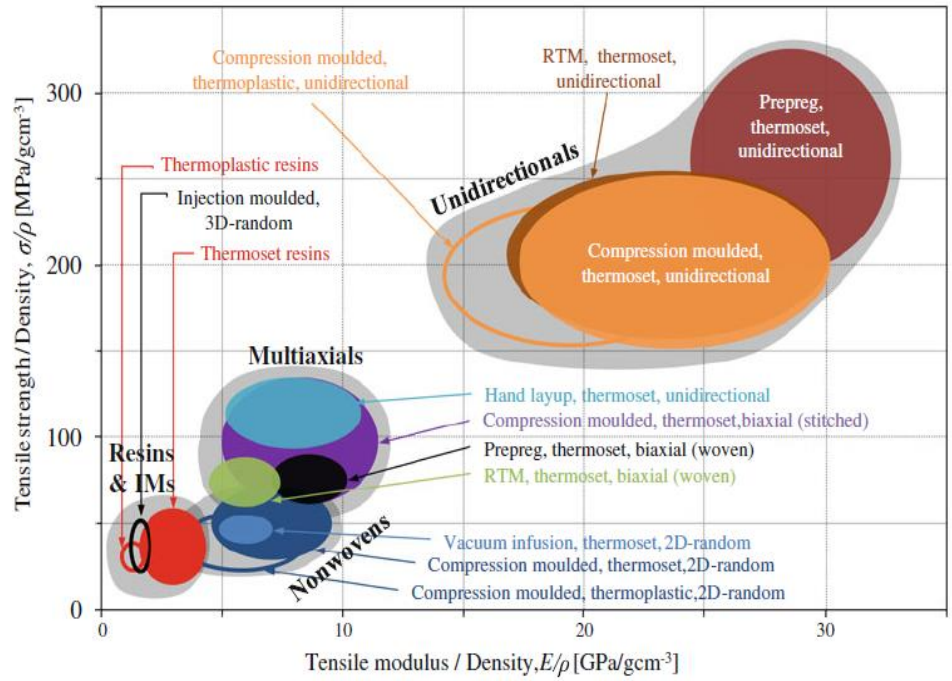


Fig. 2.10: Typical specific tensile properties for PFRPs manufactured with thermoplastic /thermoset resins, short-random/long-aligned fibre reinforcements and various manufacturing routes [47].

cross-linked structure and thus thermoset composites are practically not recyclable and the possibility of re-processing of thermoplastic PFRPs into new composites is also limited as repeated thermal exposure of plant fibres for remoulding deteriorates their mechanical properties severely [109].

2.5.3 Modification of fibre and matrix to improve the properties of composites

As mentioned earlier the hydrophilic nature of plant fibres is a constraint for fibre matrix interfacial bonding hence several researchers have concentrated their attention to improve this. Some of the modifications improve the properties of fibres that would improve the properties of composites. Methods include: fibre surface modification, matrix modification or using a coupling agent. Different techniques and chemicals are applied at various stages in the manufacturing process have been studied extensively to meet the challenge to produce a successful plant fibre composite. Methods studied in this context are categorised into two classes, namely physical modifications and chemical modifications.

2.5.3.(I) Physical modification of fibres

Physical modification techniques include structural and/or surface property changes. These methods do not involve any chemical modification of the fibres. Calendering [110, 111] is a simple way of physical modification, which has been found to improve the bonding between fibre and matrix. Stretching [112, 113] of plant fibre under wet conditions increases the tensile strength of fibres and hence improves the mechanical properties of composites. Thermal treatment of jute fibre [114] after retting was found to increase the compactness of fibre molecules, therefore improving fibre mechanical properties such as crystallinity and stiffness. The use of hybrid fibres in composites was studied extensively by several researchers.

Hybridisation can be performed at fibre, yarn or fabric state and a suitable composite for a specific end use may be produced at reasonable cost. Properties of various types of hybrid fibre composites such as jute/glass, jute/sisal, jute/coir, jute/bamboo, jute/cotton, jute/kenaf, jute/basalt, jute/bagasse, flax/glass, flax/carbon, flax/cordenka, hemp/glass, kenaf/ glass, hemp/nano silica, graphite/glass and other hybrids have been studied by several researchers. Corona, plasma, laser, UV (ultra-violet) and gamma radiation treatments are some ways to modify the physical surface of fibres.

Corona treatment activates the cellulosic fibre surface oxygen and changes the surface energy [115]. Due to the presence of active oxygen, corona treatment creates a stronger bond with the matrix compared to unmodified fibres. The same effects can be achieved by plasma treatment. A plasma is a partially or fully ionized gas that contains electrons, ions, and neutral atoms or molecules [116]. Various surface modifications can be obtained by varying the type and nature of gases used. Laser treatment is performed at very high temperatures (1000°C-1200°C [117]) and hence is not usually suitable for plant based fibres. However UV and gamma ray treatment can be performed successfully on cellulose fibres and researchers have noticed improvements of mechanical properties of composites due to these treatments [118].

Removal of yarn hairs (called singeing) is another method of physical modification. Elimination of hairs enhances the aesthetic property of yarn/fabric. Thus removal of hairs is a common practise for textiles. In this work yarn hairs were removed to understand the influence of hairs on the mechanical properties of composites by manufacturing and testing composites manufactured with hairy and hairless yarns. Yarn hairs can be

removed by burning [119], using a flame in a controlled way or using infra-red radiation. Bio polishing is another technique of singeing (method of removing hairs) available in textile processing to remove hairs [120] where a certain type of enzyme is used to dissolve protruding hairs. In this method, processing parameters and time need to be controlled accordingly; otherwise serious strength and weight loss would occur. However no literature was found to observe the influence of hairiness on the properties of composites.

Cellulose nano whiskers/particles (CNP/CNP) may be used to modify the surfaces of plant fibres as a surface coating material. The most common method to produce CNP is acid hydrolysis [121] and a less common method is mechanical disintegration [122]. In the mechanical method CNP is found in a long and flexible form and highly crystalline ribbon like CNP are obtained via an acid hydrolysis method. The first reported use of CNP in composites was by Favier et al. [123] in 1995 and since then numerous works have been published on cellulose nano composites. CNP was fabricated successfully with different types of matrices such as latex, starch-pectin blend, plasticised starch, waterborne epoxy resin, polyethylene oxide (PEO), polyvinyl acetate (PVAc), polyvinyl alcohol (PVA), phenol formaldehyde (PF) and poly lactic acid (PLA). CNW reinforced PLA composite material showed 35%, 90% and 35% improvement of tensile modulus, tensile strength and elongation at failure respectively with respect to PLA only [124]. CNP reinforced composites also showed higher water uptake [125]. However no published work has been found on coating of plant fibres by CNP as a fibre surface treatment.

2.5.3.(II) Chemical modification of fibres

Chemical modification of a fibre brings some change in the fibre's chemical composition or in the reactive groups attached to the fibre's surface. This change may affect mechanical properties of the fibres or it may help to build a stronger bond between fibre and matrix.

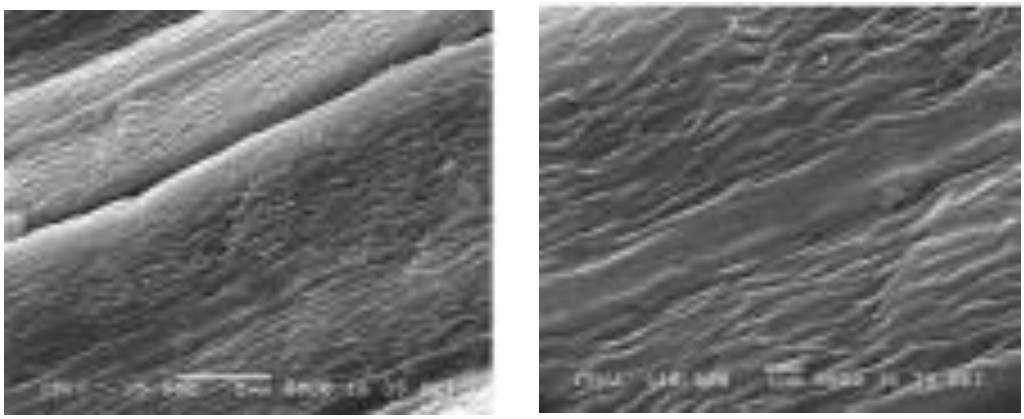
(II).a Alkali/NaOH treatment

A familiar way to modify fibres chemically is by alkali treatment known as mercerisation, which has been performed extensively on cotton fibres.

This process removes the wax and some other impurities from the fibre's surface and roughens the fibre's surface (Fig. 2.11) [126, 127] which facilitates a stronger interfacial bond with the matrix. By roughening the surface of a fibre, the interfacial area of fibre is increased [128]. A higher interfacial area provides better stress transfer from matrix to fibre and the result is higher mechanical properties of composites [129]. Ray et al. [130] found a 23% increase in flexural modulus and 20% increase in flexural strength for 4 hours alkali treated (5% solution) jute vinyl-ester composites with respect to raw jute vinyl-ester composites at 35% fibre volume fraction. Sever et al. [131] observed a 6.7% increase in tensile modulus, 8.6% increase in tensile strength, 9.1 % increase in flexural strength and 9.7% increase in flexural modulus of 5% alkali treated (treatment time 1 hour) jute polyester composites with respect to raw jute composites.

Alkali treatment on jute fibres reduces the lignin [132] and thus increases the cellulose ratio. As mechanical properties of cellulose are superior to lignin, the reduction of lignin improves the mechanical properties of fibres. (see Table 2.6 and Fig. 2.12) [133, 134]. However, extraction of excessive

lignin can deteriorate the tensile properties of fibres [68, 133]. Mercerisation of jute also increases the reactive groups of fibres and those groups can form stronger bonds with the matrix. Removal of lignin softens the inter-fibrilar matrix of fibres which reduces the stress transfer among



(A) (B)

Fig. 2.11: SEM micrographs of: (A) untreated jute, (B) 5% NaOH treated jute [127]

the fibrils as well as overall stress growth in the fibre. Gradual removal of lignin separates the bundle of jute fibres into single fibres thus increases effective fibre surface area to react with the matrix [128].

Table 2.6: Amount of lignin in native and treated wood fibres [68]

Sample	Amount of lignin (%)
Native sample	33.61
Treatment A*	29.76
Treatment B**	25.37
Treatment C***	0.40

* 4 h treatment of wood fibre at 80°C in an aqueous solution of 0.3% $NaClO_2$ buffered with glacial acetic acid at pH 4.4.

** 8 h treatment of wood fibre at 80°C in an aqueous solution of 0.3% $NaClO_2$ buffered with glacial acetic acid at pH 4.82.

*** 8 h treatment of wood fibre at 80°C in a solution of 1g $NaClO_2$ and 2 mL glacial acetic acid in 150 mL distilled at pH 2.69.

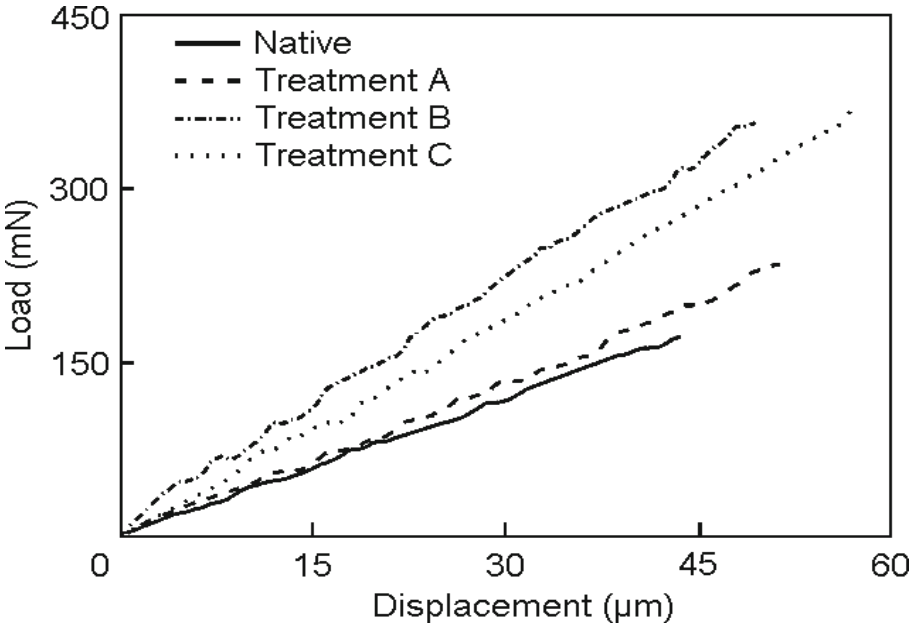


Fig. 2.12: Typical tensile load-displacement curves of single wood fibres [68]

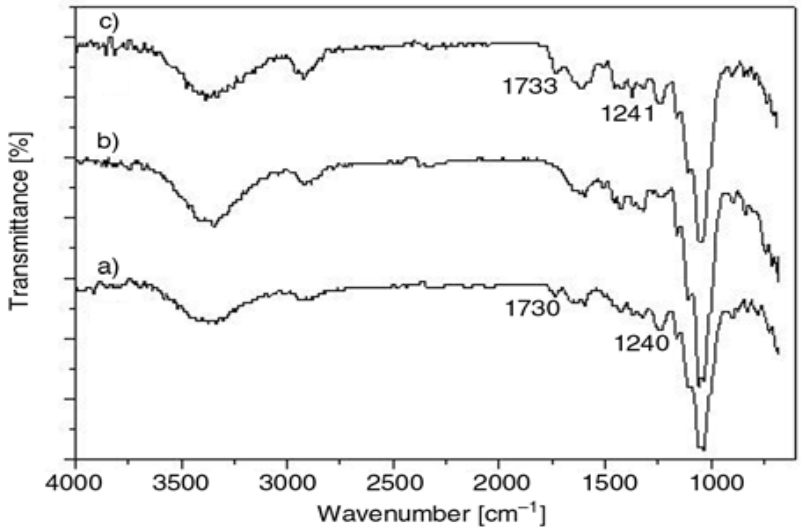


Fig. 2.13: FTIR spectra of: (a) untreated jute; (b) alkali treated jute; and (c) alkali and MAPP emulsion treated jute fibres [127]

Alkali treatment on jute fibre also hydrolyses hemicelluloses as described by Liu et al. [127] and demonstrated in Fig. 2.13. In these FTIR spectra, the peaks between 1730 cm^{-1} and 1240 cm^{-1} traced for untreated jute disappeared after NaOH treatment. These peaks were due to hemicellulose groups which dissolved in aqueous alkali solution. Removal of

hemicellulose makes the inter-fibrillar region less dense and less rigid [2]. Therefore fibrils can be rearranged to form closer packing during tensile loading. Such rearrangement among the fibrils would help to contribute to better load sharing at the moment of stretching [135]. Reduction of highly hydrophilic hemicellulose makes fibres more hydrophobic [126] (see Fig. 2.14) and hydrophobic mercerised jute helps to build a better interfacial bond with hydrophobic resin. Thus mercerised jute fibres and their composites showed less affinity to water and moisture absorption (Table 2.7) under different ageing and humidity conditions [136] compared to raw fibres and their composites. For the same reason lower strength loss was observed for composites made with mercerised jute fibres after dipping in water and exposing to humid conditions for different lengths of time (Fig. 2.15) with respect to composites made with raw fibres.

Table 2.7. Water and moisture absorptions of untreated and alkali-treated jute and jute composites [136].

	Water absorption %		Moisture absorption % after 10 days exposing at		
	24 hour room temperature	2 hour boiling water	75% RH	85% RH	90% RH
Raw Jute	133	140	7.22	7.86	8.24
Alkali treated jute	118	123	5.88	6.24	6.78
Raw jute composites	67	70	5.12	5.37	5.88
Alkali jute composites	55	62	4.12	4.38	4.52

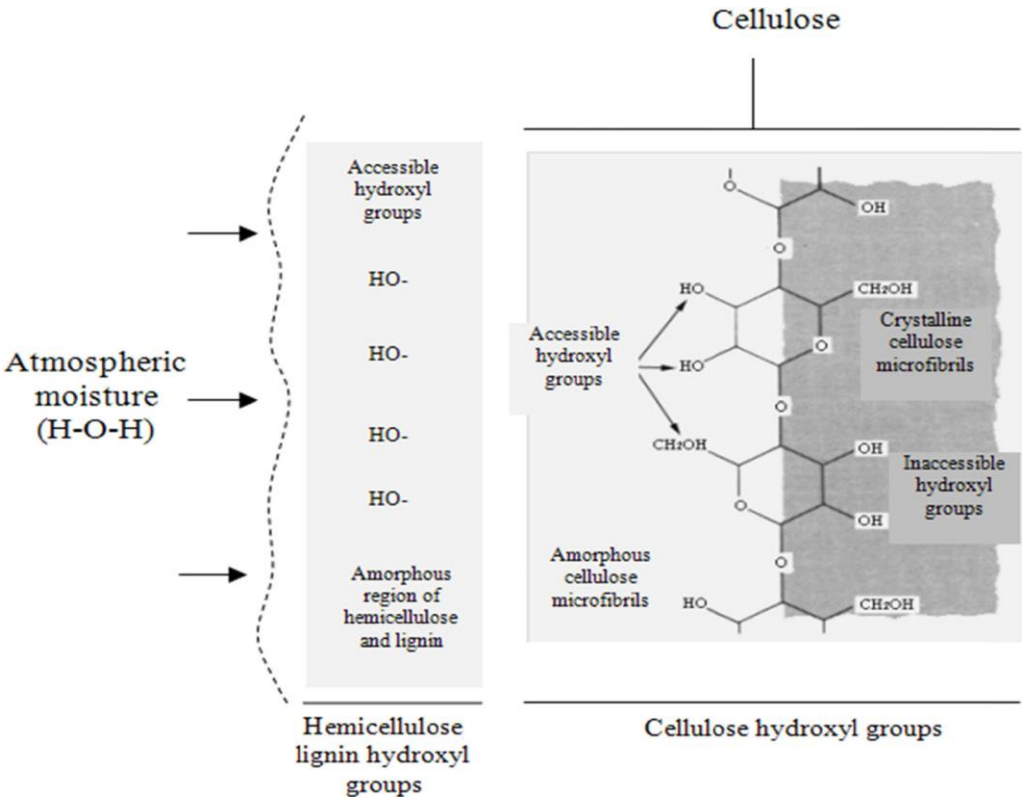


Fig. 2.14: Schematic presentation of the orientation of fibre constituents that absorb moisture [137].

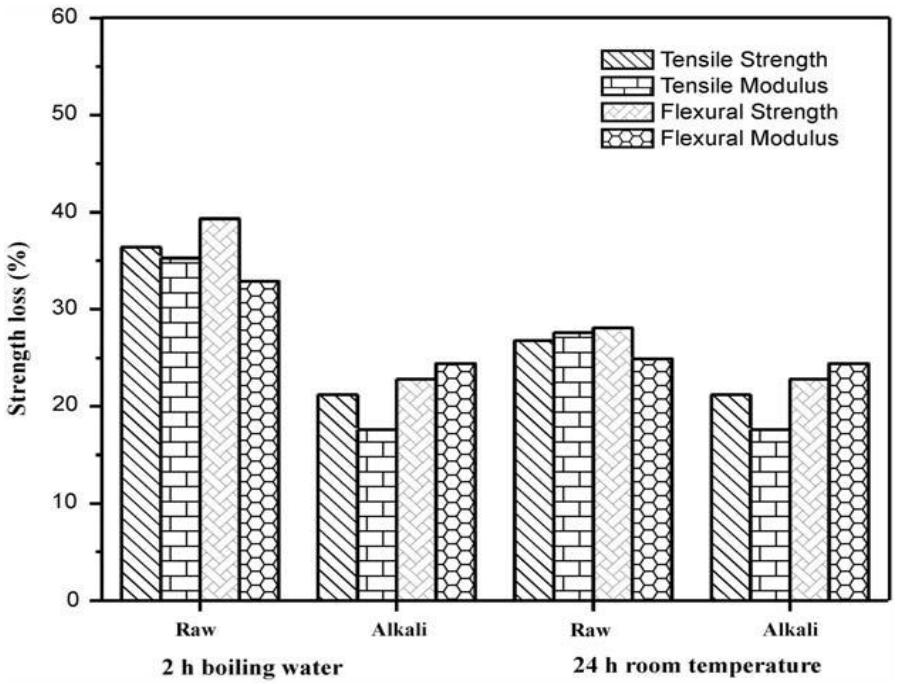


Fig. 2.15: Loss of strength of jute composites after dipping in boiling water for 2 hours and at room temperature for 24 hours [136].

NaOH treatment removes lignin, hemicellulose, wax and some other impurities associated with jute fibre that reduces the fibre weight as reported in several research articles [130, 138, 139]. A decrease of jute linear density from 33 Denier (D) to 14.5 D was measured by Ray et al. [138] for raw fibre and after 6 hours NaOH treatment respectively as presented in Fig. 2.16. They also mentioned that major changes of jute fibres occurred in 5% NaOH solution within 2 - 4 hours of treatment.

Alkali treatment on jute fibres increased the crystallinity [126, 130, 138-141] and subsequently increased the tensile modulus of fibres. The same effect was also observed for coir [142] and flax [143] fibre. Removal of cementing material due to alkali treatment improves the packing of cellulose chains, which provides a higher degree of crystallinity. NaOH treatment on fibres decreases the spiral configuration and increases the

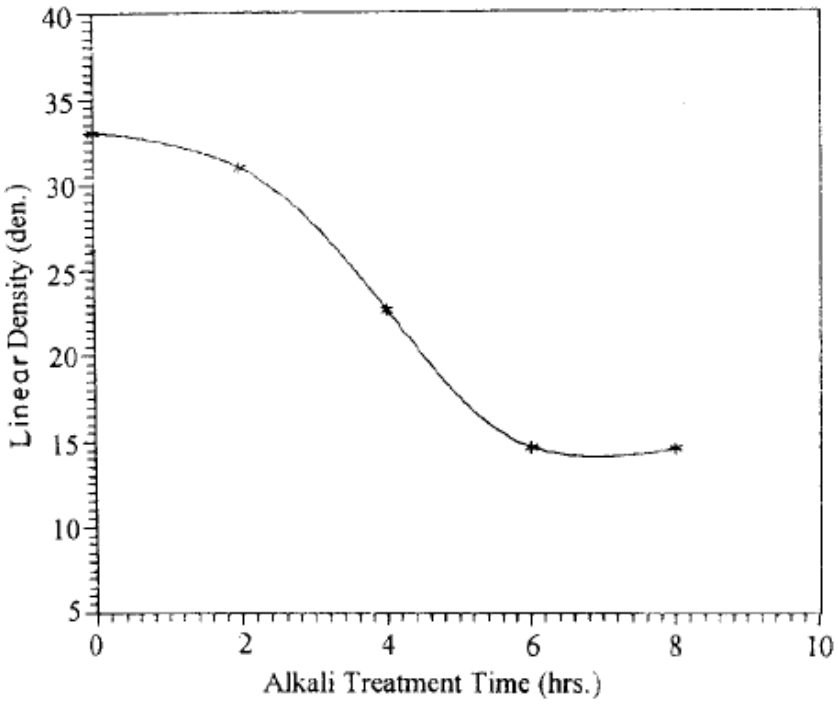


Fig. 2.16: Change of linear density of jute fibres with alkali treatment [138].

molecular orientation [127] which improves the fibre mechanical properties. Jute fibre treated for 24 hours in 0.5% concentrated alkali solution at

ambient temperature improved tensile strength by 82% and elongation at failure by 35% [139] with respect to raw jute. 5% alkali treated (for 4 hours) jute/vinylester pultruded composites (35% fibre volume fraction) showed 23% increase in flexural modulus and 35% increase in flexural strength with respect to these properties of composites manufactured with raw fibre [130].

Variation of treatment time was found to be an influential factor. For a certain concentration of alkali, increasing treatment time can improve the mechanical properties of fibres (Fig. 2.17). However, prolonged treatment has a negative effect. Ray et al. [130] studied the effect of variation of treatment time on jute in a 5% concentrated alkali solution. A treatment time of four hours was found to be optimal in terms of composite flexural strength and modulus. Treatment of jute under the same conditions

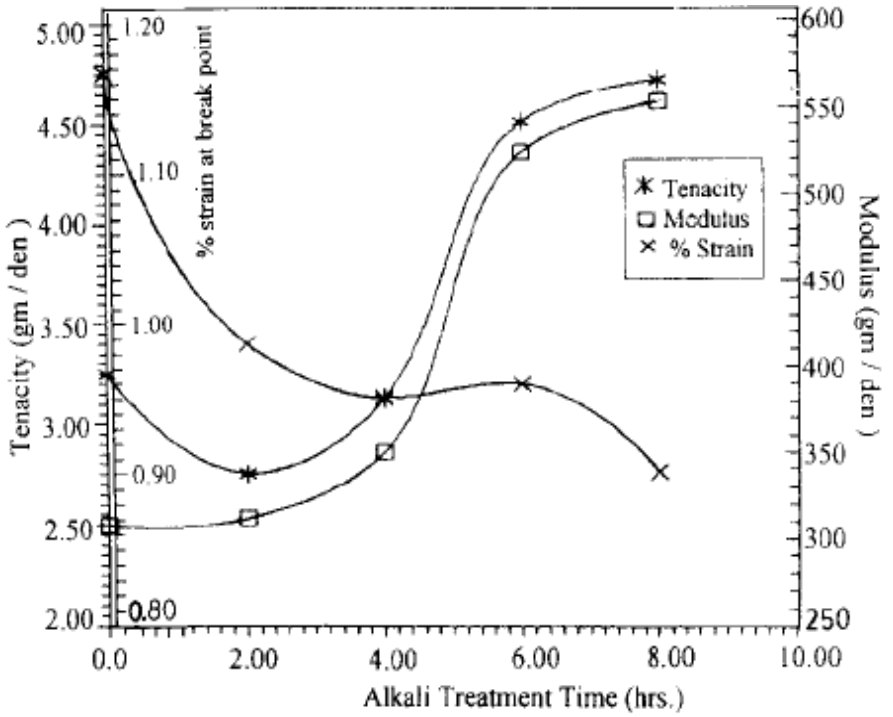


Fig. 2.17: Effect of alkali treatment on the tenacity, modulus, and strain at failure percentage of jute fibres with varying treatment time [138].

reduced the composite's tensile strength and modulus. The authors hypothesised that this reduction was due to defects incurred during the fabrication of composites. However Sutharson et al. [144] found 24 hours treatment of jute in 5% alkali solution is the optimum condition to improve tensile strength of jute-glass hybrid polyester composites.

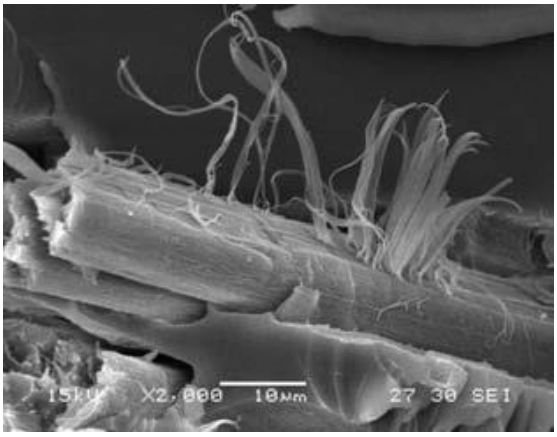


Fig. 2.18: SEM micrographs of the fracture surface of composites reinforced with untreated jute [133]

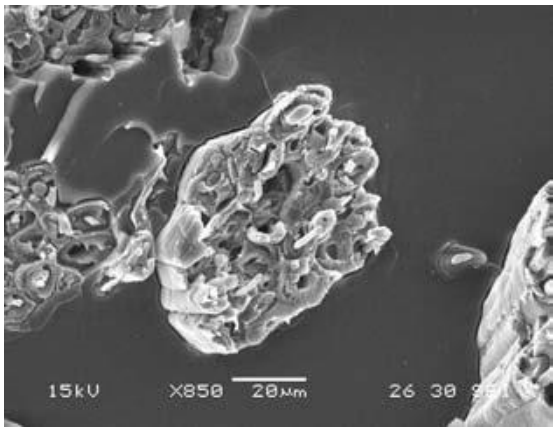


Fig. 2.19: SEM micrographs of the fracture surface of composites reinforced with alkali treated jute [133]

Mercerisation of jute fibres has also been studied by varying the concentration of alkali content and maintaining a constant treatment time [137]. The results showed that alkali treatment removed hemicellulose and lignin and that provided better interfacial adhesion between the fibres and the matrix which affects fibre pull-out during failure. Due to a weaker

interfacial bond for untreated fibres, more fibre pull out was observed (Fig. 2.18) than for alkali treated fibres (Fig. 2.19). A stronger interfacial bond also provides higher stress transfer [145]. Mercerised fibres also exhibited higher thermal stability. However the concentration of alkali, reaction time and temperature needs to be optimised.

Shrinkage is an important side-effect of the alkali treatment process (see Fig. 2.20) studied by Gassan et al. [146]. In that study, the shrinkage of jute fibres in relation to alkali concentration and the subsequent effect of shrinkage on yarn strength and modulus (Fig. 2.21) were reported. It was noted that the concentration of NaOH increases the shrinkage which has a negative effect on yarn strength and modulus.

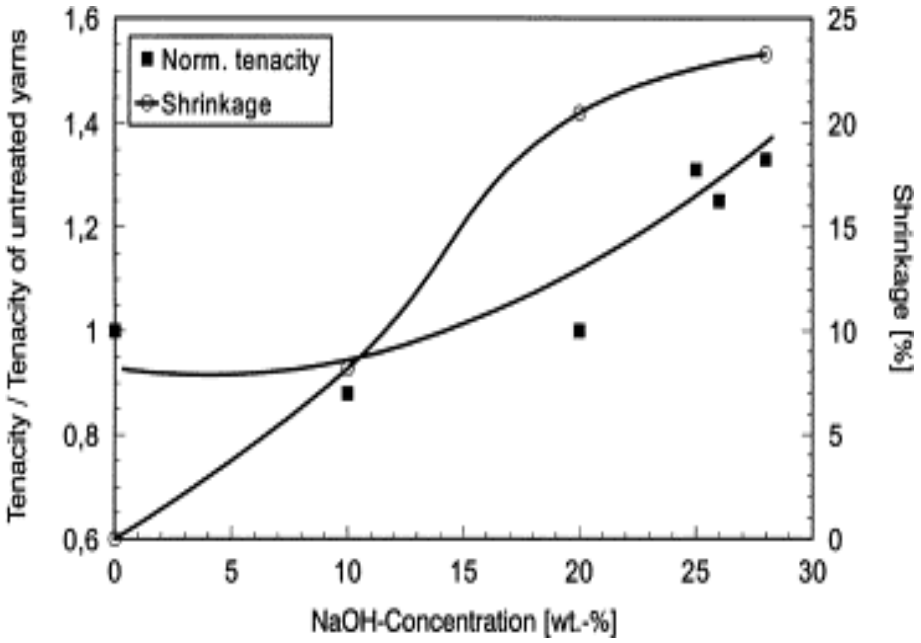


Fig. 2.20: Influence of NaOH concentration on tenacity (isometric conditions) and shrinkage of jute (treatment time 20 minutes and treatment temperature =20°C) [146].

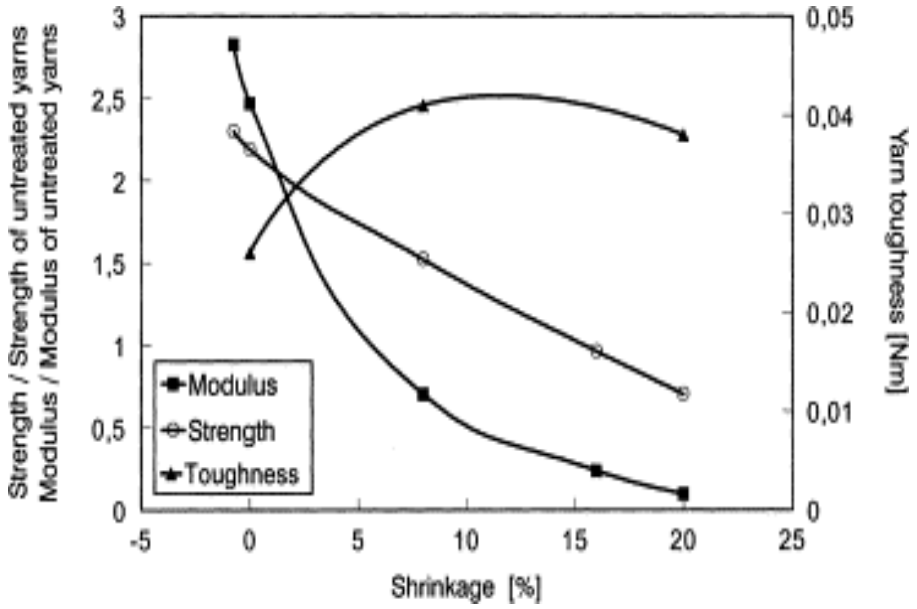


Fig. 2.21: Influence of shrinkage during alkalinisation process on the change of jute yarns tensile strength, tensile modulus and toughness (25% NaOH solution by weight, treatment time =20 minutes, treatment temperature =20°C) [146].

Generally plant fibres are treated in an aqueous solution of alkali. However Kabir et al. [137] used water and ethanol of 4:1 ratio as solvent to mercerise jute fibre. The treated jute fibre showed better thermal stability and composites made with alkali treated fibre showed up to 32% higher flexural strength and up to 48% higher strain to failure compared to untreated jute composites.

(II).b Acetylation

Moisture absorption makes plant fibres dimensionally unstable, hence limits their application in composites. Generally the fabrication temperature of plant fibre thermoplastic composites starts from 170°C and the reaction temperature of thermoset matrix is often higher than 100°C. Moisture adhering with plant fibres evaporates at this temperature and the probable effect is to generate voids [135]. Voids prohibit bond formation between fibre and matrix hence weaker interfacial strength may be obtained. Thus affinity to moisture of plant fibres need to be reduced which can be

performed in several ways. Acetylation is one of the most studied ways to reduce moisture absorption of plant fibres.

The acetylation of cellulose was discovered in 1865 [46]. Partially acetylated cellulose known as acetate rayon fibres and cellulose acetate plastics were commercialised in the early 1900s [147]. The theory of this method is to react fibre bearing hydroxyl groups (-OH) with acetyl groups (CH_3CO-) and hence removes the moisture. This reaction is known as esterification and it is performed by acetic acid and/or acetic anhydride [97]. It is a slow reaction process and can be accelerated using different types of catalyst such as sulphuric acid, pyridine, gamma-rays, potassium and sodium acetate [147]. Reactions with and without sulphuric acid catalyst are shown in equations 2.1 and 2.2.

$$\text{Fibre - OH} + CH_3COOH \xrightarrow[\text{Conc. } H_2SO_4]{(CH_3CO)_2} \text{Fibre-O-C-CH}_3 + H_2O \qquad (2.1)$$

Acetylation with acid catalyst [97]

$$\text{Fibre-OH} + CH_3\text{–C(=O)–O–C(=O)–CH}_3 \longrightarrow \text{Fibre–O–C(=O)–CH}_3 + CH_3\text{–C(=O)–OH} \qquad (2.2)$$

Acetylation without acid catalyst [97]

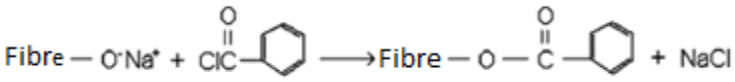
Acetylation not only improves the fibre hydrophobicity but also dimensional stability and fibre weight gains and reduces biological attack on fibres [148]. Acetylation on different fibres such as flax, hemp, jute, sisal, kapok, coir, empty fruit bunch were studied extensively by several researchers.

(II).c Benzoylation

Benzoyl chloride (C_6H_5COCl) is used in this method to modify the plant fibre surface. Generally this treatment is performed on alkali pre-treated

cellulosic fibre and the reactive hydroxyl (-OH) groups of plant fibres are converted to a benzoyl group (– $C_6H_5C=O$) (see reactions in equations 2.3

$$\text{Fibre} - \text{OH} + \text{NaOH} \longrightarrow \text{Fibre} - \text{O}^- \text{Na}^+ + \text{H}_2\text{O} \qquad (2.3)$$



(2.4)

Benzoylation of cellulosic fibre [149]

and 2.4) which makes fibres hydrophobic and creates a rough surface topography [150] that improves interfacial bonding properties with hydrophobic matrix and enhances the mechanical properties of composites [151]. Benzoylated sisal fibres also exhibited higher thermal stability [150].

(II).d Peroxide treatment

Organic peroxides containing the functional groups ROOR can be decomposed easily to free radical form as RȮ; RȮ. These free radicals react with hydrogen groups of fibres and low density polyethylene (LDPE) matrix and initiate a free radical reaction between them, offering improved mechanical properties of composites. Benzoyl peroxide (BP, $((C_6H_5CO)_2O_2)$ and dicumyl peroxide (DCP, $(C_6H_5C(CH_3)_2O)_2$) are used to modify the surfaces of plant fibres. Generally pre alkalised fibres are used for peroxide treatment. For peroxide treated sisal fibre Paul et al. [152] observed less – OH intensity in FTIR spectra with respect to raw fibre. They also demonstrated that the reaction between cellulose fibre and LDPE occurred due to peroxide-initiated free radical according to equations 2.5 to 2.9 [152].

$$\text{RO} - \text{OR} \longrightarrow 2\text{RȮ} \qquad (2.5)$$

$$\text{RȮ} + \text{Cellulose} - \text{H} \longrightarrow \text{ROH} + \text{Cellulosė} \qquad (2.6)$$

$$\text{RȮ} + \text{PE} - \text{H} \longrightarrow \text{ROH} + \text{PĖ} \qquad (2.7)$$

Probable reactions during composite preparation are as follow:

Cross linking of LDPE polymer matrix:

$$PĖ + PĖ \longrightarrow PE - PE \quad (2.8)$$

Combining cellulose and polyethylene radicals by grafting of PE on cellulose:

$$PĖ + Cellulosė \longrightarrow PE\text{-}Cellulose \quad (2.9)$$

Joseph et al. [153] treated pre-alkalised sisal fibre in 6% solution of DCP in acetone for 30 minutes and found 50% increase in tensile strength of peroxide treated sisal fibre composites with respect to untreated fibre composites. Peroxide treated oil palm empty fruit bunch/phenol formaldehyde composites showed 45% higher flexural strength and 29.5% higher flexural modulus with respect to raw fibre composites [154].

(II).e Sodium chlorite treatment

Sodium chlorite ($NaClO_2$) treatment of plant fibres is practically a bleaching process and is performed in an acid medium. Acidified $NaClO_2$ liberates chloric acid ($HClO_2$) and is oxidised to form chlorine dioxide (ClO_2) [97]. ClO_2 reacts with constituents of lignin and hemicellulose depending on treatment condition and decomposes them [155]. Removing hemicellulose makes fibres more hydrophobic [156] and discarding lignin makes fibres more flexible [157]. Composites made with $NaClO_2$ bleached sisal fibres provided inferior tensile properties, but better flexural and impact strength properties [157].

(II).f Acrylation and acrylonitrile grafting

In this method acrylic acid (CH_2=CHCOOH) is used to react with a hydroxyl group of cellulose, discharge a water molecule and form an ester linkage which improves moisture resistance properties of plant fibres [97].

2.5.3.(III) Use of coupling agent/matrix modification

As described earlier the principal compositions of plant fibres (cellulose and hemicellulose) contain highly reactive hydroxyl groups and they create hydrogen bonds thus showing less affinity to react with a hydrophobic matrix [135]. Hence a coupling agent is helpful to modify the fibre surface. Coupling agents have two different types of functional groups to make chemical bridges between functional groups of fibres and functional groups of matrix [151].

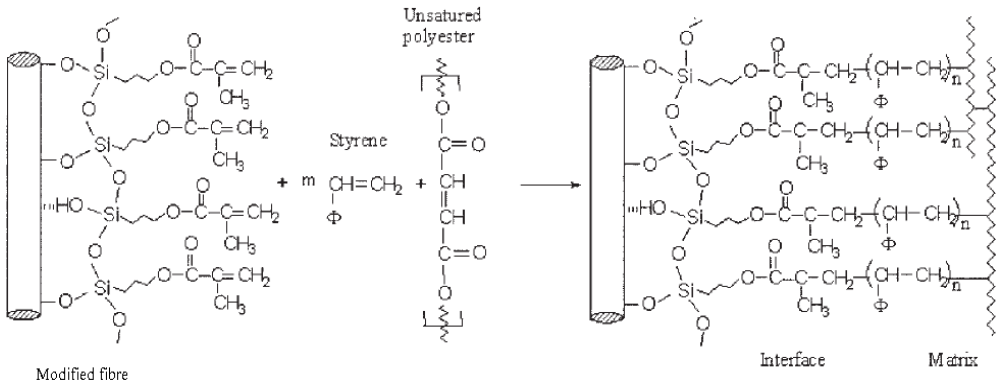
(III).a Silane treatment

Silane is a well-known coupling agent and several researchers studied its influence on the plant fibres composites. Silane treatment of plant fibres includes different stages of reactions such as hydrolysis, condensation and bond formation [97] (see equation 2.10 to 2.12). During hydrolysis functional groups of silanes called silanoles become activated in the presence of moisture and form hydrolysable alkoxy groups. At the

$$CH_2CHSi(OC_2H_5)_3 \xrightarrow{H_2O} CH_2CHSi(OH)_3 + 3C_2H_5OH \quad (2.10)$$

$$CH_2CHSi(OH)_3 + HO - Fibre \longrightarrow CH_2CHSi(OH)_2O - Fibre + H_2O \quad (2.11)$$

Silanisation [149]



-- (2.12)

Reaction between modified fibre and resin [158]

condensation stage silanes create chemical bonds with fibres and finally create bonds with the matrix. Tensile properties of untreated jute/polyester composites (JP), alkali treated jute/polyester composites (AJP), alkali and 0.1% silane treated jute/polyester composites (AJP-0.1%S), alkali and 0.3% silane treated jute/polyester composites (AJP-0.3%S), alkali and 0.5% silane treated jute/polyester composites (AJP-0.5%S) were studied by Sever et al. [159] as presented in Table 2.8. In this work the best mechanical properties were found for alkali and 0.3% silane treated fibre reinforced composites. Due to stronger bond formation, alkali and silane treated jute fibre composites showed higher tensile properties with respect to untreated fibre composites or only alkali treated fibre composites. Seki [160] also found better mechanical properties for alkali silane treated jute/epoxy and jute/polyester composites with respect to untreated and alkalised jute/epoxy and jute/polyester composites. Doan et al. [161] reported similar behaviour for jute/epoxy composites. Less fibre pull out was also noticed at failure for alkali and silane treated composites with respect to raw fibres or alkali treated fibre reinforced composites, which proves that there is a stronger interfacial bond for alkali and silane treated fibre composites (Fig. 2.22) compared to raw fibre composites [160].

Table 2.8: Mechanical properties of jute fibre/polyester composites (the data quoted are all average results taken from a minimum five tests) [159].

Composite	Fiber volume fraction V_f (%)	Tensile modulus (GPa)	Tensile strength (MPa)	Elongation at break (%)	Flexure modulus (GPa)	Flexure strength (MPa)	ILSS (MPa)
JP	38.2 ± 1.6	4.35 ± 0.18	47.64 ± 2.16	1.18 ± 0.06	3.54 ± 0.21	63.71 ± 3.16	8.91 ± 1.31
AJP	34.2 ± 1.8	5.57 ± 0.23	49.55 ± 2.03	1.28 ± 0.08	3.99 ± 0.31	62.25 ± 2.16	9.92 ± 1.51
AJP-0.1%S	36.5 ± 1.8	6.29 ± 0.31	65.56 ± 3.20	1.78 ± 0.15	5.13 ± 0.37	74.76 ± 2.67	14.67 ± 1.62
AJP-0.3%S	35.7 ± 1.5	6.30 ± 0.22	74.59 ± 3.71	1.95 ± 0.17	5.93 ± 0.34	81.54 ± 2.54	16.77 ± 1.66
AJP-0.5%S	36.6 ± 1.2	6.12 ± 0.24	54.68 ± 2.41	1.39 ± 0.09	4.56 ± 0.39	68.77 ± 1.78	11.54 ± 1.57

Wang et al. [162] found better tensile properties for composites made with alkali treated jute/polyester than alkali and silane treated jute/polyester composites. However alkali and silane treated jute/polyester composites exhibited superior tensile properties to untreated jute/polyester composites. Impact toughness was highest for alkali and silane treated jute/polyester composites with respect to other composites and was 43.2% higher than composites made with untreated jute/polyester. Kenechi et al. [163] noticed a 21% increase in tensile strength and nearly a 56% decrease in creep strain of silane treated jute/PLA composites with respect to untreated jute/PLA composites. Better creep behaviour of silane treated composite is also evidence of improved interfacial bonding between fibre and matrix. Khan et al. [164] also found better interfacial properties of silane treated jute/polycarbonate composites compared to untreated jute fibre composites.

Highly hydrophilic plant fibres swell due to moisture absorption and swelling of fibres develops micro-cracking of the composites which deteriorates composite mechanical properties [151]. Silane treatment can reduce swelling of fibres by forming chemical bonds with hydroxyl groups and hence improve the mechanical properties of composites. Silane treatment of plant fibres also fills up the micro pores at the fibre surface and develops mechanically interlocked coatings. Superior composite properties were also found with silane treated flax, henequen, coir, hemp and lyocell fibres with respect to untreated fibre composites.

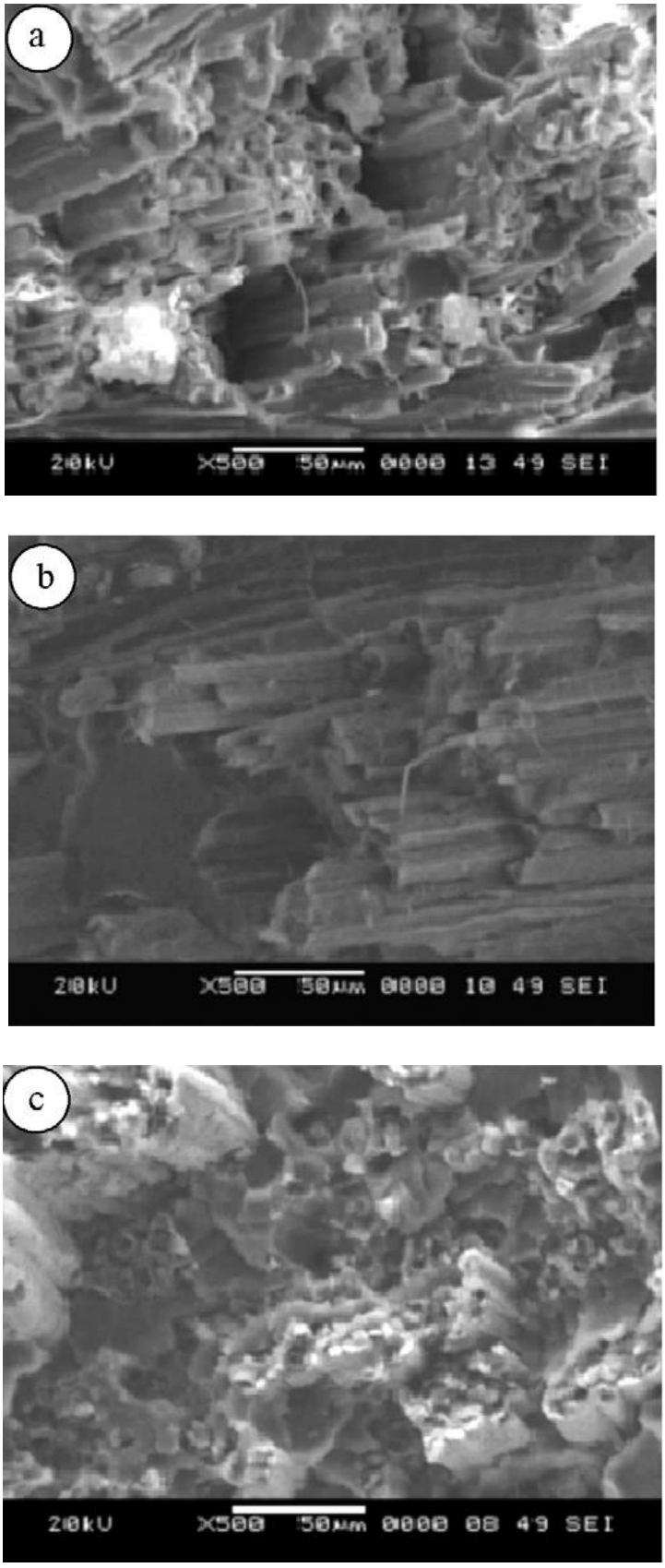
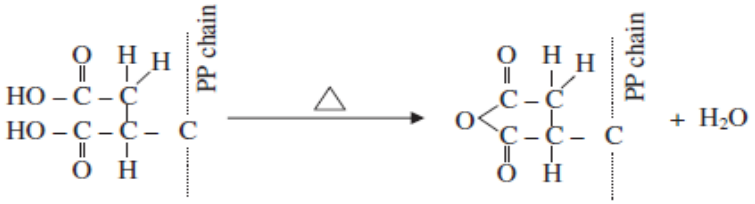


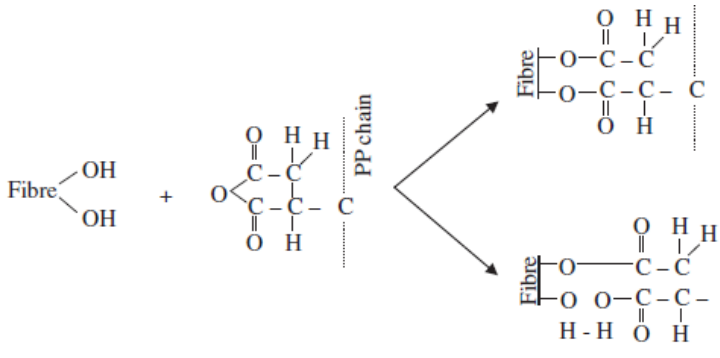
Fig. 2.22: SEM image of fracture surface of (a) untreated jute/epoxy (b) alkalised jute/epoxy and (c) alkalised silane treated jute/epoxy composites [160].

(III).b Maleated coupling agents

These are widely used substances to improve the mechanical properties of composites. Maleated coupling agents modify both fibre surface and polypropylene (PP) matrix that contributes to stronger interfacial bonding thus enhancing the mechanical properties of composites. Maleic anhydride builds as covalent bond with matrix and fibres and the reaction is completed in two steps [165] (see equation 2.13 and 2.14). In the first step activation of copolymer is achieved at 170°C, creating a covalent bond with the PP matrix leaving water molecules. The second step is an esterification reaction with the fibre. This treatment enhances the surface energy of the fibre bringing it closer to the surface energy of the matrix [165] which provides better wettability and stronger interfacial adhesion.



(2.13)



(2.14)

Mohanty et al. [166] treated jute fibre in maleic anhydride grafted polypropylene (MAPP) (0.3%, 0.5% and 1%) solution for 5 minutes at 100°C and obtained superior composite properties for jute treated with 0.5% solution. They observed 30%, 72% and 24% increase in tensile, flexural and impact strength respectively with respect to untreated fibre

composites at 30% fibre volume fraction. Maleic anhydride treated banana, hemp and sisal reinforced novolac composites absorbed less water as observed by Mishra et al. [167]. They also noticed higher mechanical properties with respect to untreated fibre composites in terms of Young's modulus, flexural modulus, hardness and impact strength.

2.5.4 Processing of plant fibre reinforced composites

Almost all traditional composite manufacturing processes can be used for plant fibre polymer composites [168]. Hand lay-up, bag moulding process, compression moulding, resin transfer moulding, filament winding, pultrusion and injection moulding can be used to fabricate PFCs.

Hand lay-up is the oldest, simplest and most common method of composite manufacturing [2] and several plant fibres such as jute, flax, hemp, pineapple, kenaf and banana fibres were processed using this method successfully. Bag moulding is also a very common method used to infuse both natural and synthetic fibres. Bag moulding is a successful method to produce plant fibre polymer composites. Various researchers utilised this method to infuse jute, flax, sisal and other plant fibres.

Pultrusion is a continuous method to produce a constant cross-section profile [169]. Plant fibres such as jute, flax, kenaf, hybrid hemp/wool and others can be processed using this technique.

Compression moulding is a popular and an appropriate method to produce PFCs due to its high reproducibility and low moulding time [102]. It is a high volume and high pressure method suitable for different forms of fibre assembly such as unidirectional (UD), woven fabric, non-woven fabric, random fibre mat to produce small to large composite components. Jute, bagasse, kenaf, wheat straw and other plant fibres can be processed by

this method. Resin Transfer Moulding (RTM) is a positive pressure process and various plant fibres such as jute, hemp, flax, kenaf, sisal and other plant fibres are infused using this method.

Injection moulding is suitable for short fibre reinforced composites. Different complex shaped parts can be produced with this method using a range of plant fibres. Jute, sisal, abaca, pineapple leaf fibre, wheat straw and other fibres were processed successfully using this method. However the fibre volume fraction is low here (usually not greater than 20% [170]). Filament winding is a continuous method suitable for PFCs manufacturing. Several researchers used filament winding to manufacture different plant fibre composites based on flax, jute, hemp and others.

From Fig. 2.9 and 2.10 [47] it can be noticed that pre-preg UD composites are the best in respect of tensile modulus and tensile strength followed by compression moulded and RTM UD thermoset composites. Random composites showed the lowest values of tensile modulus and tensile strength and the tensile properties of biaxial composites are in between random and UD composites. However selection of fibre and matrix is limited in pre-pregging and the RTM method is suitable to infuse yarn and long staple (fibre longer than 100 mm) plant fibres to produce UD thermoset composites using liquid resin and hence this process was chosen for this study.

2.5.5 Influence of yarn twist on the properties of composites

By nature plant fibres are short [171] and several techniques are available to manufacture composites using short fibres [172-174]. However some techniques such as filament winding or pultrusion need continuous fibres [175]. Continuous fibres are also required to produce UD composites [176]. Yarn is a very common form of continuous supply of fibres [11, 177, 178].

Formation of yarn needs to bind continuously a certain number of fibres together. Binding of fibres can be performed in different ways such as true twisting, false twisting, wrapping by constituent fibres or an external filament and adhesive bonding [179]. Introducing twist is the primary and most common binding mechanism [180] to hold fibres together, which increases inter fibre friction and provides strength to yarn [181]. However twist brings fibre obliquity in the yarn axis (Fig. 2.23) [182, 183] and described by Hearle et al. [184] considering the following assumptions.

- Yarns are circular in cross-section thus cylindrical in shape.
- Fibres remain in different radii of this concentric cylinder.
- Every single fibre follows uniform helical path around a concentric cylinder.
- Helix angle of fibre gradually increases from centre to surface of the yarn due to constant twist length.
- Fibre packing in yarn is constant.
- A yarn cross-section is consists of large number of fibres.

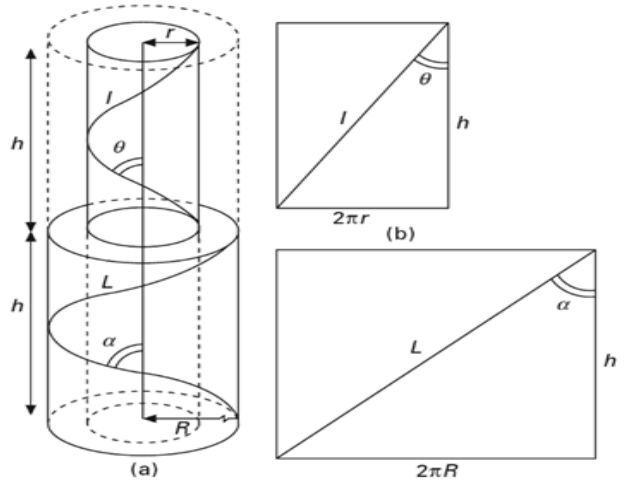


Fig.2.23 Structure of an idealised staple twisted yarn [185]

Here,

R = yarn radius

r = radius of the cylinder containing helical path of a specific fibre

T = yarn twist per metre

H = length of one turn of twist (cm)

α = angle of twist at yarn surface

θ = twist angle at radius r

l = fibre length (cm) in one turn of twist at radius r

L= fibre length (cm) in one turn of twist at radius R

Thus,

$$H = 1/T \quad (2.15)$$

$$l^2 = h^2 + 4\Pi^2 r^2 \quad (2.16)$$

$$L^2 = h^2 + 4\Pi^2 R^2 \quad (2.17)$$

$$\tan\theta = 2\Pi(r/h) \quad (2.18)$$

$$\tan\alpha = 2\Pi(R/h) = 2\Pi RT \quad (2.19)$$

Thus increasing the value of T increases the value of α that reduces the mechanical properties of yarn due to higher off axis loading [13] on fibres in addition higher yarn compactness [186]. More compact yarns restrict resin penetration [18, 187] and there is a possibility of forming voids [18, 178]. Twist not only introduces fibre obliquity in the yarn but also fibre bending and bending curvature (s) is presented according to the eq. 2.20 [185] .

$$S = \sin^2\alpha/r \quad (2.20)$$

Bending curvature of fibre is also realised by the angle of twist and bending of fibres could possibly introduce micro stretching at the outer sides of fibres and micro contraction at the inner sides of fibres. This can cause micro damage of fibres that may reduce the failure strength of yarns [188].

Hence low twisted or twist-less fibrous materials are preferable for composites manufacture [187, 189] and twist-less wrap spun flax polypropylene composites exhibited 30% increase in flexural modulus than

twisted flax yarn polypropylene composites when fibre weight fraction was more than 40% [190]. Goutianos and Peijs [189] mentioned 0.76 TPI is the optimum level of twist for plant fibres that doesn't deteriorate the mechanical properties of composites. This is a very low amount of twist for textile processing, where around 2.27 TPI [189] is an optimum twist level for manufacturing textile products suitable for composite fabrication.

Yarn manufacturing is a labour intensive and expensive process. Shah et al. [13] mentioned that yarn can be 10-30 times more expensive with respect to raw fibres and the higher the level of twist per unit length the lower will be the yarn production according to the equation 2.15.

Production per unit time= spindle speed/ twist per unit length (2.15)

Hence twisting increases cost and the use of twist-less UD fibrous material such as slivers for manufacturing composites can reduce fibre processing cost as well as better utilising of tensile properties of fibre in composites. Hydroxyethyl cellulose (HEC), a natural binding agent was used by Shah et al. [180] as an alternative method of twist and results in a 230% increase in tensile strength and 75% increase in stiffness of HEC treated low twisted yarn with respect to untreated yarn.

2.5.6 Mechanical properties of plant fibre composites

The mechanical properties of PFCs and synthetic fibre composites were studied extensively by several researchers. Researchers analysed tensile, flexural, impact, compression, fibre/matrix interfacial strength, fatigue, fragmentation, dynamic mechanical properties, water retention, ageing behaviour and others for PFCs.

Among the PFCs presented in Table 2.9 flax composites showed the highest tensile modulus (42.5±3.8 GPa [191]) followed by curaua

composites (36 GPa [192]) and then jute composites (30.8 GPa [193]). The tensile modulus of these composites is comparable to the tensile modulus of glass fibre composites [194, 195] however the tensile strength of glass fibre composites is superior to PFCs. Thus uses of PFCs are suitable to tensile stiffness oriented applications and limited in tensile strength intensive applications.

Table 2.9: Tensile properties of some common plant fibre and glass fibre composites.

Fibre	Form of fibre	Treat-ment	Method	Matrix	V_f/W_f (%)	Tensile modu-lus GPa ±Std dev.	Tensile streng-th MPa ±Std dev.	Refe-ren-ces
Jute	UD	Untrea-ted	Compre-ssion	PBS[$]	W_f =60	30.8	147.5	[193]
Jute	UD	Untrea-ted	Compre-ssion	Poly-ester	V_f=50	27.2	225	[196]
Jute	UD	Untrea-ted	RTM	Poly-ester	V_f =31.7	16.1 ±0.8	175.1 ±10.3	[21]
Jute	Quasi-UD	Untrea-ted	Filament winding	Epoxy	V_f =40	16	160	[146]
Jute	UD	Untrea-ted	VARI[!]	Epoxy	V_f=25	14.59 ±2.28	112.69 ±18.31	[197]
Jute	UD*	Untrea-ted	Compre-ssion	Polypro-pylene	V_f =21.2	9.3	142	[198]
Jute	UD*	Untrea-ted	Compre-ssion	Poly lactic Acid	V_f=38	8.5	78.5	[198]
Jute	UD	Untrea-ted	Film stacking	Polypro-pylene	V_f =49.2	6	107	[177]
Jute	UD	Untrea-ted	Compre-ssion	Poly-ester	W_f =50	5.7	165	[199]
Jute	UD	Alkali treated	Compre-ssion	PBS[$]	W_f =50	33	230	[193]
Jute	Quasi-UD	Alkali treated	Filament winding	Epoxy	V_f = 40	24	220	[146]
Jute	UD	PVA and PP coated	Film stacking	Polypro-pylene	V_f =49.2	6.6	122	[177]
Jute	Woven	Untrea-ted	Hand lay-up	Poly-ester	V_f=36	9.6	80	[200]
Jute	Woven	Untrea-ted	Hand lay-up	Poly-ester	V_f =40	9.58 ±0.22	80.19 ±5.10	[201]

Jute	Woven	Untrea-ted	Pre-preg	Epoxy	W_f =43	9.24	86.71	[202]
Jute	Woven	Untrea-ted	Hand lay-up	Poly-ester	V_f =45	7.00 ±1.12	60 ±2.80	[203]
Jute	Woven	Untrea-ted	Hand lay-up	Vinyl-ester	W_f = 40	6	41	[204]
Jute	Woven	Untrea-ted	Hand lay-up	Epoxy	V_f=35	5.46 ±0.26	61.41 ±3.49	[205]
Jute	Woven	Untrea-ted	Compre-ssion	Poly-ester	V_f=37	5.5	58	[131]
Jute	Bidirec-tional	Untrea-ted	Hand lay-up	Epoxy	W_f =48	4.5	112	[206]
Jute	Ran-dom	Untrea-ted	Compre-ssion	Poly-ester	V_f=32	5.8 ±0.11	35.4 ±2.52	[207]
Jute	Woven	2% Alkali +3% Silane treated	Hand lay-up	Epoxy	V_f=35	9.28 ±0.23	86.73 ±4.02	[205]
Jute	Woven	Alkali 4hr tension	Hand lay-up	Vinyl-ester	W_f =40	7.7	60	[204]
Jute	Woven	Silicon treated	Compre-ssion	Poly-ester	V_f=37	7	85	[131]
Jute	Woven	Fluoro-carbon treated	Compre-ssion	Poly-ester	V_f=37	6.9	92	[131]
Jute	Woven	5% Alkali +1% Silane	Hand lay-up	Poly-ester	V_f=37	6.75 ±0.8	62.49 ±3.0	[160]
Jute	Woven	2% Alkali treated	Hand lay-up	Epoxy	V_f=35	6.39 ±0.42	66.49 ±3.20	[205]
Jute	Woven	2% Alkali + 0.3% silane	Compre-ssion	Poly-ester	V_f=35	6.30 ±0.22	74.59 ±3.71	[208]
Jute	Woven	Blea-ched	Hand lay-up	Vinyl-ester	V_f =19.8	6.3	44	[209]
Jute	Woven	5% Alkali	Hand lay-up	Epoxy	V_f=35	6.28 ±0.8	65.35 ±2.6	[160]
Jute	Woven	5% Alkali	Compre-ssion	Poly-ester	V_f=37	6	60.5	[131]
Jute	Woven	Alkali 24hr tension	Hand lay-up	Vinyl-ester	W_f =40	5.9	45	[204]

Jute	Woven	5% Alkali + 1% Silane	Hand lay-up	Epoxy	V_f=30	5.74 ±0.8	67.06 ±2.5	[160]
Jute	Woven	2% Alkali	Compre-ssion	Poly-ester	V_f=35	5.57 ±0.23	49.55 ±2.03	[208]
Jute	Woven	5% Alkali	Hand lay-up	Poly-ester	V_f =34	5.57 ±0.8	45.55 ±2.0	[160]
Jute	Woven	Blea-ched	Hand lay-up	Epoxy	V_f=46	4.8	57.5	[210]
Jute	Ran-dom	MAHgP P^+	Injection moulding	Polypro-pylene	V_f=32	4.4	49	[161]
Jute	Ran-dom	Benzene diazoniu m salt	Injection moulding	Polypro-pylene	W_f =35	2.2	28	[211]
Jute	Ran-dom	Untrea-ted	Compres sion	Poly-ester	W_f =30	2	50	[199]
Jute	Ran-dom	Glass: Jute 50:50	Injection moulding	Polypro-pylene	W_f =40	1.88	29	[212]
Flax	UD	Untrea-ted	Compre-ssion	Poly-ester	W_f =55	42.5 ±3.8	157±2 8	[191]
Flax	UD	Untrea-ted	Autocl-ave	Epoxy	V_f =40	28	133	[213]
Flax	UD	3% Alkali	Autocl-ave	Epoxy	V_f =40	35	173	[213]
Flax	Woven	Untrea-ted	Hand lay-up	Epoxy	V_f =55	4.5	53.7	[214]
Flax	Woven	5% Alkali	Hand lay-up	Epoxy	V_f =55	5	71	[214]
Flax	Ran-dom	Untrea-ted	Compre-ssion	Poly-ester	W_f =50	11.5 ±1.0	115±6	[191]
Flax	Ran-dom	Untrea-ted	Compre-ssion	PLA	V_f =30	7.95	100	[215]
Flax	Ran-dom	Untrea-ted	Injection	$MAPP^+$	W_f =30	5.00	39.50	[147]
Flax	Ran-dom	18% Acetyla ted	Injection	$MAPP^+$	W_f =30	5.00	48.5	[147]
Hemp	UD	Untrea-ted	Compre-ssion	$PET^£$	V_f =48	27.6	227	[11]
Hemp	Ran-dom	Untrea-ted	Compre-ssion	PP	V_f =30	3.5	35	[215]
Hemp	Ran-dom	Blea-ched	Hand lay-up	Epoxy	V_f =43	5.2	60	[210]
Sisal	UD	Untrea-ted	Extru-sion	LDPE	V_f =30	3.09	31.12	[153]

Sisal	UD	Merce-rised under tension	Compre-ssion	Soy protein	V_f =54.4	3.80	188.5	[216]
Sisal	UD	Merceri-sed	Extru-sion	LDPE	V_f =30	3.33	34.27	[153]
Sisal	Ran-dom	Untrea-ted	Compre-ssion	PP	V_f =30	3.0	24	[215]
Sisal	Ran-dom	Untrea-ted	Compre-ssion	PF**	W_f =40	1.15	37	[154]
Sisal	Ran-dom	Untrea-ted	Extru-sion	LDPE	V_f =30	0.78	14.7	[153]
Sisal	Ran-dom	Alkali	Compre-ssion	PF**	W_f =40	1.3	35	[154]
Sisal	Rando m	Merceri-sed	Extru-sion	LDPE	V_f =30	1.14	18.8	[153]
Sisal	Ran-dom	Acety-lated	Compre-ssion	PF**	W_f =40	0.8	19	[154]
Sisal	Ran-dom	Silane	Compre-ssion	PF**	W_f=40	0.7	15	[154]
Sugar Palm	UD	Untrea-ted	Hand lay-up	Epoxy	V_f =10	3.78	43	[217]
Sugar Palm	UD	0.25M alkali	Hand lay-up	Epoxy	V_f =10	3.33	50	[217]
Cura-ua	UD	Untrea-ted	Compre-ssion	Corn starch	V_f =70	36	327	[192]
Coir	UD	Untrea-ted	Compre-ssion	PBS[$]	W_f =25	1.4	43	[218]
Coir	UD	5% Alkali Treated	Compre-ssion	PBS[$]	W_f=25	2.2	52	[218]
Bana-na	Woven	Untrea-ted	Hand lay-up	Epoxy	W_f =16	0.95	20	[219]
Bana-na	Woven	1% Alkali Treated	Hand lay-up	Epoxy	W_f =16	2.04	42.15	[219]
Bana-na	Ran-dom	Untrea-ted	Hand lay-up	Epoxy	W_f =16	0.73	14.51	[219]
Bana-na	Ran-dom	1% Alkali Treated	Hand lay-up	Epoxy	W_f =16	1.68	33.6	[219]
Bam-boo	Woven	Untrea-ted	Hand lay-up	Epoxy	V_f =55	9.2	120.1	[214]
Bam-boo	Woven	5% Alkali	Hand lay-up	Epoxy	V_f =55	10.5	150	[214]
Pine-apple	Ran-dom	Untrea-ted	Compre-ssion	Poly-styrene	W_f =50	0.82 ±0.16	22.64 ±1.25	[220]

Pine-apple	Ran-dom	4% Alkali	Compre-ssion	Poly-styrene	W_f =50	1.28 ±0.13	29.95 ±1.36	[220]
Glass	UD	Untrea-ted	Hand lay-up	Epoxy	V_f = 55	23	730	[195]
Glass	Fabric	Untrea-ted	Hand lay-up	Poly-ester	V_f =37	15.03 ±3.4	341.5 ±20.6	[194]
Glass	Fabric	3% silane treated	Hand lay-up	Poly-ester	V_f =37	18.0 ±3.0	395.8 ±23.9	[194]

*Micro-braid yarn V_f=Fibre volume fraction W_f=Fibre weight fraction, !Vacuum Assisted Resin Infiltration +Maleic Anhydride Grafted Polypropylene, **Phenol formaldehyde, $ Poly(butylene Succinate) £ Polyethylene terephthalate

According to the data presented in Table 2.9 different surface modifications of fibres improved the tensile properties of composites and various techniques of physical and chemical fibre surface modifications were analysed by researchers as described in Section 2.5.3.

Hybridisation of different types of plant fibres [221-227] as well as plant and synthetic fibres [228-233] was characterised by a number of researchers to understand its influence on the mechanical properties of composites. Hybridised plant and glass fibre composites were positive as the mechanical properties were superior to sole PFCs. Gujjala et al. [229] studied the mechanical properties of woven jute and glass mat (50:50 ratio) epoxy hybrid composites manufactured by hand lay-up maintaining 17.5% fibre volume fraction. They found the tensile modulus and tensile strength of glass composites and jute composite were 6 GPa, 116 MPa and 2 GPa, 52 MPa respectively. However the tensile modulus and tensile strength of jute/glass hybrid composites were 4.8 GPa and 85 MPa respectively. They found the flexural strength and flexural modulus of jute/glass hybrid composites were very similar to glass fibre composites.

Fibre volume fraction is a key factor that affects the mechanical properties of composites. Composites fail at low load if the fibre volume fraction is low, since higher percentage of resin increases the brittleness of composites [234]. Very high fibre volume fraction (70% or more for plant fibres) decreases the mechanical properties of composites due to improper wetting of fibres by resin [196]. Achieving higher fibre volume fraction is very important for plant fibre composites to reduce the use of fossil based matrices and UD composites are favourable to achieve higher fibre volume fraction [11]. However it is very difficult to achieve a high fibre volume fraction for yarn reinforced composites. As a jute yarn is made with 50-100 [235] fibres in cross section, yarn diameter may be 10-15 times higher than fibre diameter [236-238]. Thus even for closely packed yarns in composites an aperture would be created among them which would lead to resin rich regions in composites, which limits the attainable fibre volume fraction for composites made with plant fibre yarn to be between 35-50% [239]. Using loose fibres in composites manufacturing may overcome this problem, giving a better distribution of fibres as well as higher fibre volume fraction (60% [196]).

Similar to fibre volume fraction, fibre distribution also an important parameter for determining the mechanical properties of composites [234]. Uniform distribution of fibres reduces resin rich regions and hence reduces the brittleness of composites. Good dispersion assists the wetting of fibres. UD composites made with sliver [196], roving [240] and low twisted yarn [241] have the facility to disperse fibres more evenly with respect to some other form of textile products such as woven fabric or highly twisted yarn.

Voids are an inherent characteristic that deteriorates the mechanical properties of composites. Voids may be created due to several reasons

such as trapping of air during resin mixing, moisture absorption of reinforcement fibres during storage or processing and poor placement of fibres into the mould [242]. It is reported that PFCs may have high void content [12]. It can be up to 5% for composites having below 40% fibre volume fraction [243, 244] and can increase up to 25% with increased fibre volume fraction [12, 245]. However less than 1% void content is the requirement for aerospace applications whilst up to 5% void content may be acceptable for other applications such as automotive and marine [246]. The composite moulding process affects the void content according to the following sequence: Hand lay-up > Compression moulding > Infusion technique (vacuum infusion > RTM > vacuum assisted RTM) > Pre-preg (with autoclave curing) [21].

Physical properties of fibres such as length, strength, aspect ratio, fineness affect the tensile properties of composites. Longer fibre reinforced composites showed better tensile properties with respect to shorter fibre reinforced composites [247, 248]. However too long fibres may be intermingled with each other hence orientation would be disrupted and poor mechanical properties of composites may be obtained [234]. The tensile properties of composites are mostly dependent on the tensile properties of fibres and hence higher tensile properties of reinforcement fibres are desirable. Takagi and Ichihara [248] commented that fibres having lower aspect ratio (e.g. 20 for bamboo fibres) can be used as filler only and the aspect ratio needs to be higher than 40 for sufficient stress transfer from matrix to fibres [234]. Generally finer fibres are stronger than coarser fibres [249] and have higher aspect ratio [250]. Finer fibres have more surface area to wet by resin and thus better bonding with resin and higher stress transfer from matrix to fibre is possible. Hence finer fibres are more suitable for composite manufacture.

Interfacial bonding strength between fibre and matrix affects the mechanical properties of composites. Weak interface composites propagate damage more quickly than strong interface composites [251]. During application of load to a composite, stress is first generated in the matrix and is transferred to the fibres through interfacial adhesion [234], thus a strong interfacial bond is very important to enhance the mechanical properties of composites. Fibre pull-out occurs for weaker interfacial bond and the result is premature composite failure [252].

2.6 Conclusions

At the end of this chapter several features can be highlighted related to optimising the tensile properties of plant fibre composites.

Plant fibres have good prospects for composite reinforcement due to their low weight, very good tensile modulus, abundant supply and low cost. However to meet these prospects there is a need to overcome some challenges such as reducing the hydrophilic nature and improving interfacial bonding.

Bast fibres have good mechanical properties and are suitable for manufacturing of composites. They have high cellulose content, high aspect ratio and low micro-fibril angle which are desirable properties for composite production.

Mechanical properties of thermoset PFCs are better than thermoplastic PFCs, hence for load bearing applications thermoset resins may be the first choice. Thermoset resin such as polyester can be cured at room temperature which is energy saving with respect to thermoplastic resin. Generally thermoplastic resins are solid at room temperature and require

heat to melt which consumes energy, and melting at high temperature (typically around 200°C or more) can cause thermal damage of plant fibres.

Several physical modifications of fibres such as corona treatment, plasma treatment, hybridisation of reinforcement material and stretching; chemical modifications of fibres such as alkali treatment, acetylation, benzoylation, peroxide treatment, sodium chlorite treatment and acrylation; and modification of matrices which includes the use of several types of coupling agents such as silane and maleated coupling agents are considered to improve the mechanical properties of composites.

The prepregging technique produces the best quality PFCs followed by compression and RTM/vacuum infusion techniques. However in prepregging the selection of fibre as well as matrix is limited. Compression moulding is not very suitable for plant fibres due to application of high pressure and often high temperature which may cause damage to plant fibres. RTM is a very suitable method to infuse plant fibres where a wide range of fibres and resin can be chosen. It is a suitable method to infuse bast fibre such as jute using liquid resin such as polyester. Good quality composites can be manufactured in this process and hence this is used in this work.

Processing of fibres needs to be optimised and compatible with composite production where minimum processing, high level of alignment and minimum or no twist would be employed. Reduction of processing steps is very important to save time and money, for example using slivers instead of yarn to manufacture UD composites. However this approach should not deteriorate the quality of composites.

Higher fibre volume fraction improves the quality of composites, however very high fibre volume fraction can have a detrimental effect due to improper wetting of fibres with resin.

CHAPTER 3

INFLUENCE OF YARN HAIRINESS ON THE MECHANICAL PROPERTIES OF COMPOSITES

3.1 Summary

The influence of the hairiness of jute yarn on the mechanical properties of UD composites is studied in this chapter. Tossa jute yarn as received from the supplier is denoted as 'hairy yarn' and after removing hairs, yarn is denoted as 'hairless yarn'. Hairs are removed manually by burning using a candle flame. Tensile properties of hairy and hairless yarn are measured. Differential scanning calorimetry (DSC), X-ray photoelectron spectroscopy (XPS) and fourier transform infrared (FTIR) analysis are performed to understand the difference in properties between hairy and hairless yarns. UD matt of yarn is prepared by wrapping it on a drum and applying hydroxyethyl cellulose (HEC, a water based adhesive) as binder. UD composites are manufactured using hairy and hairless yarns separately. Unsaturated polyester (UP) resin is used as matrix and composites are manufactured via resin transfer moulding (RTM). Scanning electron microscopy energy-dispersive X-ray spectroscopy (SEM-EDX) is performed to investigate the lumen content of jute fibre fabricated in composites to find its contribution to void content of composites. Optical microscopy is used to view the physical appearance of hairy and hairless yarns. Composites are tested for tensile properties.

3.2 Introduction

'Hairiness is a property which indicates the amount and length of fibre ends and loops protruding from the body of the yarn' [253]. Hairiness is an inherent character of a spun yarn composed of staple fibres (fibres of

limited length) and is considered as a fault in textiles. Hairiness is undesirable for most textiles, but is desirable for certain types of cloths (such as flannels, soft knitwear, towels). Thus yarn with low hairiness is a common demand. Yarn hairiness influences fabric pilling, handle, comfort and appearance [254].

As a protruding fibre from the yarn surface, one end of the hair is embedded and twisted into the yarn body and the other end (which is open) is known as the hair. Most of the hairs are affixed strongly to the yarn. Hairs can be wetted easily by resin during infusion and bonding between hairs and resin may influence the interfacial properties of composites with respect to hairless. Thus mechanical properties of composites manufactured with hairy yarn may be different from the mechanical properties of composites manufactured with hairless yarn. To investigate the influence of hairiness on the mechanical properties of composites hairy and hairless yarns are used to fabricate in composites.

Staple yarn (yarn consisting of staple fibres) consists of 60-200 fibres in cross-section [14-16] and a small number of these fibres may contribute to hairs. Thus the influence of hairiness on the mechanical properties of composites may not be significant in the longitudinal direction of yarn. Hence hairiness may not be a dominating factor to modify the mechanical properties of composites in the longitudinal direction of the yarn and the influence of hairiness may be more pronounced in the transverse direction of the yarn. Tensile tests of composites are performed in the longitudinal as well as the transverse direction to assess this.

Section 3.3 contains a description of the material used, removal of hairiness and manufacturing method, Section 3.4 describes the testing of yarns and composites. Section 3.5 presents the results obtained from

testing as well as discussion of the results. Conclusions from this chapter are included in Section 3.6.

3.3 Materials and Methods

3.3.1 Materials

Tossa jute yarn is used as reinforcement material in this experimental work, supplied by Janata Jute Mills Ltd. Bangladesh. This is a single ply jute yarn having 8 lbs/spyndle (280 Tex, 1 lbs/spyndle = 35.090 Tex) linear density and 4.65 TPI. The yarn is supplied in spool form (see Fig. 3.1) and no extra finishes or coatings are used except the emulsion applied during spinning that contains jute batching oil. Unsaturated polyester, trade name POLYLITE® 420-100 by Reichhold UK Ltd. is used as the matrix material. Methyl ethyl ketone peroxide 35% in phthalate plasticizer trade name Butanox Catalyst by K & C Mouldings (England) Ltd. is used as catalyst and cobalt (11) (2-ethylhexanoate) 1% CO in diisobutyl phthalate trade name Accelerator NL-40P by K & C Mouldings (England) Ltd. is used as accelerator. The mould release agent is Chem lease® PMR-90 manufactured by Chem-Trend (Deutschland) GmbH. Hydroxyethyl cellulose (HEC) from Dow Europe GmbH, commercial name Cellosize HEC QP-52000H is used as binding material for the UD fibrous mat of yarn.



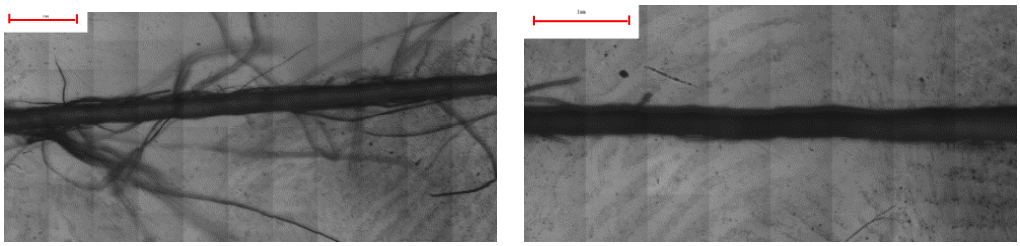
Fig. 3.1 Jute yarn spool

3.3.2 Preparation of yarn

As hairs are considered as a fault, several techniques are practised to reduce or remove hairs. Yarn hairiness decreases with increasing twist per unit length [255, 256]. However increase in twist has a number of detrimental effects such as enhancing the cost of yarn, bringing higher fibre obliquity as well as misalignment of fibres in the axis of the yarn, increasing yarn crimp in woven fabric and hindering penetration of resin inside the yarn during composite manufacturing [13]. Thus this is not a suitable way to reduce hairiness. In textiles some established methods are practised to remove hairs. A popular method of removing hairs is burning [257]. Burning can be performed using a flame [258] or using hot plate(s) or hot roller(s) [259]. Hairs can also be removed by enzyme treatment [260].

In this work yarn hairs are removed by burning and the burning of yarn hairs is performed manually using a candle flame. This task is performed carefully to avoid any damage to the yarn. Images of hairy and hairless yarns are presented in Fig. 3.2 (A) and (B). A large number of hairs is

noticed on the surface of the hairy yarn (Fig. 3.2 (A)) whilst the surface of hairless yarn (Fig. 3.2 (B)) is relatively smooth.



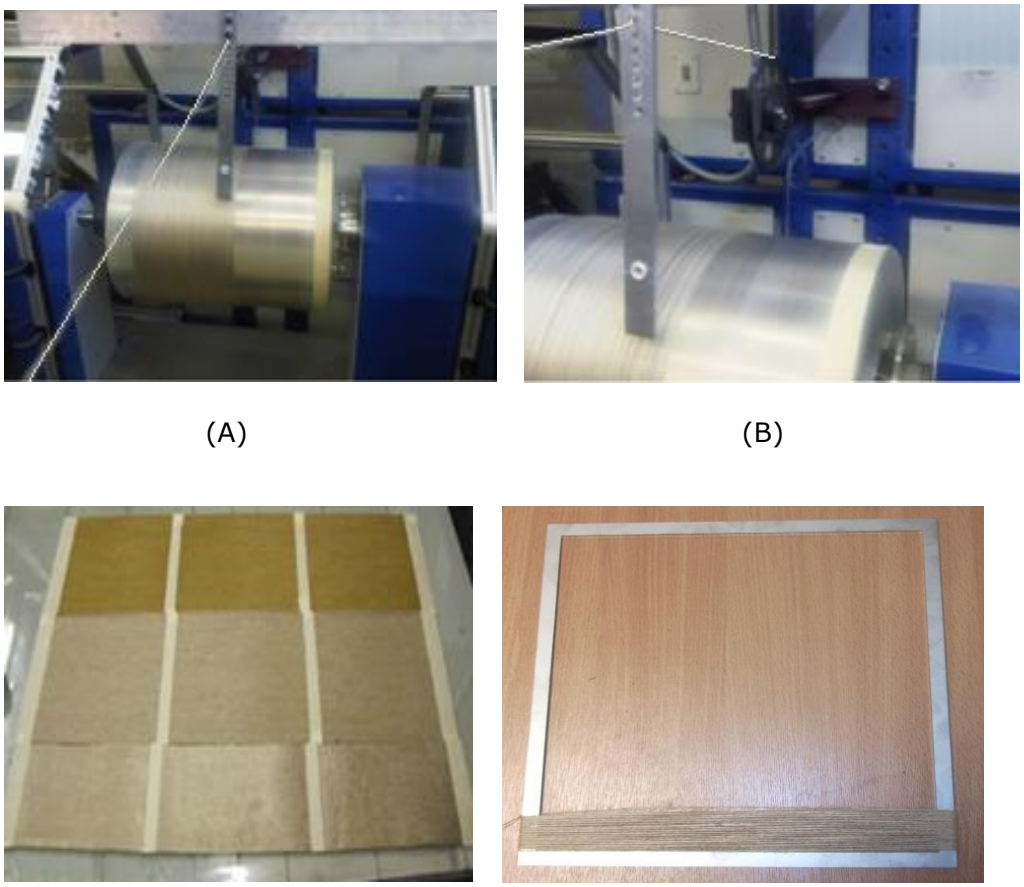
(A) (B)

Fig. 3.2 Image of hairy yarn (A) and image of hairless yarn (B) (scale bar 1 mm)

Both hairy and hairless yarns are wound on a motor operated drum separately to produce UD yarn mat. The drum speed is set at 25 revolutions per minute and the drum diameter is 32 cm. A fixed position yarn guide is used to facilitate winding (see Fig. 3.3 (A & B)) and the winding drum gives traverse motion. The drum rotation and traversing speed are adjusted to wind yarns on the drum close together avoiding any gap between yarn coils as well as any overlapping. Care is taken to avoid slackness or stretching of yarns during winding.

Tap water is used to make 0.6% (by weight) HEC solution. A homogenous solution is produced with the help of a mechanical stirrer. Stirring is performed for 10 minutes at 200 revolutions per minute. This solution is applied by hand to the yarns wrapped on the drum using a brush. Yarns were dried in an oven at 105°C [261] on the drum for one hour. After drying, yarns are allowed to cool and are removed from the drum surface by cutting. The sheets are flattened by adding some weight. These yarns are stored at ambient condition for at least 48 hours before using for composites manufacturing. Application of the binding agent is undertaken to produce the UD mat (see Fig.3.3 (C)). The area density of the UD mat is

400-500 gm/m^2. The binding agent take-up is measured as 1.5-1.8% (by weight) of the yarn.



(A) (B)

(C) (D)

Fig. 3.3 Winding jute yarn on drum (A), closer look (B), HEC sized yarn sheet after taking off from cylinder (C), yarn wrapped on square frame (D).

HEC forms a film around the yarn (discussed in Section 3.3.3) that lays the hairs on yarn resulting comparatively smooth yarn surface (Fig. 3.4 (A) and (B)). Hence the influence of hairiness on the mechanical properties of composites might not be prominent for the composite plaques manufactured with HEC sized hairy yarn and composite plaques are manufactured with unsized hairy and hairless yarn.



(A) (B)

Fig. 3.4 Unsized yarn (A) and HEC sized yarn (B)

UD mat of unsized yarns is produced by wrapping yarn on a square shape steel frame (see Fig. 3.3 (D)). The dimensions of the frame are 260x260x1 mm. The width of the arms of the frame is 10 mm and yarns are wrapped on it closely together. Care was taken to avoid under tension to minimise slackness and over tension to avoid stretching of yarns. This frame including the yarn wound on it was infused to manufacture composites and the frame was removed from the composites after curing by cutting the edges of the composite plaque.

3.3.3 Binding mechanism between HEC and fibre

HEC was manufactured by reacting the alkali cellulose and ethylene oxide and the reaction is shown in equation 3.1 [262], where 'R' represents the cellulose skeleton.

$$R(ONa)_n + CH_2CH_2\text{(O)} \longrightarrow R(OCH_2CH_2OH)_3 \qquad (3.1)$$

Hydroxyethyl cellulose

The idealised structure of HEC is presented in Fig. 3.5. The surface reactive hydroxyl (-OH) groups of HEC create hydrogen bonds with cellulose chains [263] and it can be used as a binding agent to form UD mat from yarn.

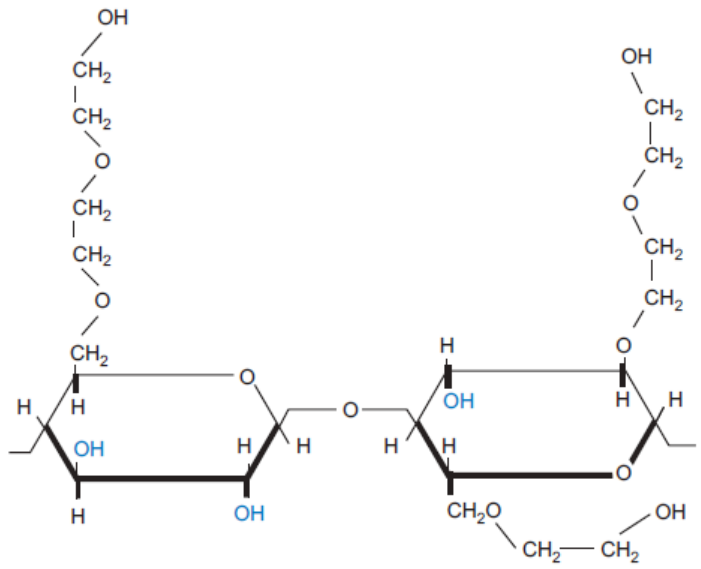


Fig. 3.5 Chemical structure of hydroxyethyl cellulose [264]

The energy introduced during stirring enables water molecules to enter into the compact stratified layers as shown in Fig.3.6. When water molecules begin to enter into the granules, they swell and the viscosity of the solution increases [15]. The granules continuously swell and gradually absorb most of the water provided. Stirring helps to achieve a gel type HEC solution and it becomes ready for application. Sizing yarns with this HEC gel forms a coating around the yarns and the schematic diagram of the film forming technique is presented in Fig.3.7. When this film becomes dry, a strong interlocked chain structure is produced among the HEC chains with the cellulose chains of the yarns due to hydrogen bonding, and the layer of yarns becomes a UD sheet transferrable to a suitable device for infusion.

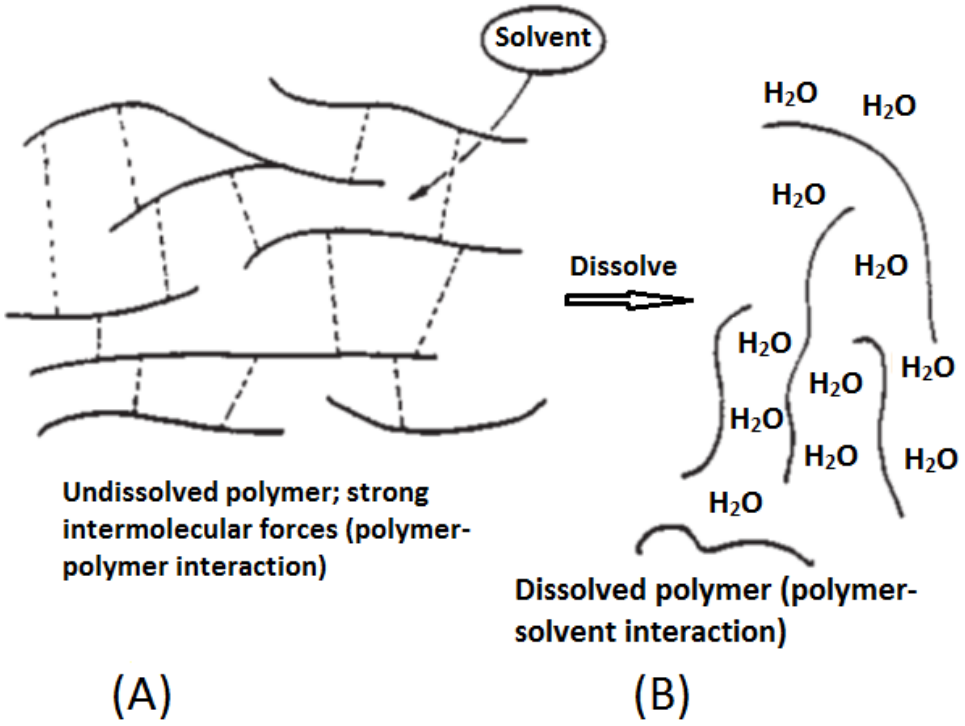


Fig. 3.6 Breaking polymer-polymer interactions (A) and replacing polymer-solvent interactions (B) [15].

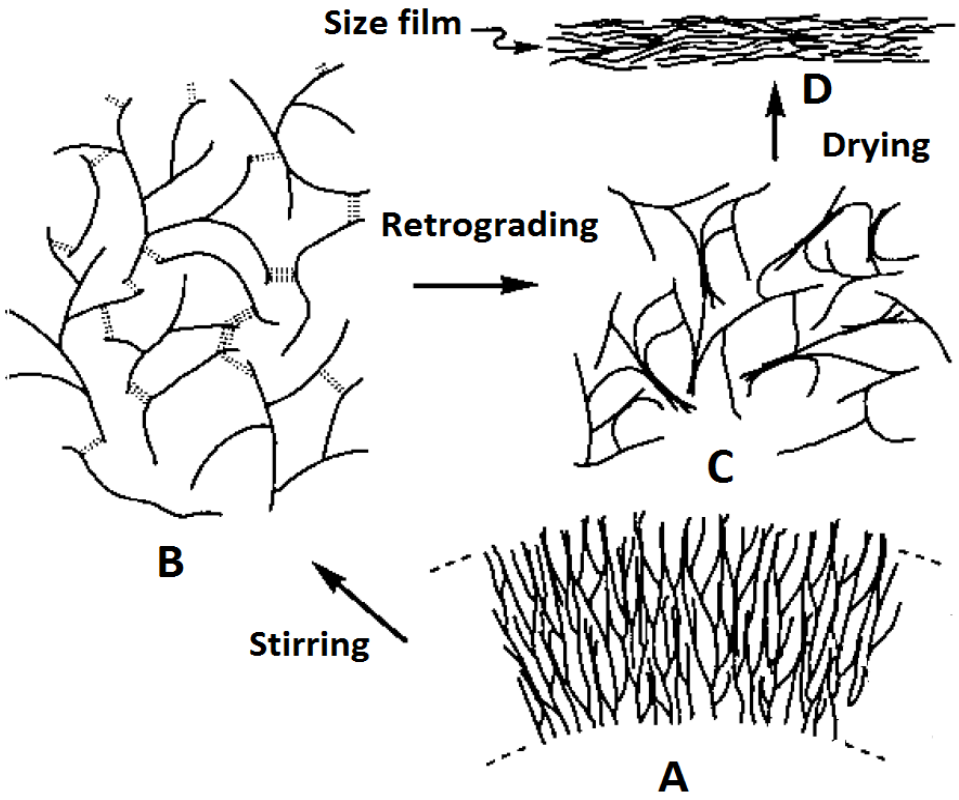


Fig. 3.7 Film forming technology of size solution [15].

3.3.4 Manufacturing of composites

UD mat of sized yarn is cut according to the inside dimension of a picture frame (250x250 mm). A slightly larger (260x260 mm) picture frame is used to infuse unsized yarn. Yarn weight is noted before infusion to calculate the fibre volume fraction. The technical data of the matrix (unsaturated polyester resin) is presented in Table 3.1. Resin infusion is performed at ambient temperature perpendicular to the yarn axis to

Table: 3.1 Properties of UP resin

Resin	Supplier	Viscosity (mPas)	Gel time at 25°C (mins)	Tensile modulus E_m (GPa)	Tensile strength σ_m (MPa)	Failure strain %
UP	Reichhold Norpol	210	30	3.7	70	3.5

generate uniform resin flow. A schematic diagram of the infusion mould is shown in Fig. 3.8. Before infusion, mould release is applied (4-5 times) to the mould and the picture frame (Fig. 3.9) and allowed to dry.

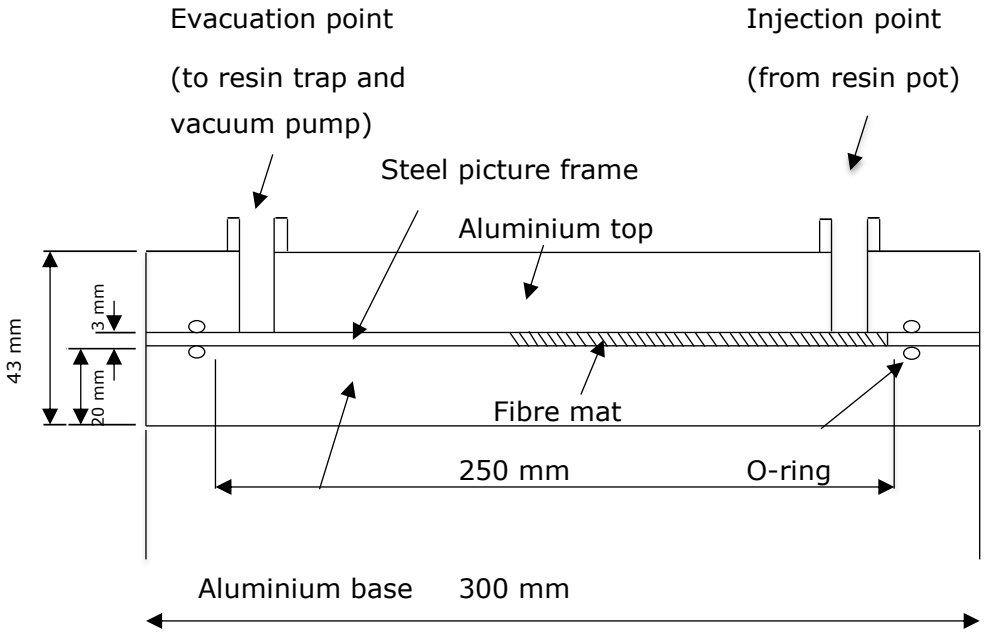


Fig. 3.8 Schematic diagram of infusion mould

After infusion, the tool is kept closed overnight for solidification (Fig. 3.10) of resin to form composites. The composite plaques are then post-cured for 6 hours at 60°C inside an oven, placed between two pieces of glass fibre composites with some weight added to avoid warping.

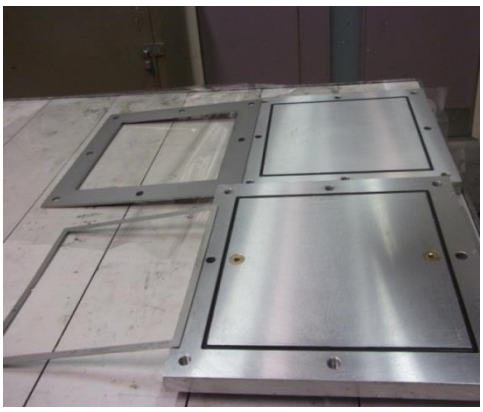


Fig. 3.9 Infusion mould



Fig. 3.10 Infusion set up

3.3.5 Preparation of composite specimens for microscopy analysis

Composite specimens are cut toward the cross-section of the fibres having around 15 mm width of the samples using a high speed circular diamond cutter keeping extraction fan on. Three samples are taken from each plaque for casting into polyester resin in a pot. The specimens are fastened temporarily on the bottom of the casting pot using u-pol easy one adhesive mixed with u-pol hardener. 2% butanox catalyst and 0.8% accelerator are mixed with resin using a mechanical agitator. After curing overnight, the cast specimens are removed from the pot and cut to a height of around 14 mm which allowed the specimens to be focused properly under the microscope. The surfaces of composites in contact with the bottom of the pot are polished using a Struers® Dap-7 machine equipped with a mechanical sample holder (Struers® Pedemin-S). The specimens are polished using water proof grit papers in several steps with gradual use of finer grit paper of 240 (58.5μ), 600 (25.8μ), 800 (21.8μ), 1200 (15.3μ),

2500 (8.4μ) and 4000 (5μ) grade. Polishing is carried out with each grit paper for 3-5 minutes [242, 265, 266]. The grinding action is carried out at 125 rpm. 2.5% alumina (1μ particle size) in water is used as final polishing material for 5-10 minutes spraying the solution onto the specimen at one minute intervals. At the end, the polished specimens are washed and preserved carefully in a sample box.

Preparation of specimens for SEM micrographs includes mounting them on a 12 mm support stub and coating. Mounting is performed using double sided adhesive conductive carbon discs. The composite specimens cast in resin are wrapped using aluminium foil and then aluminium coating is performed using an Edwards Coating System E306A.

3.3.6 Preparation of composites for tensile testing

The composite plaques are cut using a high speed circular diamond cutter using a suction tube to avoid excessive dust flying in the cutting room. The dimension of the specimen is 250x15 mm for the longitudinal direction and 250x25 mm for the transverse direction. Cut edges of the composite strips are polished using sand paper and measurement of width and thickness is performed at three points (both ends excluding gripping length by jaws of the tensile testing machine and the middle) to find the mean value to determine the tensile strength and tensile modulus. PLA film is affixed at both sides and both ends of the strips similar to the gripping length of the jaws applying araldite and dried overnight to avoid breakage near the gripping line during tensile testing.

3.4 Testing

3.4.1 Single yarn tensile test

Unsized hairy and hairless yarns were tested for tensile properties. The test was performed with reference to BS ISO 3341:2000 using a Hounsfield Series-S machine (Fig. 3.11) with a cross head speed of 1 mm min^{-1}, gauge length 250 mm and a 500N load cell (Software-QMAT). Double sided adhesive tape was wrapped around the circular jaw and yarn was wrapped on this for gripping. The distance between centres of the jaws was considered as the gauge length. 10 tests were performed for each type of yarn to find the mean value of tensile properties. The highest value of gradient of force-displacement curve of yarn was calculated every 0.5 mm extension [180] to find the tensile modulus. Mean diameter of yarns was determined from ten readings of images at or above 1 m length intervals taken by a Zeiss optical microscope. Yarn cross-section was enumerated considering a circular cross-section of yarn and packing

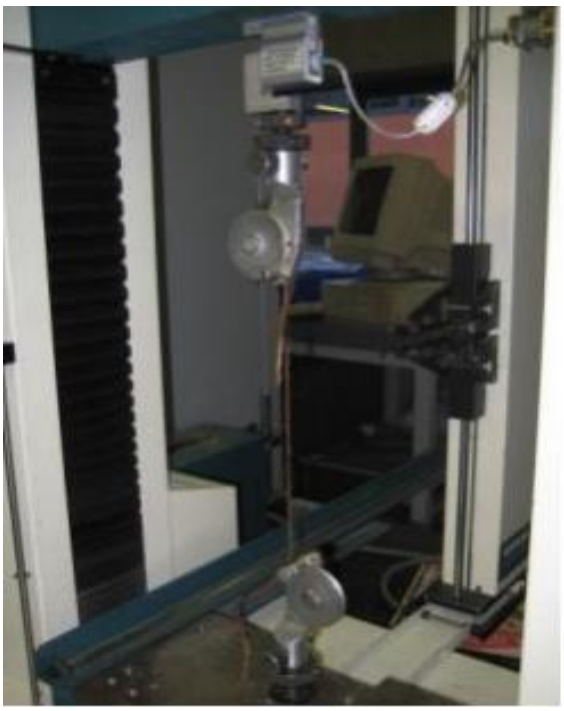


Fig. 3.11 Single yarn tensile test.

fraction of yarn (the ratio between apparent cross-section and actual cross-section of yarn) is considered to be 0.6 (was determined by Shah et al. for the same yarn [180]). The failure load of the yarn obtained from the tensile test was converted to tenacity (cN/Tex) which is a unit to explain the strength of yarn [267]. Failure load of yarn was obtained in Newtons (N) and transformed to cN/Tex according to the equation 3.2

$$cN/Tex = \frac{Nx100}{\text{Yarn count in Tex}} \qquad (3.2)$$

The supplier provided the yarn count in lbs/spyndle and is converted to Tex following the relation mentioned in Section 3.3.1.

3.4.2 X-ray photoelectron spectroscopy (XPS)

XPS analysis of hairy and hairless yarns was performed to observe any surface chemical changes and to examine the effects of burning the fibres. A VG Scientific ESCALab Mark II x-ray photoelectron spectrometer was used in order to characterise the fibre surfaces, utilising non-monochromatic Al Kα X-rays at an anode potential of 15kV and filament emission current of 20mA. Survey (wide scan) spectra were collected at an electron pass energy of 50 eV within the 0-1200 binding energy range in order to ensure all peaks in the samples are present in the spectra. Subsequent data processing and peak labelling of spectra were performed using CasaXPS software.

3.4.3 Differential scanning calorimetry (DSC) analysis

DSC thermograms were taken using TA instruments, USA, model DSC Q10 in a nitrogen atmosphere (gas flow rate 50ml min^{-1}) for hairy and hairless yarns to understand any thermal degradation of fibres due to burning hairs. A constant heating rate of $10^{O}C\ min^{-1}$ was used from $0^{O}C$ to $400^{O}C$ for

fibres and post-cured resin and $0^{O}C$ to $350^{O}C$ for liquid resin and composites. A sample size of approximately 10 mg was used in a Tzero aluminium pan crimped with a pinhole. Data acquisition and processing was performed using TA Universal Analysis 2000 software. A blank pan measurement was conducted for background study. Liquid resin, post cured composites made with unsized hairy and hairless yarns were also analysed to obtain DSC thermograms to understand their curing behaviour.

3.4.4 Fourier transform infrared (FTIR) spectroscopy analysis

Identification of functional groups of the hairy and hairless yarns was performed using FTIR (Tensor-27, Bruker). All spectra were analysed with Opus^{TM} software version 5.5. Both yarn types are scanned in transmittance mode in the region of 4000 cm^{-1} and 550 cm^{-1} (wave numbers) using a standard pike attenuated total reflectance (ATR) cell (Pike Technology, UK). After every test the spectrometer is cleaned using acetone.

3.4.5 Microscopy

A Philips XL-30, FEI scanning electron microscope (SEM) was used to analyse the lumen and cell wall of the jute fibre as well as the matrix (UP resin). This experiment was performed to investigate the character of the lumen of fibres fabricated in composites to find the contribution of lumen to the void content. 12 kV voltage beam with a 10 mm working distance was used as a working environment. The diameter of a single jute yarn was measured using a Zeiss optical microscope. The resolution was 2150 pixel/min and the mean value was calculated from ten readings using image processing software (Image Pro 6.2). The diameter of the yarn was measured at least once per meter interval of length.

3.4.6 Composite tensile test

Composite plaques were stored at ambient conditions for at least 48 hours after post-curing prior to conducting any test. Before performing tensile tests, the fibre volume fraction of the composites was measured. As mentioned in Section 3.3.4 the weight of the fibres was measured before infusion and the volume of the fibres is calculated considering the density of jute fibre (1.45 g/cc [201, 268]). The volume of the composite plaque was determined by measuring the length, width and thickness. At least three readings were taken for each dimension and the mean values were considered for calculating the volume of the composite plaque. Fibre volume fraction of composites was obtained by dividing the volume of fibres by the volume of the composite plaque. As the fibre volume fraction of composites was measured for the whole plaque thus standard deviation is not available.

The tensile tests of composites were conducted with reference to BS EN ISO 527-4:1997 using an Instron 5969 fitted with a 50 kN load cell (software Bluehill®) and a mechanical extensometer. The cross-head speed used is 1mm min^{-1}. Plaques were tested along the longitudinal and transverse direction of yarns. Five tests were performed for each plaque to find the mean values of tensile properties. Tests were performed under ambient conditions (15-25°C and 60-90% RH).

3.4.7 Statistical hypothesis test

A statistical interference test was performed to draw the conclusion of random variation between two sets of data and the result is known as statistical significance. Each test of significance starts with a null hypothesis H_O which describes that there is no variation between data sets. The alternative hypothesis, H_1 is configured to establish the null hypothesis.

The result of the test is always concluded in terms of the null hypothesis and denoted by 'P', either H_O is rejected in favour of H_1 or it is accepted. Rejecting the null hypothesis means that variation exists between data sets.

For a given hypothesis test the significance level α is a value considered for which a P-value is less than or equal to α that is to be as statistically significant. Typically the values of α are 0.1, 0.05 and 0.01, corresponding to the probability of observation for an extreme value by chance [269]. For example if a P-value of the test is 0.042, the probability of finding this value by chance is less than 0.05 and the result is significant at the level of 0.05 [270] and denoted by $P<0.05$. For accepting or rejecting the null hypothesis two different types of test can be performed, a one-tailed test (i.e. one direction of interest) and a two-tailed test (i.e. the level of significance is divided into two halves). In this work a two tailed test was performed i.e. testing the possibility of a relation in both directions and the data were considered as two-samples of unequal variance.

3.4.8 Fibre and matrix volume fraction in composite

Mechanical properties of composites depend on the relative volume of reinforced fibres and matrix and the fibre volume fraction (V_f) in composites is defined according to the equation 3.3.

$$V_f = \frac{\text{Volume of fibre}}{\text{Volume of composite}} \qquad (3.3)$$

Volume fraction of matrix (V_m) is defined according to the equation 3.4.

$$V_m = \frac{\text{Volume of matrix}}{\text{Volume of composite}} \qquad (3.4)$$

As volume of composite is the sum of the fibre volume and the matrix volume, thus

$$V_f + V_m = 1 \qquad (3.5)$$

3.4.9 Longitudinal modulus

Longitudinal modulus of composites in fibre direction is calculated by the formula called the rule of mixtures (ROM). Important assumption to calculate modulus using ROM is that the strain in the matrix and the fibre is same i.e. bond between fibre and matrix is perfect [271]. It means during stretching of composite in the fibre direction equal elongation would occur for fibre and matrix.

According to definition strain,

$$\varepsilon_1 = \frac{\Delta L}{L} \qquad (3.6)$$

where L = original length and ΔL = elongation

As both fibre and matrix are elastic thus stress on fibre (σ_f) and stress on matrix (σ_m) can be defines according to the equation 3.8 and equation 3.9

$$\sigma_1 = E_1\varepsilon_1 \qquad (3.7)$$

$$\sigma_f = E_f\varepsilon_1 \qquad (3.8)$$

$$\sigma_f = E_f\varepsilon_1 \qquad (3.9)$$

Load carried by lamina (P), fibre (P_f) and matrix (P_m) can be defined as-

$$P = \sigma_1 A \qquad (3.10)$$

$$P_f = \sigma_f A_f \qquad (3.11)$$

$$P_m = \sigma_m A_m \qquad (3.12)$$

Where A, A_f and A_m are the area of cross section of composite, fibre and matrix respectively.

The total load carried by the composite is the sum of load carried by the fibre and the matrix.

$$P = P_f + P_m = \sigma_f A_f + \sigma_m A_m = \sigma_1 A \qquad (3.13)$$

Then

$$E_1\varepsilon_1 A = E_f\varepsilon_1 A_f + E_m\varepsilon_1 A_m \quad (3.14)$$

Divide through by $\varepsilon_1 A$

$$E_1 = E_f(A_f/A) + E_m(A_m/A) \quad (3.15).$$

Thus

$$E_1 = E_f V_f + E_m V_m \quad (3.16)$$

Where V_f (fibre volume fraction) = A_f/A and V_m (composite volume fraction) = A_m/A

And the rule of mixtures formula comes to

$$E_1 = E_f V_f + E_m(1-V_f) \quad (3.17)$$

Equation 3.17 is used to determine the back calculated tensile modulus of fibre.

3.4.10 Transverse modulus

To determine the modulus in the transverse direction of fibres the main assumption is similar to longitudinal direction i.e. stress on fibre and stress on matrix is same. This assumption implies perfect bonding between fibre and matrix.

Since both fibre and matrix are considered as linear elastic materials thus the strains in the fibre and matrix are-

$$\varepsilon_f = \sigma_2/ E_m \quad (3.18)$$

$$\varepsilon_m = \sigma_2/E_m \quad (3.19)$$

However $\varepsilon_2 \neq \varepsilon_f \neq \varepsilon_m \quad (3.20)$

The net strain across the lamina is the weighted average of the fibre and matrix strains-

$$\varepsilon_2 = V_f\varepsilon_f + (1-V_f)\varepsilon_m \quad (3.21)$$

substitute for the fibre and matrix strains

$$\varepsilon_2 = V_f\sigma_2/E_f + (1-V_f)\sigma_2/E_m \quad (3.22)$$

$$E_2 = \frac{E_f E_m}{V_f E_m + (1-V_f)\, E_f} \quad (3.23)$$

This equation can be written as-

$$\frac{1}{E_2} = \frac{V_m}{E_m} + \frac{V_f}{E_f} \qquad (3.24)$$

Equation 3.24 Is known as the inverse rule of mixtures.

3.4.11 Longitudinal tensile strength

To develop the model for longitudinal tensile strength for continuous fibre (σ_f), it is assumed that all fibres possess same fibre strength. The second assumption is that both reinforced fibre and matrix show linear behaviour up to failure. The third assumption is that the fibres are brittle than matrix and the last assumption is the fibres are stiffer than the matrix [].

According to the assumptions mentioned above the composite is supposed to break when the stress in the fibres is equal to strength (σ_f). When fibres break, matrix also fails to carry load. Thus elongation at failure of composites (ε_c) and fibre (ε_f) is same. Under these circumstances it is assumed that the longitudinal strength of composite is powered by fibre strength (σ_f) and can be written as –

$$F_1 = \sigma_f V_f + \sigma^*_m(1-V_f) \qquad (3.25)$$

and is used to determine the back calculated tensile strength of fibre.

Where the failure stress on matrix is

$$\sigma^*_m = \sigma_f(E_m/E_f) \qquad (3.26)$$

Hence the tensile strength is

$$F_1 = \sigma_f \left[V_f + (E_m/E_f)(1-V_f)\right] \qquad (3.27)$$

3.4.12 Transverse tensile strength

Transverse tensile strength of fibres (F_2) depends on the strength of matrix, fibre-matrix interface and defects in the matrix. Models developed up to

date cannot predict this property accurately. Thus the empirical formulae [] are presented here due to the lack of more accurate formulae.

$$F_2 = \sigma_m C_v (1-V_f^{1/3}) E_2/E_m \qquad (3.28)$$

Where σ_m is the tensile strength of matrix and C_v is reduction co-efficient to account for voids.

$$C_v = 1- \sqrt{\frac{4V_v}{\Pi(1-V_f)}} \qquad (3.29)$$

where V_v is the void volume fraction. Another empirical formula [272] is

$$F_2 = \sigma_m C_v \left[1 + (V_f - \sqrt{V_f})(1 - E_m/E_f) \right] \qquad (3.30)$$

3.5. Results and Discussion

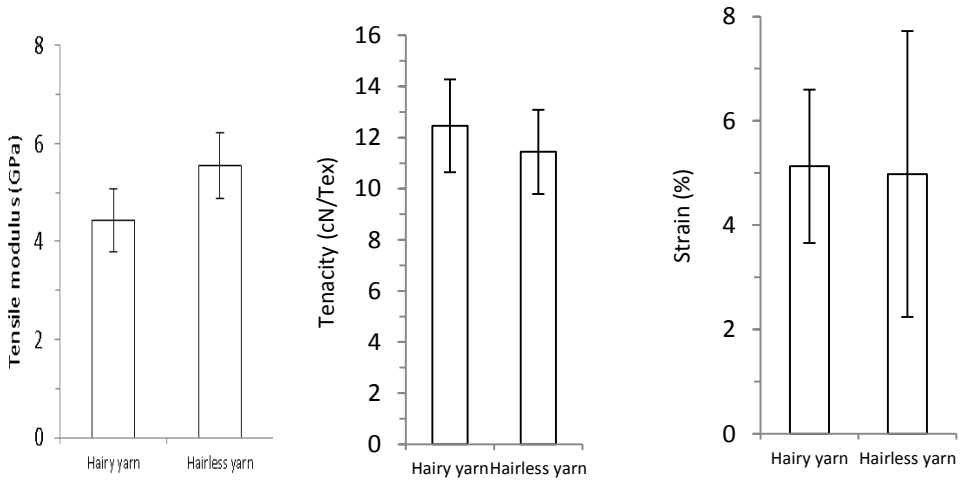
3.5.1 Single yarn test results

The removal of yarn hairs by burning brings the possibility of deteriorating the tensile properties of hairless yarn due to possible damage of thermally unstable constituents of jute fibre at higher temperatures [273-275]. Thus studying tensile properties of hairy and hairless yarns is of interest, since any damage of yarn would affect the mechanical properties of composites. However tensile modulus of hairless yarn is found to be 24% higher than that of a hairy yarn (Table 3.2 and Fig. 3.12 (A)) and this difference is

Table:3.2 Tensile properties of yarn

	Tensile modulus (GPa)	Standard deviation	Tenacity (cN/Tex)	Standard deviation	Strain at failure (%)	Standard deviation
Hairy yarn	4.4	0.7	12.5	1.8	5.1	1.5
Hairless yarn	5.6	0.7	11.4	1.7	5.0	2.7

statistically significant ($P<0.05$). No specific cause is identified that could increase the tensile modulus of a hairless yarn. Very similar standard deviations are obtained for tensile moduli of yarns. A minor difference is found between tenacity of hairy and hairless yarn (Table 3.2 and Fig. 3.12 (B)) revealing that burning of hairs does not deteriorate the tensile strength. Standard deviations of tenacity of both types of yarn are also similar. Strain at failure of hairy yarn and hairless yarn is found to be very similar, however the standard deviation of strain at failure (Table 3.2 and Fig. 3.12 (C)) of hairless yarn is higher than that of hairy yarn disclosing that strain at failure of hairless yarn is more variable than strain at failure of hairy yarn and the causes of this characteristic are unknown.



(A) (B) (C)

Fig.3.12 Tensile modulus (A), tenacity (B) and strain at failure of yarn (C) (error bars indicate standard deviation).

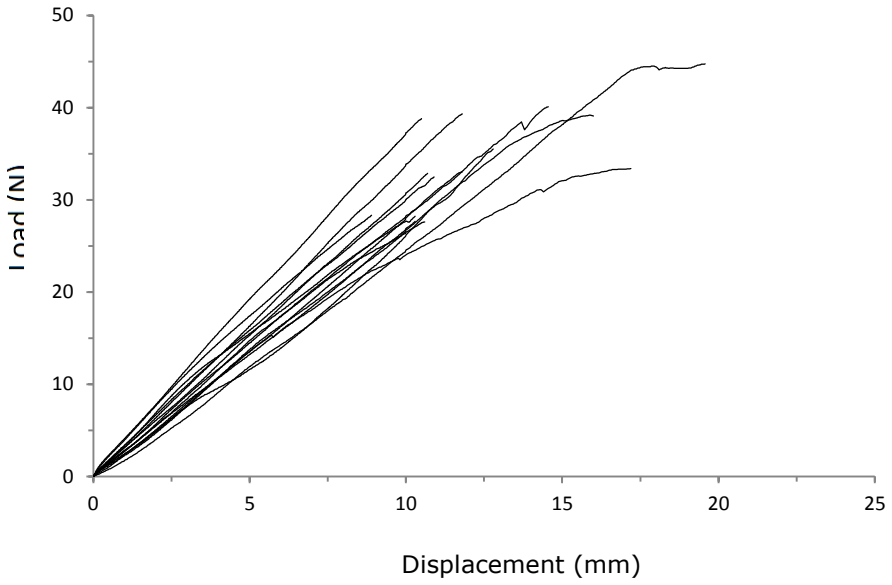


Fig.3.13 Force displacement curves of unsized hairy yarn

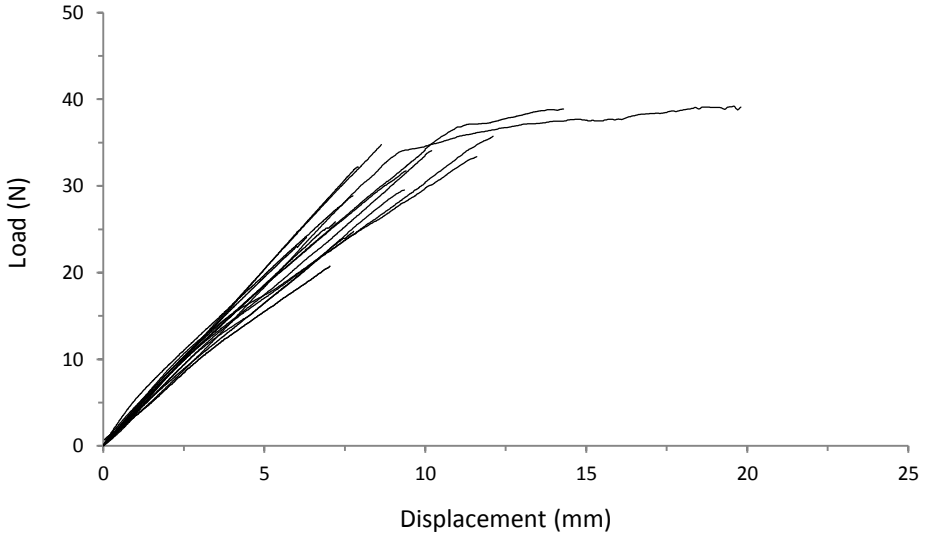


Fig.3.14 Force displacement curves of unsized hairless yarn

Very similar force-displacement curves of both hairy and hairless yarns are observed (Fig. 3.13 and Fig. 3.14) revealing that removing hairs does not influence the characteristics of force displacement curves of yarns.

3.5.2 XPS analysis for surface chemical of hairy and hairless yarns

Table 3.3 Surface chemicals of hairy and hairless yarns.

Type of yarn	Carbon%	Oxygen%	Nitrogen%	Silicon%
Hairy yarn	68.2	27	2.5	2.3
Hairless yarn	73.6	22.6	1.6	2.2

A study of x-ray photoelectron spectroscopy presents some variations of surface composition of hairy and hairless yarns. 5.4% higher carbon (C) content and 4.4% lower oxygen (O) content (Table 3.3) are observed on hairless yarn surface with respect to those of hairy yarn surface. Exposing yarn under open flame increases unburnt carbon deposition on the surface. Most likely the amount of other elements are the same before and after burning. However deposition of unburnt carbon on the yarn surface increases the carbon content hence reduces the proportions of other elements.

3.5.3 Thermal analysis

Thermal analysis helps to understand the moisture content and volatile components that affect the properties of composites [276]. DSC analysis is performed on hairy yarn, hairless yarn, composites made with hairy yarns, composites made with hairless yarns, post-cured and liquid polyester resin to understand their thermal behaviour. DSC thermograms of hairy and hairless yarns are found to be very similar (Fig. 3.15). An extended endothermic peak is observed in the temperature range 75-160°C for both the types of yarn due to vaporisation of moisture absorbed by fibres [277]. Between 160-200°C a small exothermic peak is observed and is due to decomposition of lignin [277, 278]. No peak is found between 200-260°C

reflecting the thermal stability of fibre in this region. A small exothermic peak around 260-280°C indicates the decomposition of hemicellulose [279]. Major changes start around 300°C as cellulose decomposes at this temperature [280].

The DSC thermogram of post-cured resin does not show any major change up to 325°C and around this temperature it starts to decompose and around 400°C it decomposes fully. A quasi-linear relationship up to the starting point of decomposition suggests the resin is fully cured and it can be exposed safely up to 325°C. Characteristics of DSC thermograms of composites made with hairy and hairless yarns are closer to that of jute fibres than that of post-cured resin. Endothermic peaks due to evaporation of moisture of jute fibre in DSC thermograms of composites are obvious

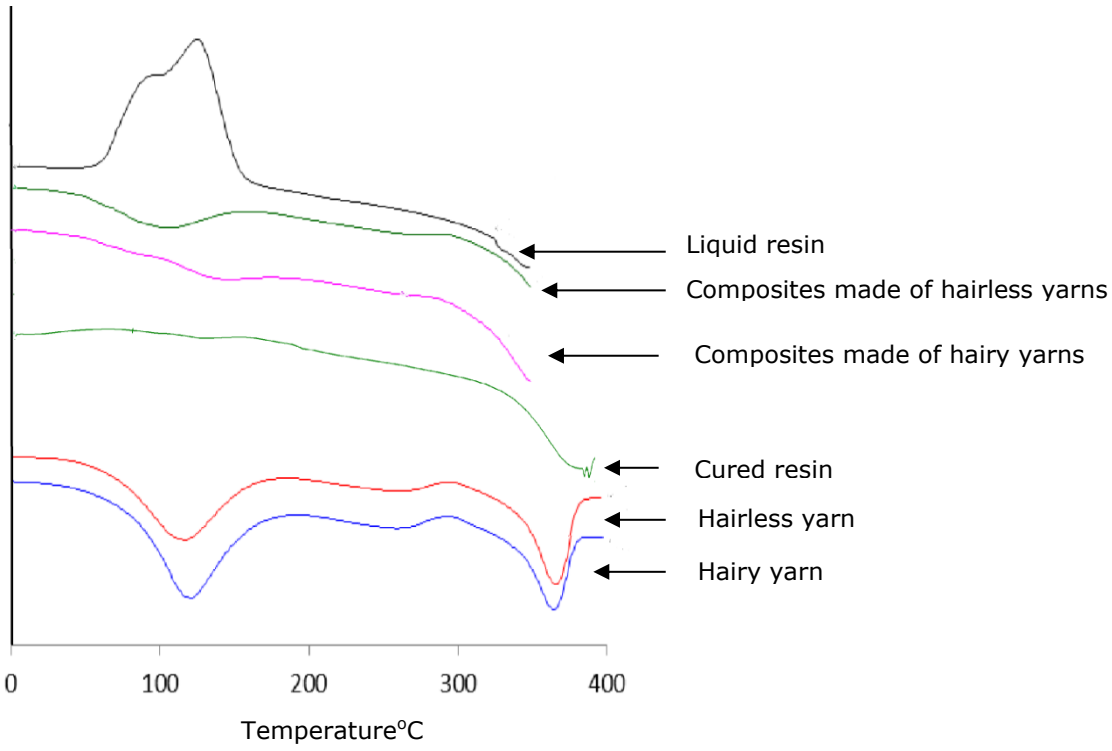


Fig. 3.15 DSC thermograms of yarns, composites and polyester resin

and composites start to decompose at a similar temperature to that for jute fibres which is lower than for post-cured resin (Fig. 3.15). Hence this

reflects that thermal change of composites is influenced mostly by jute. The DSC thermogram of liquid polyester resin shows an extended exothermal peak in the temperature range 50°C to 135°C due to the exothermic reaction of resin to get cross-linked structure.

3.5.4 Fourier transform infrared (FTIR) analysis

FTIR spectra of hairy and hairless yarns are shown in Fig. 3.16 from wave number 4000 cm^{-1} to 500 cm^{-1}. Both spectra are very similar regarding the peak position, revealing that no major change of chemical composition of fibres has occurred due to burning. The absorption band in the region 3346 cm^{-1} represents O-H stretching vibration of cellulose [126, 281]. The peak near 2900 cm^{-1} is due to C-H stretching vibration of methyl and methylene groups presents in cellulose and hemicellulose [282]. A sharp peak at 1740

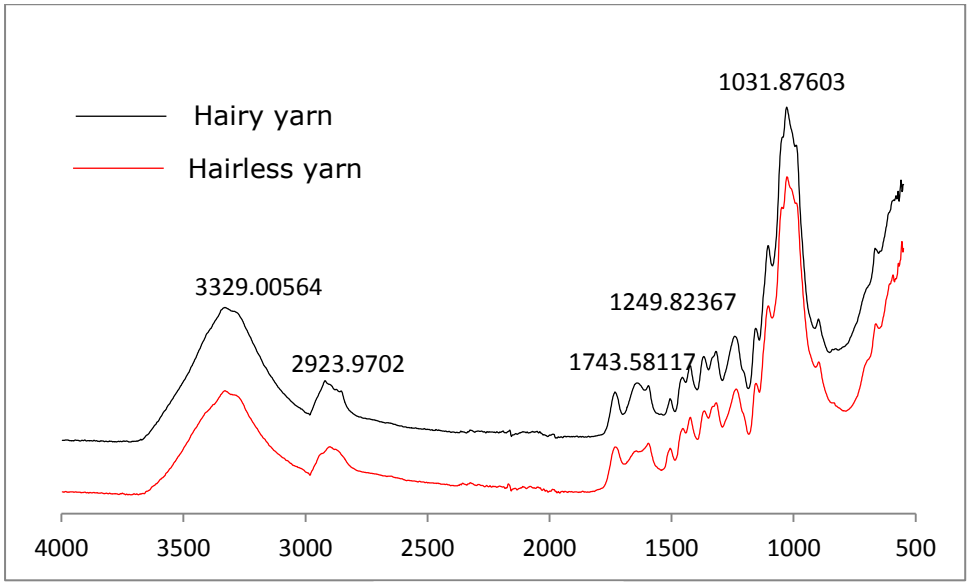


Fig. 3.16 FTIR spectra of hairy and hairless yarn

cm^{-1} is due to the C-O stretching of the carboxyl and acetyl groups in hemicellulose [126]. Vibration at 1647 cm^{-1} is assigned to aromatic rings [283] of lignin [284]. The peaks at 1596 cm^{-1}, 1510 cm^{-1} and 1323 cm^{-1} are attributed to lignin or phenolic compounds [284]. The absorbency band

at 1463 cm^{-1} represents CH_3 deformation (asymmetric) in lignin [126]. The absorbance at 1373 cm^{-1} is a characteristic absorbance associated with C-H deformation (symmetric), which may be due to lignin, cellulose and xylan [126]. The absorbency band at 1247 cm^{-1} is assigned to C-O stretching of acetyl group in xylan and the band at 1031 cm^{-1} is ascribed to aromatic C-H and C-O deformation of primary alcohol in lignin [126].

3.5.5 Contribution of lumen to void content of composites

Cross-sections of jute fibre fabricated in composites and raw fibre (un-fabricated in composites) are examined to analyse the contribution of the lumen to void content of composites. Fig. 3.18 and Fig. 3.19 show the images obtained by SEM of fabricated jute fibres in composites and un-fabricated jute fibres respectively. It is well established that plant fibre composites possess high void content [12, 245] and part of the void content is contributed by the lumen of the fibres as the lumen is not filled by resin [285]. However the lumen of fibres fabricated in composites (Fig. 3.17) and the lumen of un-fabricated fibres (Fig. 3.18) appear different under SEM. SEM EDX analysis is performed for lumen of fibre, cell wall of fibre that is fabricated in composites as well as the matrix used to manufacture composites. Spectra present very similar peaks for lumen (spectrum 1) and matrix (spectrum 3) (Fig. 3.19 and Fig. 3.20) and different types of peaks for the fibre cell wall (spectrum 2). The results reveal that the lumen of the jute fibre becomes filled with resin and does not contribute to the void content of the composites. The peaks identified as aluminium are due to aluminium being used to coat the specimens. Tungsten is identified due to the earlier use of tungsten as a coating material for other samples utilising the same coating machine.

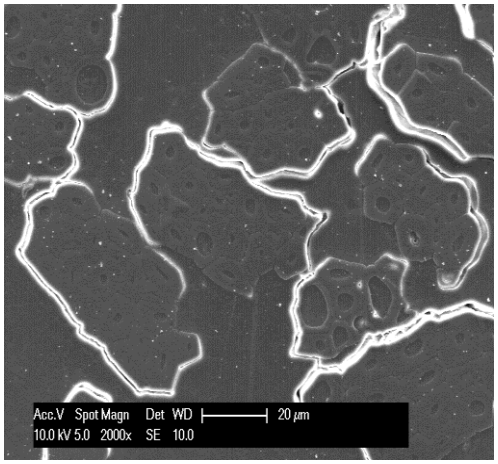


Fig. 3.17 SEM micrograph of jute fibre cross-section (fabricated in composites)

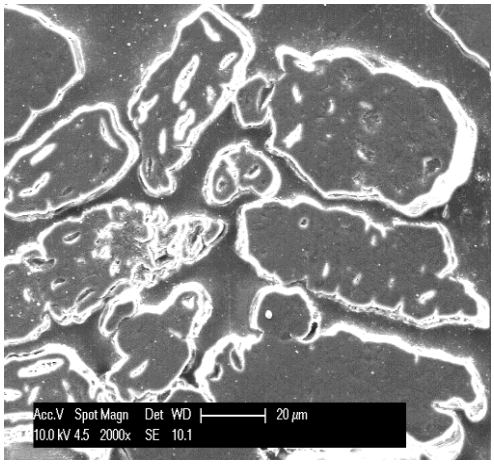


Fig. 3.18 SEM micrograph of jute fibre cross-section (not fabricated in composites)

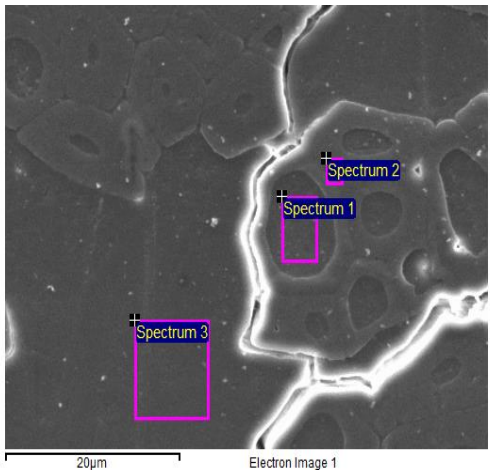


Fig. 3.19 SEM EDX micrograph denoting the position of spectra.

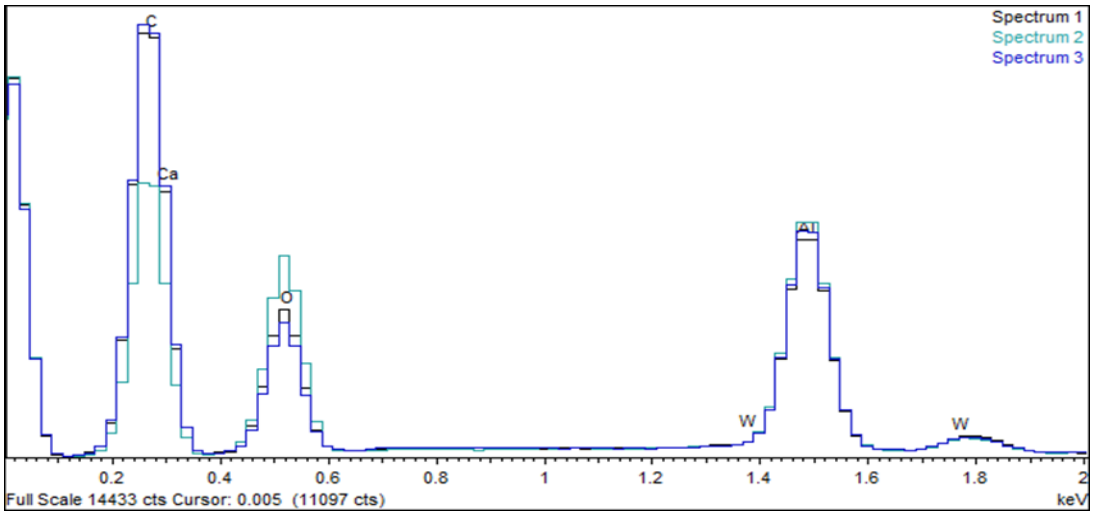


Fig. 3.20 SEM-EDX spectra of jute fibre lumen (spectrum 1), cell wall (spectrum 2) and matrix (spectrum 3)

3.5.6 Composite test results

3.5.6.(I) Longitudinal direction of yarn

Table 3.4 Fibre volume fraction and tensile test results of composites

	Fibre volume fraction (%)	Tensile modulus (GPa)	Standard deviation	Tensile strength (MPa)	Standard deviation	Strain at failure (%)	Standard deviation
Hairy yarn (unsized)	38	17.4	0.34	169	12	1.36	0.06
Hairless yarn (unsized)	38	16.5	0.58	182	8	1.47	0.03
Hairy yarn (sized)	35	18.3	0.57	187	4	1.34	0.07
Hairless yarn (sized)	36	17.7	1.02	177	14	1.39	0.05

Composites made with unsized hairy yarns show 5.5% higher tensile modulus with respect to composites made with unsized hairless yarns (see Table 3.4 and Fig.3.21) and this difference is statistically significant ($P<0.05$). Composites made with unsized hairy yarns show 7.5% lower strain at failure with respect to composites made with unsized hairless yarns (see Table 3.4 and Fig.3.25) and this difference is statistically significant ($P<0.05$). Even taking into consideration the slight different fibre volume fraction the significant difference in property is possibly due to yarn hairiness. The projected hairs from the yarn surface embedded in resin possibly contribute better initial stress transfer during tensile loading that helps to increase the tensile modulus. Strain at failure of composites (Table 3.4 and Fig. 3.23) is much less than that of polyester resin (Table 3.1) and this property of composites in the longitudinal direction of yarn is governed by the strain at failure of fibre. However the cause of increasing strain at failure of composites made with unsized hairless yarn compared to that of composites made with unsized hairy yarn is unknown. A similar

trend is observed for tensile modulus and strain at failure of composites manufactured with HEC sized hairy and HEC sized hairless yarns, however the differences are less pronounced. Hairs laid on the yarn surface due to sizing possibly do not contribute to the tensile properties of composites as much as for composites made with unsized yarn. The tensile strengths of composites manufactured with hairy and hairless yarn (Table 3.4 and Fig.3.22) do not show statistically significant differences for any of the cases (i.e. sized and unsized) studied in this chapter.

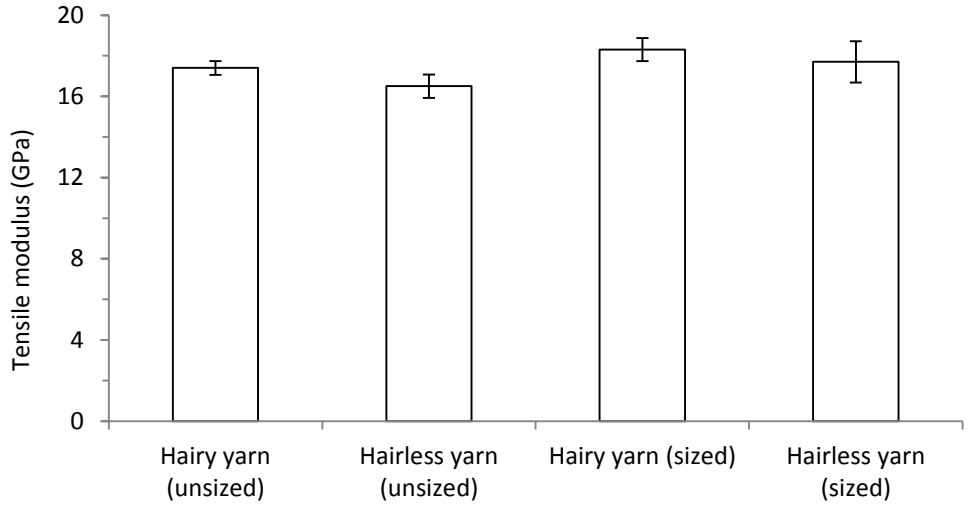


Fig.3.21 Tensile modulus of composites (error bars indicate standard deviation)

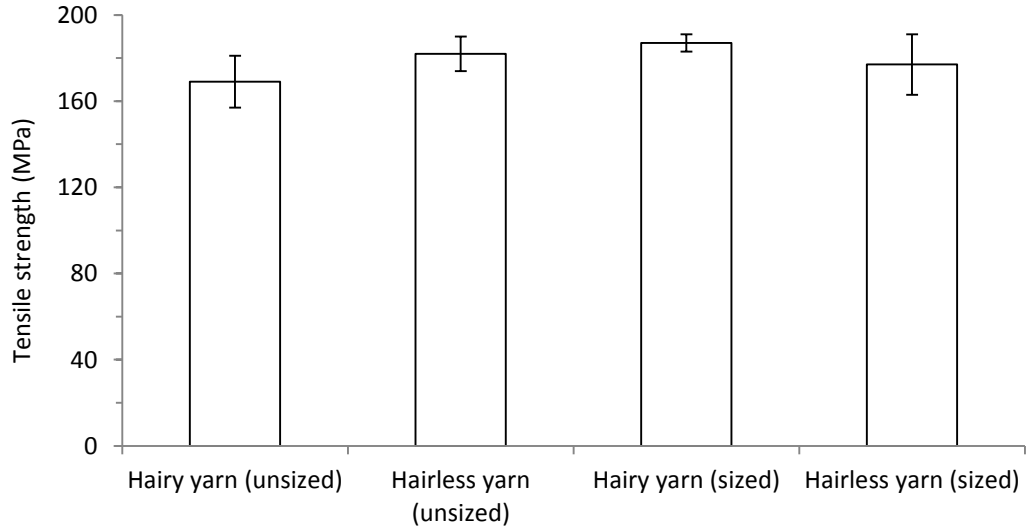


Fig.3.22 Tensile strength of composites (error bars indicate standard deviation)

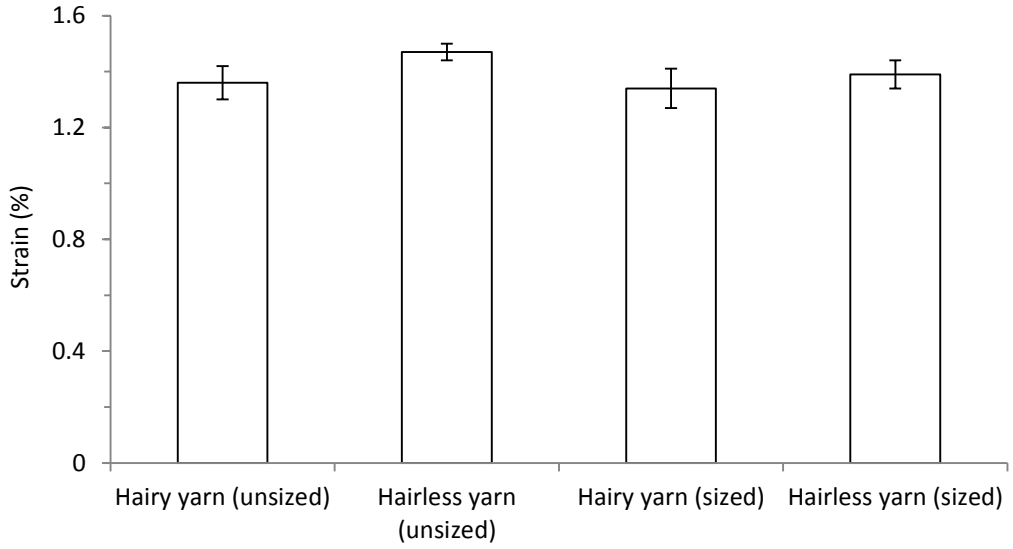


Fig.3.23 Strain at failure of composites (error bars indicate standard deviation)

Table 3.5 Back calculated tensile properties of fibres (longitudinal direction)

	Tensile modulus (GPa)	Standard deviation	Tensile strength (MPa)	Standard deviation
Hairy yarn (unsized)	39.7	0.91	329	29
Hairless yarn (unsized)	37.3	1.51	364	21
Hairy yarn(sized)	44.7	2.09	405	10
Hairless yarn(sized)	42.7	2.82	368	39

Similar to the earlier discussion only the back calculated tensile modulus of fibres from composites manufactured with unsized hairy yarn shows a significantly higher value ($P<0.05$) than that of composites manufactured with unsized hairless yarn. Back calculated tensile modulus and tensile strength of fibre of composites manufactured with HEC sized hairy yarn are similar to those of other researchers [21] however most of the literature cited inferior tensile properties of UD jute polyester composites [177, 197-199] compared to the results obtained in this work. No published work is

found to compare the tensile properties of composites manufactured with hairless jute yarn.

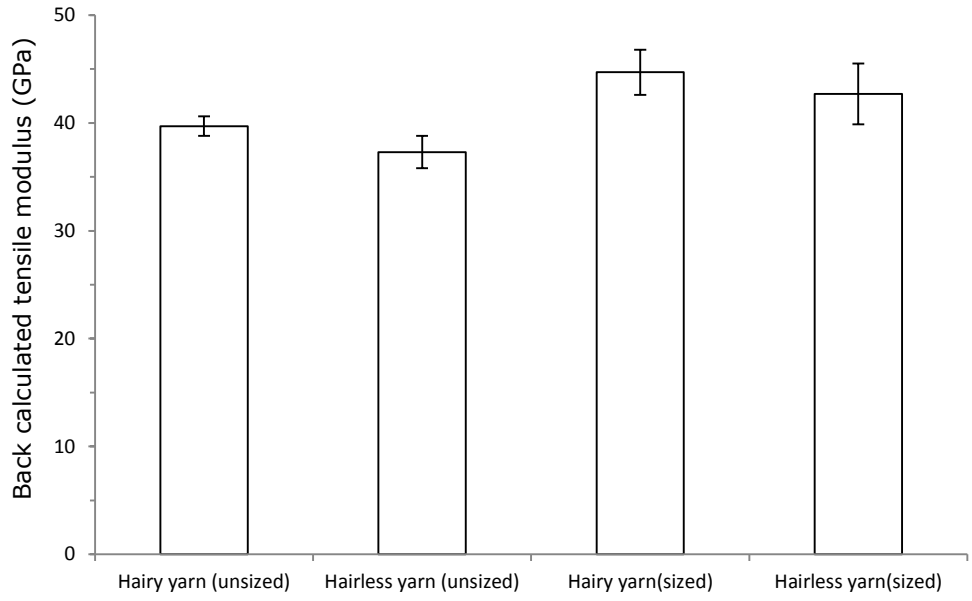


Fig.3.24 Back calculated tensile modulus of fibres (error bars indicate standard deviation)

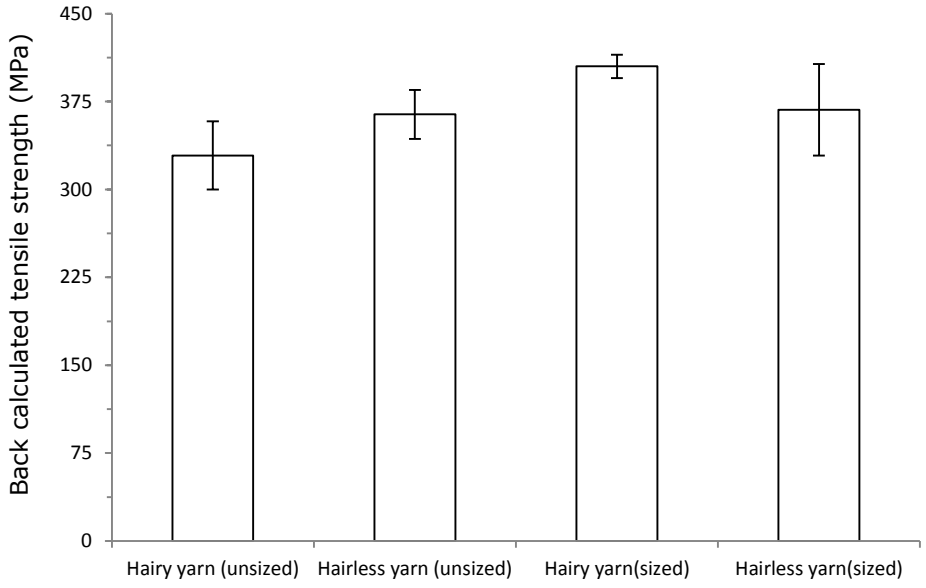


Fig.3.25 Back calculated tensile strength of fibres (error bars indicate standard deviation)

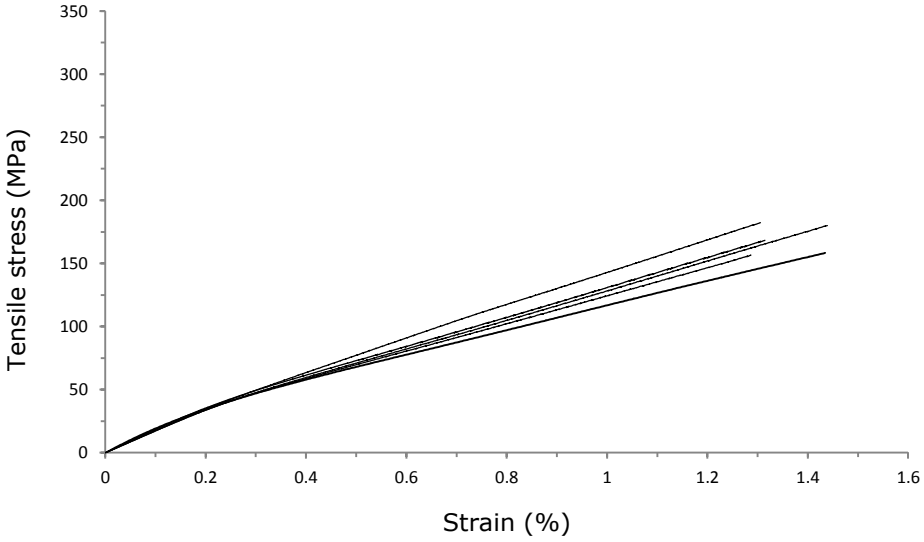


Fig.3.26 Stress-strain curves of composites manufactured with unsized hairy yarn

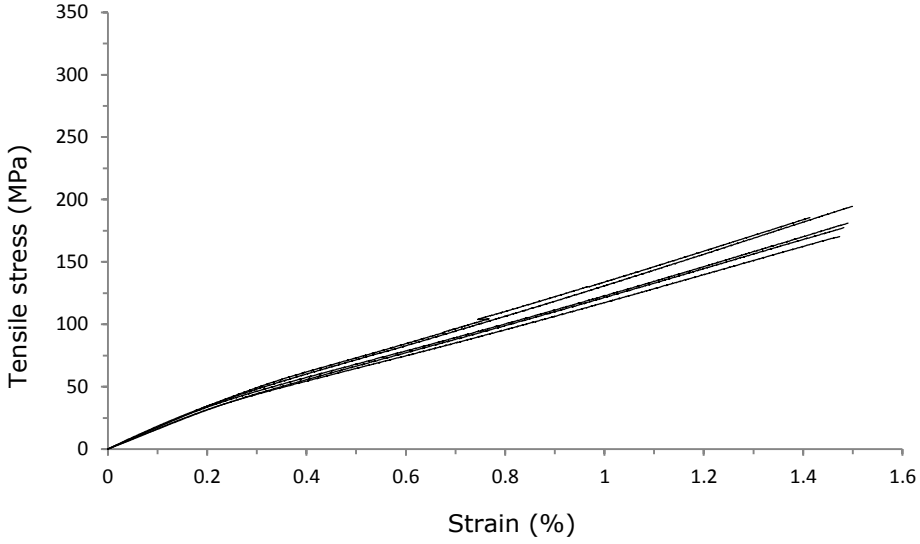


Fig.3.27 Stress-strain curves of composites manufactured with unsized hairless yarn

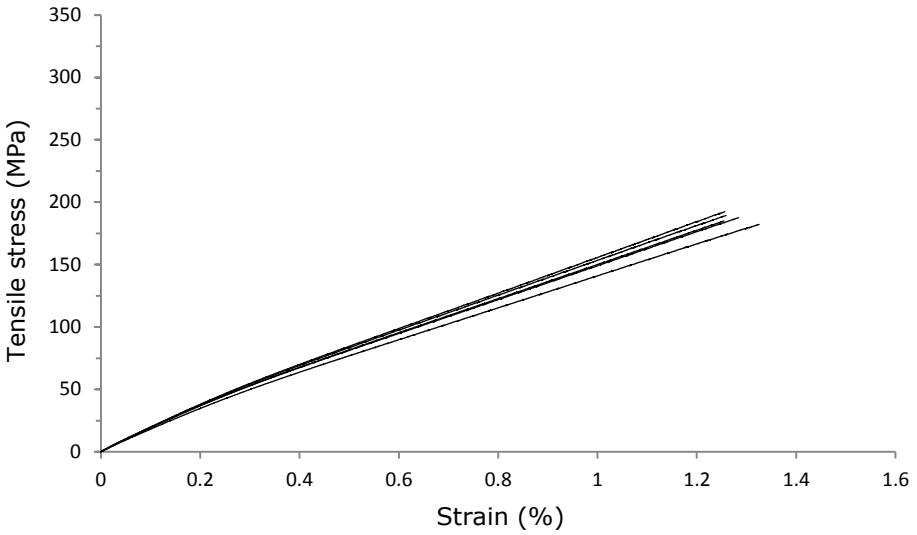


Fig.3.28 Stress-strain curves of composites manufactured with HEC sized hairy yarn

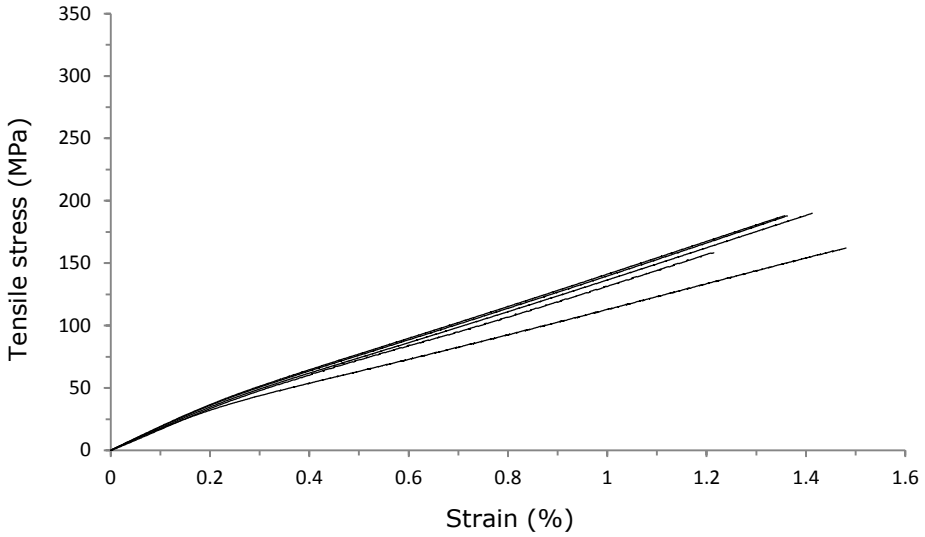


Fig.3.29 Stress-strain curves of composites manufactured with HEC sized hairless yarn

Stress-strain curves of UD jute composites in the longitudinal direction of yarn are presented in Fig.3.26 to Fig.3.29. Characteristics of the curves obtained from different plaques of composites are similar. An initial linear portion, an extended curved region and again a linear portion with lower slope are observed for each plaque of composite tested. Similar behaviour

of stress-strain curves of UD composites were found by Dwaikat et al. [286] and Rozite et al. [287]. The initial linear portion of the stress-strain curve during tensile loading is clearly visible, and when micro cracking of the matrix starts, the stiffness of the composites falls. This micro cracking happens over a longer period and during micro cracking of the matrix there may be an extension of composites due to small straightening of helically configured fibres, resulting in an effective reduction in stiffness. Propagation of cracking of matrix continues with increasing load and the composite fails. After the curved region stress and strain are become proportional again.

3.5.6.(II) Transverse direction of yarn

Table 3.6 Fibre volume fraction and tensile test results of composites

Sample name	Volume fraction (%)	Tensile modulus (GPa)	Std. dev.	Tensile strength (Mpa)	Std. dev.	Strain at failure (%)	Std. dev.
Hairy yarn (unsized)	36	2.4	0.27	7.0	0.73	0.80	0.08
Hairless yarn (unsized)	36	3. 2	0.79	7.6	0.96	0.43	0.13
Hairy yarn (sized)	36	3.6	1.03	7.3	1.06	0.29	0.02
Hairless yarn (sized)	34	3.9	0.52	9.2	0.30	0.25	0.02

Std. dev.= Standard deviation

Composites manufactured with unsized hairy and hairless yarn possess similar fibre volume fraction (Table 3.6). Tensile modulus (Table 3.6 and Fig. 3.30) and tensile strength (Table 3.6 and Fig. 3.31) of these composite plaques do not show any statistically significant difference ($P<0.05$). However composites made with unsized hairy yarns shows 86% (see Table 3.6 and Fig. 3.32) higher strain at failure with respect to

composites manufactured with unsized hairless yarns in the transverse direction and this difference is statistically significant ($P<0.05$). Yarn hairiness is a possible reason for the increase in strain at failure. From Fig. 3.33 it is clear that after a certain level of strain (around 0.5%, which is very close to strain at failure of composites manufactured with unsized hairless yarn) there is almost no increase in stress. The increase in strain after this level is possibly due to bonding between yarn hairs and resin. Very similar tensile modulus and similar strain at failure is observed for composites manufactured with sized hairy and hairless yarn.

Composites manufactured with sized hairless yarn show 26% increase in tensile strength with respect to composites manufactured with sized hairy yarn (Table 3.6) which is statistically significant. As sizing is performed for both types of yarns, resulting in a smooth surface as if the yarn is hairless, no specific reason is identified as a cause of significant variation of tensile properties.

Highly anisotropic plant fibres [288] cannot improve the tensile properties of composites in the transverse direction and the tensile modulus of composites in the transverse direction is very similar to the tensile modulus of resin and the tensile strength of composites is much lower than that of resin (see Table 3.1). For this reason back calculated fibre tensile properties are not determined for the transverse direction. Tensile modulus and tensile strength of UD jute polyester composites in transverse direction are found very similar (3.25 GPa tensile modulus, 11 MPa tensile strength and 0.35% strain at failure) to those of other researchers [289].

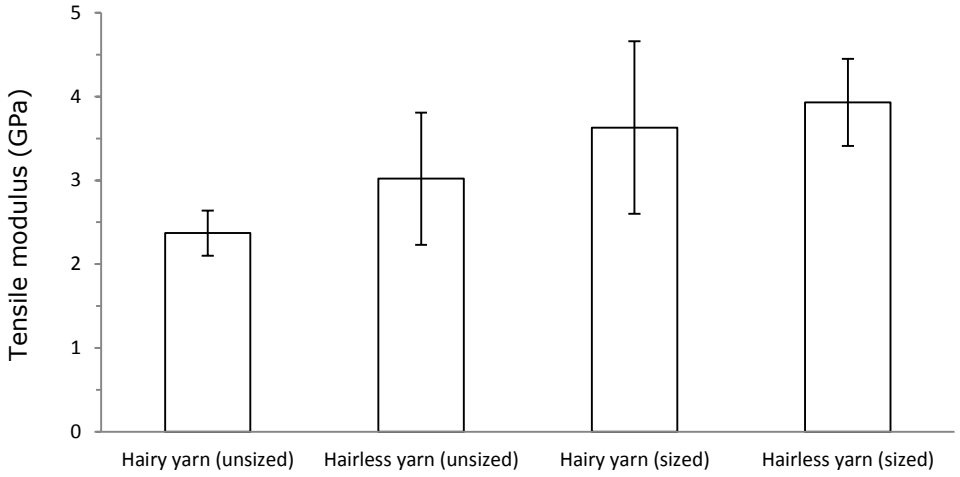


Fig. 3.30 Tensile modulus of composites (error bars indicate standard deviation)

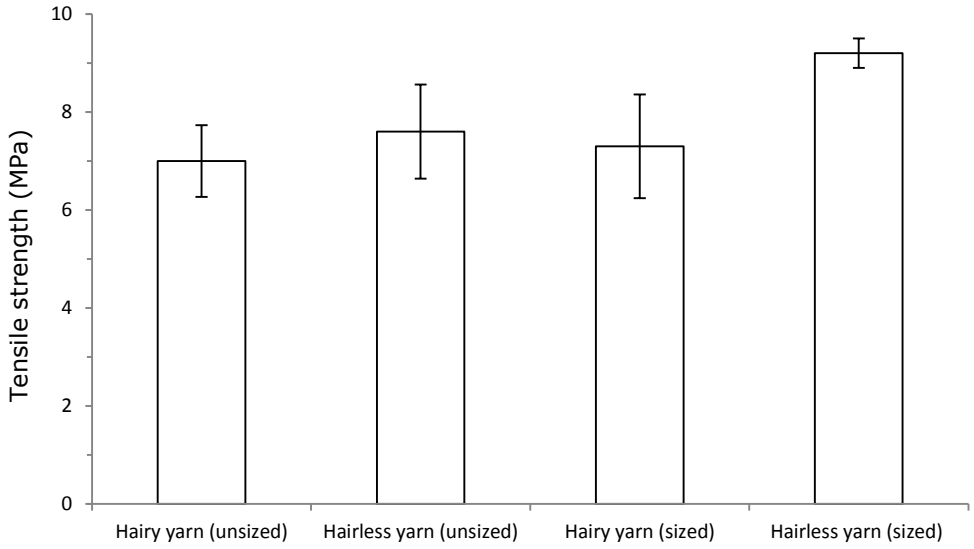


Fig.3.31 Tensile strength of composites (error bars indicate standard deviation)

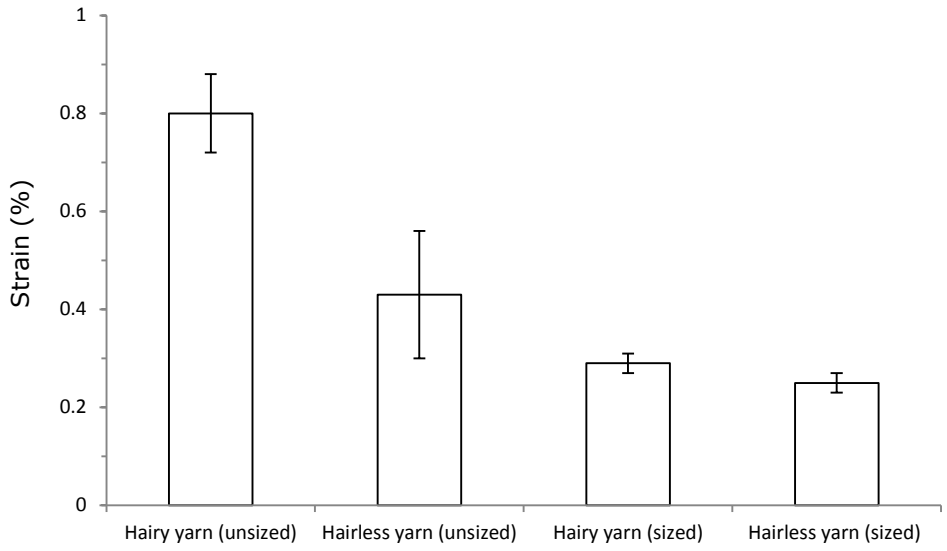


Fig.3.32 Strain at failure of composites (error bars indicate standard deviation)

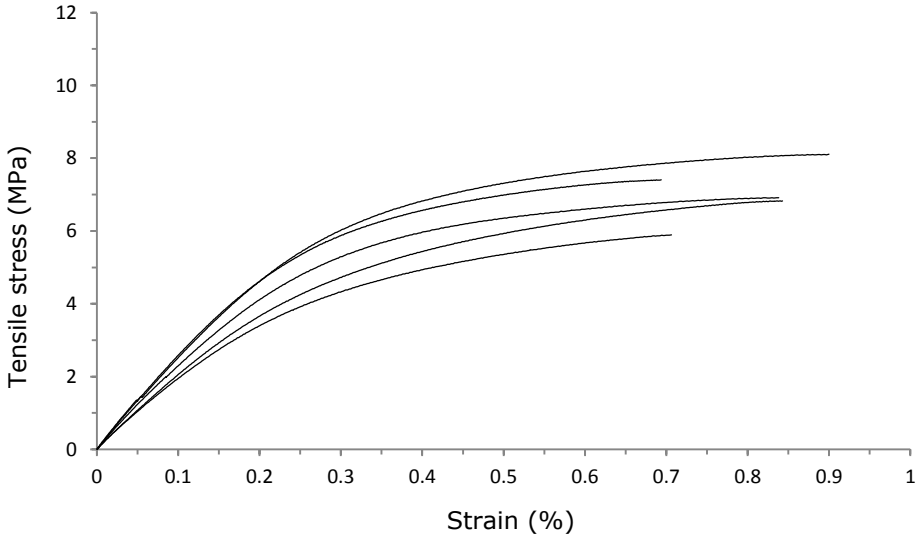


Fig.3.33 Stress-strain curves of composites manufactured with unsized hairy yarn

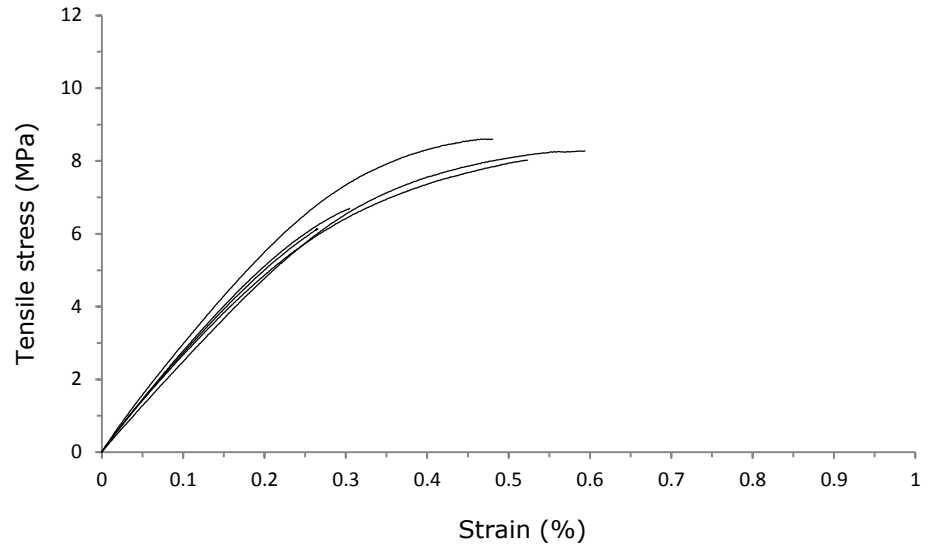


Fig.3.34 Stress-strain curves of composites manufactured with unsized hairless yarn

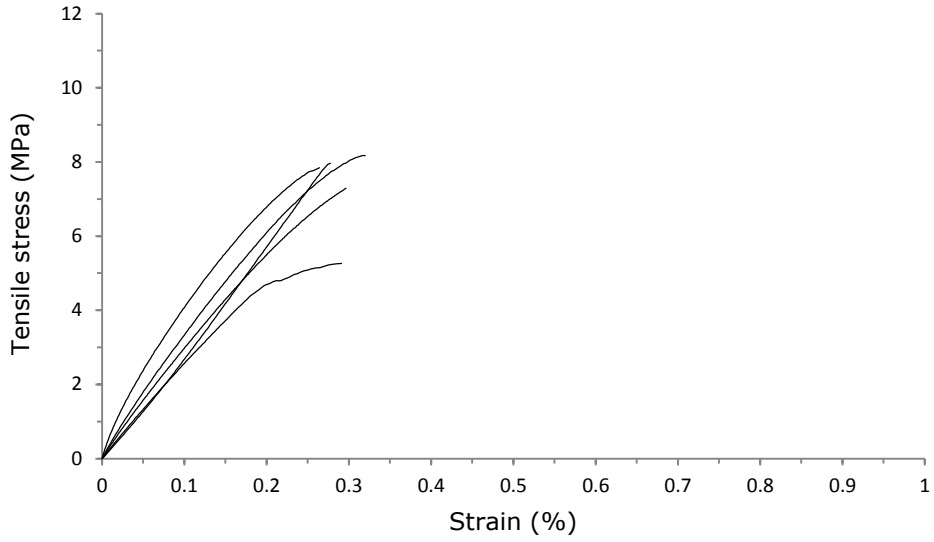


Fig.3.35 Stress-strain curves of composites manufactured with HEC sized hairy yarn

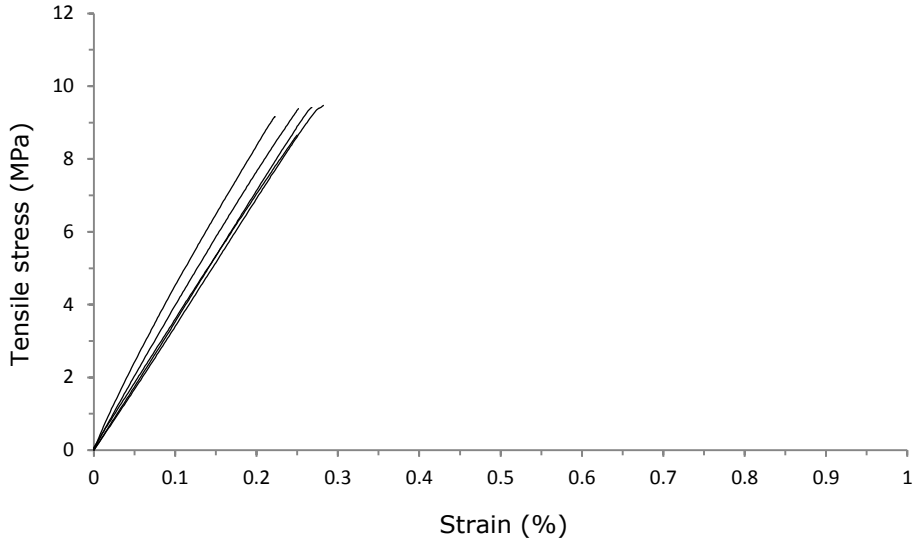


Fig.3.36 Stress-strain curves of composites manufactured with HEC sized hairless yarn

Stress-strain curves of composites manufactured with unsized hairy yarns (Fig. 3.33) show an initial linear portion, an extended curved region and again a linear portion which is similar to the curves obtained in the longitudinal direction (Section 3.5.7.(I)). Thus a similar explanation is also valid here, however the curved region and second linear portion in the transverse direction are due the bonds between hairs and resin. As bonds between hairs and resin are unable to carry significant stresses, at the end of the curved region (around 0.5% strain) an increase in strain is observed with a small increase in stress. Stress-strain curves of composites manufactured with unsized hairless yarns (Fig. 3.34) show an initial linear portion and an extended curved region. As the manual burning process cannot remove all hairs thus bonds between remaining hairs and resin may be responsible for the curved region for composites manufactured with unsized hairless yarn. However stress-strain curves of composites manufactured with sized yarns (Fig. 3.35 and Fig. 3.36) show only linear behaviour. The surface of sized yarns (hairy and hairless) becomes smooth (see Fig. 3.4) thus linear stress-strain curves occur with low strain at

failure for composites manufactured with these yarns. Large standard deviations of the tensile properties of composites in the transverse direction are observed, which is very common for plant fibre composites due to natural variations of plant fibres.

3.6 Conclusions

At the end of the chapter the following findings can be summarised.

- Burning hairs by candle flame does not affect the failure load of yarn.
- DSC and FTIR analysis of hairy and hairless yarns show no major change of properties due to burning hairs.
- XPS study presents differences of surface chemicals between hairy and hairless yarn such as hairless yarn show higher carbon, less oxygen and less nitrogen content than those of hairy yarn. Burning hairs using open candle flame makes these changes.
- SEM EDX spectra reveals that lumens of jute fibres in composites are filled with resin and do not contribute to the void content of jute/polyester composites.
- Yarn hairiness affects the tensile modulus and strain at failure of UD composites in the longitudinal direction and strain at failure in the transverse direction of yarns. An increase in tensile modulus as well as a decrease in strain at failure of composites manufactured with unsized hairy yarn is observed in the longitudinal direction with respect to those properties of composites manufactured with unsized hairless yarn. An increase in tensile modulus of composites manufactured with hairy yarn could be of interest for composite manufacturers and may reduce the work load of the spinner, as

spinners need to pay attention to reduce the hairiness of yarn which is a prime requirement for most applications of textiles.

CHAPTER 4

INFLUENCE OF WATER AND HYDROXYETHYL CELLULOSE (HEC) TREATMENT ON THE MECHANICAL PROPERTIES OF JUTE FIBRES, YARNS AND COMPOSITES

4.1 Summary

The influence of water and HEC treatment on the tensile properties of jute fibres, yarns and composites is studied in this chapter. Jute yarns are treated by tap water with and without tension at room temperature for 20 minutes and then dried. The tensile properties of fibres and yarns are measured before and after treatment. Unidirectional (UD) composites are manufactured with treated and untreated yarns with and without applying HEC as a binder. All the composite plaques are tested for tensile properties.

4.2 Introduction

Surface modification of plant fibres using different chemicals is a common practice in composites as discussed in Section 2.5.3. Generally chemical treatment on plant fibres is performed in a water based solution. As a primary element of plant fibre, cellulose has a large amount of hydroxyl groups (-OH) on its surface and these groups have high attraction or interaction with water molecules that make them hydrophilic in nature [147]. Due to this property plant fibres absorb large amount of water as an effect of dipping them in water or aqueous solution and they swell [290]. The higher the amount of water absorption, the higher would be the swelling. Swelling increases the fibre volume [290] and affects the dimensional stability of fibres. At the same time longitudinal shrinkage of fibre can happen [291] affecting the tensile modulus and tensile strength of fibres [146]. As water plays an important role on the mechanical properties of treated fibres (swelling and shrinkage) thus water is applied

on jute yarn and the influence of water treatment on jute fibre, yarn and jute reinforced composites is of interest.

In this work jute yarn is received in spool form (Fig. 3.1) from the supplier and HEC is used as a binding agent to form UD mat from yarn. Preparation of UD mat and application of HEC on the yarn are discussed in Section 4.3.2 and Section 3.3.2 respectively. It is discussed in Section 3.3.3 that HEC makes a film around the yarn, building hydrogen bonds with fibres that help to form UD mat and facilitate transfer of fibres into an infusion mould. As HEC film coated yarn is used to manufacture UD composites thus the influence of HEC coating on the mechanical properties of composites, yarn and fibres is of interest. Coating of yarns by HEC may affect fibre-matrix adhesion, and is observed in this chapter by studying the mechanical properties of composites manufactured with both HEC sized and unsized yarns. The influence of the interlocked structure of HEC after drying and hydrogen bonding between fibre and HEC may affect the properties of yarns, and this is investigated by studying the tensile properties of yarn. Tensile properties of fibres extracted from HEC sized yarn and from unsized yarn are also studied to understand any change to tensile properties of single fibres.

Section 4.3 contains a description of materials and methods of composites manufacturing and Section 4.4 describes the mechanical tests performed on fibres, yarns and composites. Results are discussed in Section 4.5 and Section 4.6 draws some conclusions.

4.3 Materials and Methods

4.3.1 Materials

Information regarding fibre, matrix, catalyst, accelerator and mould release is provided in Section 3.3.1

4.3.2 Preparation of yarn for water treatment, sizing and composites

Yarns are manually wound on a perforated plastic tube under light tension applied by hand. The length of the tube is approximately 250 mm and the diameter is 50 mm. After water treatment, these yarns are described as water treated yarn with tension. Loose yarns (Fig. 4.1(A)) dipped in water are described as water treated yarn without tension. All yarns to be sized are wrapped closely together on a glass fibre composite sheet in a single ply (Fig. 4.1(B)). Sizing enables yarns to form a UD mat (the mechanism of forming the mat with HEC is discussed in Section 3.3.3). UD mat of unsized yarns was made by wrapping it on a square steel frame as described in Section 3.3.2.



(A) (B)

Fig. 4.1 (A) Yarn at loose condition before dipping and (B) yarn wrapped on glass fibre composite board.

4.3.3 Water treatment and preparation of composites

Loose yarns and yarns wrapped on the perforated tube are dipped in tap water at room temperature for 20 minutes. 10 times the weight of water to that of yarn was used to dip yarns. Yarns are then taken out of the water bath, squeezed and pressed gently and dried in an oven at 105^0C [261] for one hour. These yarns are stored at ambient condition for at least 48 hours before using for composites manufacturing.

Water treated yarns with and without tension are sized (yarn preparation for sizing is mentioned in Section 4.3.2) with HEC to produce a UD fibrous mat. The preparation of HEC and the procedure of sizing are discussed in Section 3.3.2. Composite plaques are manufactured using RTM and the manufacturing technique is explained in Section 3.3.4. One metre length of yarn is taken to determine the shrinkage due to water treatment without tension. Both the ends of the yarn are wrapped by a piece of thread (Fig. 4.2 (A)) to minimise the opening of fibres due to untwisting. These yarns are wrapped loosely on the plastic tube (Fig. 4.2 (B)) and dipped. Wrapping is performed to minimise the tangling of yarns during wetting (Fig. 4.2 (B)). Before dipping into water, both the ends of the yarn are tied



(A) (B) (C)

Fig.4.2 (A) Bound ends of yarn (B) yarn wound loosely on the tube before dipping in water (C) treated yarn after drying.

together loosely to avoid untwisting. After wetting for 20 minutes yarns are taken out of water and dried in an oven at 105°C as mentioned earlier followed by conditioning at ambient atmosphere for two days. The length of the treated yarn is measured to calculate the yarn shrinkage. Care is taken to avoid any stretching to remove tangling (Fig. 4.2 (C)) during measuring the length. Five samples are treated to find the mean shrinkage of the yarn.

4.3.4 Single fibre preparation for tensile test

Tensile strength of single fibre is affected by test length due to weak link theory. Virk et al. [292] showed that with increasing gauge length tensile strength of fibres decreased as expected. For example 557 MPa tensile strength was found for 6 mm length fibre tested however it was measured 154 MPa when fibre length was 300 mm. They tested jute fibres of 6 mm, 10 mm, 20 mm, 30 mm, 50 mm, 100 mm, 150 mm, 200 mm, 250 mm and 300 mm. Fidelis [86] and Anderson [87, 293] also found similar trend for jute and flax fibre respectively. Gradual decrease of tensile modulus of jute fibre also reported by Virk et al. [292] with increasing fibre length and was the highest for 6 mm fibre (32.5 GPa) and the lowest for 300 mm fibre (26.4 GPa). Fracture strain of fibres also decreased with increasing fibre length (1.79% for 6mm fibre and 0.58% for 300 mm fibre). However no specific trend is found for mean diameter of fibre depending on the fibre length.

Yarns are untwisted to separate fibres for tensile testing. Separation of fibres from yarns is performed using tweezers. This operation is performed carefully to minimise the fibre damage.

To facilitate the tensile testing of single jute fibres, a fibre is attached to a rectangular paper frame. The dimension of the paper frame is 35x20 mm.

Inside the frame a rectangular hole is cut. The dimension of the hole is 25x10 mm. Each individual fibre is attached on a separate paper frame (see Fig. 4.3) with araldite and is allowed to dry. The diameter of each fibre is measured using a Zeiss optical microscope at three places (both the ends and approximately midpoint) and the mean is considered as the fibre diameter. Practically the width of the fibre is considered as diameter



(A) (B)

Fig. 4.3: (A) Rectangular paper template and (B) fibre attached on the frame.

and the cross-section of the fibre is assumed circular. This is a well-established method and practised by other researchers for jute fibres [294, 295]. In this work only jute fibre (a kind of bast fibre) is used for tensile testing as well as for experimental work.

4.4 Testing

4.4.1 Single fibre tensile test

Tensile testing of single fibres is performed according to BS ISO 11566:1996 using a Hounsfield Series-S machine (Software-QMAT) with a cross head speed of 1 mm min^{-1}, gauge length 25 mm and a 5N load cell. Fibre gripping in this machine is performed by screw mounting jaws as shown in Fig. 4.4. At least 20 specimens are tested to obtain the mean value.



Fig. 4.4 Fibre testing on Hounsfield Series-S machine

4.4.2 Single yarn and composites tensile test

Unsized raw yarn, HEC sized raw yarn, unsized water treated yarn without tension, HEC sized water treated yarn without tension, unsized water treated yarn with tension and HEC sized water treated yarn with tension are used for tensile testing. The testing procedure is discussed in Section 3.4.1. Composites manufactured with unsized and HEC sized raw and water treated yarns are tested for tensile properties as described in Section 3.4.6. To measure the strain at failure of composite strips a mechanical extensometer (for some cases) and a non-contact video gauge (software: Imetrum) extensometer (Fig. 4.5) (for other cases) is used. To measure the tensile modulus as well as strain at failure of composites using the video gauge extensometer, composite strips are painted with a white background and then black dots are applied by spraying liquid paint. After painting, the strips are allowed to dry at room temperature to avoid any removal of paint during mounting of samples in the tensile testing machine.



Fig. 4.5 Video gauge extensometer.

4.5 Results and Discussion

4.5.1 Fibre test results

Tested results of jute fibre diameter, tensile modulus, tensile strength and strain at failure are presented in Table 4.1 and shown in Fig. 4.6 to Fig. 4.9. A minimum of 20 single fibres are tested for each sample and all the test results are contained in Appendix B. The mean values of fibre diameter (Table 4.1 and Fig. 4.6) presents minor variations among the fibres extracted from unsized raw yarn, HEC sized raw yarn, unsized water treated yarn without tension and HEC sized water treated yarn without tension. Fibres obtained from HEC sized water treated yarn with tension show 17% larger fibre diameter with respect to fibres separated from

unsized water treated yarn with tension and this variation is statistically significant ($P<0.05$, explained in Section 3.4.9). As HEC sizing of yarn does

Table 4.1: Fibre test results (mean values and standard deviation)

	Dia.** (µm)	Std. dev.	Tensile modulus (GPa)	Std. dev.	Tensile strength (MPa)	Std. dev.	Strain at failure (%)	Std. dev.
Raw	52	12	23.7	6.9	385	247	1.47	0.59
Raw HEC	54	15	18.4	6.2	374	179	1.87	0.53
Wet no ten* no HEC	54	14	21.7	8.8	398	229	1.70	0.64
Wet no ten HEC	60	13	19.8	5.6	329	152	1.39	0.44
Wet ten no HEC	48	7	24.3	8.7	386	188	1.54	0.45
Wet ten HEC	56	12	19.4	7.8	344	147	1.71	0.50

* ten=tension, ** Dia.=Diameter Std. dev.=Standard deviation

not increase the diameter of fibres significantly with respect to fibres extracted from a similar type of unsized yarn in the other two cases thus it cannot be said that the diameter increase of fibres extracted from HEC sized water treated yarn with tension with respect to fibres extracted from unsized water treated yarn with tension is due to HEC adhering as size

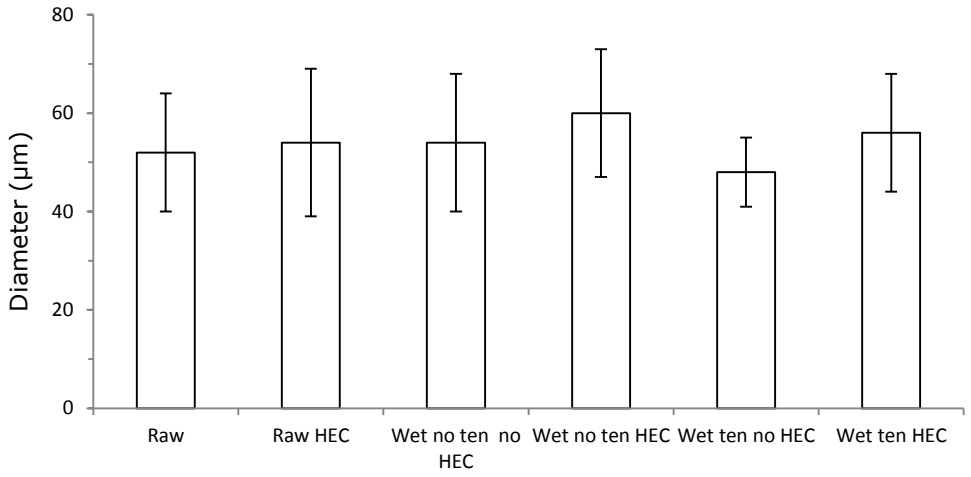


Fig. 4.6 Mean diameter of single fibre (error bars indicate standard deviation)

Table 4.2 Tensile properties of single jute fibre obtained by several researchers

Diameter* (µm)	Tensile* modulus (GPa)	Tensile* strength (MPa)	Elongation* at failure (%)	References
5-25	7.2	187-773	1.5-3.1	[46]
48.15	-	340.74	2.63	[295]
53.38±5.93	38.92±10.52	369±97	1.18±0.30	[134]
200	26.5	393-773	1.5-1.8	[296]
160-185	30	400-800	1.8	[297]
61.4±13.5	31.0±7.0	403.3±140	1.11±0.34	[294]
40	30±14	505±165	-	[24]
31.5±3.7	103±16	1233±406	1.3±0.4	[298]
52±12	23.7±6.9	385±247	1.47±0.59	This work

*Mean±standard deviation

take up by yarns is measured 1.5-1.8%. Natural variation plays the role to increase the mean diameter of fibres stated here and the large variation of jute fibre diameter also noted by other researchers (Table 4.2) and the fibre diameter is observed in this work is similar to that of noticed by other researchers [134, 291, 292]. High values of standard deviation are also due to inherent character of single jute fibres (Table 4.2).

Single fibres extracted from unsized yarns show higher tensile modulus with respect to fibres extracted from similar type of HEC sized yarns in all cases and fibres separated from unsized raw yarns show 28% higher tensile modulus with respect to fibres extracted from HEC sized raw yarn and fibres extracted from unsized water treated yarns with tension show 25% higher tensile modulus with respect to fibres extracted from HEC sized water treated yarns with tension (see Table 4.1 and Fig. 4.7). These variations are statistically significant ($P<0.05$) revealing that yarn sizing with HEC decreases the tensile modulus of fibres. Manual extraction of single fibres from HEC sized yarn may affect the tensile properties. The adhesive character of HEC binds fibres together in the yarn and separation of single fibres from this yarn can deteriorate the tensile properties of fibres as the separation process is performed against the adhesive character of HEC. Breaking the HEC film and pulling fibres against the bond between fibre and HEC can deteriorate the fibre properties. However a large variations of tensile modulus of single jute fibre is observed in previous studies [134, 292]. A large range of tensile modulus of fibre also tabulated by several researchers (Table 4.2) and tensile moduli of jute fibre observed by some researchers [289, 287] is similar to those of this work. The standard deviation of tensile moduli found in this chapter agrees with data obtained by Virk et al. [292] and Sayeed et al. [134].

Tensile strength of fibres studied in this chapter (Table 4.1 and Fig. 4.8) follows the trend of tensile modulus however the variations are minor. High values of standard deviations are also found for tensile strength of jute fibres studied in this chapter, which are similar to the data obtained by other researchers [134, 292]. Tensile strength of jute fibre found in this chapter agrees with the results obtained by other researchers [134, 287].

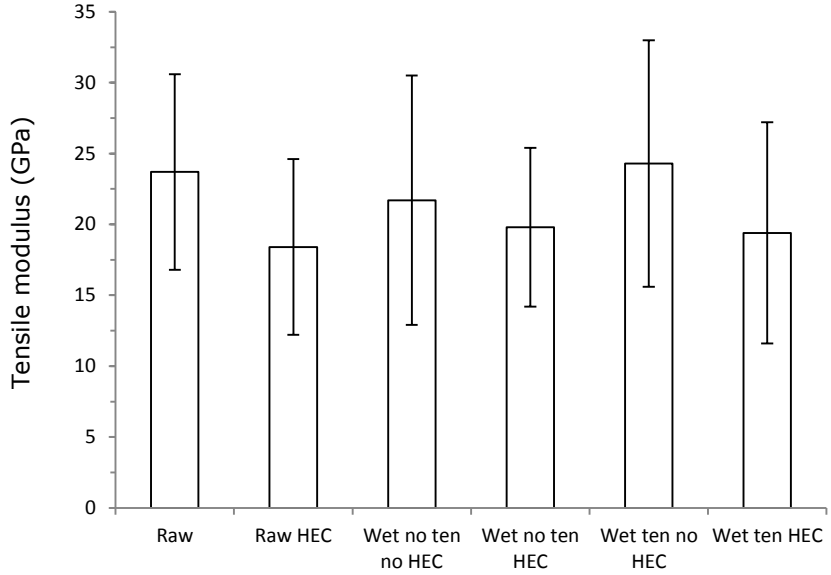


Fig. 4.7 Tensile modulus of single jute fibre (error bars indicate standard deviation)

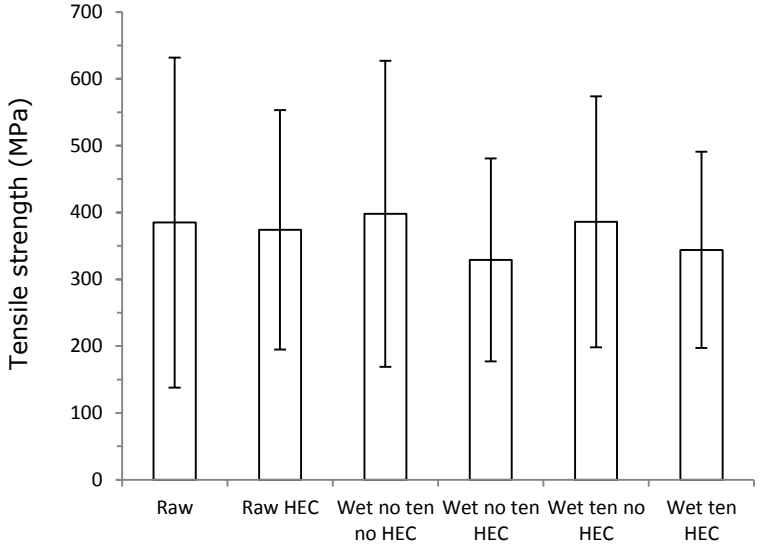


Fig. 4.8 Tensile strength of single jute fibre (error bars indicate standard deviation)

No specific trend is observed for strain at failure of fibres studied in this chapter (see Table 4.1 and Fig. 4.9). However fibres extracted from HEC sized raw yarn show 21% higher strain at failure with respect to fibres obtained from unsized raw yarn and this difference is statistically significant ($P<0.05$) however the cause of this variation is unknown. The differences in strain at failure of fibres extracted from unsized and sized

water treated yarns with and without tension are minor. However large standard deviations are also observed for strain at failure of fibres, similarly to other properties studied here.

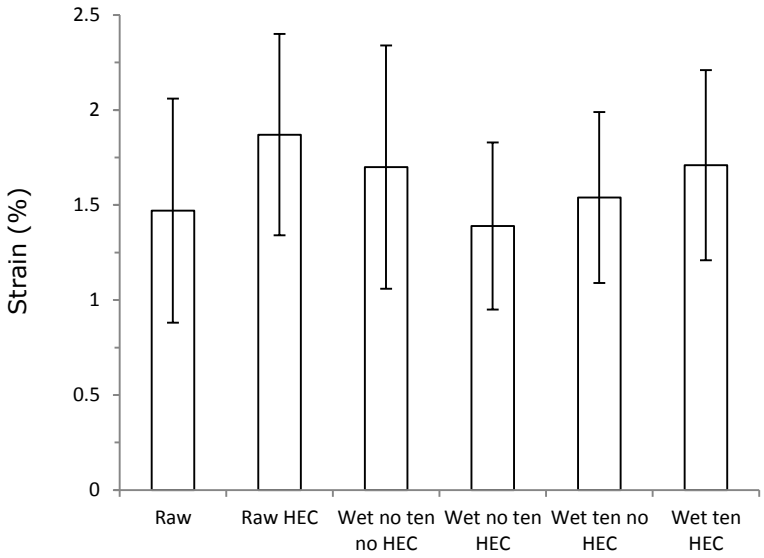


Fig. 4.9 Strain at failure of single jute fibre (error bars indicate standard deviation)

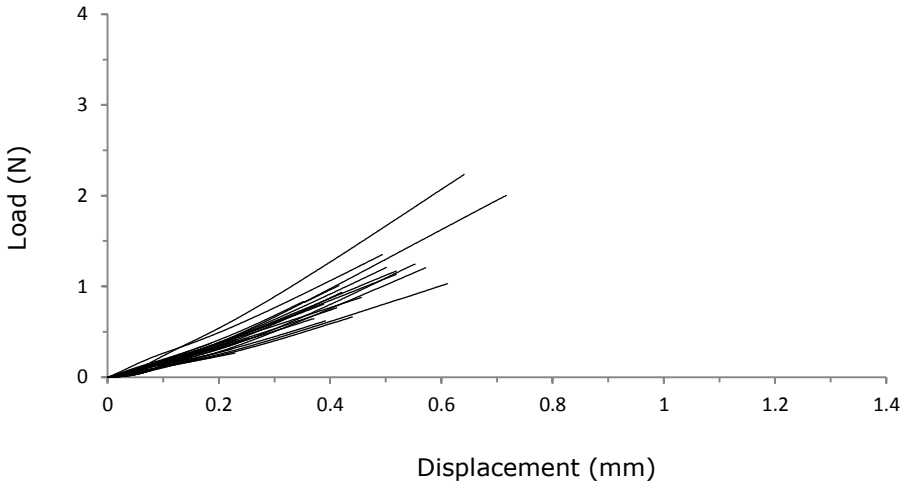


Fig. 4.10 Force-displacement curves of single jute fibres extracted from unsized raw yarn

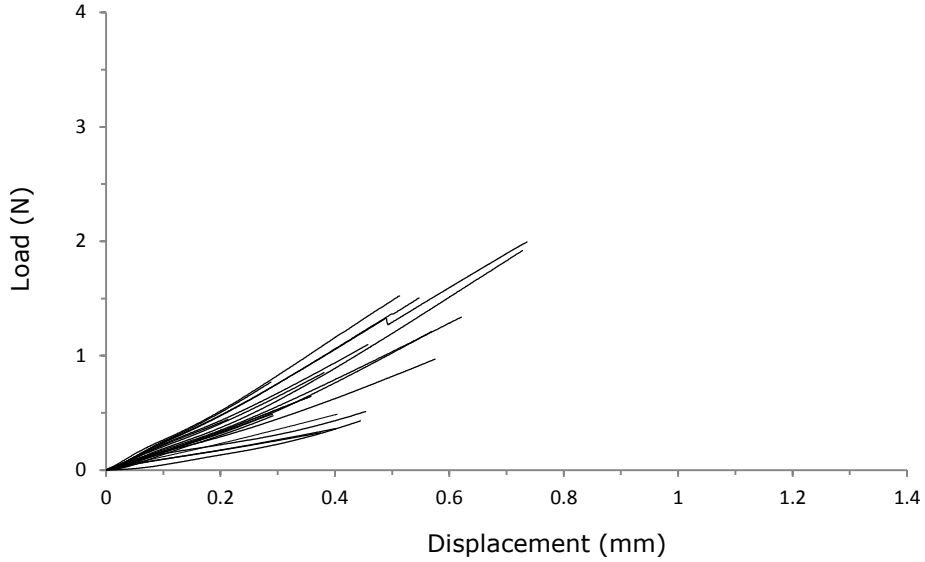


Fig. 4.11 Force-displacement curves of single jute fibres extracted from HEC sized raw yarn

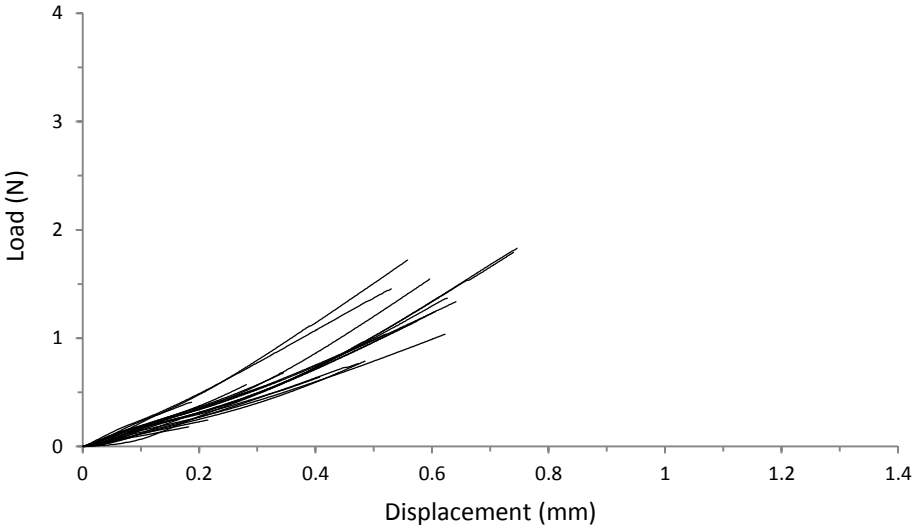


Fig. 4.12 Force-displacement curves of single jute fibres extracted from unsized water treated yarn without tension

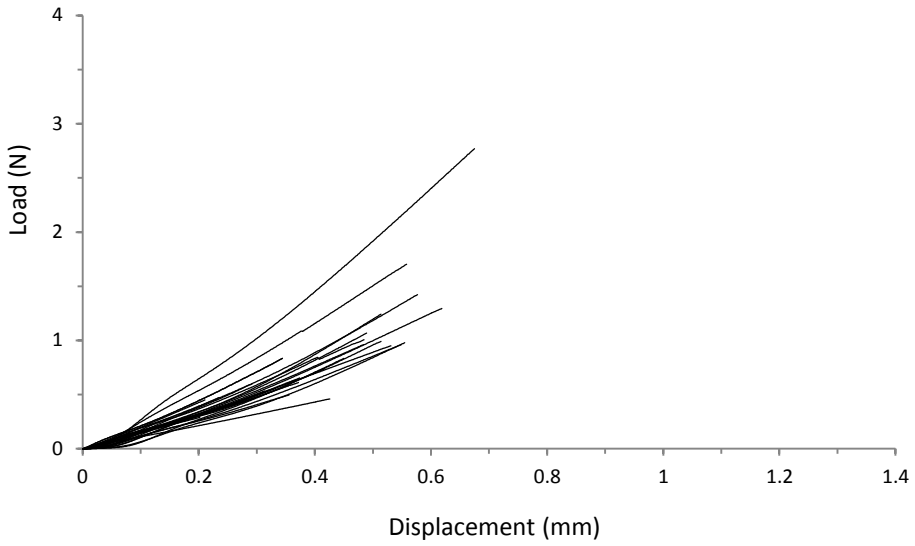


Fig. 4.13 Force-displacement curves of single jute fibres extracted from HEC sized water treated yarn without tension

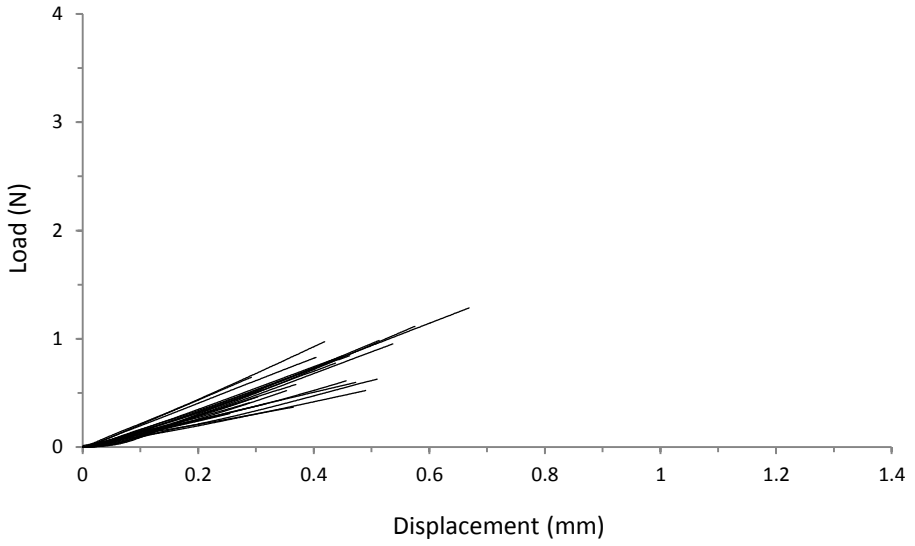


Fig. 4.14 Force-displacement curves of single jute fibres extracted from unsized water treated yarn with tension

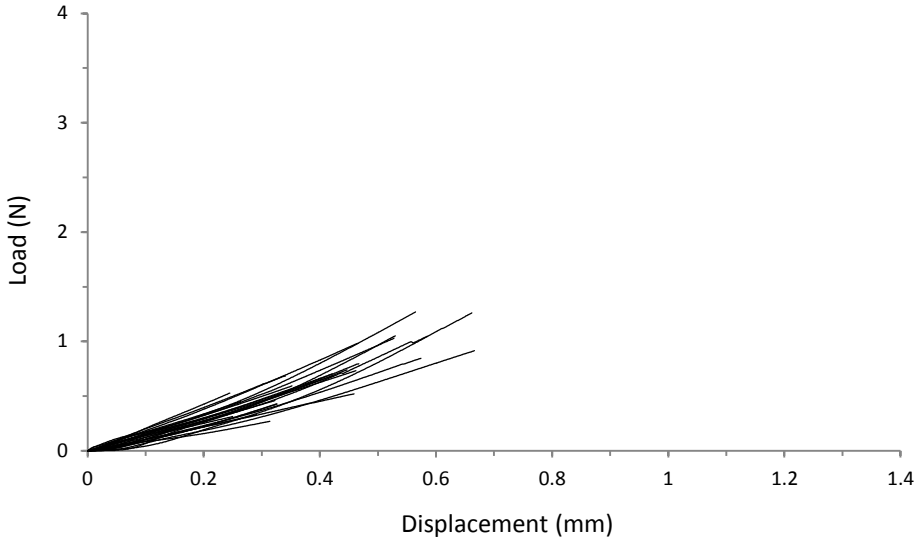


Fig. 4.15 Force-displacement curves of single jute fibres extracted from HEC sized water treated yarn with tension

Force-displacement curves of single fibres studied in this chapter are presented in Fig. 4.10 to Fig. 4.15 and the characteristics of curves seems very similar revealing that the water treatment and HEC sizing do not affect the force displacement curves of single jute fibre. However the nature of force-displacement curves of jute fibres tested in this chapter is different from that of other common textile fibres such as cotton [299, 300] and flax [301]. The force-displacement curves of common textile fibres has an initial elastic region, a yield point and quasi-linear plastic region. However the force-displacement curves of most of the jute fibres obtained in this chapter show initially a very small linear region and then the slope of the curves increases presenting stiffer behaviour with increasing load. The initial linear region observed here is very small, nearly 0.2% of total extension and possibly this region is due to extension of hemicellulose and lignin. In this region the curves show a low gradient as these are low modulus materials [46]. With increasing load fibres show an increase in tensile modulus due to untwisting of cellulose microfibrils, which remain helical with respect to the longitudinal axis of fibres [2, 45]. Untwisting and

stretching of microfibrils occurs simulataneously and continues up to fibre failure.

In most cases the distribution of curves for fibres extracted from HEC sized yarns is wider with respect to that of fibres extracted from unsized yarns. This may be due to uneven sizing that includes faults as well as deterioration of fibre tensile properties during fibre extraction from yarns as discussed earlier in this section.

4.5.2 Yarn test results

Tensile modulus and tenacity (calculation method explained in Section 3.4.1) of jute yarns studied in this chapter are presented in Table 4.3, Fig. 4.17 and Fig. 4.18. Unsized water treated yarn without tension shows 20% lower tensile modulus and 23% lower tenacity with respect to unsized raw

Table 4.3 Tensile test results of single yarns

	Tensile modulus (GPa)	Standard deviation	Tenacity (cN/Tex)	Standard deviation	Strain at failure (%)	Standard deviation
Raw	4.5	0.7	12.5	1.8	5.1	1.5
Raw HEC	4.4	0.4	12.0	1.7	4.7	0.8
Wet without tension unsized	3.7	0.6	9.6	1.7	6.1	3.5
Wet without tension HEC sized	4.5	0.6	10.8	2.3	5.5	1.4
Wet with tension unsized	5.3	0.8	13.2	1.9	6.5	2.8
Wet with tension HEC sized	5.5	0.7	14.6	2.3	6.9	2.6

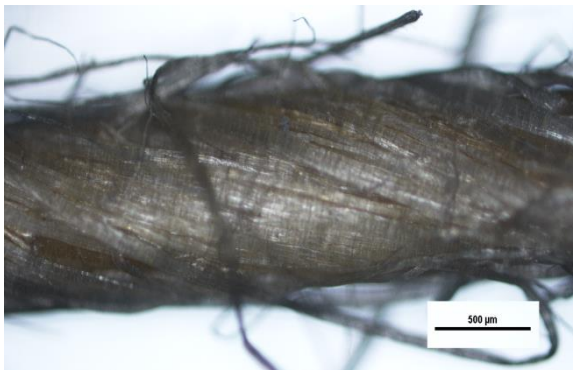
Table 4.4 Shrinkage percentage of water treated yarn without tension

Specimen number	1	2	3	4	5	Mean	Standard deviation
Shrinkage in %	4.5	5.5	4	4.5	4	4.5	0.55

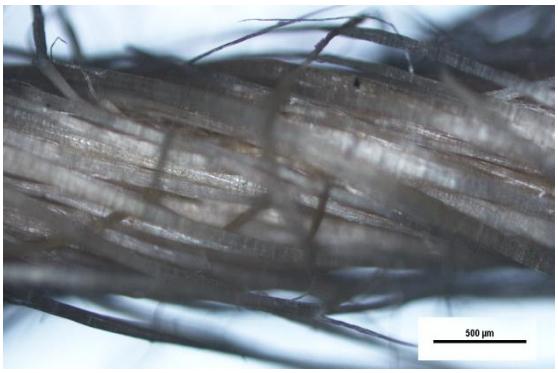
yarn. These variations are statistically significant ($P<0.05$). The principal cause of deterioration in tensile properties of water treated yarn without tension is shrinkage. When dipping cellulose yarn in water in a loose condition shrinkage occurs and it is measured in this chapter at 4.5% (Table 4.4)**.** The jute spinning process involves a series of operations including drawing (pulling of fibres due to higher speed of front rollers or gills with respect to that of back rollers or gills), drafting (attenuating the fibre bunch) and twisting. All of these operations apply external stresses due to implementation of tension that induces a level of stretching to fibres [302] and stretched fibres tend to relax when tension is removed. At unloaded condition (when externally applied load is removed) fibres try to release strain and achieve a minimum energy level [303], thus shrinkage occurs which is really a contraction of yarn. In addition to releasing stretch, wetting of hydrophilic yarn causes shrinkage due to fibre swelling as explained by Collins [304]. The amount of swelling depends on the amount of water absorption, voids in fibres and proportion of amorphous and crystalline regions [290]. Yarn diameter increases due to fibre swelling. Swollen fibres undulate as they pass around the yarn resulting in shrinkage of the yarn [304] and shrinkage causes misalignment of fibres which reduces the tensile strength of yarn.

Due to variation of position of fibres in yarn (from yarn centre to surface) all fibres cannot shrink to the same extent, thus rearrangement of fibres occurs which can cause some internal change of yarn structure and the

result is a slightly open structure of yarn (Fig. 4.16). Fibres in this yarn face less cohesive force which also contributes poorer mechanical properties. Gassan and Bledzki [146] also mentioned that shrinkage of jute yarn reduces tensile modulus and tensile strength.



(A)



(B)

Fig. 4.16 Raw yarn (A) and water treated yarn without tension (B)

Tensile modulus of unsized water treated yarn with tension increases 18% compared to that of unsized raw yarn and this difference is statistically significant ($P<0.05$). During water treatment of yarn under tension, yarns are prevented from shrinking, which may have helped fibres to become oriented along the yarn axis [146]. That helped to increase the tensile modulus of water treated yarn with tension with respect to that of raw yarn.

Tensile modulus of HEC sized water treated yarn without tension increased by 28% compared to unsized water treated yarn without tension (Fig. 4.17) and this difference is statistically significant ($P<0.05$). Increase in tensile properties of sized yarn with respect to unsized yarn is a well cited [305, 306] character of spun yarn since the size material has an adhesive property which binds fibres in the yarn together, preventing fibre slippage during axial loading making the yarn stiffer and stronger. The trend change in tensile modulus (Fig. 4.17) due to treatment studied on yarn in this chapter is similar to the change in tenacity (Fig. 4.18); however the intensity of variation is more prominent for modulus than tenacity.

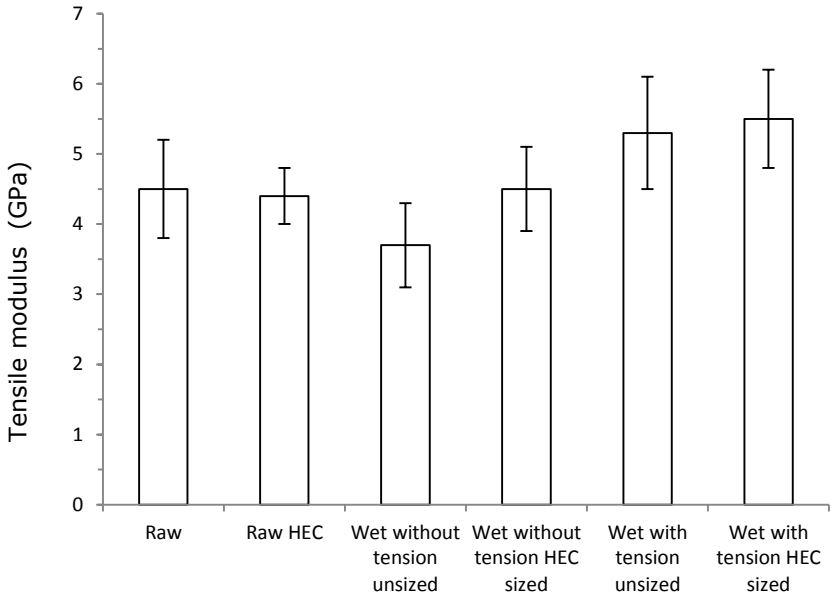


Fig. 4.17 Tensile modulus of single jute yarn (error bars indicate standard deviation)

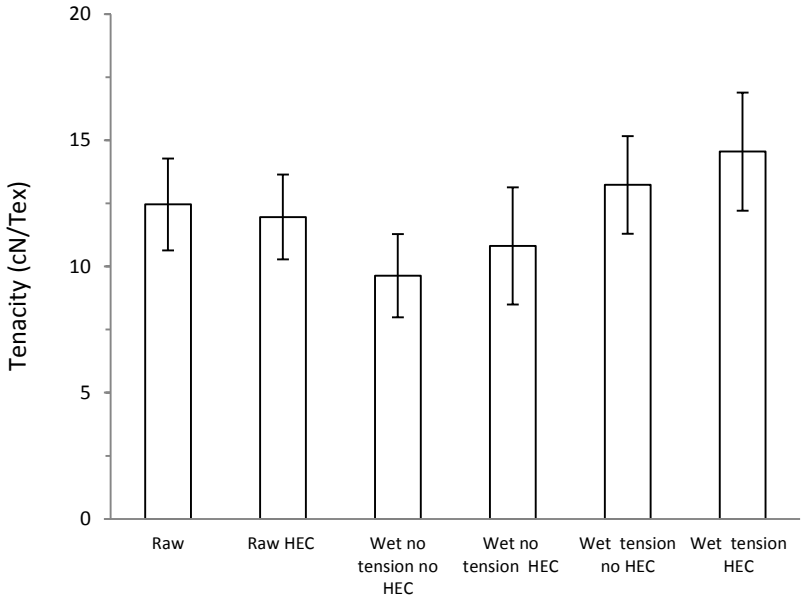


Fig. 4.18 Tenacity of single jute yarn (error bars indicate standard deviation)

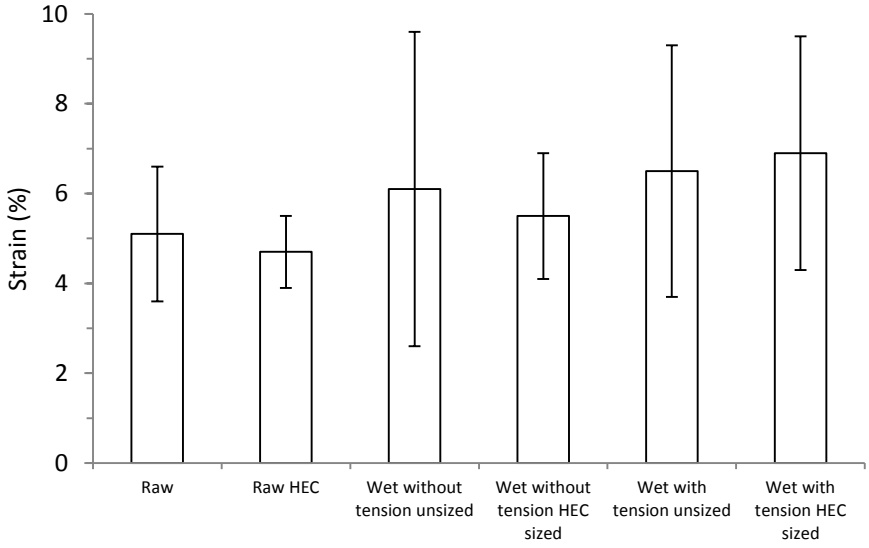


Fig. 4.19 Strain at failure of single jute yarn (error bars indicate standard deviation)

No specific trend of strain at failure of unsized and sized yarns is observed (Table 4.3 and Fig. 4.19) and the differences are minor for most of the cases. Variation between HEC sized raw yarn and HEC sized water treated yarn with tension (32%) is statistically significant ($P<0.05$) and is probably not due to the effect of water treatment but may be related to the poor performance of HEC sized raw yarn. Water treatment and HEC sizing of yarn may not have any effect on strain at failure of yarn.

Fibres extracted from unsized raw and unsized water treated yarns with and without tension show similar values of tensile modulus and similar values of tensile strength (Fig. 4.7 and Fig. 4.8) revealing that water treatment does not affect the tensile properties of fibres. Water treatment at room temperature followed by drying at $105^{O}C$ is not supposed to affect the tensile properties of fibres. However water treatments affects the tensile properties of yarn due to changing the yarn structure as discussed earlier, nevertheless no change of fibre properties is noticed. Again similar values of tensile modulus and similar values of tensile strength are observed for fibres extracted from HEC sized raw and HEC sized water treated yarns also revealing that water treatment does not affect the tensile properties of fibres. The reduction in tensile modulus of fibres extracted from HEC sized yarn occurred during separation of fibres from yarn, as discussed earlier. On the other hand sizing increases the tensile properties of yarn due to the reasons discussed earlier of this section and no relationship between tensile properties of fibres and tensile properties yarn is observed. No specific relationship is found between the treatment studied in this chapter and strain at failure of fibres as well as strain at failure of yarn.

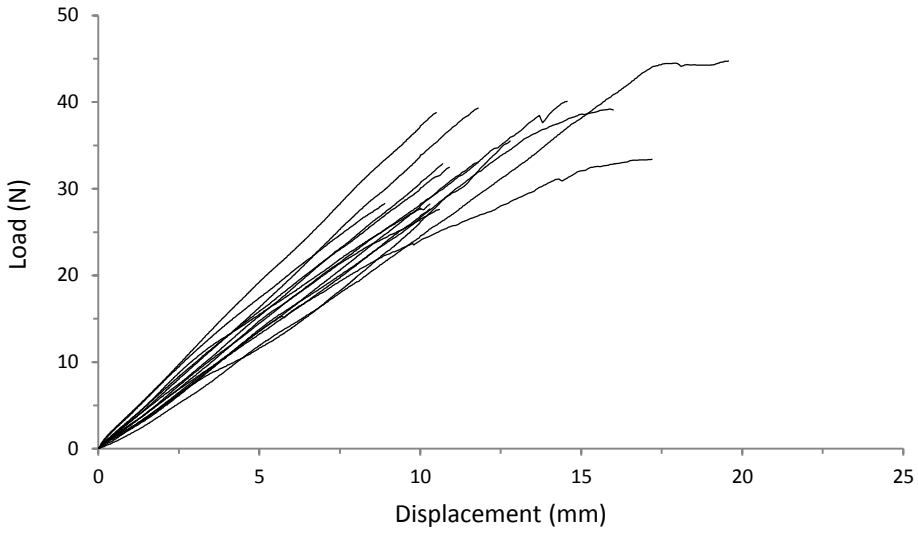


Fig. 4.20 Force displacement curves of unsized raw yarn

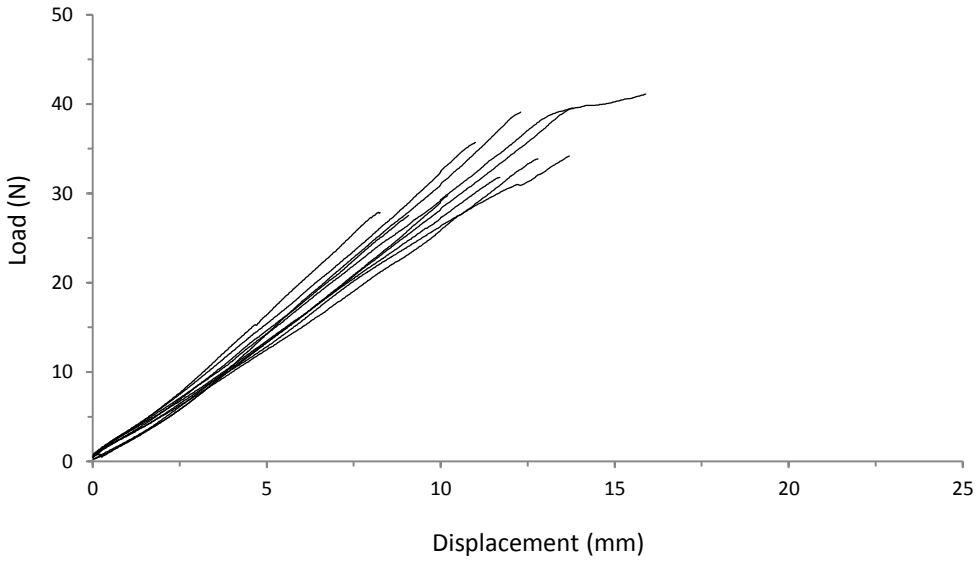


Fig. 4.21 Force displacement curves of HEC sized raw yarn

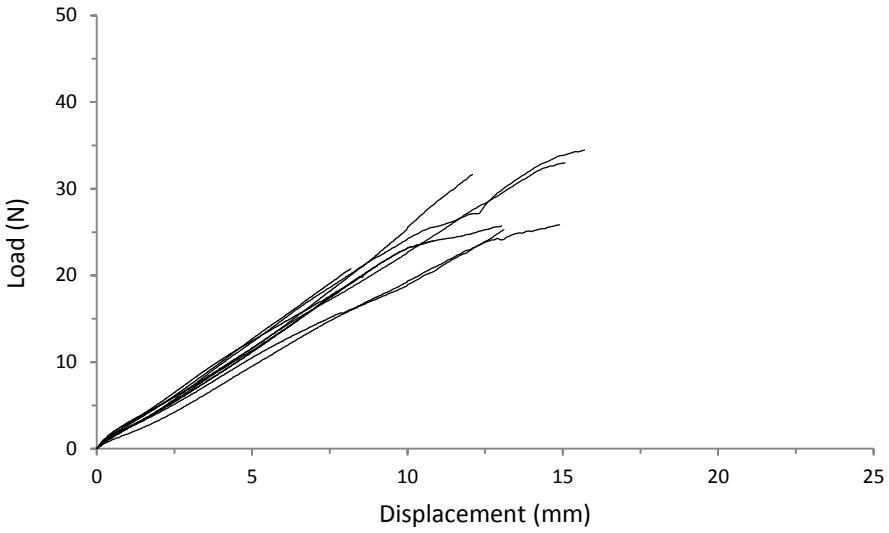


Fig. 4.22 Force displacement curves of unsized water treated yarn without tension

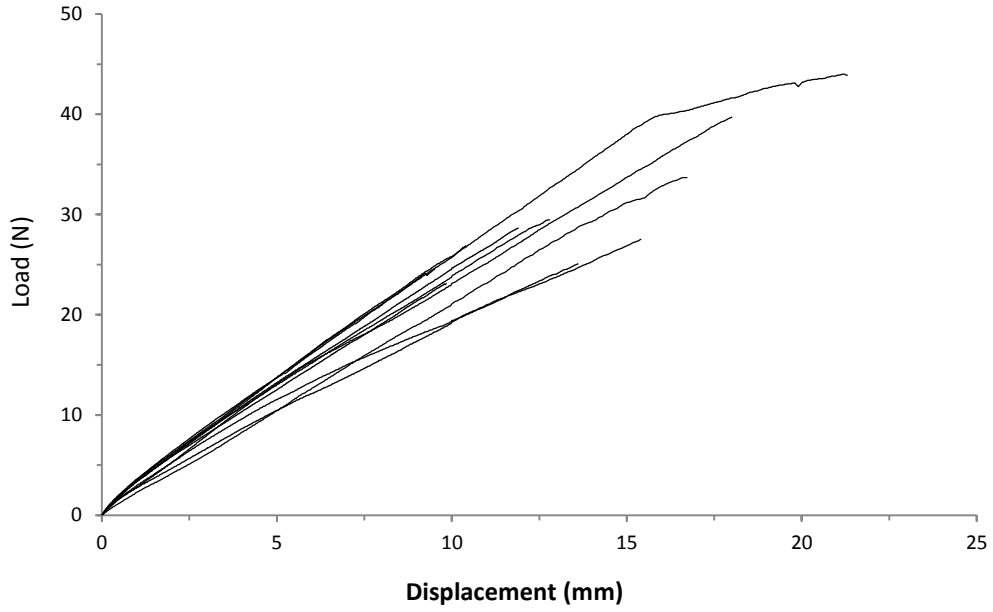


Fig. 4.23 Force displacement curves of HEC sized water treated yarn without tension

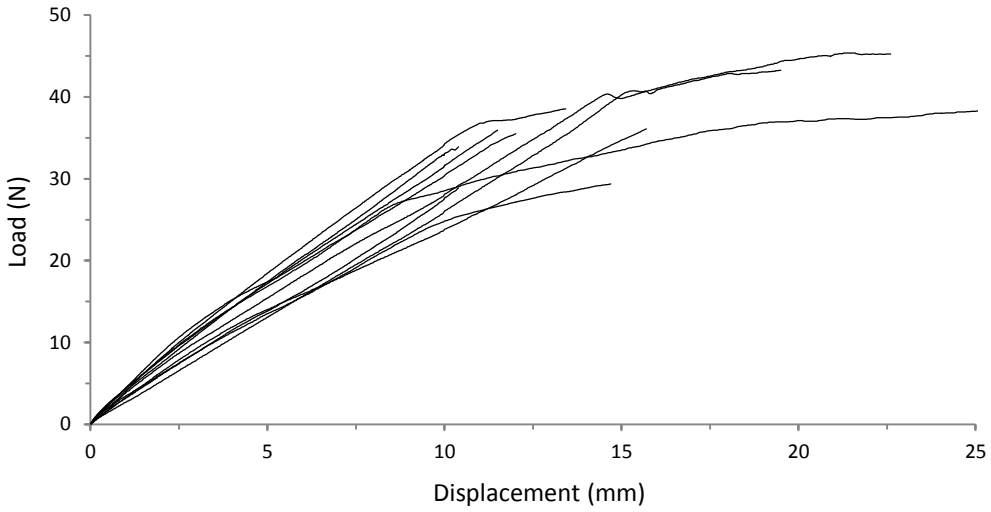


Fig. 4.24 Force displacement curves of unsized water treated yarn with tension

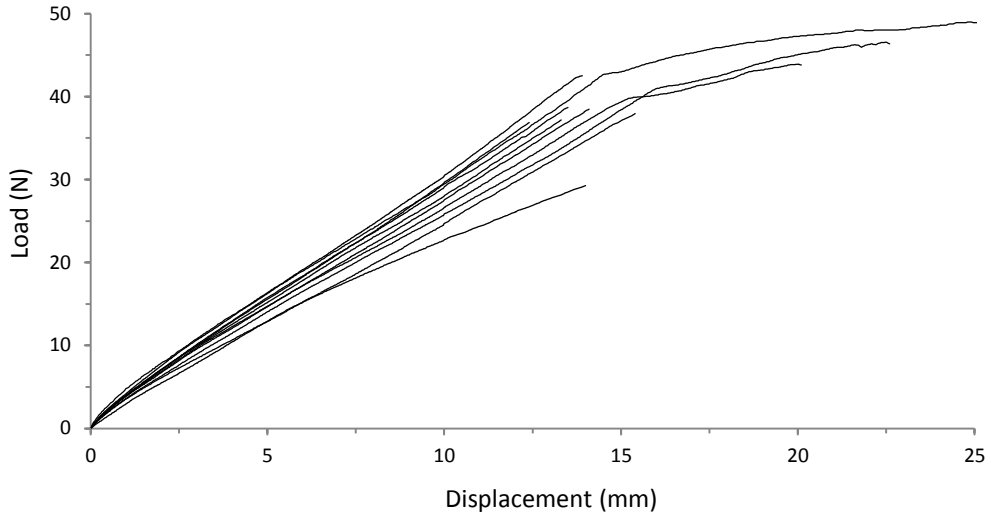


Fig. 4.25 Force-displacement curves of HEC sized water treated yarn with tension

Force displacement curves of yarns tested in this chapter (Fig. 4.20 to Fig. 4.25) show a very small initial linear region, a yield point and again a linear portion for maximum cases which are similar to the curves found by other researchers for spun yarns [307, 308]. Possibly initial straightening of fibres in yarn is the cause of initial linear portion of the curve. Cause of yield point is possibly fibre slippage due to the axially applied load which reduces the slope of the curve. Most likely the slippage of fibres occurs up

to yarn failure and thus a straight line after yield point continues up to breaking point. However in some tests yarn show linear force-displacement curve which may be due to faults associated with yarn mounting in the tensile testing machine e.g. stretching during fixing of yarn into the jaw. Sudden failure of yarn is found for most of the cases revealing that multiple fibre rupture happens at the same time. However in some tests, after approximately 10 mm extension yarn extends with very little or no increase in force. This occurred due to slippage of fibres in yarn without breaking the majority of fibres.

From Table 4.5 it can be observed that approximately 20% of fibre tensile modulus could be utilised in yarn however approximately 45% of tensile strength of fibre is utilised. There may be several causes of reduction in tensile properties of yarn with respect to those of fibres. In staple yarns, fibres are combined by twist which introduces cohesive forces (generated by friction) among the fibres which provide the stiffness and strength of yarns. During axially applied load on yarns these fibres can slip resulting in inferior tensile properties meaning poor utilisation of fibre properties in yarn. Twist introduces a helical configuration of fibres in yarn that causes off-axis loading of fibres also reducing tensile properties. There is a possibility to introduce some processing faults during spinning that might also be responsible for reduced tensile properties of yarn. Slippage of fibres in a yarn during application of load causes higher strain at failure for yarn with respect to that of fibres as slippage phenomenon is not applicable for fibres.

Table 4.5 Comparative tensile properties of fibre and yarn

	Tensile modulus of yarn (GPa)	Tensile modulus of fibre (GPa)	Ratio (yarn/ fibre)	Tensile strength of yarn (MPa)	Tensile strength of fibre (MPa)	Ratio (yarn/ fibre)	Yarn failure load (N)	Yarn** failure load (N)	Ratio (yarn/ fibre)	Strain at failure of yarn (%)	Strain at failure of fibre (%)	Ratio (yarn/ fibre)
Raw	4.5	23.7	0.19	178	385	0.46	34.9	82.3	0.42	5.1	1.47	3.5
Raw HEC	4.4	18.4	0.24	171	374	0.46	33.5	86.8	0.39	4.7	1.87	2.5
Wet no ten* no HEC	3.7	21.7	0.17	137	398	0.34	27.0	91.3	0.30	6.1	1.70	3.6
Wet no ten HEC	4.5	19.8	0.23	154	329	0.47	30.3	92.4	0.33	5.5	1.39	4.0
Wet ten no HEC	5.3	24.3	0.22	189	386	0.49	37.0	73.8	0.50	6.5	1.54	4.2
Wet ten HEC	5.5	19.4	0.28	208	344	0.60	40.7	84.8	0.48	6.9	1.71	4.0

* tension **Calculated from fibre failure load (in this work total number of fibres in yarn cross section is found to be 106 which is the mean value of five readings. Fibre failure load is multiplied by mean number of fibres found in yarn cross section to calculate the failure load of yarn from the failure load of fibre). Yarn strength is calculated from yarn failure load discussed in Section 3.4.1 based on data found in this chapter.

4.5.3 Composites test results

Table 4.6 Fibre volume fraction and tensile properties of composites

	Fibre volume fraction (%)	Tensile modulus (GPa)	Std. dev.	Tensile strength (MPa)	Std. dev.	Strain at failure (%)	Std. dev.
Raw	38	17.4	0.3	169	12	1.36	0.06
Raw HEC	35	18.3	0.6	187	4	1.34	0.07
Wet without tension unsized	36	15.5	0.2	154	6	-	-
Wet without tension HEC sized	36	17.4	1.2	180	12	1.36	0.11
Wet with tension unsized	36	17.6	0.6	185	4	-	-
Wet with tension HEC sized	37	19.3	0.6	215	4	1.4	0.04

Std. dev.=standard deviation

The fibre volume fractions of composite plaques studied in this chapter are presented in Table 4.6 and a large variation of fibre volume fraction is not found. The tensile moduli and tensile strengths of composites tested in this chapter are presented in Table 4.6, Fig. 4.26 and Fig. 4.27. Tensile moduli and tensile strength of composites manufactured with HEC sized yarns increase 5-12% and 9-17% respectively compared to composites made with respective unsized yarns. These differences are statistically significant ($P<0.05$) revealing that sizing of yarns by HEC increases the tensile properties of composites. It is likely that surface active –OH groups of HEC helps to build a better interfacial bond resulting in better quality composites. Thus to form UD mat from yarn or even from fibre, HEC can be applied as a binding agent

and this also has the capability to enhance the tensile properties of composites.

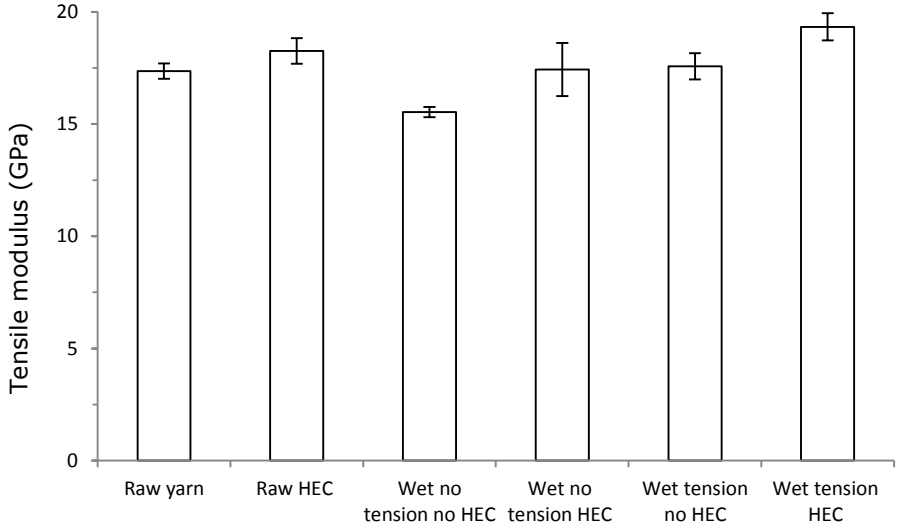


Fig. 4.26 Tensile modulus of composites (error bars indicate standard deviation)

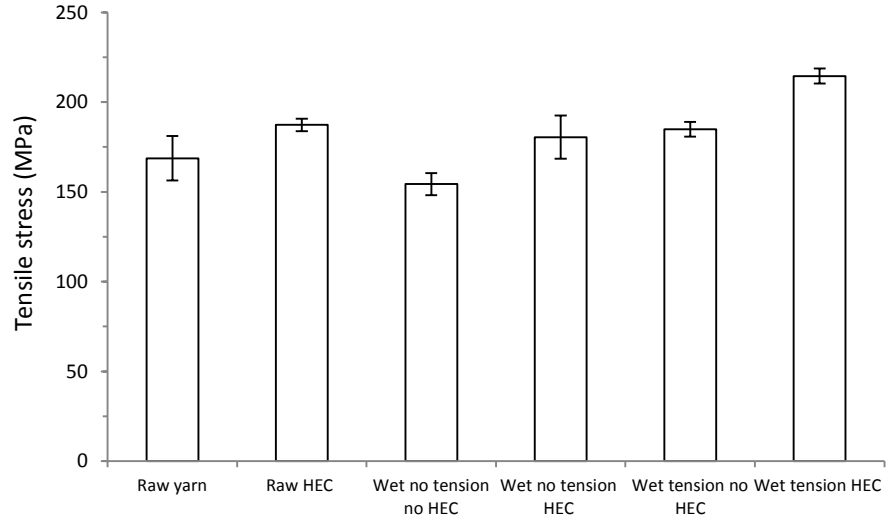


Fig. 4.27 Tensile strength of composites (error bars indicate standard deviation)

Composites made with unsized water treated yarn without tension show 11% lower tensile modulus and 10% lower tensile strength with respect to composites made with unsized raw yarn and these differences are statistically

significant (P<0.05). Dipping yarn in water in a loose condition (Fig. 4.28 (A)) enables tangling (yarn twisted within, Fig. 4.28 (B)) and drying this tangled yarn sets this configuration. The stiffness of the 1 mm thick steel frame (yarn is wrapped on it to manufacture composites with unsized yarn, as discussed in Section 3.3.2) is not sufficient to remove this tangling effect, thus alignment of yarn in composites is poor and this obviously affects the tensile properties of composites manufactured with unsized water treated yarn without tension.



(A) (B)

Fig. 4.28 The moment of dipping yarn in water at loose condition (A), tangling of yarn after wetting (B)

Composites made with unsized water treated yarn with tension show 8% higher tensile strength with respect to composites made with unsized raw yarn (which is untreated yarn thus no tension is applied) and this difference is statistically significant (P<0.05). As discussed previously (Section 4.5.2) water treatment of yarn with tension enhances the fibre alignment in the yarn axis which increases the strength of composites. Composites made with unsized water treated yarn with tension show 13.5% higher tensile modulus and 20% higher tensile strength with respect to those of composites made

with unsized water treated yarn without tension and these differences are statistically significant ($P<0.05$). Composites made with HEC sized water treated yarn with tension show 11% higher tensile modulus and 19% higher tensile strength with respect to composites made with HEC sized water treated yarn without tension and these differences are statistically significant ($P<0.05$). Composites made with HEC sized water treated yarn with tension show 15% increase in tensile strength than composites manufactured with HEC sized raw yarn and this difference is statistically significant ($P<0.05$). Possible cause of these variations is acquired alignment of fibres in yarn due to water treatment with and without tension is discussed in Section 4.5.2.

Composites made with HEC sized yarns present an increase in tensile modulus and tensile strength with respect to composites made with similar type of unsized yarns. Composites made with water treated yarn with tension show significantly higher tensile modulus and tensile strength with respect to composites made with water treated yarn without tension as well as raw yarn for most of the cases. Thus both treatments are found to be important to influence the tensile properties of composites.

Back calculated tensile properties of fibres are calculated following the rule of mixtures (as explained in Section 3.5.6(I)) and these are presented in Table 4.7, Fig. 4.29 (tensile modulus) and Fig 4.30 (tensile strength). Tensile properties of composites and back calculated tensile properties of fibres show similar trends. Darshil et al. [178], studied the tensile properties of UD jute polyester composites manufactured with HEC sized yarn and found similar value of back calculated tensile modulus (43.4 GPa) and back calculated

tensile strength (442 MPa) of fibre. No study is found on the tensile properties of composites manufactured with water treated jute yarn.

Table 4.7 Back calculated tensile properties of composites

	Tensile modulus (GPa)	Std. dev.	Tensile strength (MPa)	Std. dev.
Raw	39.7	0.91	329	29
Raw HEC	45.2	1.64	404	10
Wet without tension unsized	36.7	0.66	304	16
Wet without tension HEC sized	41.8	3.28	377	34
Wet with tension unsized	42.2	1.64	390	12
Wet with tension HEC sized	46.0	1.61	460	11

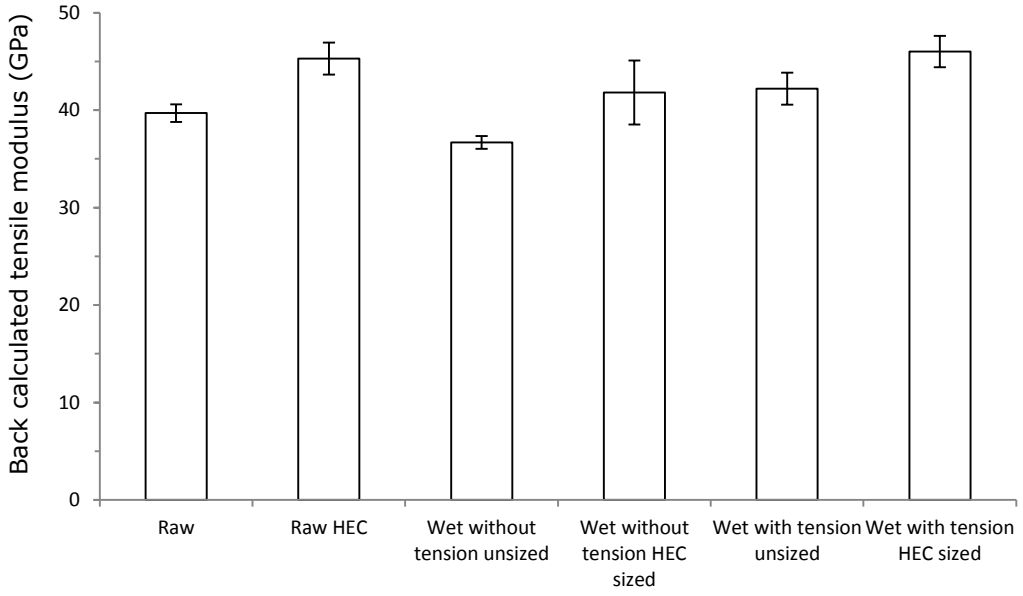


Fig. 4.29 Back calculated tensile modulus of fibres (error bars indicate standard deviation)

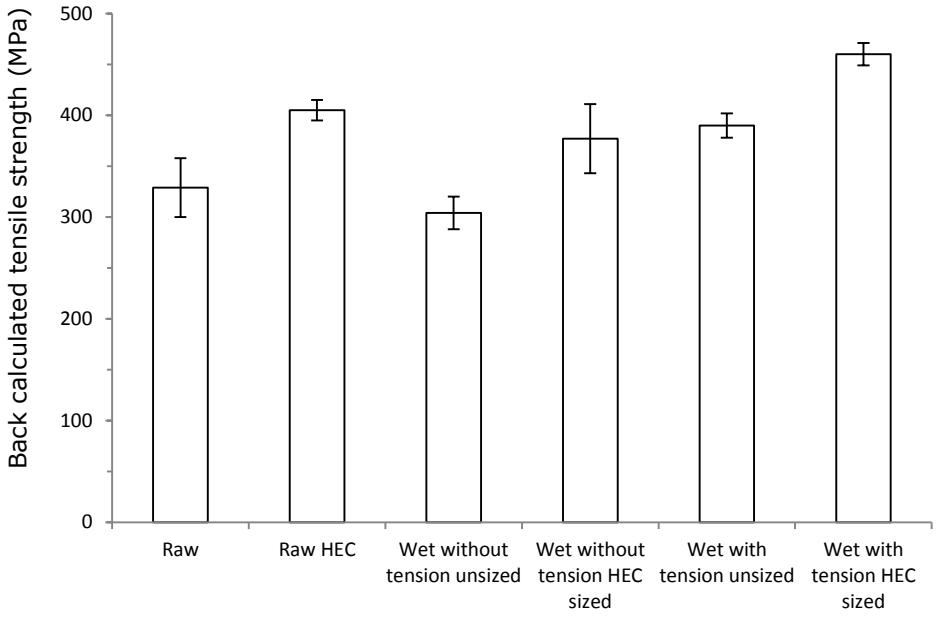


Fig. 4.30 Back calculated tensile strength of fibres (error bars indicate standard deviation)

Strain at failure of composites is presented in Table 4.6 and Fig. 4.31. Strain at failure of composites manufactured with unsized water treated yarn with and without tension are missing as strain at failure of these composite plaques were not noted due to constraints associated with the extensometer. The extensometer is removed from the sample soon after 1% strain of the composite samples. For the same reason force displacement curves for these two composite plaques are not presented. Strains at failure of the other four composite plaques are very similar revealing that water treatment and HEC sizing do not affect the strain at failure of composites. Strain at failure of these composite plaques agrees with that of found by Shah et al. [178].

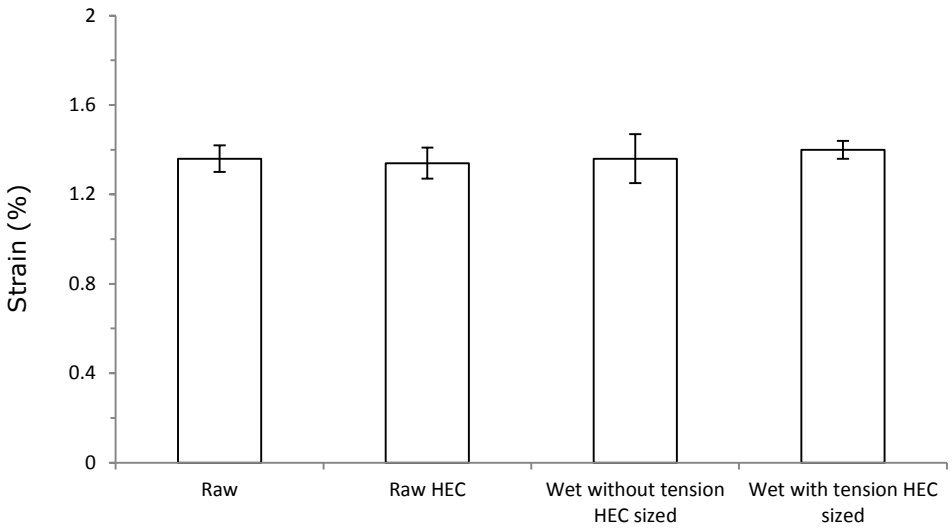


Fig. 4.31 Strain at failure of composites (error bars indicate standard deviation)

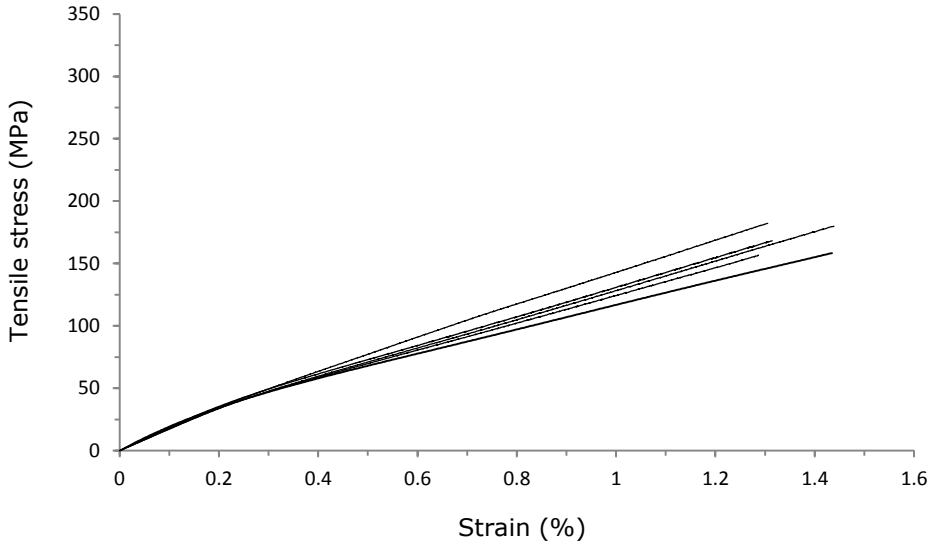


Fig. 4.32 Stress-strain curves of composites manufactured with unsized raw yarn

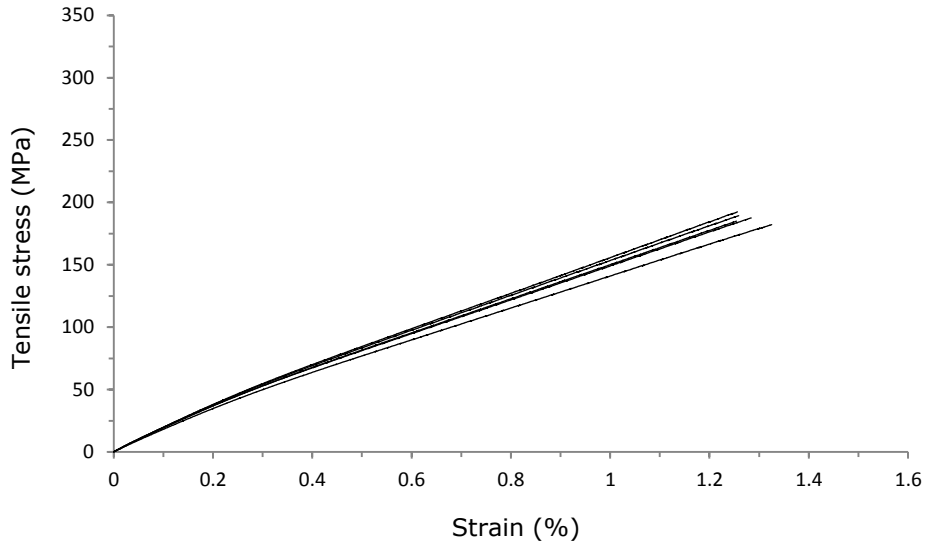


Fig. 4.33 Stress-strain curves of composites manufactured with HEC sized raw yarn

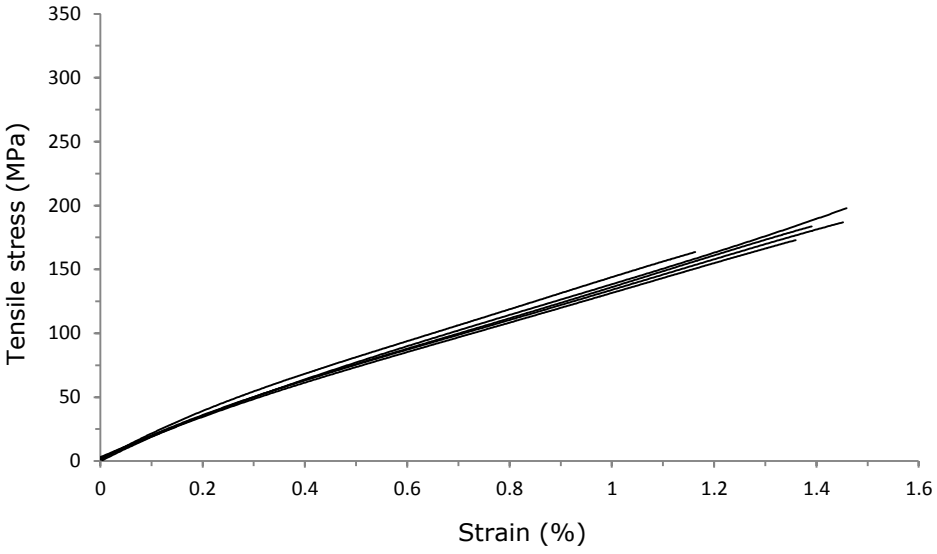


Fig. 4.34 Stress-strain curves of composites manufactured with water treated yarn without tension and HEC sized

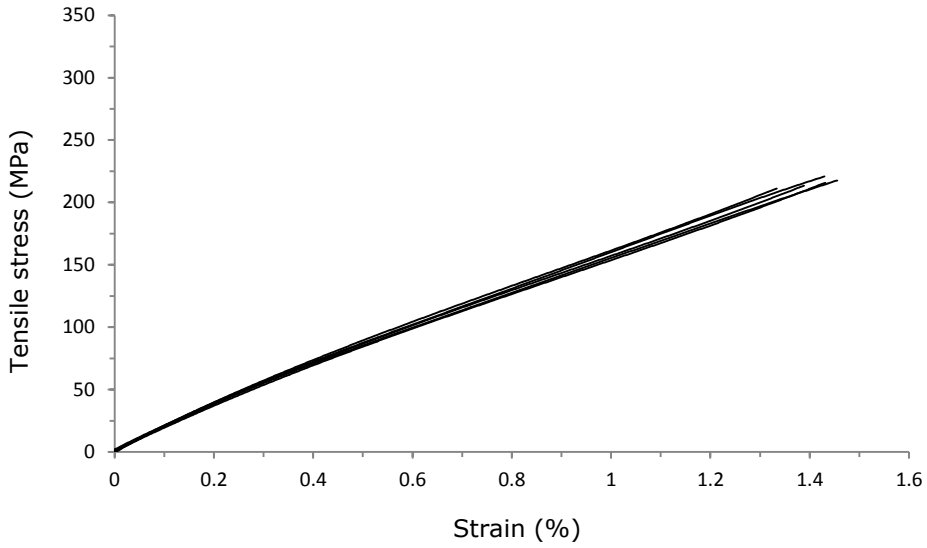


Fig. 4.35 Stress-strain curves of composites manufactured with water treated HEC sized yarn with tension

Characteristics of stress-strain curves (Fig. 4.32 to 4.35) of composites studied in this chapter are similar to those of studied in Chapter 3 and discussed in Section 3.5.6.(I) and same explanation is also apply here.

Comparing the tensile properties of fibre (Table 4.1), yarn (Table 4.3) and composites (Table 4.6) it can be concluded that tensile properties of composites are more related to the tensile properties of fibre than that of yarn. Yarns show very poor tensile modulus and tensile strength with respect to the properties of composites. Strain at failure of composites is also very close to the strain at failure of fibres. However the alignment of fibres in yarn affects the tensile properties of composites.

4.6 Conclusions

Single jute fibre extracted from sized yarn show significantly higher mean fibre diameter with respect to fibres extracted from unsized yarn in one case. However 1.5-1.8% size pick-up by yarn is not expected to result in an increase in diameter of fibres and these differences are due to inherent variability of plant fibres. Tensile modulus and tensile strength of jute fibres extracted from HEC sized yarns are low with respect to tensile modulus and tensile strength of jute fibres extracted from unsized yarns in all cases. This is due to difficulties faced to separate fibres from HEC sized yarn due to the adhesive character of HEC, where removing single fibres during separation damages fibres. All the properties of fibres studied in this chapter show a high value of standard deviation. The characteristic of force-displacement curves of treated and untreated fibres is very similar. After a small initial linear portion an upward curvature of force-displacement curves is observed for the majority of fibres tested, revealing that stiffness of jute fibres increases with increasing load, possibly caused by the straightening of micro-fibrils during axially applied load.

Tensile modulus and tenacity decrease for water treated yarn without tension and increase for water treated yarn with tension compared to those of raw yarn. This means that there is a need to be careful during water treatment of hydrophilic twisted yarn. However application of HEC increases the tensile modulus and tenacity of yarn with respect to that of unsized yarn which is a common character of sized yarn. After studying the influence of water treatment and HEC sizing on the tensile properties of jute yarn it can be concluded that both treatments are influential to increase or decrease the

tensile properties of yarn and the influence of water treatment is found to be more important than HEC sizing. These findings are important as generally chemical modifications of plant fibres are performed in aqueous solution and these results help to understand the influence of water on plant fibres composites. However water treatment does not influence the tensile properties of single fibres.

From Table 4.5 it can be observed that approximately 20% of tensile modulus and 45% of tensile strength of fibres are utilised in yarn. Major constraints of utilising tensile properties of constituent fibres in yarn are (i) frictional forces among fibres that develop due to twist and provides tensile properties of yarn, which restricts proper utilisation of fibre properties in yarn (ii) helical configuration of fibres in the yarn contributes to off-axis loading on fibres which reduces the properties of yarn and (iii) bending of yarn due to twist cause micro stretching of outer surface of fibres and micro contracting of inner surface of fibres that may introduce micro cracking of fibres.

Despite the lack of effect of HEC size on yarn properties, composites made with HEC sized yarns show significantly higher tensile modulus and tensile strength in all cases with respect to composites made with similar type of unsized yarn. This reveals that HEC size influences the tensile properties of composites positively. Possibly surface active –OH groups of HEC create hydrogen bonds between fibre and matrix that enhance the tensile properties of composites. Composites made with water treated yarn without tension show lower tensile modulus and lower tensile strength and composites made with water treated yarn with tension show higher tensile modulus and higher tensile strength with respect to composites made with raw yarn. Dipping yarn

in a loose condition causes tangling of yarns which increases misalignment of fibres. Swelling and shrinkage are also responsible for fibre misalignment. Misalignment of fibres reduces the tensile properties of composites manufactured with water treated yarn without tension. However yarn entangling or yarn shrinkage is not possible for water treated yarn with tension. Moreover the tendency shrinkage under tension can improve the fibre alignment which improves the tensile properties of composites manufactured with water treated yarn with tension. This behaviour is very important for water based chemical treatment of plant fibre yarn for composite manufacturing. Yarn properties and composite properties are clearly affected due to wetting of yarn with and without tension. HEC sizing and water treatment do not affect the strain at failure of composites.

A comparative study of fibre, yarn and composites suggest that the tensile properties of composites are more influenced by the tensile properties of fibres than that of yarn. However fibre alignment in yarns plays very important role in influencing by the tensile properties of composites.

CHAPTER 5

INFLUENCE OF ALKALI TREATMENT OF JUTE FIBRE WITH AND WITHOUT TENSION ON THE TENSILE PROPERTIES OF UD COMPOSITES

5.1 Summary

In this chapter the influence of alkali treatment on the tensile properties of jute fibre as well as jute composites are studied. Alkali treatment is performed with and without applying tension on jute yarn. Tensile properties of treated fibre as well as composites are measured to understand the influence of alkali treatment on these substrates.

5.2 Introduction

Cellulose is the chief element of jute fibre (Table 2.3) and it has very high mechanical properties (Section 2.4.1). However low tensile properties of jute fibres (with respect to cellulose) e.g. 7.2 [46] – 38.9 [134] GPa tensile modulus and 187 [46] – 800 [297] MPa tensile strength were observed by many researchers. Various natural defects [289, 309], processing faults and the chemical composition play a major role to reduce the tensile properties. For example hemicellulose, the second major component of jute fibre (see Table 2.3) possesses 5-8 GPa tensile modulus and the third important component lignin has 2.5-3.7 GPa tensile modulus and 25-75 MPa tensile strength [310] which are much lower than the properties of cellulose. Hence, the removal of hemicellulose and lignin improves the tensile properties of cellulose fibres [68]. Thus the intention of this chapter is to remove lignin and hemicellulose to understand the effect on the properties of jute fibres as well as the properties of the resulting composites. Hemicellulose is hydrophilic in

nature [64] and hydrophilic materials create poor bonding with hydrophobic matrices [311]. Thus removal of hemicellulose is studied in this chapter. Alkali treatment is a well-established technique to remove hemicellulose, lignin, pectin, wax and other oily substances of plant fibres [48, 131, 312]. As alkali treatment is performed in an aqueous medium and treatment of jute yarn in water with and without tension significantly affects the tensile properties of yarn as well as composites (Section 4.5.2 and Section 4.5.3), hence alkali treatment is performed on jute yarn with and without tension to understand the influences of these treatment on the mechanical properties of composites. Similarly to Chapter 3 and Chapter 4 HEC is used to form the UD mat from untreated and treated yarns.

Section 5.3 contains a description of the material, alkali treatment performed and methods of composites manufacturing, Section 5.4 describes the mechanical tests performed on fibres and composites. Results are discussed in Section 5.5 and Section 5.6 draws some conclusions.

5.3 Materials and Methods

5.3.1 Materials

Information regarding fibre, matrix, catalyst, accelerator and mould release is provided in Section 3.3.1. Laboratory grade NaOH ($\geqslant$ 97%) from Sigma-Aldrich, pellet form is used for alkali treatment. $\geqslant$ 99.7% acetic acid from Sigma-Aldrich is used to neutralise the alkali solution after treatment.

5.3.2 Preparation of yarn for alkali treatment, sizing and composites

Yarn preparation for alkali treatment is similar to yarn preparation for water treatment without tension as discussed in Section 4.3.2. Alkali treatment of

jute yarn with tension is performed by wrapping it on a glass fibre composites board (similar to Fig. 4.1) in a single ply. A single ply of yarn is preferred to ease the reaction of alkali.

5.3.3 Alkali treatment

1%, 3% and 5% NaOH solution (by weight) is made with tap water. Different yarn samples are dipped in these solutions separately at loose condition (yarn at coil form without any support Fig.4.1(A)) and using tension (yarn wrapped on glass fibre composite board Fig.4.1(B)) for 20 minutes at room temperature. Yarns are then neutralised by adding acetic acid, washed several times, squeezed gently (for loose fibre) and dried in an oven at $105^{O}C$ [261] for one hour keeping the extraction fan on. Fibres are then conditioned for 48 hours at room temperature before further processing.

5.3.4 HEC sizing and manufacturing of composites

Alkali treated yarn with and without tension are sized with HEC to produce a UD fibrous mat. The preparation of HEC for sizing and the procedure of sizing are discussed in Section 3.3.2. Composite plaques are manufactured using RTM. The manufacturing technique is explained in Section 3.3.4.

5.3.5 Preparation of materials for testing

Single fibre separation and preparation for testing are discussed in Section 4.3.4. Measuring fibre diameter at several places and taking the mean as the fibre diameter is an established method and practised in this work except in this chapter. Here the thinnest width of the fibre is taken as the fibre diameter and the cross-section is considered circular. The thinnest diameter is

considered to compare the results with mean diameter of fibres as well as composites. These comparisons are performed in Chapter 7.

5.4 Testing

Single jute fibre extracted from raw yarn, 1% alkali treated yarn (with and without tension) and 5% alkali treated yarn (with and without tension) are used for tensile testing. The testing procedure is discussed in Section 4.4.1. Composites manufactured with HEC sized raw yarns, 1% alkali treated yarn (with and without tension), 3% alkali treated yarn (with and without tension), 5% alkali treated yarn (with and without tension) are tested for tensile properties as described in Section 3.4.6. A non-contact video gauge (software: Imetrum) extensometer (Fig. 4.5) is used to measure tensile modulus of the composite plaques.

5.5 Results and Discussion

5.5.1 Fibre test results

Fibre diameter studied in this chapter (Table 5.1 and Fig 5.1) do not show any significant differences ($P<0.05$). Tensile moduli of raw and alkali treated jute fibres do not show any specific trend or relation and no significant ($P<0.05$) differences (Table 5.1 and Fig. 5.2, raw data are presented in Appendix C) are observed. Large values of standard deviations are observed here due to the inherently variable properties of jute fibre.

Table 5.1 Tensile properties of fibres

Type of fibre	Fibre diameter (μm)	Standard deviation	Tensile modulus (GPa)	Standard deviation	Tensile strength (MPa)	Standard deviation	Strain at failure (%)	Standard deviation
Raw	32	8	45.4	16.3	1108	403	2.03	0.46
1% NaOH no tension no HEC	35	8	41.6	17.8	1050	442	2.09	0.52
1% NaOH with tension no HEC	33	9	44.6	14.8	973	445	1.92	0.62
5% NaOH no tension no HEC	32	9	43.5	16.2	932	378	1.95	0.52
5% NaOH with tension no HEC	29	7	46.2	20.1	953	406	2.29	0.68

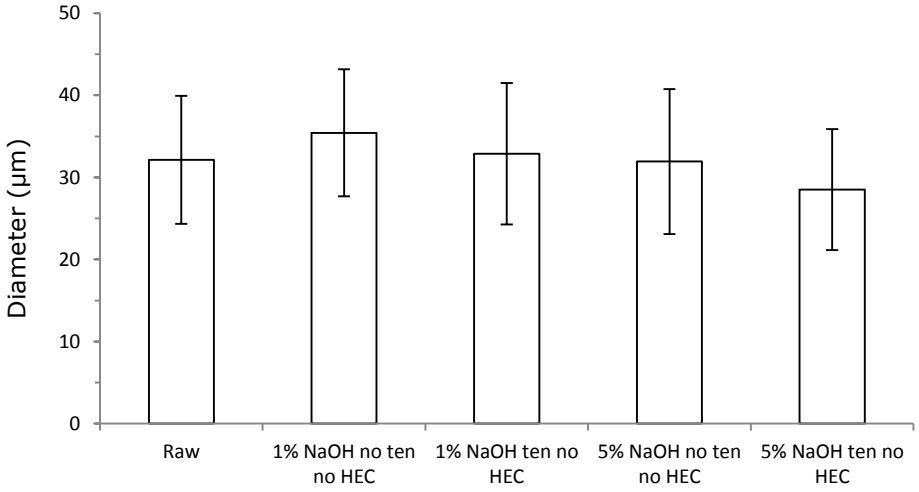


Fig. 5.1: Diameter of fibres (error bars indicate standard deviation)

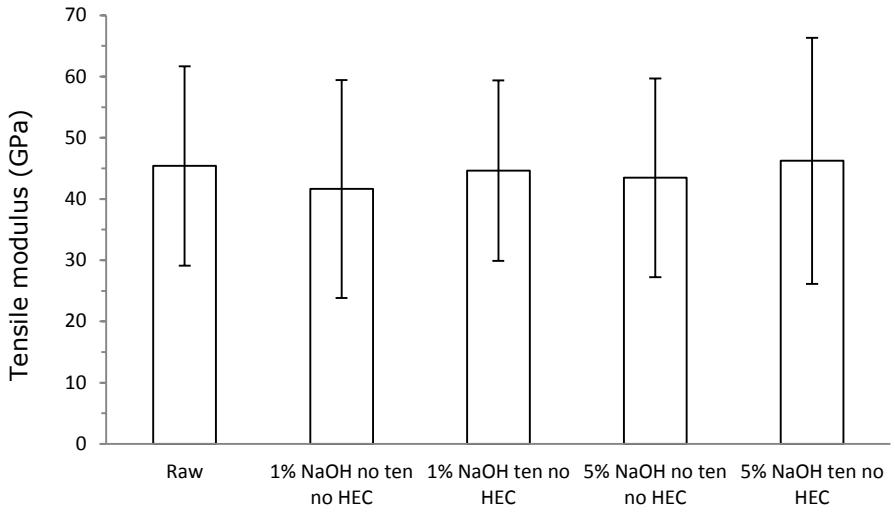


Fig. 5.2: Tensile moduli of fibres (error bars indicate standard deviation)

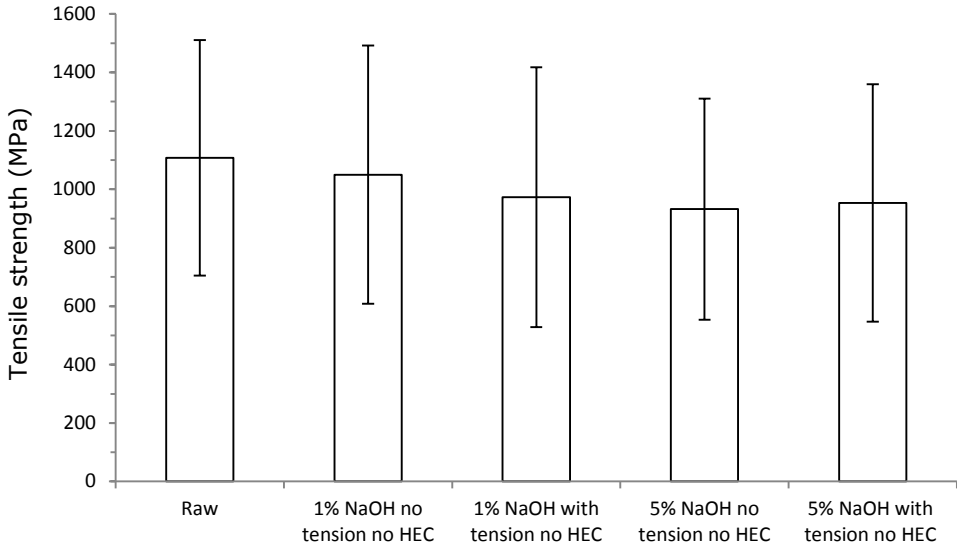


Fig. 5.3: Tensile strength of fibres (error bars indicate standard deviation)

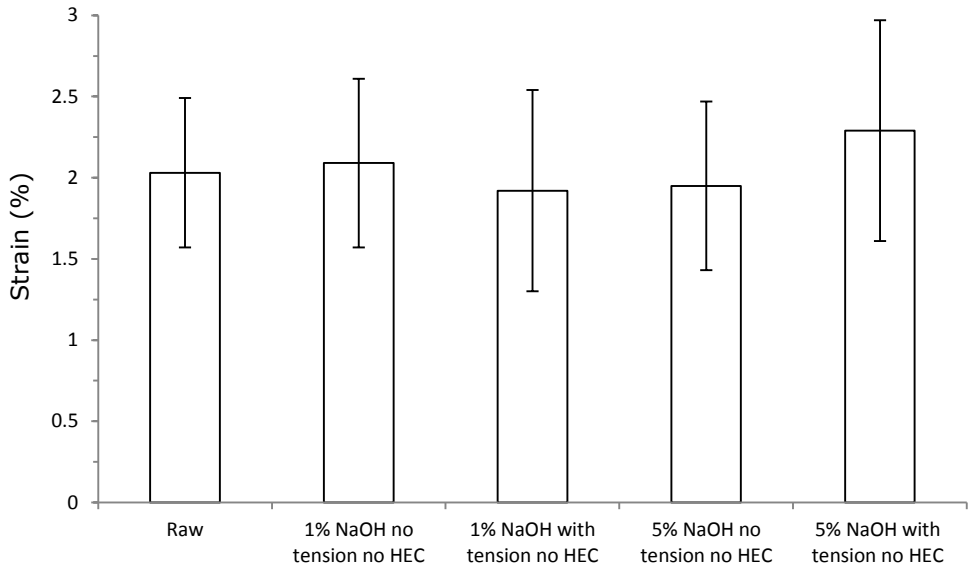


Fig. 5.4: Strain at failure of fibres (error bars indicate standard deviation)

Tensile strength of raw and alkali treated fibres present no significant ($P<0.05$) differences (Table 5.1 and Fig. 5.3, raw data are presented in Appendix C). Scattered results are observed for strain at failure of single jute fibres (Table 5.1 and Fig. 5.4) and the variations are not statistically significant ($P<0.05$). Fibre test results indicate that the chemical treatment as well as the treatment conditions studied in this chapter do not affect the diameter and tensile properties of jute fibre.

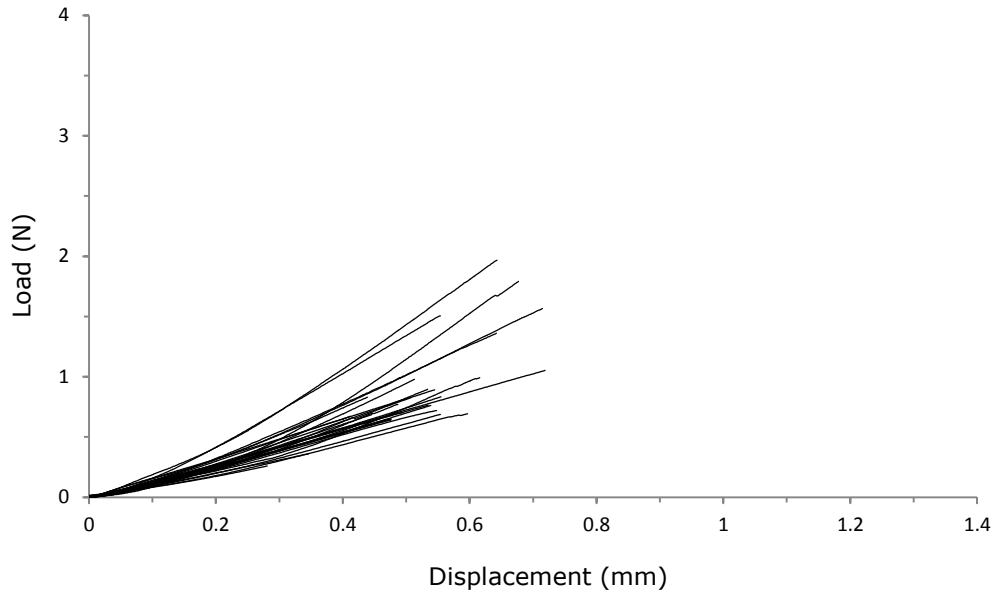


Fig. 5.5 Force displacement curves of single jute fibres extracted from unsized raw yarn

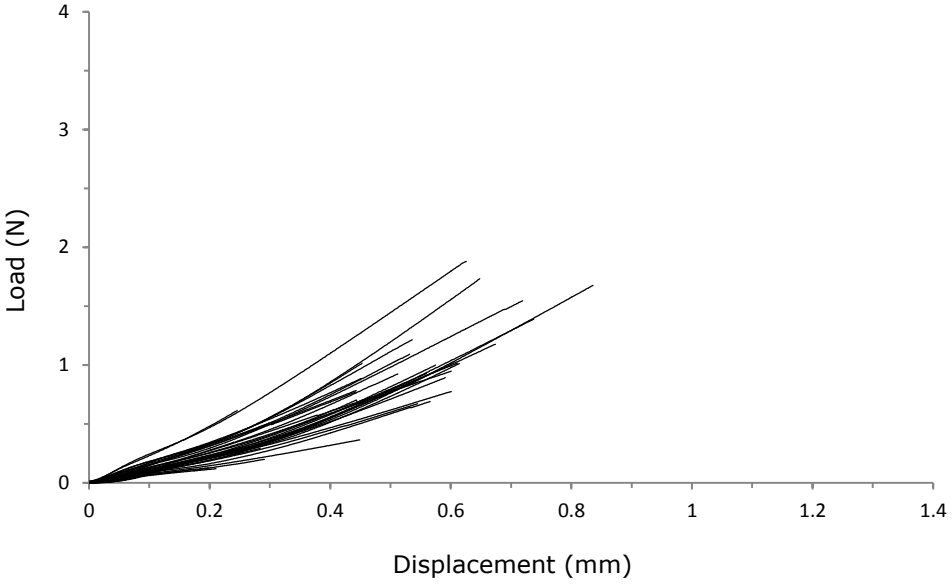


Fig. 5.6 Force displacement curves of single jute fibres extracted from unsized and 1% NaOH treated yarn without tension

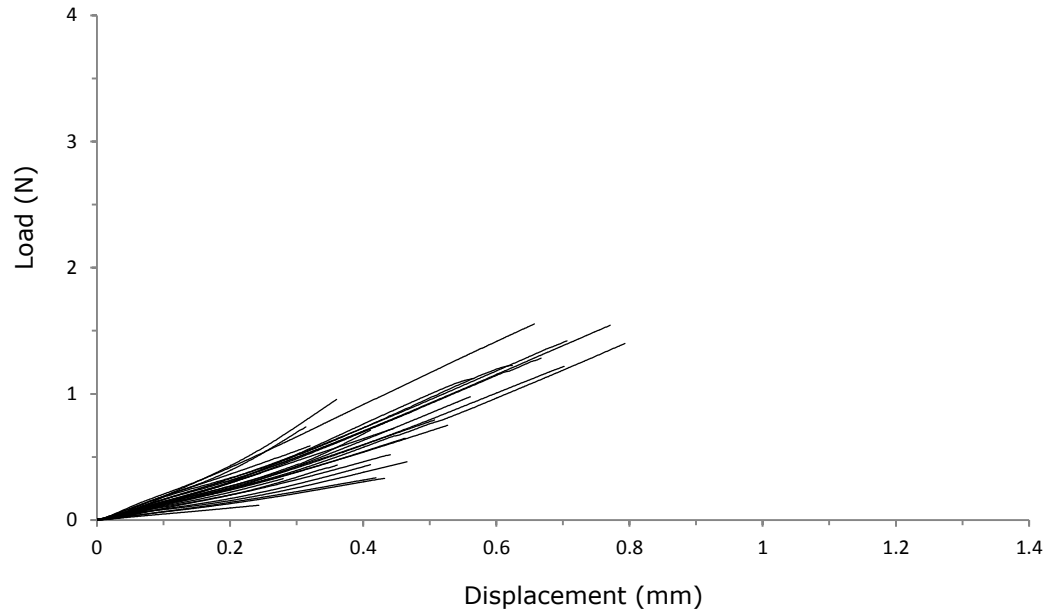


Fig. 5.7 Force displacement curves of single jute fibres extracted from unsized and 1% NaOH treated yarn with tension

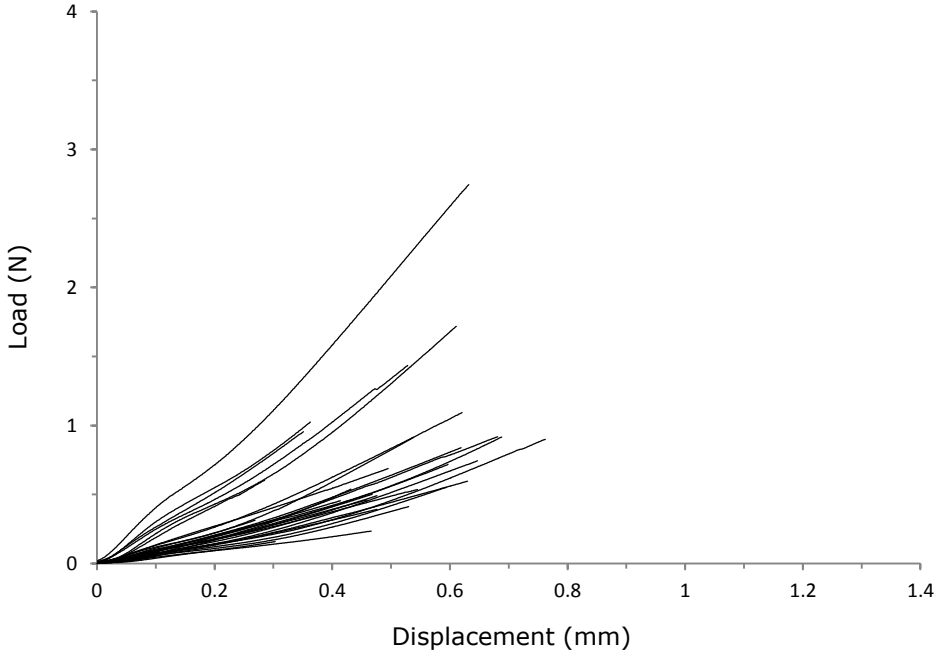


Fig. 5.8 Force displacement curves of single jute fibres extracted from unsized and 5% NaOH treated yarn without tension

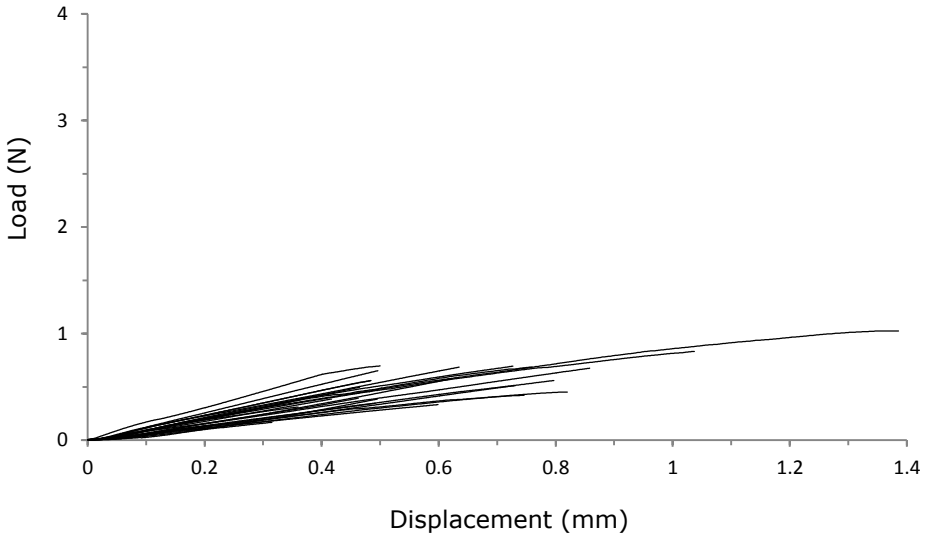


Fig. 5.9 Force displacement curves of single jute fibres extracted from unsized and 5% NaOH treated yarn with tension

Force-displacement curves of single jute fibres are presented in Fig. 5.5 to Fig. 5.9. The majority of force-displacement curves of fibres show an upward curvature after a small initial linear portion similar to Chapter 4 and possible causes are discussed in Section 4.5.1. However the characteristics of single fibres extracted from 5% NaOH treated yarn with tension showed a quasi-linear froce-displacement curve and the reason is unknown.

5.5.2 Composites test results

The influence of alkali treatment with and without tension on the tensile properties of jute composites is presented in Table 5.2, Fig. 5.10 (tensile modulus, raw data are presented in Appendix C), Fig. 5.12 (tensile strength, raw data are presented in Appendix C) and Fig. 5.14 (strain at failure, raw data are presented in Appendix C). The composite plaques possess large variation in fibre volume fraction (minimum fibre volume fraction, V_f=35% and maximum V_f=46%) due to a change of mould. The old mould was made

of aluminium and it became thinner due to polishing. The new mould is made of steel which is thicker than aluminium mould and using the same number of UD yarn plies (i.e. four) a higher fibre volume fraction of composites is found. Thus back calculated tensile modulus and tensile strength of fibres are determined to compare the results. The calculation method is explained in Section 3.5.6.(I) and the data are presented in Table 5.3, Fig. 5.11 (tensile modulus) and Fig. 5.13 (tensile strength).

Table. 5.2 Fibre volume fraction and tensile properties of composites

Name of the composite plaque	Fibre volume fraction (%)	Tensile modulus (GPa)	Standard deviation	Tensile strength (MPa)	Standard deviation	Strain at failure (%)	Standard deviation
Raw yarn	35	18.3	0.6	187	4	1.34	0.07
1% NaOH no tension	36	18.1	0.8	160	13	1.30	0.06
1% NaOH with tension	36	19.9	1.1	196	8	1.34	0.03
3% NaOH no tension	37	19.0	0.8	167	5	1.29	0.08
3% NaOH with tension	38	20.3	1.0	207	10	1.33	0.06
5% NaOH no tension	46	22.7	1.3	214	7	1.44	0.08
5% NaOH with tension	43	23.1	1.6	242	11	1.36	0.07

Table. 5.3 Back calculated values of tensile properties of fibres

Name of the composite plaque	Tensile modulus (GPa)	Standard deviation	Tensile strength (MPa)	Standard deviation
Raw yarn	45.3	1.6	405	10
1% NaOH no tension	43.5	2.3	319	36
1% NaOH with tension	48.1	3.0	420	24
3% NaOH no tension	45.5	2.2	331	14
3% NaOH with tension	47.3	2.6	429	26
5% NaOH no tension	44.8	2.8	383	15
5% NaOH with tension	48.5	3.7	469	28

Alkali treatments on jute yarn as studied in this chapter (the concentration of alkali solution as well as the treatment condition) do not show any significant change of back calculated tensile modulus (Table 5.3 and Fig. 5.11) of fibres and these are similar to the back calculated tensile modulus of fibres manufactured with raw yarn. This means that the alkali treatment studied (the concentration of alkali solution as well as the treatment condition) has no effect on the tensile modulus of composites. These results are comparable to those for tensile modulus of fibres as alkali treatment does not affect the tensile modulus of fibres. Composites manufactured with HEC sized raw yarn, HEC sized water treated yarn with and without tension studied in Chapter 4, (Section 4.5.3) also do not show any significant change in back calculated tensile modulus of fibres. Back calculated tensile modulus of fibres of composites manufactured with water treated yarn with tension as well as HEC sized (Chapter 4, Section 4.5.3) is similar to that of composites manufactured

with alkali treated yarn with tension and back calculated tensile modulus of fibres of composites manufactured with water treated yarn without tension and HEC sized (Chapter 4, Section 4.5.3) is similar to that of composites manufactured with alkali treated yarn without tension.

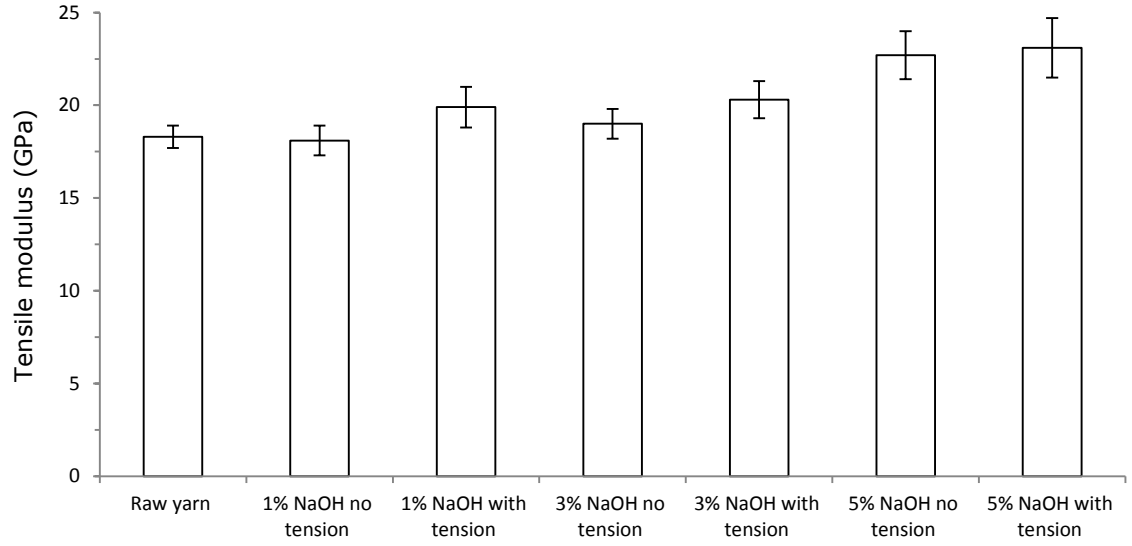


Fig. 5.10 Tensile modulus of composites (error bars indicate standard deviation)

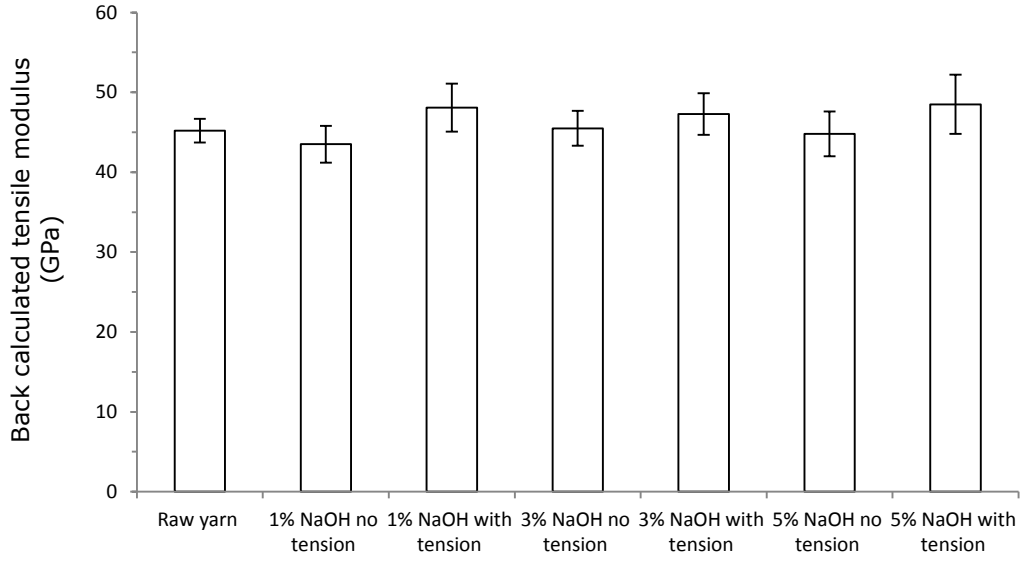


Fig. 5.11 Back calculated tensile modulus of fibres (error bars indicate standard deviation)

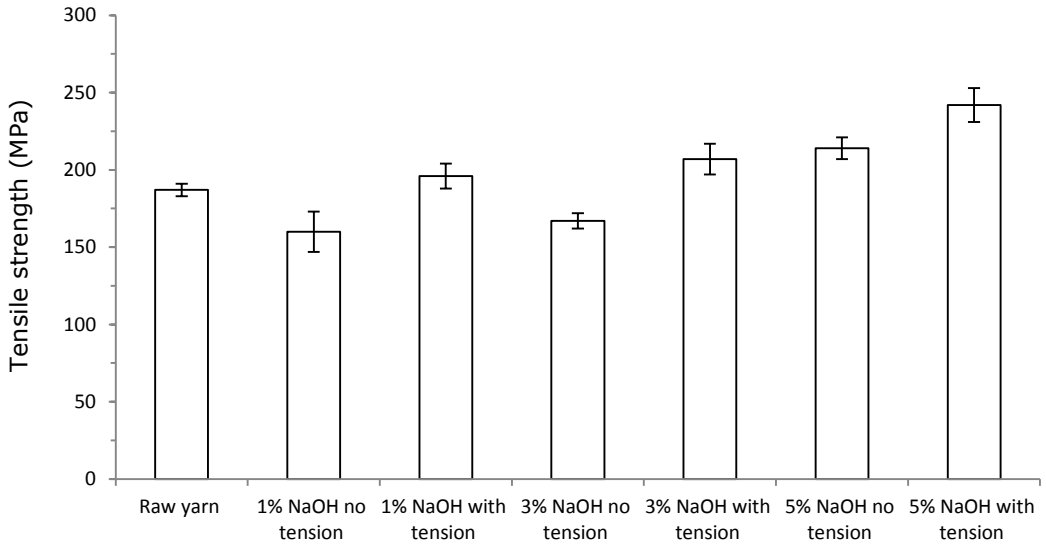


Fig. 5.12 Tensile strength of composites (error bars indicate standard deviation)

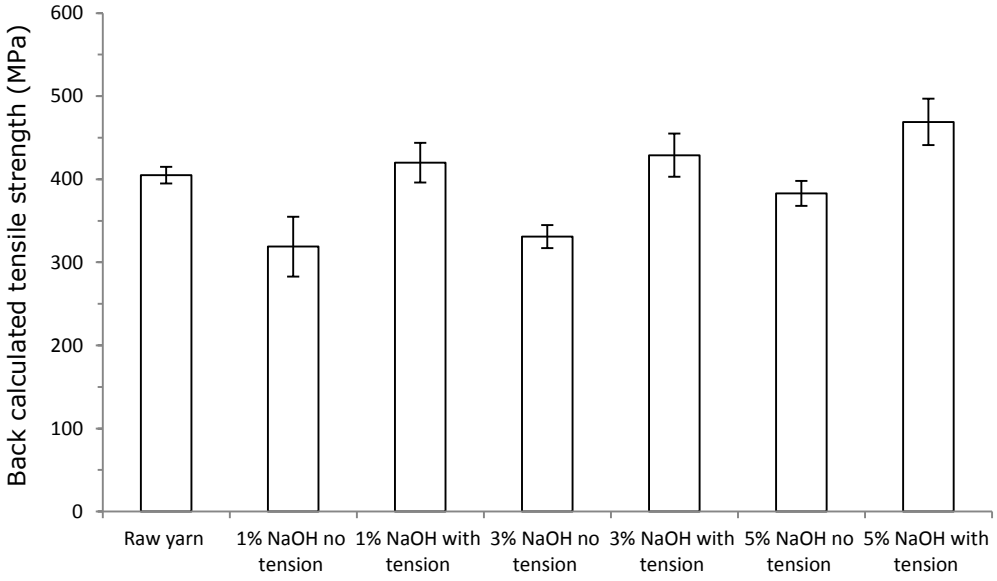


Fig. 5.13 Back calculated tensile strength of fibres (error bars indicate standard deviation)

Composites manufactured with 1% alkali treated yarn with tension, 3% alkali treated yarn with tension and 5% alkali treated yarn with tension show 32%,

30% and 22% increases in back calculated tensile strength respectively (Table 5.3 and Fig. 5.13) with respect to that of composites manufactured with 1% alkali treated yarn without tension, 3% alkali treated yarn without tension and 5% alkali treated yarn without tension. All of these differences are statistically significant ($P<0.05$) revealing that yarn treatment in aqueous solution with and without tension can significantly influence the tensile strength of composites and the causes mentioned in Section 4.5.3 possibly also apply here. Similar trend is also found in Chapter 4 (Section 4.5.3) i.e. composites manufactured with water treated yarn with tension show significantly higher ($P<0.05$) back calculated tensile strength of fibres than that of water treated yarn without tension. Back calculated tensile strength of fibres of composites manufactured with water treated yarn with tension as well as HEC sized (Chapter 4, Section 4.5.3) is similar to that of composites manufactured with alkali (1%, 3% and 5%) treated yarn with tension and back calculated tensile strength of fibres of composites manufactured with water treated yarn without tension as well as HEC sized (Chapter 4, Section 4.5.3) is similar to that of composites manufactured with 5% alkali treated yarn without tension and significantly higher ($P<0.05$) than that of composites manufactured with 1% and 3% alkali treated yarn without tension.

Minor differences in back calculated fibre tensile strength are observed for composites manufactured with 1% NaOH treated yarn without tension and composites manufactured with 3% NaOH treated yarn without tension. Tensile strengths of these two composite plaques are significantly lower ($P<0.05$) than those of composites manufactured with raw yarn. This trend matches with Chapter 4 (Section 4.5.3) where the back calculated tensile strength of fibre for composites manufactured with water treated yarn without tension is

lower than that of composites manufactured with raw yarn. However the difference is not statistically significant ($P<0.05$). No significant differences ($P<0.05$) of back calculated fibre tensile strength are found for composites manufactured with 1% NaOH treated yarn with tension and composites manufactured with 3% NaOH treated yarn with tension and tensile strengths of these two composite plaques are very similar to those of composites manufactured with raw yarn. Possibly lower concentrated (1% and 3%) alkali solutions are not able to influence the tensile strength of composites and the lower tensile strength of composites manufactured with 1% and 3% NaOH treated yarn without tension with respect to composites manufactured with raw yarn is due to water treatment of yarn at loose condition as discussed in Section 4.5.3. However a significant improvement ($P<0.05$) of back calculated fibre tensile strength is found for composites manufactured with 5% alkali treated yarn without tension with respect to composites manufactured with 1% alkali treated yarn without tension (20%). Again composites manufactured with 5% alkali treated yarn with tension show a significant increase ($P<0.05$) in back calculated fibre tensile strength with respect to that of composites manufactured with 1% alkali treated yarn with tension (12%). However the differences between composites made with raw yarn and composites made with 5% alkali treated yarn without tension is not statistically significant ($P<0.05$). Composites made with 5% alkali treated yarn with tension shows 16% higher back calculated fibre tensile strength with respect to that property of composites manufactured with raw yarn which is statistically significant ($P<0.05$).

After comparing the back calculated tensile strength of fibres, composites made with 5% alkali treated with tension show a statistically significant

increase with respect to that of composites manufactured with raw yarn. Other composite plaques made with treated yarn show either inferior or similar values of back calculated tensile strength with respect to composites manufactured with raw yarn. Thus no clear effect of alkali treatment on the back calculated tensile strength of fibre is noticed in this chapter and it is not obvious that an increase in back calculated tensile strength (only observed for one case with respect to composites manufactured with raw yarn) is due to NaOH treatment. It may be due to treatment of yarn in the aqueous medium under tension or some other unidentified cause. Back calculated tensile strength of fibres is very low with respect to tensile strength of fibres obtained from fibre tests and the comparison is discussed in Chapter 7.

Composites manufactured with raw yarn as well as alkali treated yarns with or without tension do not show a marked change in strain at failure (Table 5.3 and Fig. 5.14) revealing that the treatments performed in this chapter do not affect the strain at failure of composites. Water treatment of yarn also do not influence the strain at failure of composites studied in Chapter 4 (Section 4.5.3). Almost similar values of standard deviations are observed for all composite plaques tested here and similar to that of composite plaques tested in Chapter 4 (Section 4.5.3).

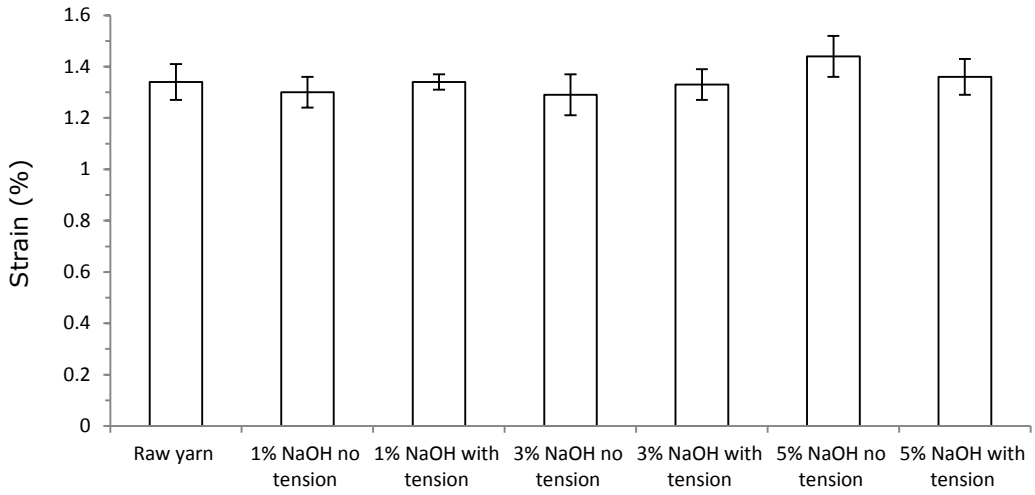


Fig. 5.14 Strain at failure of composites (error bars indicate standard deviation)

No published work is found on the tensile properties of alkali treated UD jute polyester composites to compare the results of this chapter.

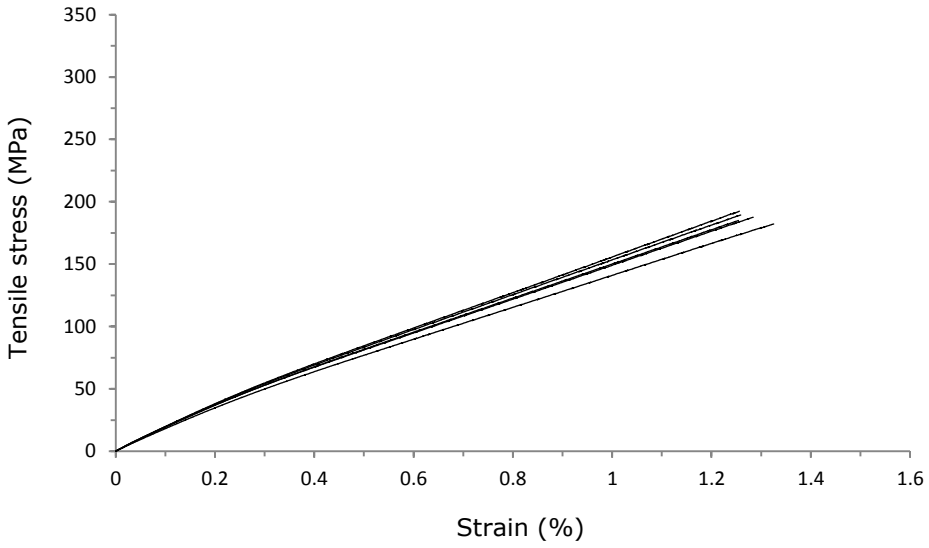


Fig. 5.15 Stress-strain curves of composites manufactured with HEC sized raw yarn

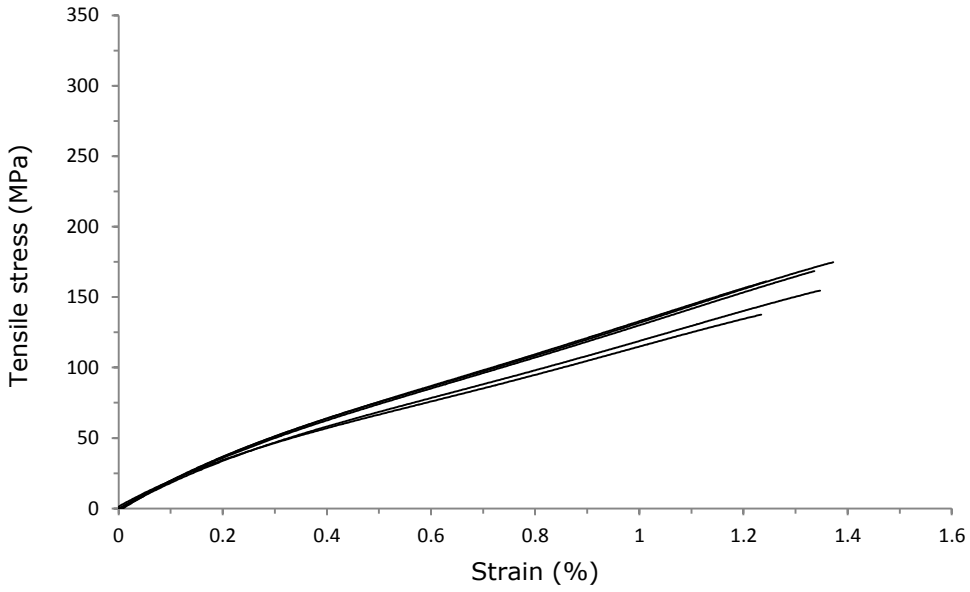


Fig. 5.16 Stress-strain curves of composites manufactured with HEC sized 1% NaOH treated yarn without tension

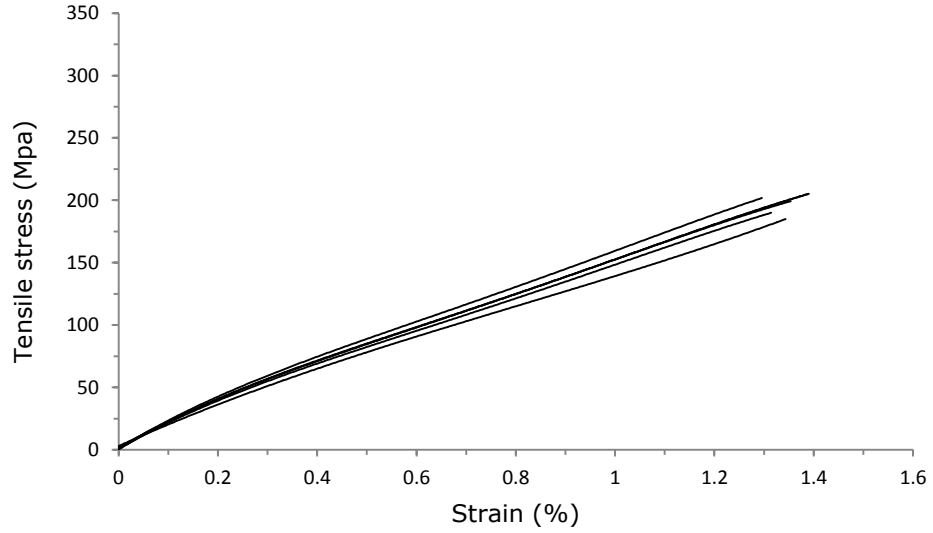


Fig.5.17 Stress-strain curves of composites manufactured with HEC sized 1% NaOH treated yarn with tension

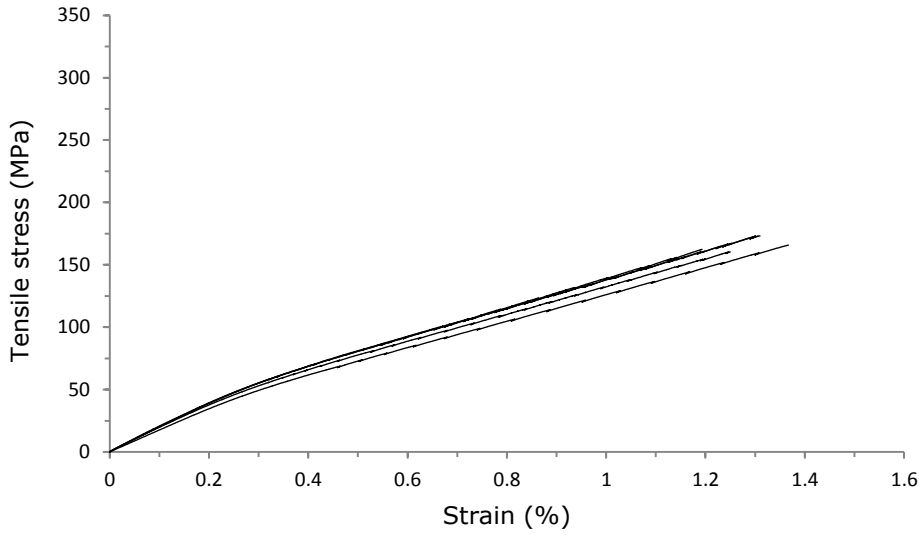


Fig. 5.18 Stress-strain curves of composites manufactured with HEC sized 3% NaOH treated yarn without tension

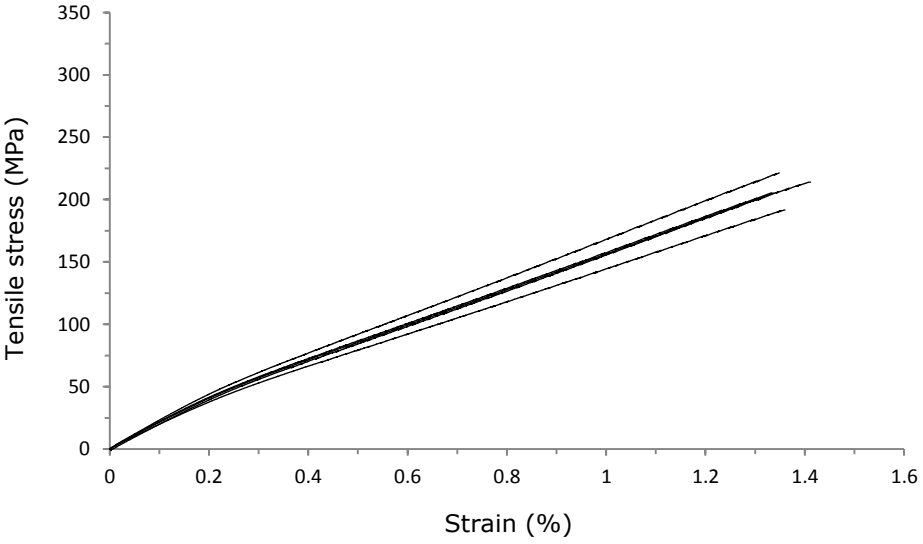


Fig. 5.19 Stress-strain curves of composites manufactured with HEC sized 3% NaOH treated yarn with tension

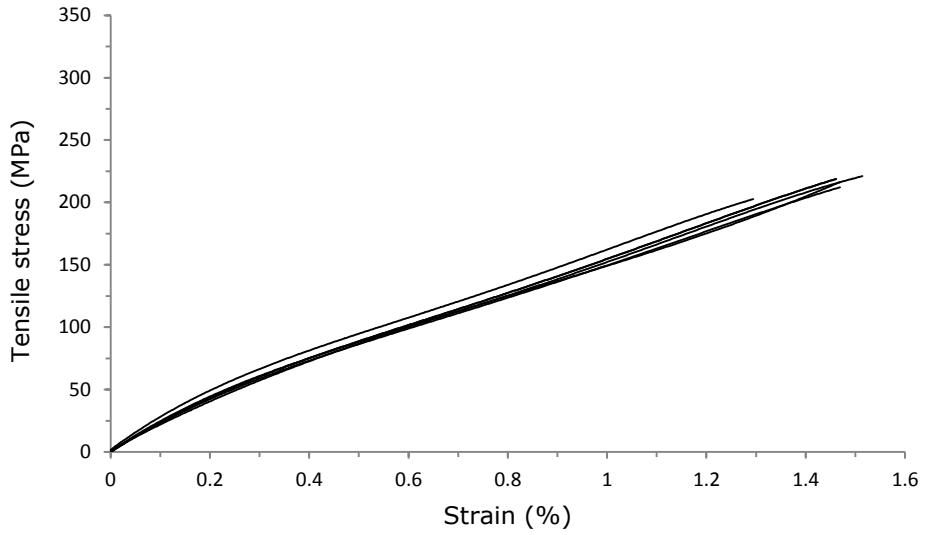


Fig. 5.20 Stress-strain curves of composites manufactured with HEC sized 5% NaOH treated yarn without tension

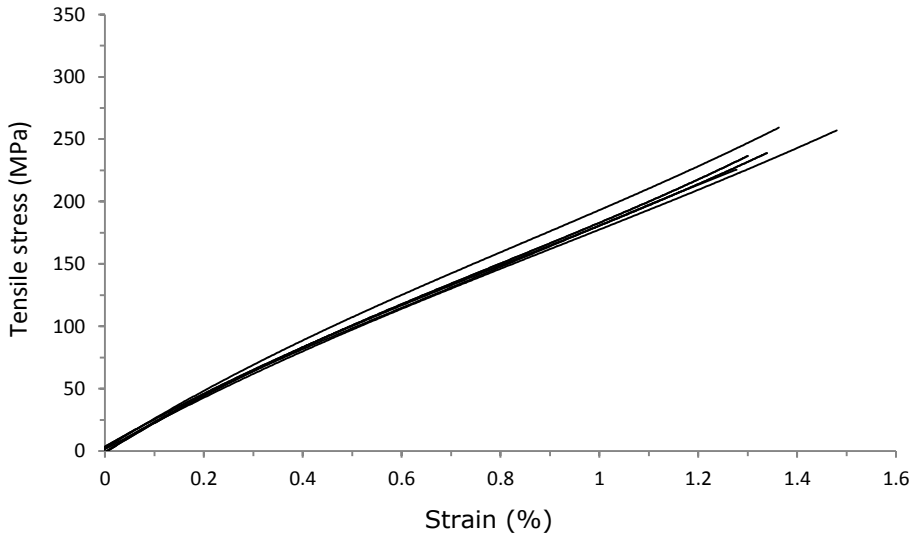


Fig. 5.21 Stress-strain curves of composites manufactured with HEC sized 5% NaOH treated yarn with tension

The stress-strain curves of composites discussed in this chapter are presented in Fig. 5.15 to Fig. 5.21. The characteristics of the curves are very similar to

the curves obtained in Chapter 3 (longitudinal direction of yarn) and Chapter 4 (Section 4.5.3) and the arguments in Section 3.5.6.(I) are also valid here.

5.6 Conclusions

Diameter of single fibres does not change due to alkali treatments performed in this chapter. The alkali treatment of jute fibre studied here does not affect the tensile properties irrespective of the alkali concentration as well as the condition of the treatment. Similarly to Chapter 4, force-displacement curves of single jute fibres show an upward bending after initial linear portion revealing that with increasing load the stiffness of fibres increases and the alkali treatment does not affect the behaviour of the curves. However force-displacement curves of fibres treated in 5% NaOH solution behave differently and the cause is unknown.

Composites manufactured with yarns treated in alkali solution of different concentration with and without tension do not influence the back calculated fibre tensile modulus. However tension during alkali treatment is found to be influential on back calculated tensile strength of fibres. Back calculated fibre tensile strength increases for composites made with alkali treated yarn with tension with respect to that of composites made with a similar type of alkali treated yarn without tension for each case and this effect is possibly due to the treatment of yarn in aqueous solution with and without tension as discussed in Section 4.5.3. Composites manufactured with 5% alkali treated yarn with tension show significantly higher back calculated fibre tensile strength with respect to composites manufactured with raw yarn. No other composites manufactured with treated yarn show significant improvement of back calculated fibre tensile strength with respect to composites

manufactured with raw yarn. Thus it can be concluded that the alkali treatment studied in this chapter has no effect on the back calculated tensile properties of fibres and the improvement of this property for one case is probably due to water treatment with tension. No significant variation is observed for strain at failure of composites due to alkali treatment.

The stress-strain curves of composite plaques examined in this chapter show similar behaviour and are also similar to the curves studied in earlier chapters. Thus it can be said that the alkali treatments performed here have no demonstrable effect on the stress-strain behaviour of composites.

CHAPTER 6

INFLUENCE OF CELLULOSE NANO-PARTICLE (CNP) TREATMENT ON THE MECHANICAL PROPERTIES OF JUTE COMPOSITES

6.1 Summary

The influence of cellulose nano-particles (CNP) on the mechanical properties of composites is studied in this chapter. In this work CNP are extracted from cotton which are actually cellulose crystals of long cellulose chains. The amorphous portions of cotton cellulose chains have been dissolved by acid hydrolysis and the crystals are extracted. After going through a series of processes, dried CNP as well as CNP in suspension are collected to apply on jute yarn as a surface treatment material. CNP are also mixed with resin before infusion to understand the effect on the mechanical properties of composites. UD composites are made with CNP sized yarns as well as CNP mixed with resin before infusion and tested for tensile properties to understand the effect of CNP on UD jute composites.

6.2 Introduction

CNP are rod shape structures, have high aspect ratio (3-5 nm width and 50-500 nm length), are highly crystalline and 100% cellulose [313]. It is a biodegradable natural polymer obtained from trees or plants and often called cellulose nano-particle (CNP). It is also called cellulose nano-whisker (CNW) or cellulose nano-crystal (CNC). Generally CNP are manufactured using an acid hydrolysis technique [314, 315] and in fewer cases mechanical process such as ball milling [122, 316]. Nano-fibres are defined to have at least one dimension (generally diameter) equal to or less than 100 nm [317, 318].

Nano-fibres extracted from different natural resources have drawn much interest from composites researchers due to their high mechanical properties. Elimination of weaker portions such as amorphous regions and defects as well as the reduction in size improves the mechanical properties of CNP. Better reinforcement properties come from improved mechanical properties and higher surface area with respect to volume [319]. CNP has hydroxyl reactive groups (-OH) on their surface that facilitate self-assembly, controlled dispersion within a various matrix and influences particle-particle and particle-matrix bond strength [313].

Bio-based nano-composites have shown their potential uses in food packaging, biomedical and tissue engineering [320-322] due to better thermal and mechanical properties. Strong interactions between the hydroxyl-rich surface of the CNP and the ester linkages of the matrix are expected [125] to build a strong interface, with a positive impact on the mechanical properties of composite materials. Thus application of CNP is expected to influence the mechanical properties of composites and is used in this work as a surface coating material of jute yarn as well as being mixed in resin prior to infusion. However since the sole element of CNP is cellulose it creates hydrogen bonds among the CNP and agglomerations occur that can reduce the properties of composites. Thus a low concentration of CNP is used and sonication before use is necessary.

Section 6.3 contains a description of the material used, CNP treatment performed and manufacturing methods of composites, Section 6.4 describes the testing performed for composites. Section 6.5 presents the results

obtained from testing as well as discussion of the results. Conclusions of this chapter is given in Section 6.6

6.3 Materials and methods

6.3.1 Materials

Information regarding reinforcement material, matrix, accelerator, catalyst and mould release is provided in Section 3.3.1.

6.3.2 Cellulose nano-whiskers (CNP) preparation

CNP are prepared using an acid hydrolysis technique of cotton (see Fig. 6.1). Chopped cotton is hydrolysed in 64% (by weight) aqueous solution of H_2SO_4 by constant stirring for 45 min at 45^oC [323]. The suspension obtained following the above process is treated according to the technique described by Labet and Thielemans [314] to obtain cellulose nano-crystals. A brief description of the process is given here. The acid hydrolysed suspension is washed with deionised water utilising three successive centrifugations at 10,000 rpm and 10^oC for 15 minutes. The residual free acid from the suspension is removed by dialysis under running tap water for 48 hours. After that the pH of the suspension is measured (as it is required to be neutral). The CNP dispersion in water is homogenised by sonication using a Branson sonicator for 4 minutes and then filtered utilising a glass filter. Overnight stirring is performed with amberlite resin of the filtrate to remove non H_3O^+ cations from the CNP suspension and it is filtered again to get the final CNP in deionised water. At last freezing of the suspension in liquid nitrogen is performed as freeze drying gives crystalline CNP in the dry state. Prior to freeze drying, a homogeneous suspension of CNP in chloroform is prepared by stirring and sonication. The CNP are obtained after freeze drying in powder

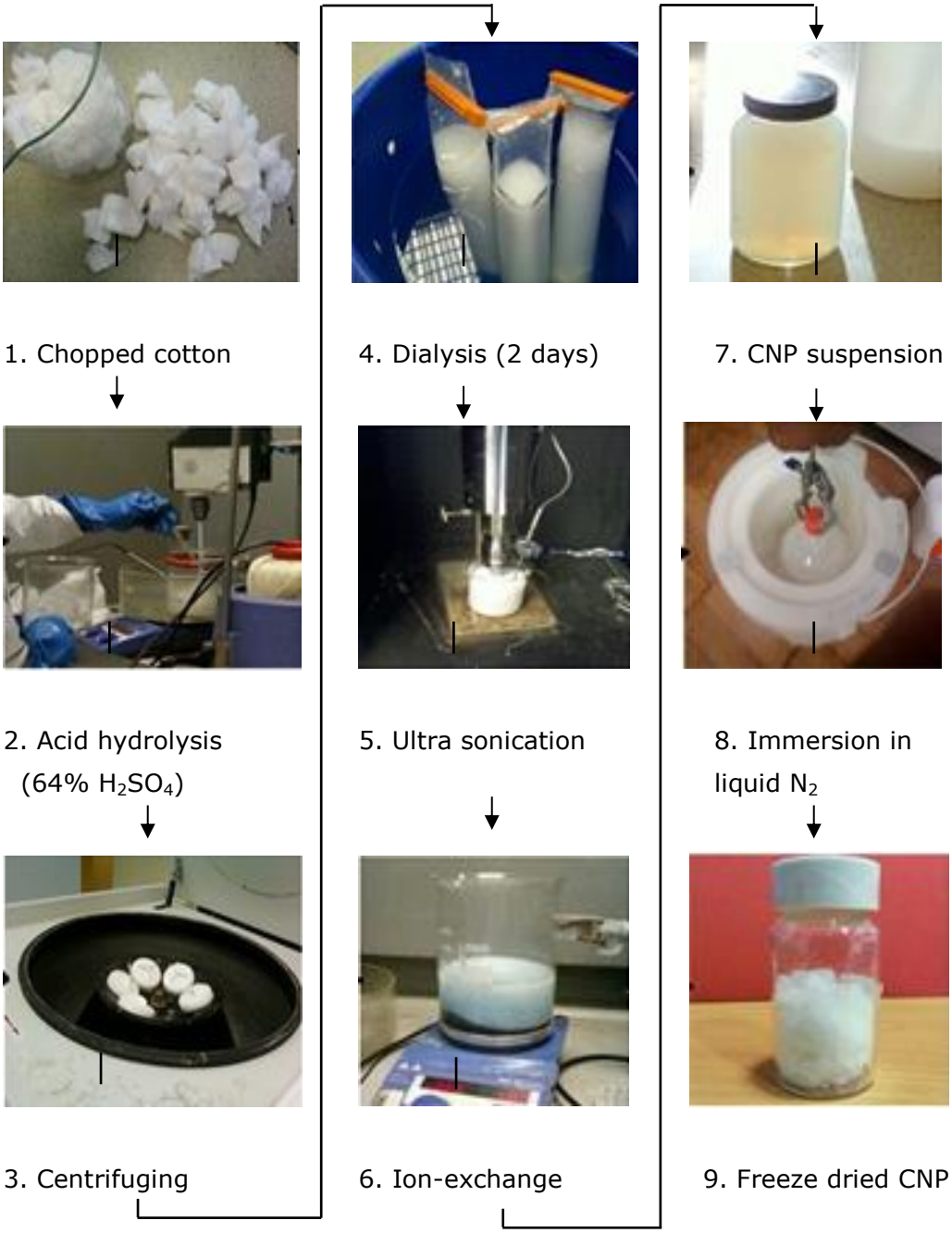


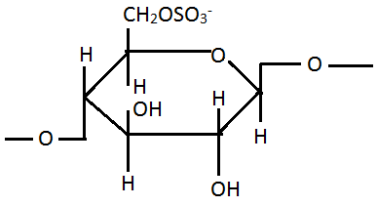
Fig. 6.1: Production of CNP from cotton using acid hydrolysis process.

form and are described as freeze dried (FD) CNP. CNP suspensions achieved before freeze drying, described as non-dried (ND) CNP, are also used in this work as a surface treatment agent of jute yarn. CNP suspension is also used

before amberlite resin treatment, described as no resin treated, non-dried (NRTND) CNP. CNP suspension after amberlite resin treatment and non-dried, described as resin treated, non-dried (RTND) CNP is used to coat yarn.

6.3.3 Dispersion of CNP and the significance of sulphate ester groups

For manufacturing CNP, sulphuric acid is widely used with respect to others such as hydrochloric or nitric acid. A major reason of using sulphuric acid is better dispersion of CNP and CNP manufactured using other acids tends to form cluster. Anionic sulphate ester group ($-OSO_3^-$) of sulphuric acid grafted with surface hydroxyl group via an esterification reaction and randomly distributed on the surface of CNP [324]. The negatively charged $-OSO_3^-$ groups form an electrostatic layer around the surface of nanocrystals thus an electrostatic repulsion is occurred that promotes to disperse CNP in solvents. They also form layer by layer assembly [325] that prevent to form sediment of CNP in solvent. Better dispersion of sulphate esterified CNP reduces the viscosity of suspension with respect to non-esterified CNP manufactured using HCl. With increase in sulphate group i.e. sulphated CNP promotes the dispersion in suspension and desulphation i.e. removal of sulphate group increases agglomeration of CNP [326].



Negatively charged esterified CNP [324]

6.3.4 CNP treatment of jute yarn

CNP treatment is performed on jute yarn wrapped on a glass fibre composite board as discussed in Section 5.3.2. Free surface –HO groups of CNP have a tendency to create strong hydrogen bonds with cellulose based jute fibres. Thus treatment of yarns with CNP suspension enables the UD sheet to achieve sufficient strength to transfer the yarns from the wrapping board to the infusion mould and CNP are used as a substitute for HEC. However the composite plaques made with CNP mixed in resin need to use HEC size for the UD sheet of yarns. The process is described in Section 3.2.2.

In this work (i) dialysed, sonicated and without amberlite resin treated CNP in suspension (NRTND); (ii) dialysed, sonicated and amberlite resin treated CNP in suspension (RTND); and (iii) amberlite resin treated as well as freeze dried (FD) CNP are used to coat the jute yarn to understand their influence on the mechanical properties of jute/polyester composites. However only freeze dried CNP are used to mix with resin before infusion to understand the influence of CNP filled resin on the mechanical properties of composites.

According to the process followed in this work CNP are obtained in suspension in the first instance. The dried CNP are found after the final step of the process called freeze drying. Freeze dried CNP are used to make a suspension to coat yarns. 0.5% and 1% CNP suspensions (by weight) are used to coat the yarns. CNP suspension of the required concentration is made using tap water. Before using the CNP suspension to coat the yarns, sonication is performed to avoid agglomeration of CNP. Jute yarn is wrapped on a glass fibre composite board and dipped in CNP suspension for coating for at least 30 minutes. 0.5% and 1% CNP suspension before and after amberlite resin

treatment as well as non-freeze dried suspension are also used to coat the yarns. Five times higher CNP suspension by weight of yarn is maintained to save CNP as the CNP manufacturing method is time consuming. Excess suspension is drained and the UD yarn wrapped on glass fibre composite board is then dried in an oven at 105°C for one hour. Finally the dried yarns are kept at room temperature for at least 48 hours before infusion.

0.5% and 1% freeze dried CNP (by weight) are mixed with resin before infusion. Mixing is performed using a mechanical stirrer at 200 RPM before adding accelerator and catalyst.

6.3.5 Preparation of composites

Composite plaques are manufactured using RTM. The manufacturing technique is explained in Section 3.3.4.

6.4 Testing

6.4.1 Composite tests

Composites manufactured with CNP coated yarn and CNP mixed in resin are tested for tensile properties as described in Section 3.4.6. A non-contact video gauge (software: Imetrum) extensometer (Fig. 4.5) is used to measure the tensile modulus of the composite plaques. At least three tests were performed for each plaque to find the mean values of tensile properties.

6.5 Results and Discussion

6.5.1 Composites test results

Tensile moduli and tensile strengths of composites manufactured and tested in this chapter are presented in Table 6.1; Fig. 6.2 and Fig. 6.4 (raw data are

presented in Appendix D). A large variation of fibre volume fraction (35-44%) is observed among the composite plaques manufactured in this chapter and the cause is mentioned in Section 5.5.2. Thus back calculated tensile moduli of fibres (Table 6.2 and Fig. 6.3) and back calculated tensile strength (Table 6.2 and Fig. 6.5) of fibres are calculated to compare the mechanical properties of these composite plaques obtained as described in Section 3.5.6.

Table 6.1 Fibre volume fraction and tensile properties of composites

Sample name	V_f %	Tensile modulus (GPa)	Std. dev.	Tensile Strength (MPa)	Std. dev.	Strain at Fail (%)	Std. dev.
Raw yarn	38	17.4	0.3	169	12	1.36	0.06
Raw yarn HEC sized	35	18.3	0.6	187	4	1.34	0.07
0.5% CNP sized (FD)	39	17.3	0.4	199	12	1.56	0.03
1% CNP sized (FD)	35	16.6	1.2	162	10	1.32	0.09
0.5%CNP sized (RTND)	40	18.6	1.3	216	12	1.47	0.09
1%CNP sized (RTND)	38	19.7	0.3	223	6	1.42	0.01
1%CNP sized (NRTND)	44	20.7	0.7	225	16	1.39	0.10
0.5%CNP mixed in resin	37	17.8	0.8	183	13	1.35	0.03
1%CNP mixed in resin	37	16.0	1.1	166	11	1.20	0.36

V_f - Fibre volume fraction FD – freeze dried, RTND – CNP are obtained after amberlite resin treatment and non-freeze dried, NRTND – CNP are neither amberlite resin treated and non-freeze dried.

Table 6.2 Back calculated tensile modulus and back calculated tensile strength of fibres

Sample name	Tensile modulus (GPa)	Standard deviation	Tensile Strength (MPa)	Standard deviation
Raw yarn	39.5	0.9	329	29
Raw yarn HEC sized	45.2	1.5	404	10
0.5% CNP sized (FD)	38.6	1.2	402	30
1% CNP sized (FD)	40.6	3.4	331	29
0.5%CNP sized (RTND)	41.1	3.2	459	30
1%CNP sized (RTND)	45.9	0.8	472	14
1%CNP sized (NRTND)	42.5	1.8	441	35
0.5%CNP mixed in resin	41.8	2.1	375	36
1%CNP mixed in resin	37.1	2.7	332	27

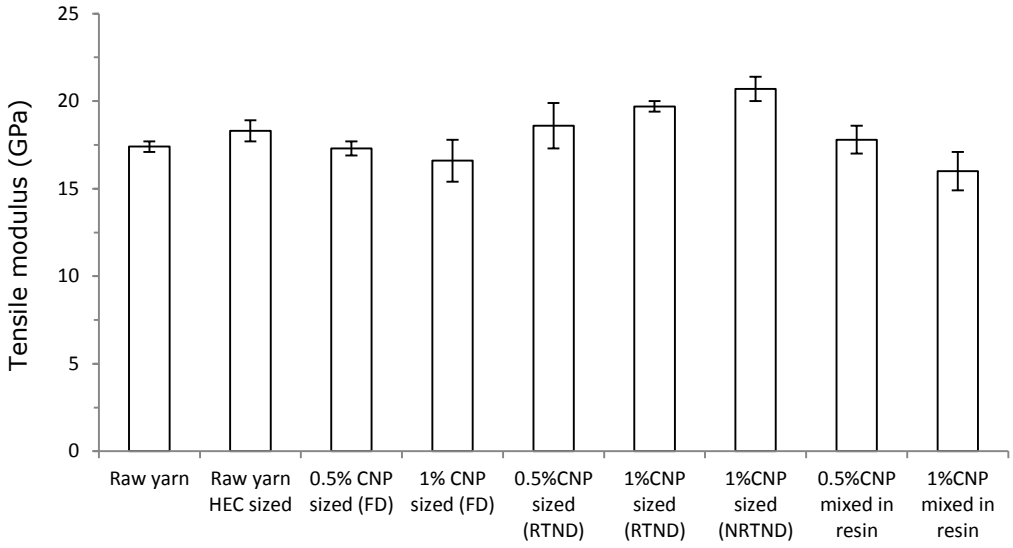


Fig.:6.2 Tensile modulus of composites (error bars indicate standard deviation)

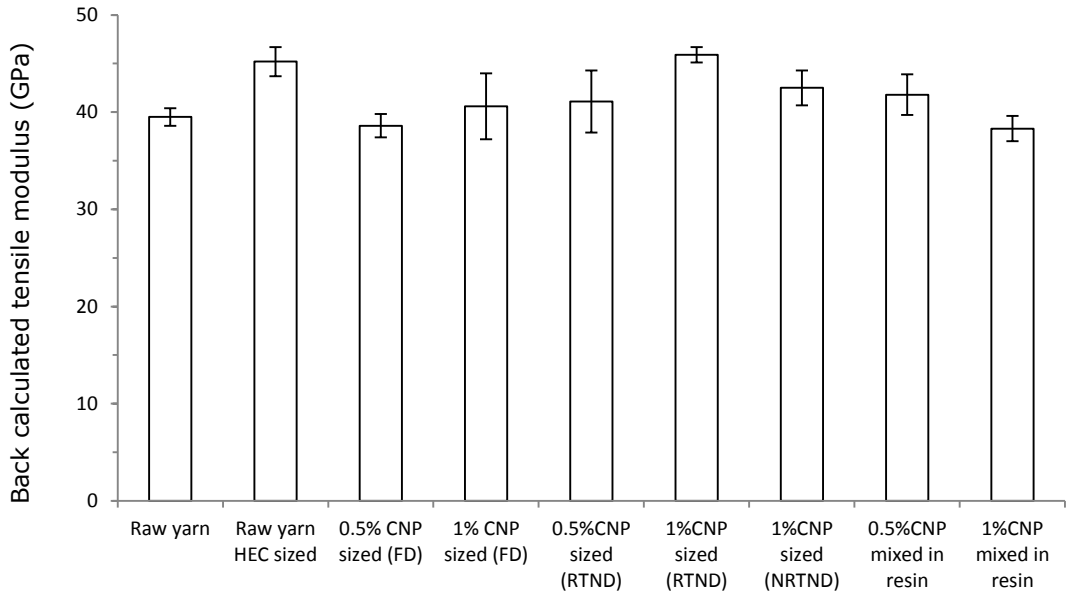


Fig.:6.3 Back calculated tensile modulus of fibres (error bars indicate standard deviation)

6.5.1.(I) Composites manufactured with freeze dried (FD) CNP treated yarn

Two composite plaques are manufactured with FD CNP treated (0.5% and 1% suspension) yarn and only back calculated tensile strength of fibres manufactured with 0.5% CNP coated yarn increases (22%) significantly ($P<0.05$) with respect to that of composites manufactured with unsized raw yarn (Table 6.2 and Fig. 6.5). On the other hand back calculated tensile modulus of fibres of composites manufactured with FD CNP treated (0.5% and 1% suspension) yarn and back calculated tensile strength of fibres of composites manufactured with FD CNP treated (1% suspension) yarn are significantly lower (modulus 17% for 0.5% CNP treated yarn, 11% for 1% CNP treated yarn and strength 22% for 1%CNP treated yarn) with respect to

those of composites manufactured with HEC sized raw yarn and these differences are statistically significant ($P<0.05$).

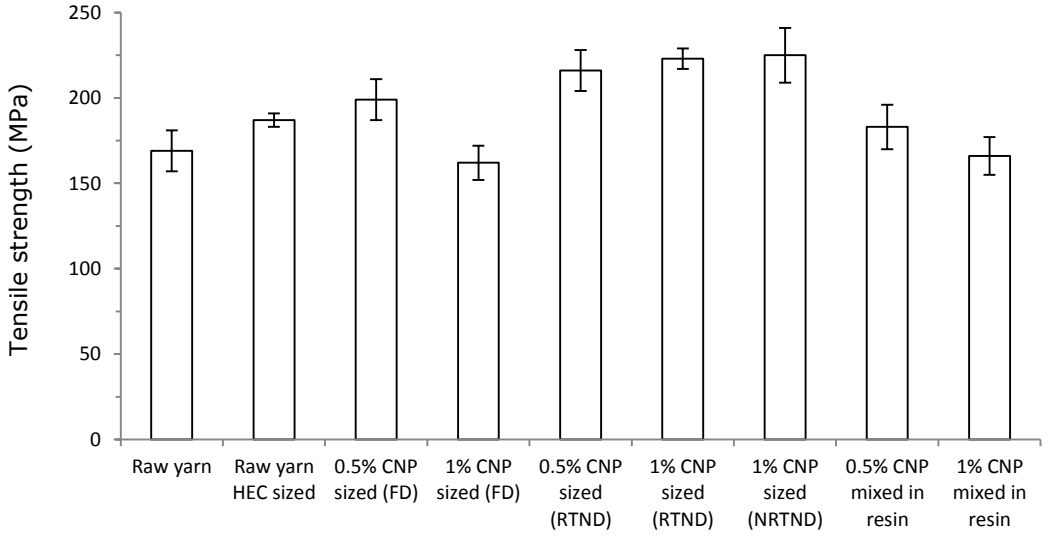


Fig. 6.4 Tensile strength of composites (error bars indicate standard deviation)

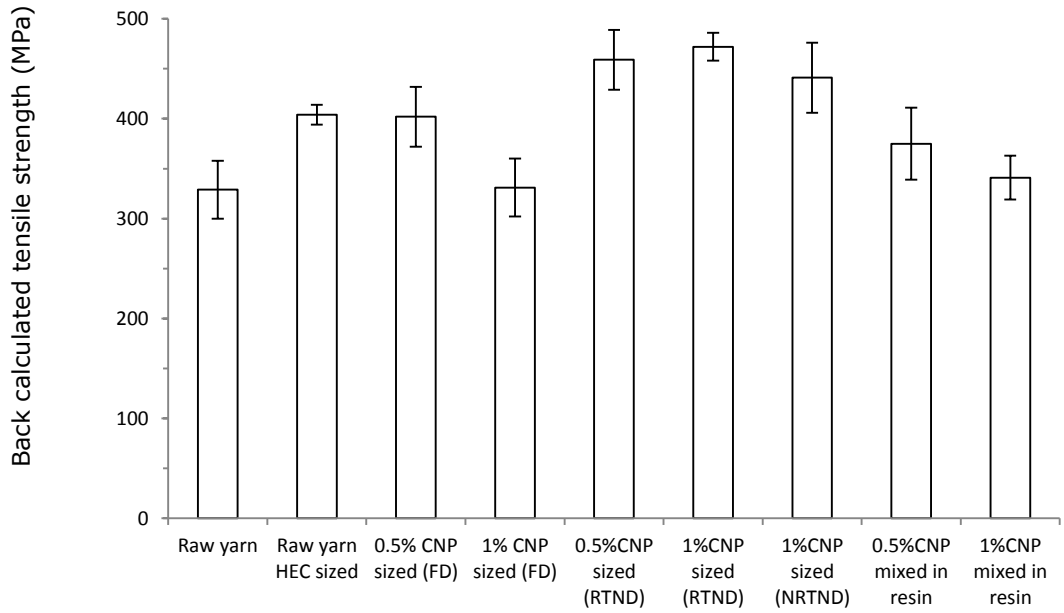


Fig. 6.5 Back calculated tensile strength of fibres (error bars indicate standard deviation)

These results reveal that CNP (FD) treatment of yarn does not improve the tensile properties of composites markedly with respect to that of composites manufactured with unsized raw yarn and CNP (FD) treatment of yarn reduces the tensile properties of composites for the majority of cases with respect to that of composites manufactured with HEC sized raw yarn. As mentioned earlier freeze dried CNP agglomerate and an agglomerated CNP deposition is found on the surface of CNP treated yarn (Fig. 6.6 and Fig. 6.7). CNP deposition is more prominent on the surface fibres with respect to inner fibres of the yarn (Fig. 6.8 and Fig. 6.9) suggesting that agglomerated CNP deposition has occurred on the yarn surface rather than inside. Possibly these CNP agglomerations do not contribute as expected to bond formation between reinforcement fibres and matrix, resulting in no improvement in mechanical properties of composites. Naturally CNP deposition would be higher for yarn treated in 1% CNP concentrated suspension than that of for 0.5% CNP concentrated suspension hence the mechanical properties of composites manufactured with yarn treated by 1% CNP concentrated suspension are found inferior to those of composites manufactured with yarn treated by 0.5% CNP concentrated suspension.

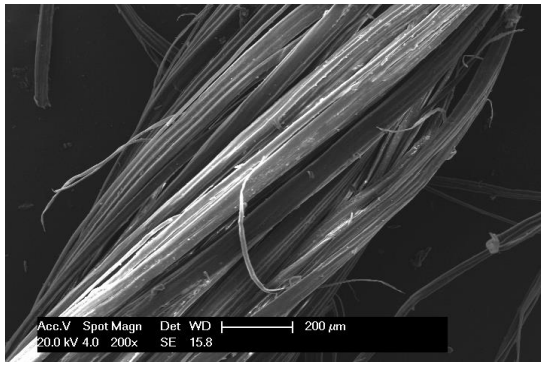


Fig. 6.6 Raw yarn under SEM

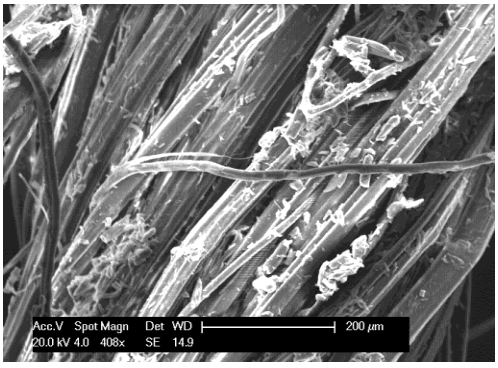


Fig. 6.7 Yarn sized by 1% CNP suspension under SEM

Back calculated tensile modulus and tensile strength of fibre of these composites plaques are significantly lower ($P<0.05$) with respect to those of composites manufactured with water treated yarns with tension as well as HEC sized (Section 4.5.3).

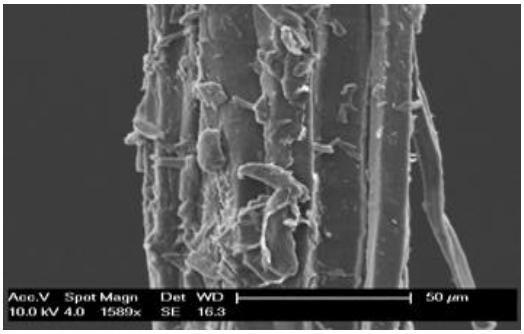


Fig. 6.8 Surface fibre of yarn sized by 1% CNP suspension

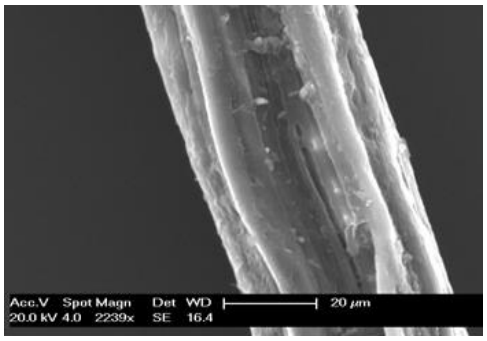


Fig. 6.9 Inside fibre of yarn sized by 1% CNP suspension

Strains at failure of composites manufactured with CNP treated yarn and composites manufactured with untreated yarn are fairly similar (Table 6.1 and Fig. 6.10) and also similar to that of studied in Chapter 4 (Section 4.5.3) revealing that CNP treatment as well as water treatment of yarn has no influence on this property of composites.

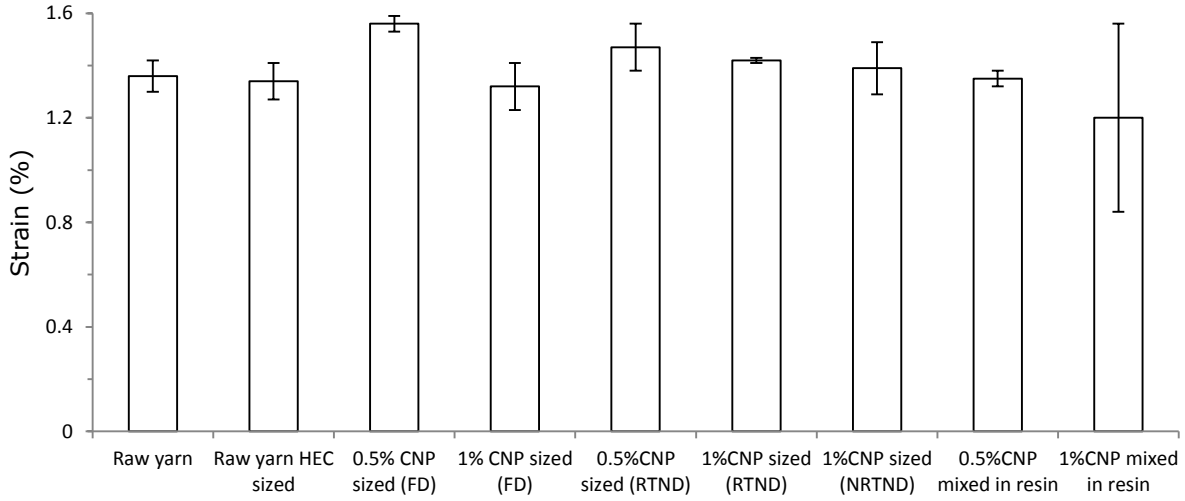


Fig. 6.10 Strain at failure of composites

6.5.1.(II) Composites manufactured with RTND CNP treated yarn

Composites made with 1% CNP treated yarn show a 16% increase in back calculated tensile modulus (Table 6.2 and Fig. 6.3) with respect to that of composites manufactured with unsized raw yarn and this difference is statistically significant ($P<0.05$). An increase in back calculated fibre tensile strength (14 - 43%) of both the composite plaques (0.5% and 1% CNP treated) is noticed with respect to that of composites manufactured with unsized raw and HEC sized raw yarns. These differences are also statistically significant ($P<0.05$) revealing that resin treated non-dried CNP applied to jute yarn has a better effect on the tensile properties of composites compared to freeze dried CNP treated jute yarn. Dispersion of CNP is better for the non-dried suspension with respect to the freeze dried CNP suspension and thus less agglomeration of CNP is expected (Fig. 6.11 and Fig 6.12) for yarn treated with non-dried CNP. Lower agglomeration and better distribution of CNP on the yarn surface possibly contribute to enhance the tensile properties of composites due to hydrogen bonding between reinforcement fibres, matrix and CNP. CNP treatment of yarn in an aqueous medium with tension is also a possible cause of increasing tensile properties.

These results are very similarity to the back calculated tensile modulus and tensile strength of fibre of composites manufactured with water treated yarn with tension as well as HEC sized studied in Chapter 4 (Section 4.5.3). Thus CNP treatment of yarn in aqueous medium (similar to water treatment with tension and HEC sized) and water treatment with tension then HEC sizing show similar effect on the tensile properties of composites. Composites manufactured with both type of treated yarn (CNP and water) show similar

differences of back calculated tensile properties of fibres with respect to those of composites manufactured with raw yarn.

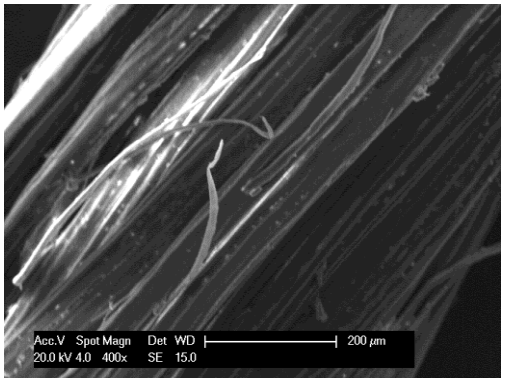


Fig. 6.11 Yarn sized by 0.5% RTND CNP suspension under SEM

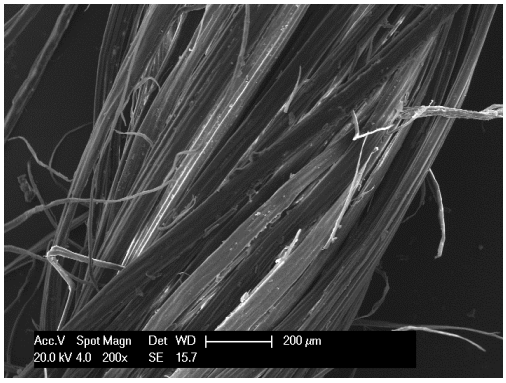


Fig. 6.12 Yarn sized by 1% RTND CNP suspension under SEM

Very similar strains at failure are observed for composites manufactured with jute yarn treated by amberlite resin treated non-dried CNP and composites manufactured with untreated jute yarn (Table 6.1 and Fig. 6.10) which are similar to composites manufactured with water treated yarn with tension as well as HEC sized (Chapter 4, Section 4.5.3). Thus it can be said that CNP treatment does not influence the strain at failure of composites.

6.5.1.(III) Composites manufactured with NRTND CNP treated yarn

Only one composite plaque was manufactured with jute yarn treated by no resin treated non-dried CNP. This composite plaque shows an increase in back calculated fibre tensile modulus (8%) and back calculated fibre tensile strength (34%) with respect to that of composite plaque manufactured with unsized raw yarn. These differences are statistically significant ($P<0.05$) and the possible causes mentioned in Section 6.5.1.(II) are also valid for this composite plaque. Very similar strain at failure is observed for composites

made with CNP treated and untreated yarn (Table 6.1 and Fig. 6.11) revealing that CNP treatment of jute yarn has no effect on the strain at failure of composites.

As the results obtained for this composite plaque is similar to results discussed in section 6.5.1(II) thus comparison with composites manufactured with water treated yarns is also similar.

6.5.1.(IV) Composites manufactured with CNP mixed in resin

Two composite plaques were manufactured using 0.5% and 1% CNP (by weight) mixed in resin prior to infusion. The back calculated fibre tensile modulus of both the composite plaques is significantly ($P<0.05$) lower (8% for 0.5% CNP mixed in resin and 18% for 1% CNP mixed in resin) with respect to that of composites manufactured with HEC sized jute yarn. The composite plaque manufactured with 1% CNP mixed in resin shows significantly lower ($P<0.05$) back calculated fibre tensile strength (18%) with respect to that of composite plaque manufactured with HEC sized raw yarn. Mixing CNP with resin makes it viscous which may cause a different to wet fibres properly and may generate faults such as voids. Possibly due to this reason composite plaques manufactured with CNP mixed in resin show inferior mechanical properties and the deterioration of mechanical properties is more prominent for composite plaques manufactured with 1% CNP mixed in resin, as this resin was even more viscous than 0.5% CNP mixed in resin. The lowest strain at failure is also noticed for composites manufactured with 1% CNP mixed in resin (Table 6.1 and Fig. 6.10) and it is also due to fault (void) incurred in the composite plaque.

Due to faults (void) in composite plaques manufactured with CNP mixed in resin the results are not comparable with composites manufactured with water treated yarn.

No published study is found on the mechanical properties of composites manufactured with CNP treated UD jute polyester composites to compare the results of this chapter.

6.5.1.(V) Stress-strain curves of composite plaques

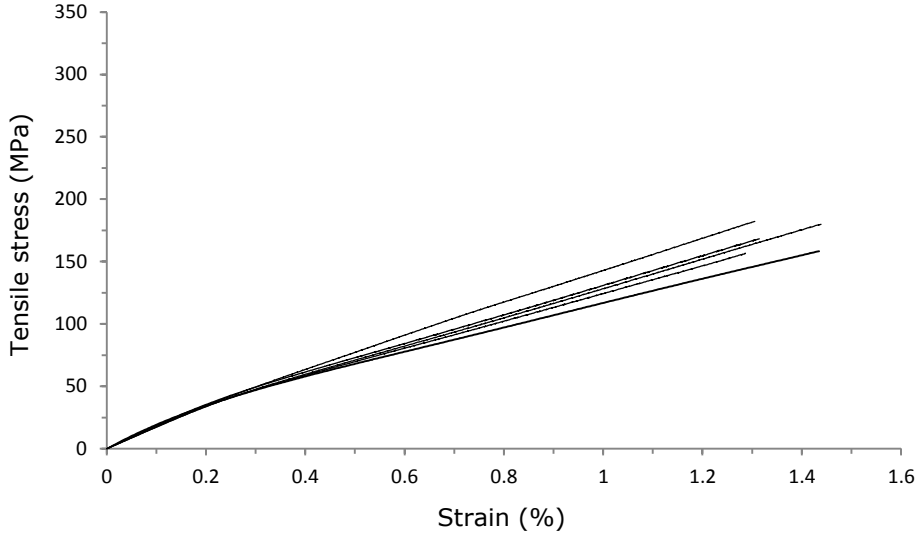


Fig. 6.13 Stress-strain curves of composites manufactured with unsized raw yarn

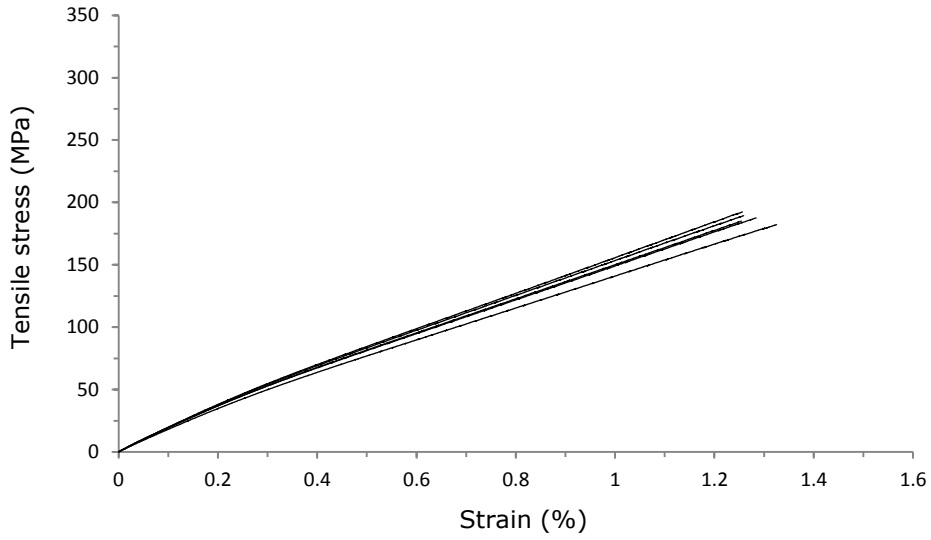


Fig. 6.14 Stress-strain curves of composites manufactured with HEC sized raw yarn

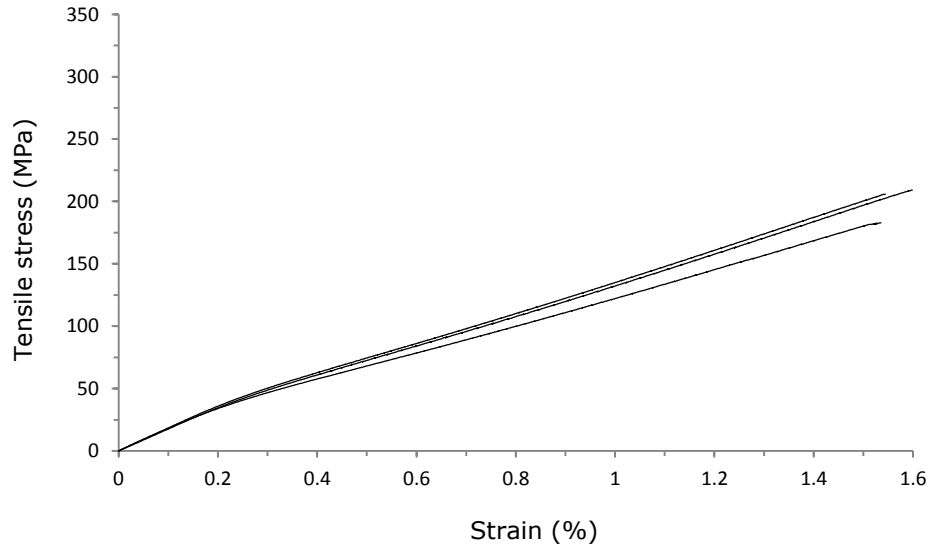


Fig. 6.15 Stress-strain curves of composites manufactured with 0.5% CNP (freeze dried) treated yarn

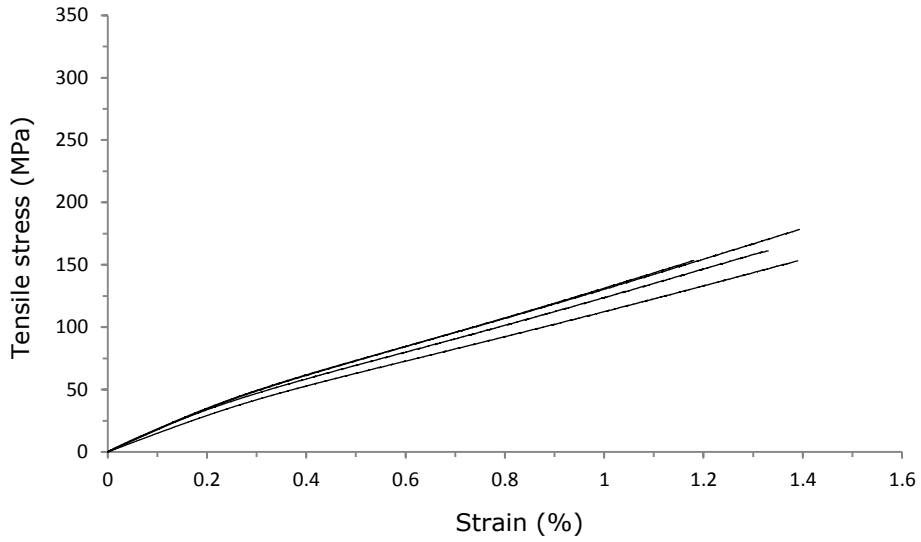


Fig. 6.16 Stress-strain curves of composites manufactured with 1% CNP (freeze dried) treated yarn

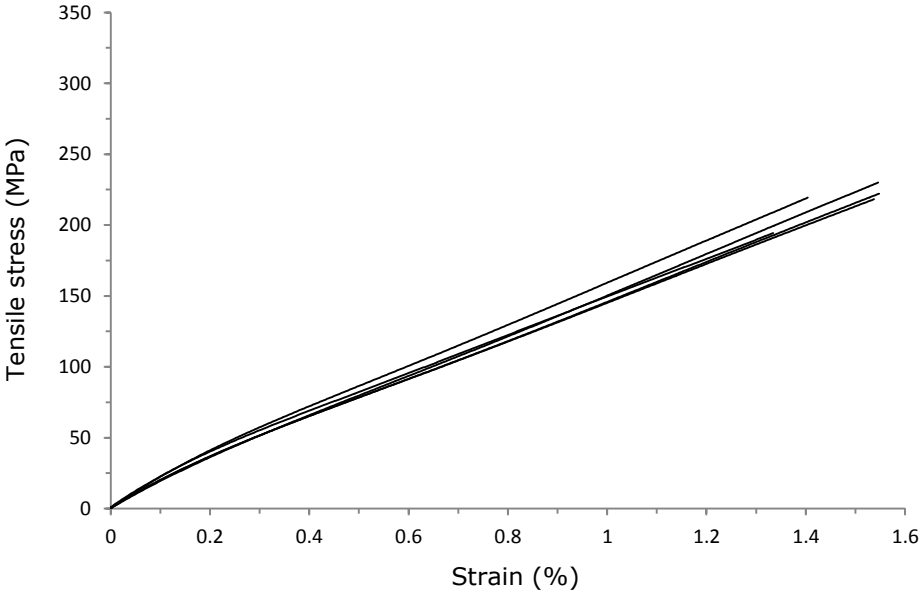


Fig. 6.17 Stress-strain curves of composites manufactured with 0.5% CNP (RTND) treated yarn

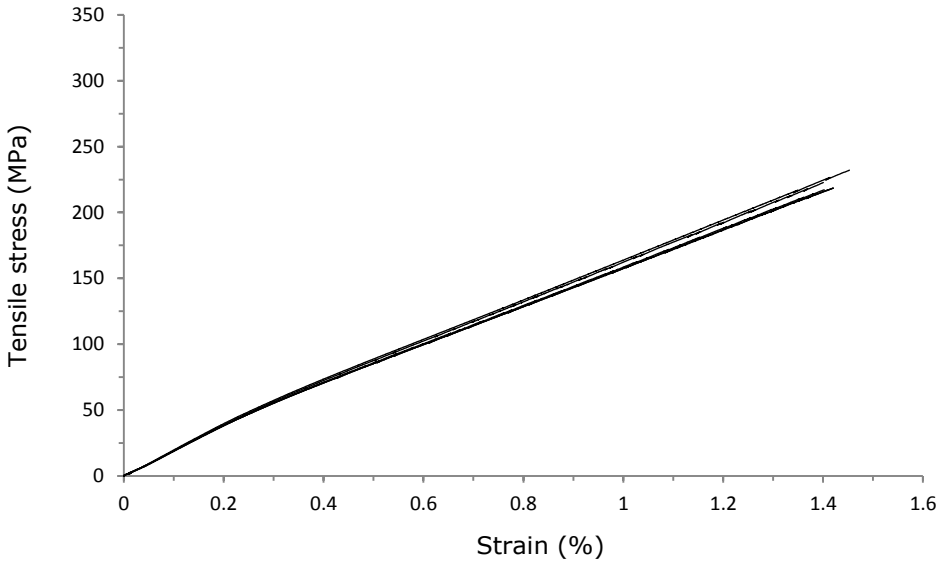


Fig. 6.18 Stress-strain curves of composites manufactured with 1% CNP (RTND) treated yarn

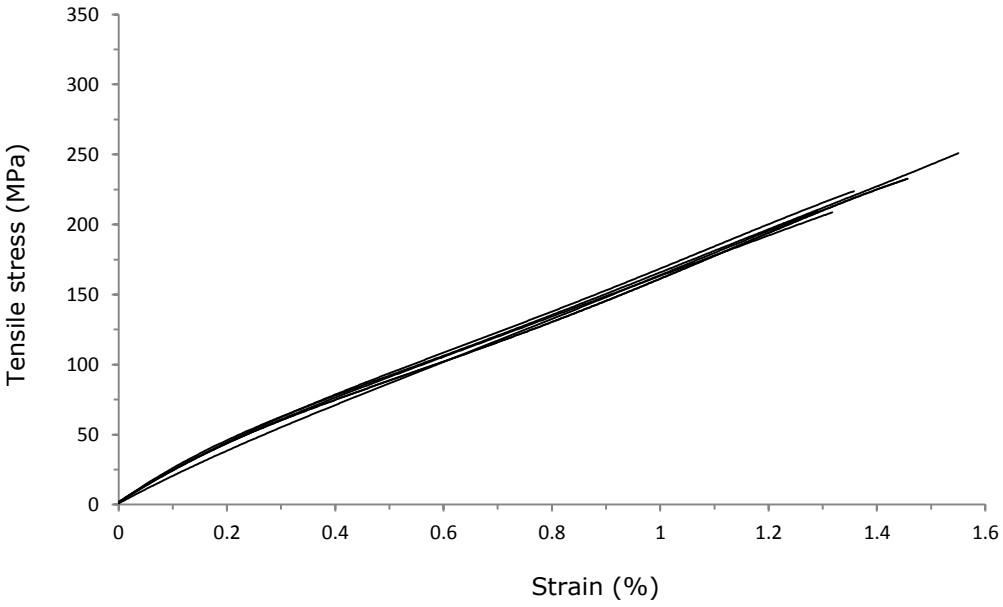


Fig. 6.19 Stress-strain curves of composites manufactured with 1% CNP (NRTND) treated yarn

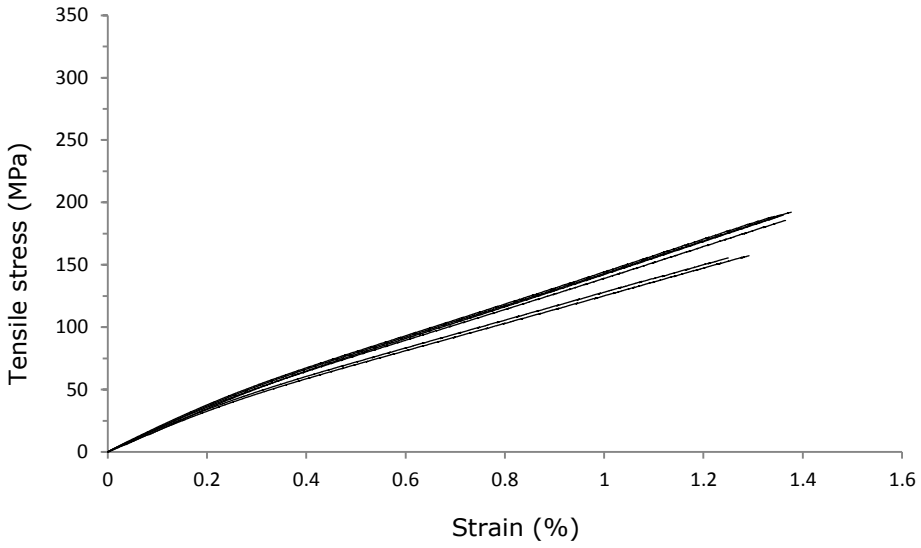


Fig. 6.20 Stress-strain curves of composites manufactured with 0.5% CNP mixed in resin

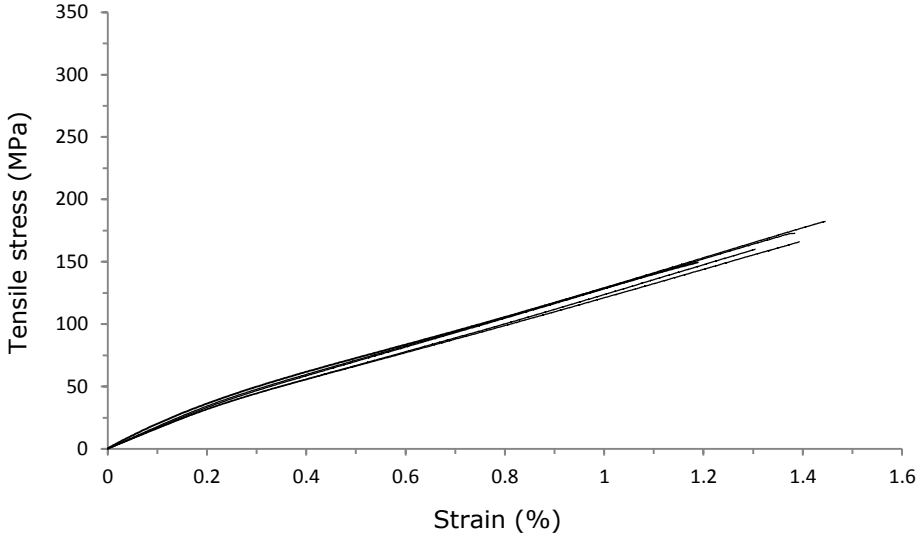


Fig. 6.21 Stress-strain curves of composites manufactured with 1% CNP mixed in resin

Stress-strain curves of composites manufactured and tested in this chapter are presented in Fig. 6.13 to Fig. 6.21. The shape of the curves for all

composite plaques is very similar and also similar to the stress-strain curves studied in Chapter 3 (Section 3.5.6(I)) and Chapter 4 (Section 4.5.3). Thus the discussion in Section 3.5.6(I) is also applicable here.

6.6 Conclusions

CNP obtained at various steps (such as FD, RTND and NRTND) of the manufacturing process are used as surface coating materials for jute yarn. Yarn is treated by these CNP for different concentrated (0.5% and 1% by weight) suspensions. The influence of these treatments on the mechanical properties of UD composites is studied in this chapter. Freeze dried CNP of different proportions (0.5% and 1% by weight) are also mixed with resin before infusion to investigate their influence on the mechanical properties of composites.

After analysing the results it is found that the mechanical properties of composites manufactured with yarn treated by freeze dried CNP are very similar to those of composites manufactured with unsized raw yarn and in most of the cases tensile properties are found inferior with respect to those of composites manufactured with HEC sized jute yarn. Agglomerated CNP deposition on the yarn surface does not contribute to improve the tensile properties of composites manufactured with FD CNP treated yarn for most of the cases. Strain at failure of composites manufactured with CNP treated yarn and untreated yarn are very similar.

Composite plaques manufactured with jute yarn treated by non-dried CNP suspension show better tensile properties for most of the cases with respect to that of composites manufactured with unsized and HEC sized raw jute yarn.

Within the CNP treatment studied in this chapter the influence of non-dried CNP is found best on the mechanical properties of composites due to low agglomeration and better distribution. CNP treatment of yarn in an aqueous medium with tension also plays an important role in increasing the tensile properties. Non-dried CNP suspension can be used as a binding material to form UD mat of yarn and at the same time it improves the mechanical properties of composites. Advantages of using these CNPs are eliminating the CNP drying process as well as avoiding preparation of CNP suspension to treat yarn. No major differences are found for strain at failure of composites manufactured with CNP treated yarn and untreated yarn.

CNP mixed in resin deteriorates the tensile properties of composites with respect to tensile properties of composites manufactured with HEC sized jute yarn. Adding CNP makes the resin more viscous which impedes fibre wetting and generates voids which deteriorate the mechanical properties of composites. A higher proportion of CNP leads to inferior mechanical properties in this work. Inferior strain at failure is also observed for composites manufactured with 1% CNP mixed in resin due to fault incurred such as voids. A binding agent is necessary to form UD mat of yarn to manufacture composites using CNP mixed in resin and HEC is used in this case which is not necessary for yarn treated with CNP suspension.

Stress-strain curves of CNP treated composites are found to be very similar to those of composites manufactured with untreated jute yarn.

CHAPTER 7

MECHANICAL PROPERTIES OF UD COMPOSITES MANUFACTURED WITH RAW JUTE FIBRE.

7.1 Summary

UD jute composites are manufactured using raw jute fibre, root end of raw jute and jute sliver (an intermediate product of the jute spinning process). Raw jute fibre is collected from cultivator and wrapped on a glass fibre composite board utilising adhesive tape to attain UD mat of fibres. Root end of fibres are cut and also wrapped on a plastic board separately. After wrapping a sufficient amount of fibres, 0.6% (by weight) HEC solution is applied (as described in Section 3.3.2) and then dried inside an oven at 105°C for 1 hour. Dried fibres are kept in ambient condition for at least 24 hours prior to infusion. Composites are manufactured with HEC sized UD mat using RTM. Breaker carded sliver (delivery material of breaker carding machine; this is an opening i.e. splitting of naturally tangled fibres, cleaning and sliver forming machine in jute spinning) and finisher carded sliver (delivery material of finisher carding machine; this is a doubling i.e. method of feeding two or more products to the machine together, opening, cleaning and sliver forming machine in jute spinning) are infused without applying HEC. Single fibres as well as composites are tested for mechanical properties and the mean values of tested results as well as the back calculated values of mechanical properties (calculated using the rule of mixtures) are discussed.

7.2 Introduction

Yarn is a common and convenient textile product to manufacture UD composites. The raw material of yarn is fibres, in either staple (fibres have

definite length) or continuous filament form. The primary requirement to construct a yarn is to hold fibres together by twisting (ring or rotor spun yarn) [327], wrapping by constituent fibre (air jet spun yarn) or external filament (wrap yarn), use of adhesive material (twilo or pavena system) [179] to obtain a continuous configuration of strands. Twist is the most common technique to form yarn from staple fibres. Twist develops inter fibre friction that brings strength to yarn [13]. For further processing of yarn, twist plays a very important role; however it has a number of unfavourable effects that influence the performance of composites.

Due to twist, fibres follow a helical path in yarn hence alignment of fibres in the direction of the yarn axis is prohibited. Thus fibres are loaded off axis which reduces the properties of composites [241]. Goutianos et al. [189] found 1.85 times higher epoxy impregnated flax roving strength when twist was reduced from 230 turns per metre to 70 turns per metre. Twist brings the compactness of yarns and this property increases with increasing twist per unit length. Compactness reduces the permeability of yarns and obstructs resin impregnation [187] resulting in voids in composites [21].

Yarn manufacturing is a labour intensive as well as an expensive process. Moreover the higher the amount of twist in yarns, the higher will be the manufacturing cost due to equation 7.1.

$$\text{Production per unit time} = \frac{\text{Spindle speed}}{\text{Twist per unit length}} \qquad (7.1)$$

As spindle speed is set to an optimum condition and generally spinners do not change this, thus increasing twist would reduce the production and due to less

production with respect to time, manufacturing cost would increase. Considering the constraints of using yarns in composites, raw fibre is chosen to understand their suitability for manufacturing of aligned composites with respect to yarns.

The total number fibres in jute yarn, used in this work, is found to be 106 (mean value from five readings). These fibres are theoretically packed in the yarn in two ways, namely (i) open packing (Fig. 7.1) and (ii) hexagonal close packing (Fig. 7.2). In open packing 93 fibres in a yarn cross-section make 6 layers and 91 fibres make the same number of layers in hexagonal close packing. Thus 106 fibres would make 6.5 layers and as the centre layer consists of a single fibre hence if a yarn cross-section contains 106 fibres, the diameter of that yarn may be considered 12 times higher than that of single fibre. Application of this yarn model in composite manufacturing would provide large gaps among yarns resulting in resin rich regions. Using raw fibres for the same purpose would provide a better distribution of fibres and

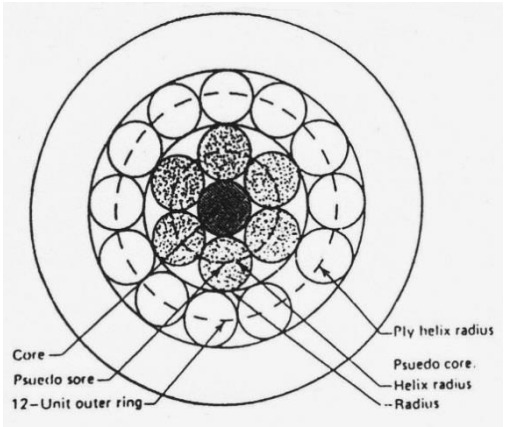


Fig. 7.1 Open packing [328]

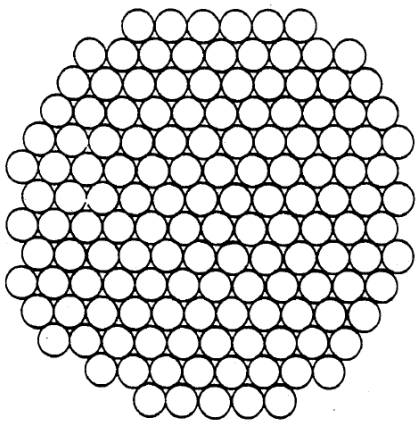


Fig. 7.2 Hexagonal close packing [329]

thus less resin rich region. Hence raw jute fibre from the cultivator is selected as a reinforcement material to investigate the mechanical properties of composites.

However some drawbacks need to be overcome to utilise plant fibres to achieve a certain fibre volume fraction for UD composites such as the challenge of continuous supply of fibres due to their limited length and manufacturing of UD mat prior to infusion. As a yarn is a well-established product to manufacture UD composites thus various techniques are available to build UD mat from yarn such as filament winding, stitching, knitting, weaving, braiding, use of adhesive materials. Prepreg tape is also a popular impregnated fibre assembly to manufacture UD composites. Application of raw jute to manufacture UD composites also needs to produce an aligned fibrous mat. The size and shape of raw jute fibre are found suitable to perform this operation and application of adhesive material (HEC) has proved successful to form UD fibrous mat. Accordingly UD composites are produced using raw jute fibre to understand their mechanical properties with respect to composites made with jute yarn.

The jute spinning process involves a series of operations by different machineries and activities. These are: Selection of batch $\longrightarrow$ Piecing up $\longrightarrow$ Softening $\longrightarrow$ Conditioning $\longrightarrow$ Breaker card $\longrightarrow$ Finisher card $\longrightarrow$ 1st draw frame $\longrightarrow$ 2nd draw frame $\longrightarrow$ 3rd draw frame $\longrightarrow$ Spinning. Several mechanical actions such as hackling, carding, drawing, doubling and twisting are involved in these operations. To understand the effect of these operations on the mechanical properties of fibres, single fibres collected from different intermediate products are tested for tensile properties.

To understand the suitability of intermediate products of the jute spinning process for composite manufacturing, breaker carded sliver and finisher carded sliver are infused to study their mechanical properties.

The length of jute fibre is equal to the length of the jute plant and it is generally 1 to 4 metres [330]. Such a long fibre may vary in mechanical properties from root to tip. To understand the variation of tensile properties of fibres, single fibres are tested from root end, tip end and the middle part of the plant. Composites are also manufactured with the root end fibres to investigate any localised effect of fibres on the mechanical properties of composites. Due to time constraints other parts are not investigated.

Section 7.3 contains a description of the material used and manufacturing methods of composites, Section 7.4 describes the testing performed on fibres and composites. Section 7.5 presents the results obtained from testing as well as discussion of the results. The conclusion of this chapter is included in Section 7.6.

7.3 Materials and Methods

7.3.1 Materials

Tossa jute fibre is used in this work, collected from the cultivator in Bangladesh. Intermediate products of the jute spinning process as well as yarns are supplied by Janata Jute Mills Ltd. Bangladesh. Unsaturated polyester resin, trade name Crystic 2446PALV by Scott Bader Ltd. UK is used as a matrix. It is a pre-accelerated resin and only catalyst (mentioned in Section 3.3.1) is needed to mix for infusion. Mechanical properties of resin are listed in Table 7.2. Information regarding mould release is discussed in

Section 3.3.1. Due to a change of manufacturer of resin a composite plaque is manufactured with HEC sized raw yarn using Crystic 2446PALV to compare the tensile properties of composite plaques manufactured with fibres and slivers in this chapter.

7.3.2 Preparation of fibres and composites

Raw fibres are wrapped on a glass fibre composite plaque (Fig. 7.3) to obtain a UD mat of jute fibres using adhesive tape. Effort is paid to wrap the fibres in parallel under tension and the operation is performed manually. After wrapping a sufficient quantity of fibres it is sized with HEC solution as described in Section 3.3.2 and dried in an oven at $105^{O}C$ for one hour. Composite plaques are manufactured using RTM and the manufacturing technique is explained in Section 3.3.4.



Fig. 7.3 Raw fibres wrapped on glass fibre composite board

7.4 Testing

A description of fibre preparation and composite preparation for tensile testing is given in Section 4.3.4 and Section 3.3.6 respectively. Tensile testing for

single fibres and composites are described in Section 4.4.1 and 4.4.2 respectively. Analysis of variance (ANOVA, a statistical tool similar to T-test is used to compare data set of more than two) test is performed to compare the tensile properties of single fibre tested in this chapter. Similar to T-test two tail 'P' value (Section 3.4.7) and 0.05 significance level (i.e. 95% confidence level) is considered to analyse the data.

7.5 Results and Discussion

7.5.1 Fibre test results

Fibres from the root end, the tip end, the middle part and from random positions; fibres from different intermediate processing stages as well as fibres from yarns are tested for tensile properties.

7.5.1.(I) Diameter of fibres

The fibre test results are shown in Table 7.1 and Fig. 7.4 (raw data are presented in Appendix E) showing the mean diameter of fibres extracted from several specimens (the method is explained in Section 4.3.4). For most of the cases raw fibres show higher fibre diameter with respect to fibres separated from intermediate materials as well as yarn. Most likely several processing stages involved in spinning help to split naturally adhered fibres which reduce the diameter. The diameter of fibres extracted from the middle part and the tip end of the plant is also lower than that of fibres from the root end. This is because separation of the fibre is found to be easier for the middle and tip end of the plant with respect to fibres from the root end. Significant difference ($P<0.05$) also found by ANOVA test among the diameter of fibres obtained

from different samples. Due to natural variation of fibre diameter standard deviation is high for each case.

Table 7.1 Tensile test results of fibres

Type of fibre ↓	Fibre diameter (μm)	Std. dev.	Tensile modulus (GPa)	Std. dev.	Tensile strength (MPa)	Std. dev.	Strain at failure (%)	Std. dev.
Raw fibre from cultivator (random)	70	14	22.2	4.5	468	133	2.18	0.57
Raw fibre from the root end of the plant	71	15	19.4	5.2	466	145	2.25	0.65
Raw fibre from the middle part of the plant	55	10	25.3	7.3	457	219	1.68	0.79
Raw fibre from the tip end of the plant	54	16	23.5	11.4	292	175	1.11	0.33
Raw fibre from * Janata Jute Mills Ltd.	76	14	21.0	6.0	380	147	1.78	0.46
Fibre from breaker* carded sliver	58	12	18.0	5.1	376	166	1.82	0.53
Fibre from finisher* carded sliver	56	19	15.6	7.8	332	186	1.76	0.59
Fibre from 1st draw* frame sliver	48	13	18.9	6.3	411	186	1.84	0.57
Fibre from 2nd draw* frame sliver	52	14	22.2	8.5	535	232	2.03	0.47
Fibre from 3rd draw* frame sliver	52	12	18.1	10.3	369	237	1.81	0.60
Fibre from yarn*	55	14	18	7	409	241	2.01	0.66

Std. dev. = standard deviation. * Raw fibre, intermediate products and yarn from same batch

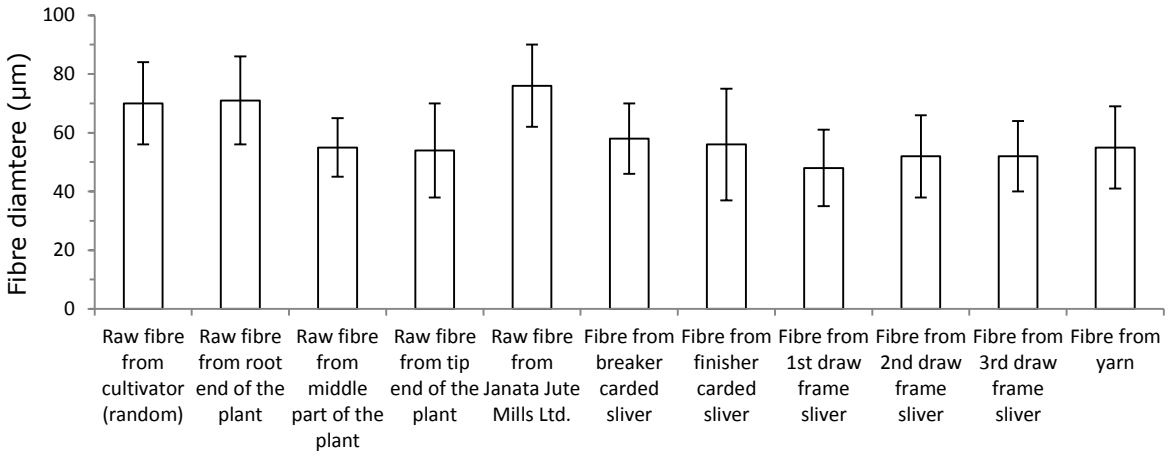


Fig. 7.4 Diameter of single jute fibres

7.5.1.(II) Tensile modulus of fibres

Tensile moduli of fibres (Table 7.1 and Fig. 7.5, raw data are presented in Appendix E) obtained in this chapter do not show any specific trend. However very similar values of tensile moduli are found for fibres collected from the cultivator and fibres supplied by Janata Jute Mills Ltd. The standard deviations of these two cases are also very similar.

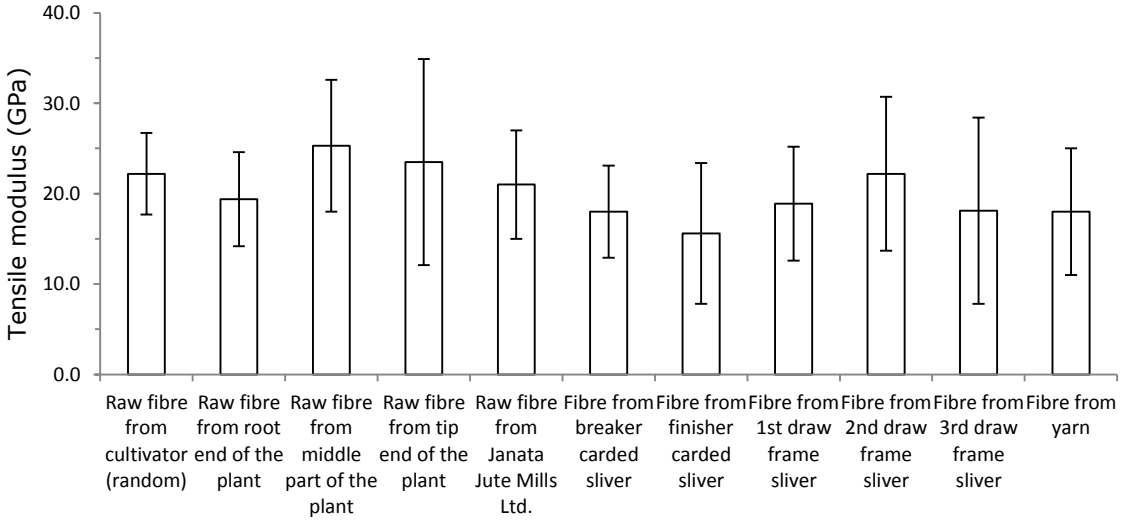


Fig. 7.5 Tensile modulus of single jute fibres

The highest tensile modulus is observed for fibres separated from the middle part of the plant. However the differences among the fibres obtained from different parts of the plant (i.e. root, middle and tip) and fibres extracted from random position of the plant are not statistically significant ($P<0.05$) revealing that tensile modulus of jute fibre does not depend on the position of fibres in the plant. The difference between the tensile modulus of fibres supplied by Janata Jute Mills Ltd. and the tensile modulus of fibres obtained from yarn is not statistically significant ($P<0.05$) revealing that application of several types of processing due to spinning does not deteriorate this property of fibres. A fall and rise of this property is noticed for different intermediate products, for example the lowest tensile modulus is found for fibres extracted from finisher carded sliver and a minor increase in tensile modulus is obtained for fibres separated from 2nd draw frame sliver. Possibly these variations are due to inherent variability of jute fibres. The tensile modulus of fibres from the cultivator is significantly higher ($P<0.05$) than that of fibres extracted from breaker carded sliver, finisher carded sliver, 1st draw frame sliver, 3rd draw frame sliver and yarn. ANOVA results confirms the singnificant variations ($P<0.05$) among the tensile modulus obtained from different fibre sample. A possible cause is a different origin of fibres (raw fibre from cultivator and others are from Janat Jute Mills Ltd.).

7.5.1.(III) Tensile strength of fibres

No specific trend is found for tensile strength of fibres studied in this chapter (see Table 7.1 and Fig. 7.6). Tensile strengths of fibres from raw jute (random position), root end and the middle of the plant show very similar values, however fibres extracted from the tip end show the lowest tensile

strength and it is significantly lower ($P<0.05$) with respect to that of fibres extracted from random position, the root end and the middle part (37%, 37% and 36% lower respectively) of the plant. The tensile strength of jute fibres from Janata Jute Mills Ltd. shows no statistically significant ($P<0.05$) difference with respect to the tensile strength of fibres extracted from intermediate products, as well as fibres extracted from yarn except fibres extracted from the 2nd draw frame sliver. These results reveal that the spinning process does not reduce the tensile strength of jute fibre. However fibres collected from the 2nd draw frame sliver show the highest tensile strength and the variation with respect to that of fibres supplied by Janata Jute Mills Ltd. (41% higher) is statistically significant ($P<0.05$). The very high tensile strength of these fibres is possibly due to localised variation. ANOVA result reveals significant variation ($P<0.05$) among the tensile strength of fibres tested here.

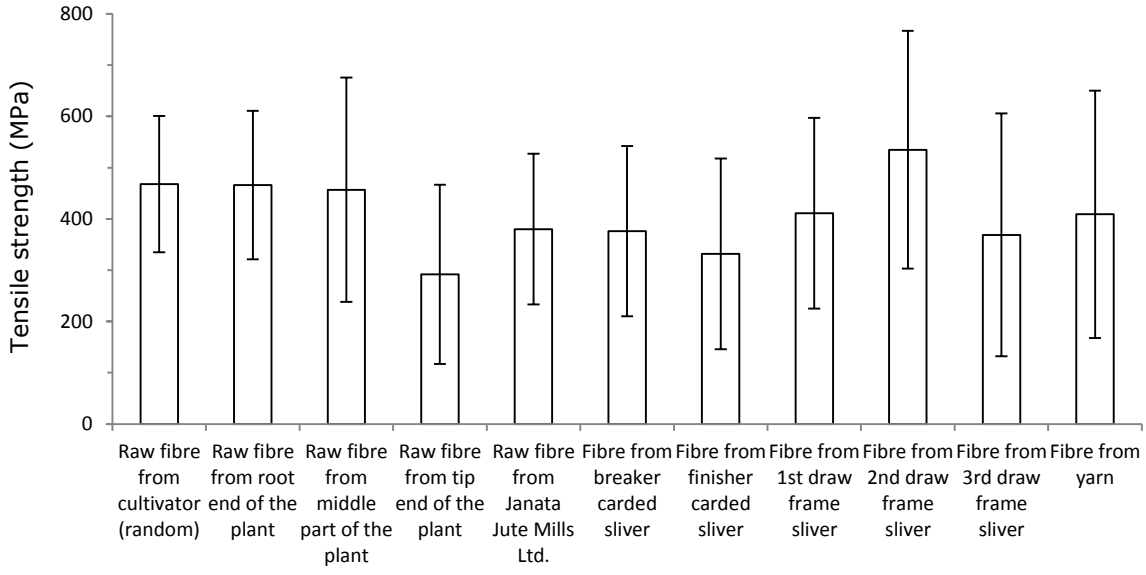


Fig. 7.6 Tensile strength of single jute fibres

Exceptionally high standard deviation is observed for tensile strength revealing that very a wide range of data is obtained for tensile strength of fibres which is due to the inherent character of jute fibres. No specific relation is observed between tensile modulus and tensile strength of fibres studied in this chapter.

7.5.1.(IV) Strain at failure of fibres

Single fibres from root end of the plant show the highest percentage strain at failure (Table 7.1 and Fig. 7.7) with respect to other fibres and fibres from the tip end present the lowest percentage strain at failure. Strain at failure of fibres from the middle of the plant is in between that of the root end and the tip end. This phenomenon shows that strain at failure of jute fibres reduces gradually from root to tip and the difference between root and tip is statistically significant ($P<0.05$). ANOVA result also show significant variations ($P<0.05$) in strain at failure of single jute fibres tested in this chapter. Strain at failure of fibres from the root end and from random position is very similar. Strain at failure of fibres supplied by Janata Jute Mills Ltd. and the property of fibres collected from intermediate products as well as fibres from yarn show no statistically significant ($P<0.05$) differences, revealing that the yarn manufacturing process does not affect this property of jute fibre. Strain at failure of single jute fibres obtained in this chapter is within the range of strain at failure of single jute fibre found by other researchers (see table 4.2). Similar to other properties studied in this chapter strain at failure also shows very high standard deviations which is due to the inherent character of jute fibre.

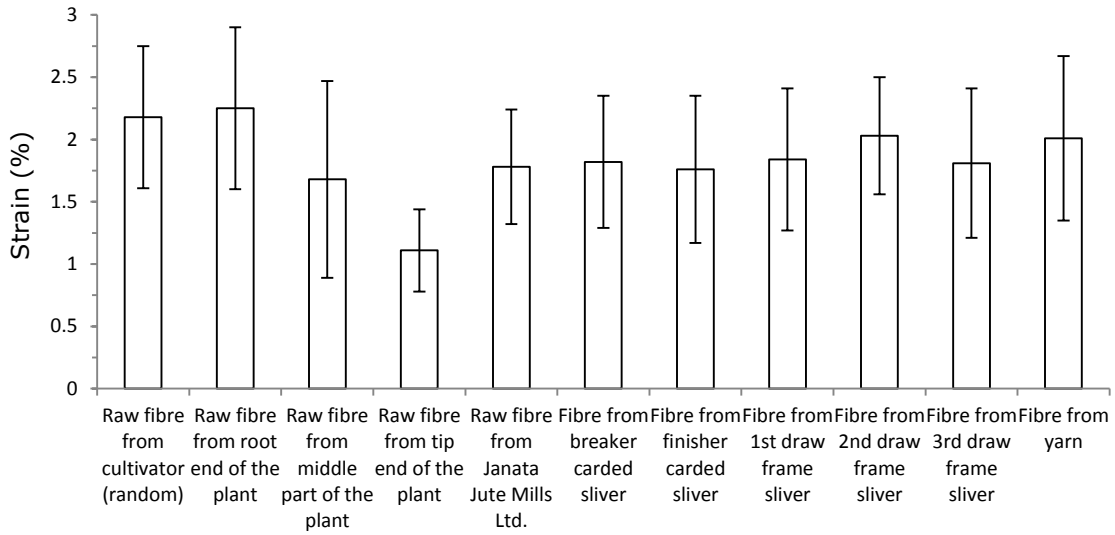


Fig. 7.7 Strain at failure of single jute fibres.

7.5.1.(V) Force-displacement curves of single fibres

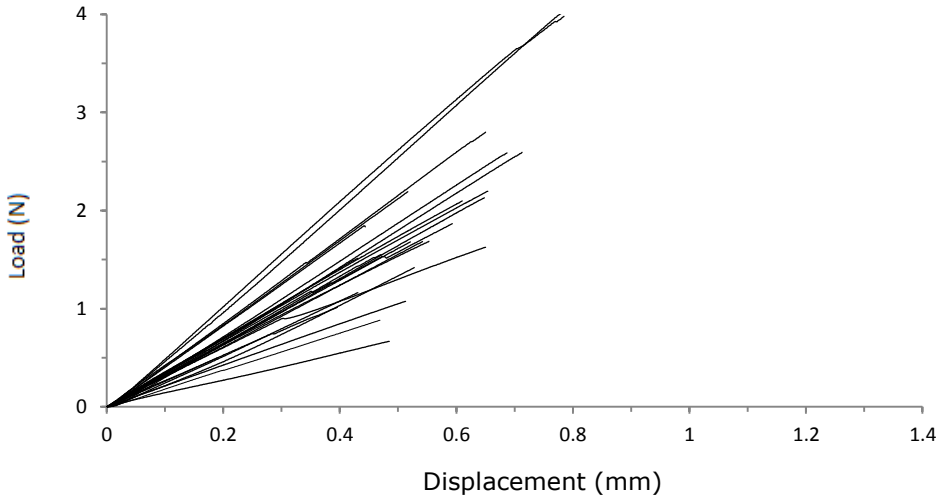


Fig. 7.8 Force-displacement curves of single fibres extracted from raw jute received from cultivator (random position).

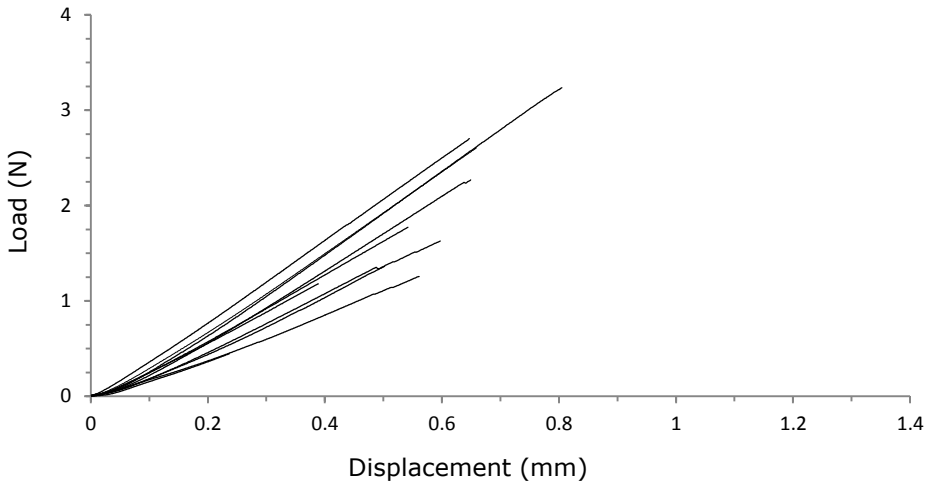


Fig. 7.9 Force-displacement curves of single fibres extracted from the root end of the plant.

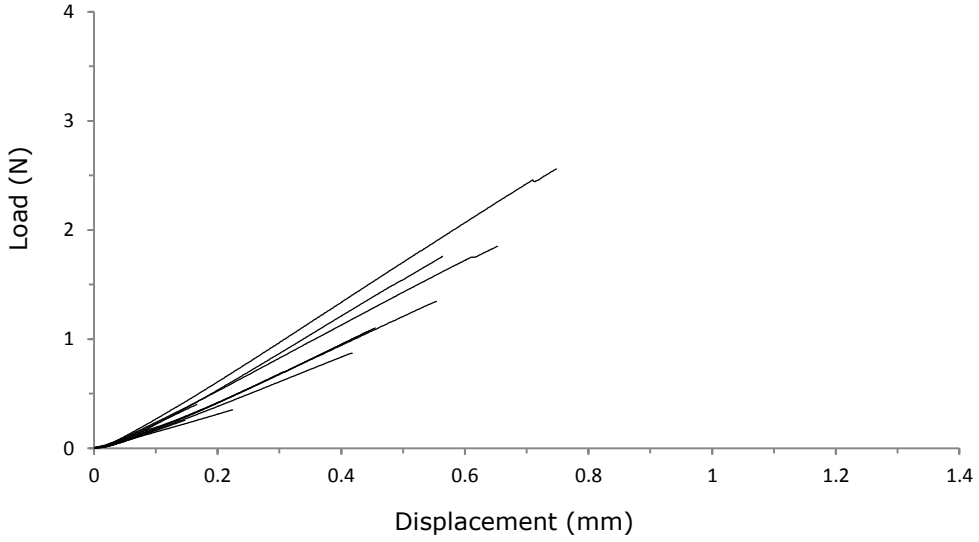


Fig.7.10 Force-displacement curves of single fibres extracted from the middle part of the plant.

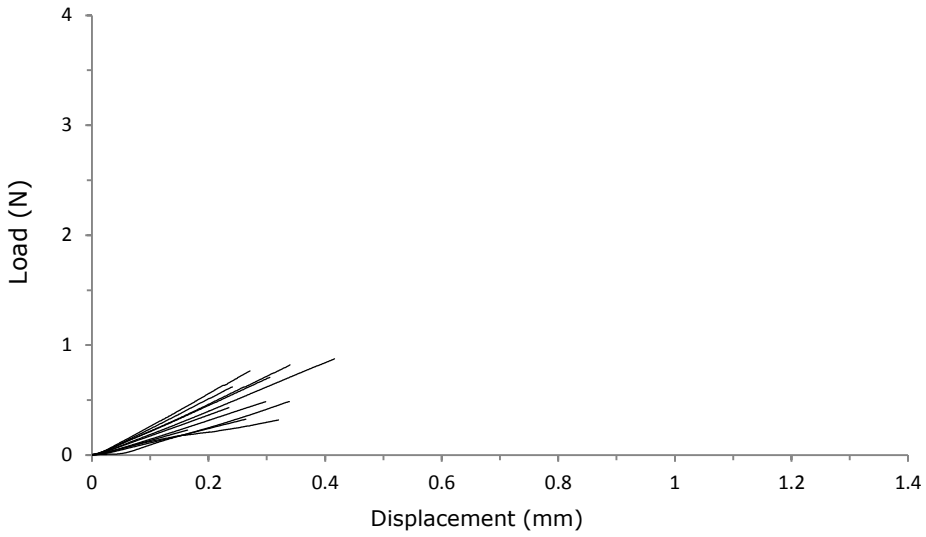


Fig.7.11 Force-displacement curves of single fibres extracted from the tip end of the plant.

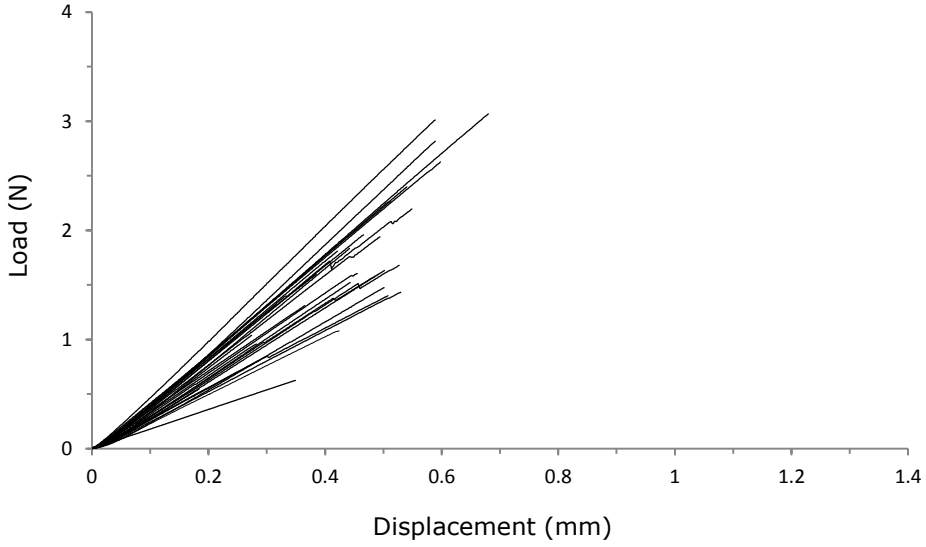


Fig. 7.12 Force-displacement curves of single fibres extracted from raw jute received from Janata Jute Mills Ltd.

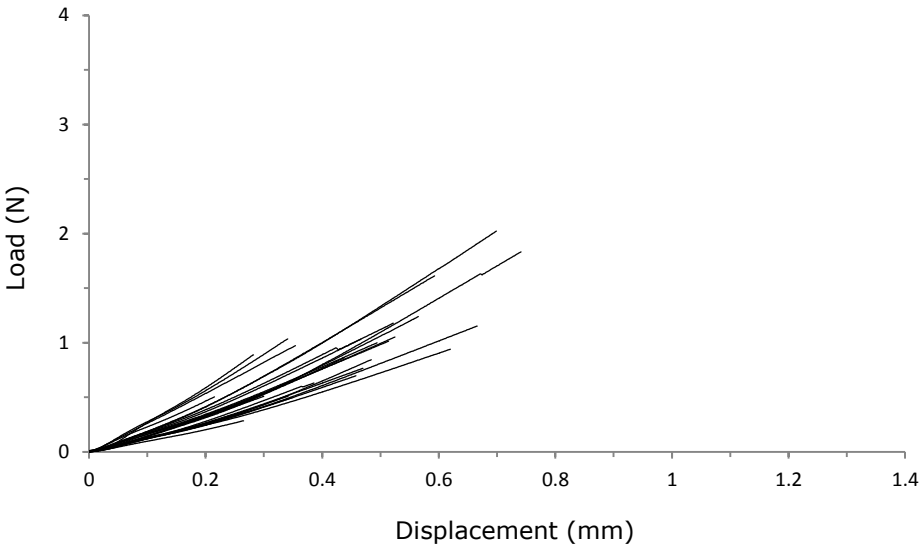


Fig. 7.13 Force-displacement curves of single fibres extracted from breaker carded sliver.

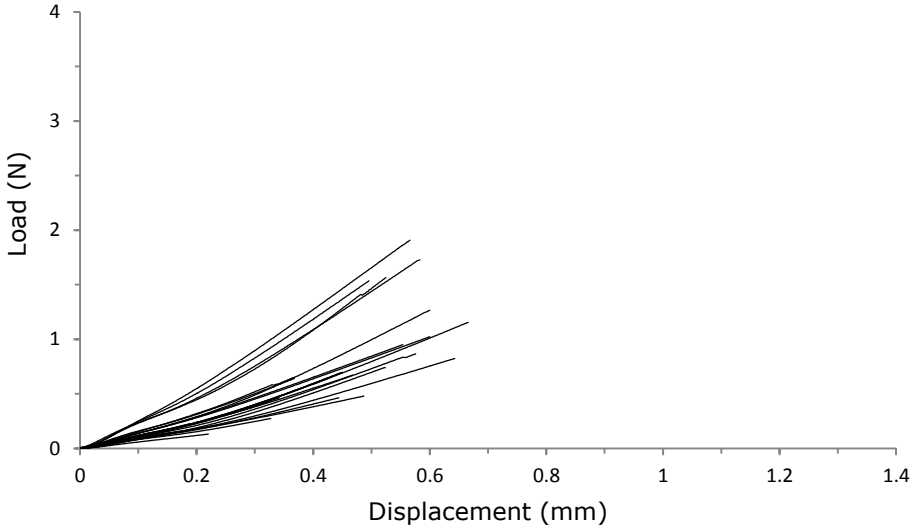


Fig. 7.14 Force-displacement curves of single fibres extracted from finisher carded sliver.

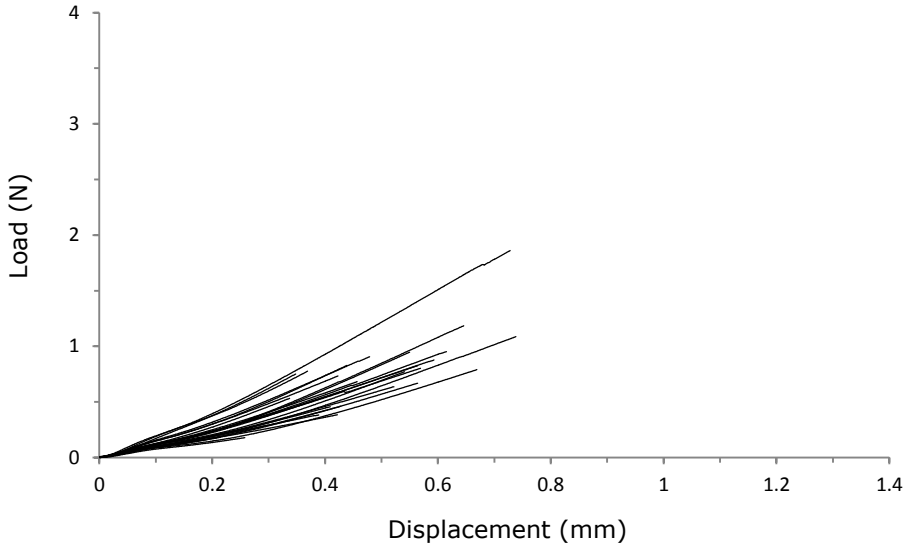


Fig. 7.15 Force-displacement curves of single fibres extracted from 1st draw frame sliver.

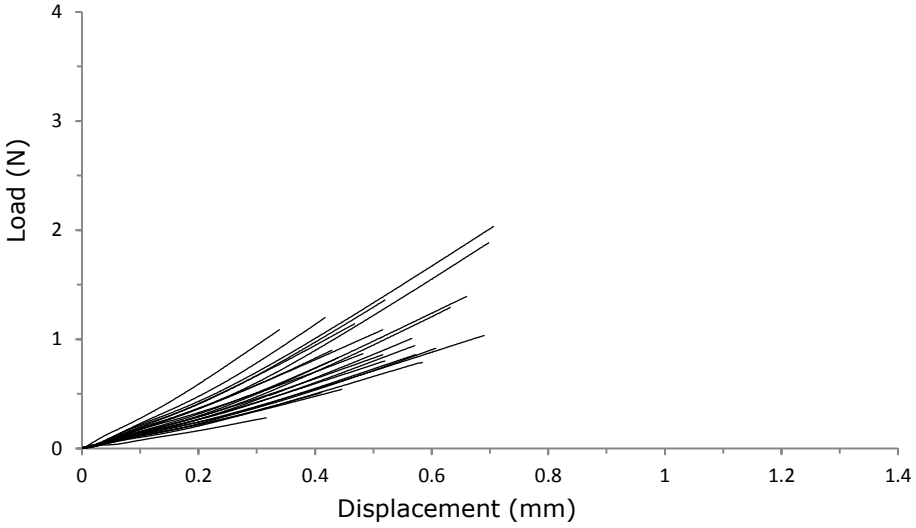


Fig. 7.16 Force-displacement curves of single fibres extracted from 2nd draw frame sliver.

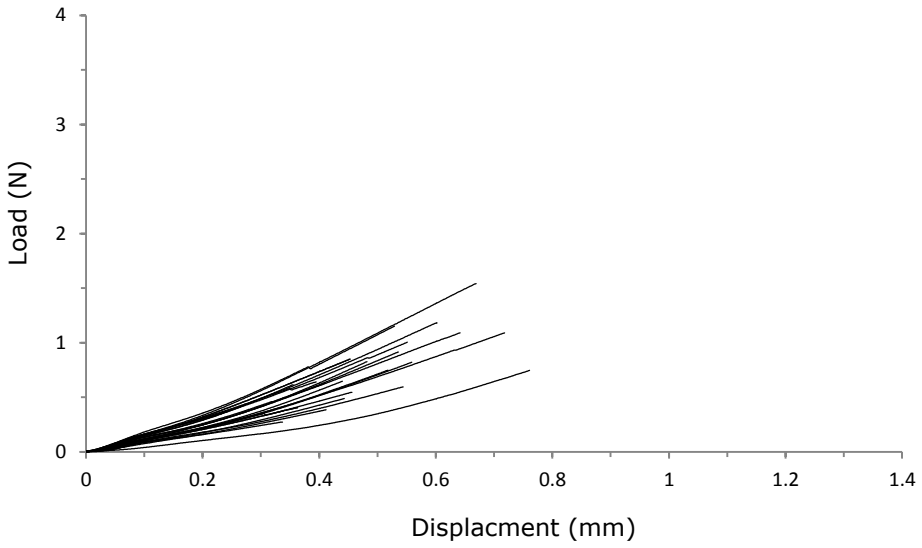


Fig. 7.17 Force-displacement curves of single fibres extracted from 3rd draw frame sliver.

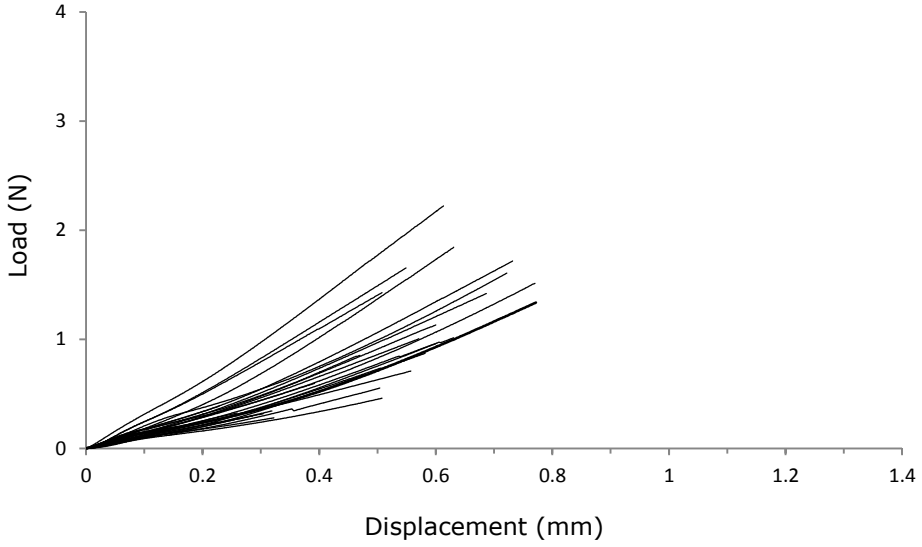


Fig. 7.18 Force-displacement curves of single fibres extracted from raw yarn

Force-displacement curves of single fibres studied in this chapter are presented in Fig. 7.8 to Fig. 7.18. Very similar force-displacement curves are obtained for fibres extracted from raw jute, intermediate products and yarn,

revealing that the spinning process does not influence the force-displacement curves of jute fibre. The characteristics of curves found in this chapter are similar to curves obtained in previous chapters, with the explanation for their shape given in Section 4.5.1 expected to apply here also.

7.5.2 Fibre diameter/cross-section correction factor

Accurate measurement of fibre diameter/cross-section is a requirement to determine the tensile properties of fibre. As mentioned in Section 4.3.4 common practice to determine the fibre diameter is to use the mean value of fibre width, which is measured at several locations along the fibre length and the cross-section of fibres is considered circular [292-295, 331]. However the cross-section of plant fibres is not circular (see Fig. 7.19) and is not uniform along the fibre length (see Fig. 7.20). Thus inclusion of an error is inevitable. Virk et al. [292, 294, 331] measured the apparent cross section of jute fibres (average width of fibre of several readings and considering circular cross-section of fibre) and the true cross-section of jute fibre (measured actual cross-section of fibres using image analysis) [332] and determined a fibre area cross-section correction factor of 1.42 [333], which is the ratio between apparent cross-sectional area and true cross-sectional area. This suggests that the cross-sectional area of jute fibre is over estimated when above mentioned procedure is followed. Thus tensile modulus and tensile strength are under estimated.

In this work images of fibre cross-sections are taken by a Zeiss optical microscope (preparation and process are explained in Section 4.3.4) and these are analysed to measure the fibre diameter/cross-section correction factor. 60 fibres are considered to measure the width and subsequently cross-

sectional area, considering the cross-sections of fibres to be circular. The actual cross-sectional area of those 60 fibres is measured using the image analysis software Image J. After calculation; diameter and cross-sectional area correction factors are found to be 1.19 (the ratio between apparent and actual diameter) and 1.42 (the ratio between apparent and actual cross-sectional area) respectively (very similar to the fibre cross-sectional area correction factor determined by Virk et al. [333]).

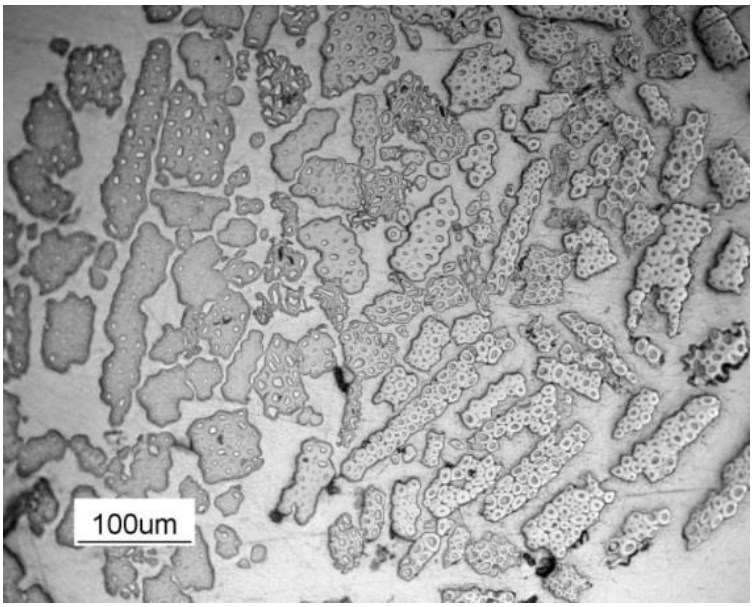


Fig. 7.19 Cross-sectional view of jute fibre

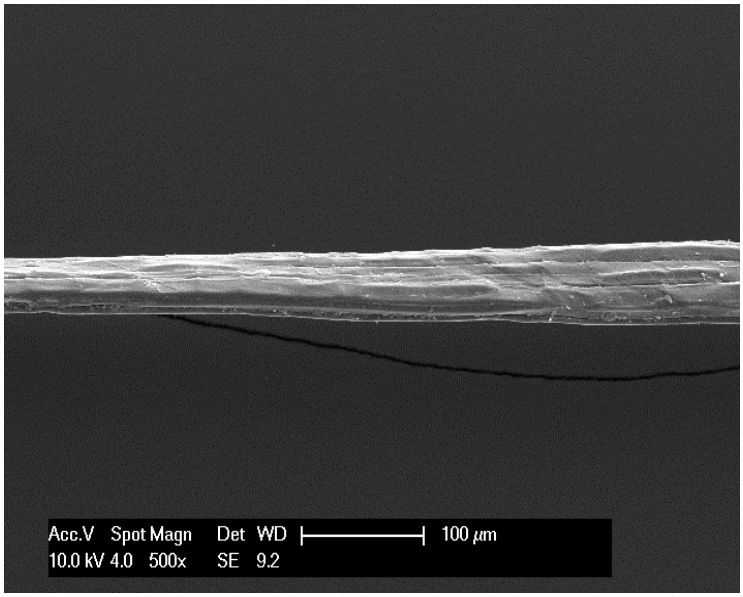


Fig. 7.20 Longitudinal view of jute fibre

7.5.3 Composites test results

Table 7.2 Fibre volume fraction, tensile test results of composites and tensile properties of resin

Composites manufactured with	V_f (%)	Tensile modulus (GPa)	Std. dev.	Tensile strength (MPa)	Std. dev.	Strain at failure (%)	Std. dev.
Root end of fibres	45	30.8	1.5	303	10	1.07	0.05
Breaker carded sliver	34	13.2	1.1	139	10	1.48	0.07
Finisher carded sliver	33	12.3	0.2	126	8	1.48	0.04
Tossa jute 1*	37	25.2	2.0	257	18	1.10	0.09
Tossa jute 2*	49	33.3	2.9	315	24	1.05	0.08
Yarn HEC sized	38	19.7	0.8	207	5	1.40	0.03
Resin	-	3.6	-	55		1.6	-

V_f = Fibre volume fraction, Std. dev. = Standard deviation, * These two composite plaques are made with the same raw fibre and the number 1 and 2 are used to notify the composites plaque.

The composite plaques manufactured and tested in this chapter have a large variation of fibre volume fraction (33-49%) and the cause is explained in Section 5.5.2. The old aluminium mould is used to manufacture composite plaques made with breaker carded sliver, finisher carded sliver, tossa jute 1 and yarn. The new steel mould is used to manufacture composite plaques made with the root end of fibres and tossa jute 2. A similar amount of fibres is used for composite plaques manufactured with root end of fibres and tossa jute 1 and composites made with root end of fibres gave higher fibre volume fraction. Very low fibre volume fraction is obtained for composites manufactued with breaker carded sliver and finisher carded sliver. This is due to the constraint associated with sliver thickness. Two plies of sliver are used to manufacture composites as three plies of sliver hinders the ability to close the tool properly. However high fibre volume fraction of composites manufactured with raw jute fibre (tossa jutw 2) is achieved to compare the results with other composite plaques. Tensile modulus and tensile strength of composites as well as the back calculated fibre tensile modulus and tensile strength (obtained using the rule of mixtures as described in Section 3.5.6) are presented in Table 7.2 (raw data are presented in Appendix E) and Table 7.3 respectively.

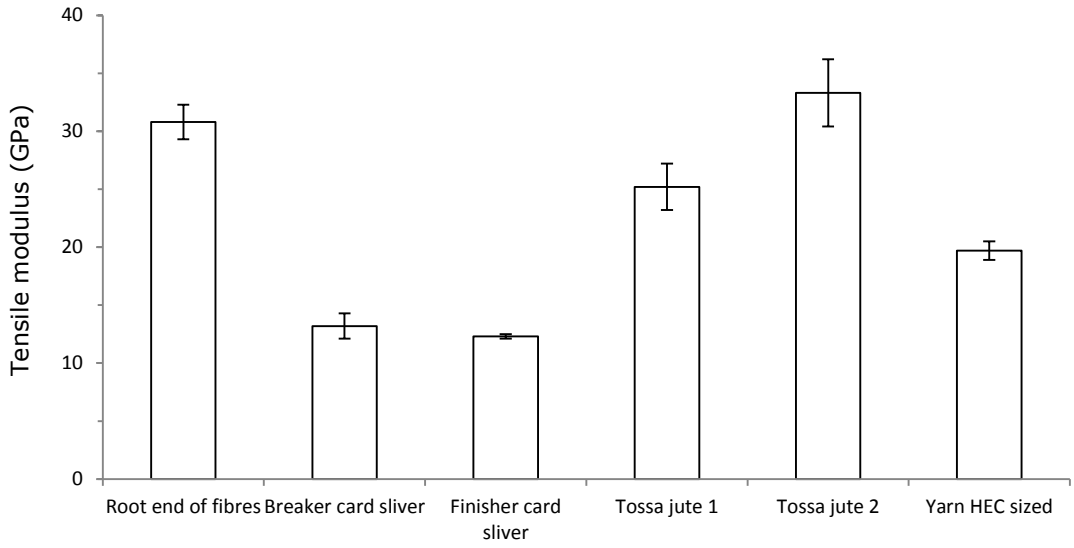
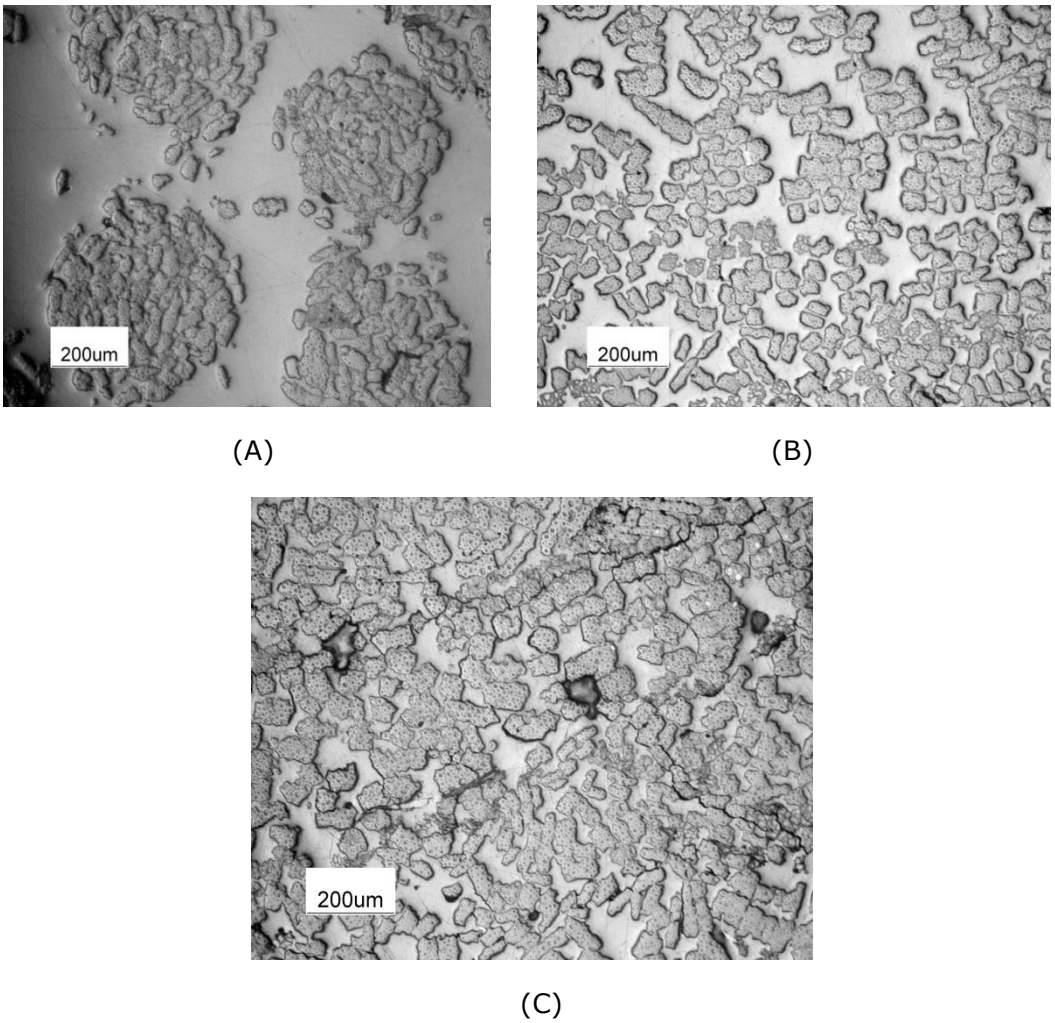


Fig. 7.21 Tensile modulus of composites (error bars indicate standard deviation)

Composites made with yarn and tossa jute 1 have very similar fibre volume fraction (Table 7.2) and composites made with tossa jute 1 show a 28% higher tensile modulus with respect to that (Table 7.2 and Fig. 7.21) of composites made with jute yarn and this difference is statistically significant ($P<0.05$). A major cause of the increase in tensile modulus is better alignment of fibres acheived by composites made with fibres with respect to composites made with yarn. Twist brings a spiral configuration of fibres in yarn, causing off axis loading which reduces tensile properties [13].

With increasing fibre volume fraction for the composite plaque made with tossa jute 1 to the root part of fibres, tensile modulus increases and a further increase in tensile modulus is also observed for composites made with tossa jute 2 which possesses the highest fibre volume fraction among the composite plaques manufactured in this work. Composites made with tossa jute 2 show the highest tensile modulus in this work. Back calculated fibre tensile modulus

of fibres for these three composite plaques show very similar values (Table 7.3 and Fig. 7.23) revealing the utilisation of fibre tensile modulus is very similar in these composites and the variation of absolute values is only due to variation of fibre volume fraction.



(A) (B)

(C)

Fig. 7.22 Cross-sectional view of composites; made with yarn (A), tossa jute 1 (B) and tossa jute 2 (C)

Composites made with breaker carded and finisher carded slivers show very low tensile moduli (Table 7.2 and Fig. 7.21) and these are approximately 50% than that of composites made with tossa jute 1. Low modulus fibre of different origin, low fibre volume fraction, off axis loading of fibres due to

misalignment and using unsized fibrous mat are some causes of reduced tensile modulus for these two composite plaques. The back calculated fibre modulus of these two composite plaques is nearly 50% lower with respect to other composite plaques made with raw fibres which is statistically significant ($P<0.05$) and the causes mentioned above (except fibre volume fraction) also apply.

Table 7.3 Back calculated tensile properties of fibres

Composites manufactured with	Tensile modulus (GPa)	Standard deviation	Tensile strength (MPa)	Standard deviation
Root end of fibres	64.0	3.4	606	23
Breaker card sliver	31.8	3.2	303	29
Finisher card sliver	30.1	0.7	269	24
Tossa jute 1	61.9	5.4	576	49
Tossa jute 2	64.3	5.9	586	49
Yarn HEC sized	46.0	2.1	455	12

Lower values of standard deviation are noticed for composites made with breaker carded sliver, finisher carded sliver and yarn with respect to composites made with raw fibres (root, tossa jute 1 and tossa jute 2). Manual preparation of UD mat with raw fibres (i.e. wrapping raw fibres on glass fibre composite board by hand) includes huge variation of fibre density (see Fig. 7.3) and thus high localised variation of fibre volume fraction in a single plaque occurs, thus a wider range of tensile modulus is observed resulting in higher values of standard deviation. Mechanically produced breaker carded sliver, finisher carded sliver and yarn are more even with respect to manually prepared UD fibrous mat and thus a lower range of standard deviations are noticed for composite plaques made with slivers and yarn.

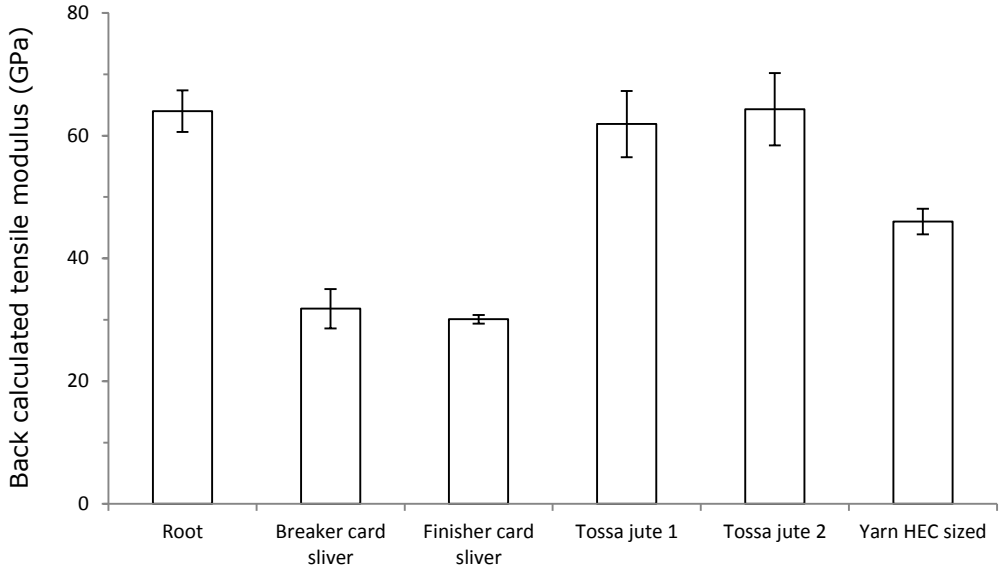


Fig. 7.23 Back calculated values of tensile modulus of fibres (error bars indicate standard deviation)

Composites made with tossa jute 2 present the highest tensile strength with respect to all other composite plaques manufactured in this chapter, followed by composites made with root end of fibres, followed by tossa jute 1, followed by composites made with yarn, followed by composites made with breaker carded sliver and composites made with finisher carded sliver (Table 7.2 and Fig. 7.24). This trend is similar to that for tensile modulus of composites. Composites manufactured with tossa jute 1 show a 24% increase in tensile strength with respect to composites manufactured with yarn and this difference is statistically significant ($P<0.05$). These two composite plaques possess very similar fibre volume fraction and the improvement in tensile properties of composites manufactured with fibre is due to better alignment, more uniform distribution (Fig.7.22) and reduction of off-axis loading with respect to composites manufactured with yarn as mentioned earlier. For the same reasons in addition higher fibre volume fraction, composites

manufactured with the root end of fibres and composites manufactured with tossa jute 2 show higher tensile strength. However the differences of back calculated values of fibre tensile strength (Fig. 7.25) of these three composite plaques (tossa jute 1, root end and tossa jute 2) are not statistically significant ($P<0.05$). Thus the variation of tensile strength is due to the variation of fibre volume fraction only and the utilisation of the tensile properties in composites is similar for all these composite plaques.

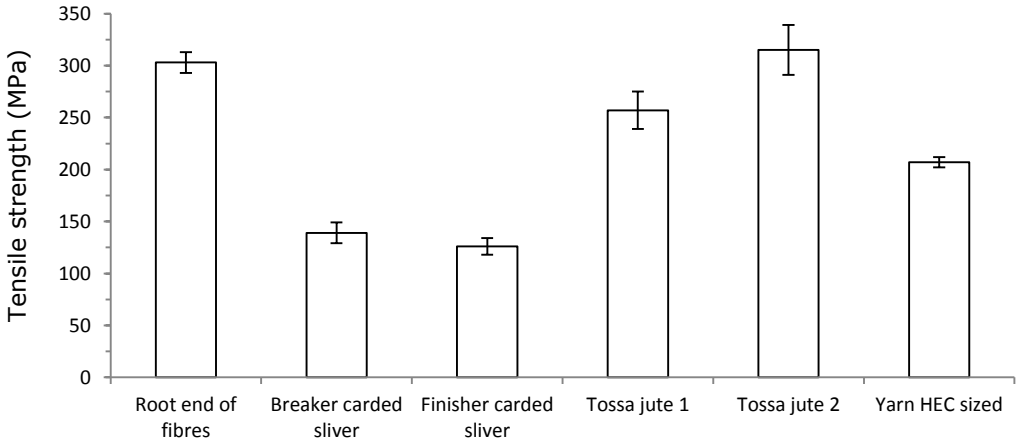


Fig. 7.24 Tensile strength of composites (error bars indicate standard deviation)

Composites manufactured with breaker carded sliver and finisher carded sliver show nearly 50% decrease in tensile strength (Table 7.2 and Fig. 7.24) and nearly 50% decrease in back calculated fibre tensile strength (Table 7.3 and Fig. 7.24) with respect to composites manufactured with tossa jute 1. These differences are due to lower fibre volume fraction (valid only for tensile strength), not using HEC, misalignment of fibres and fibres from different origin for composites manufactured with breaker carded and finisher carded sliver. On the other hand composites manufactured with raw jute (tossa jute 1, tossa jute 2 and root end of fibres) possess high fibre volume fraction and

better fibre alignment. The trend of standard deviation of tensile strength is similar to that of tensile modulus of composites and the causes explained for tensile modulus are also expected to be applicable for tensile strength.

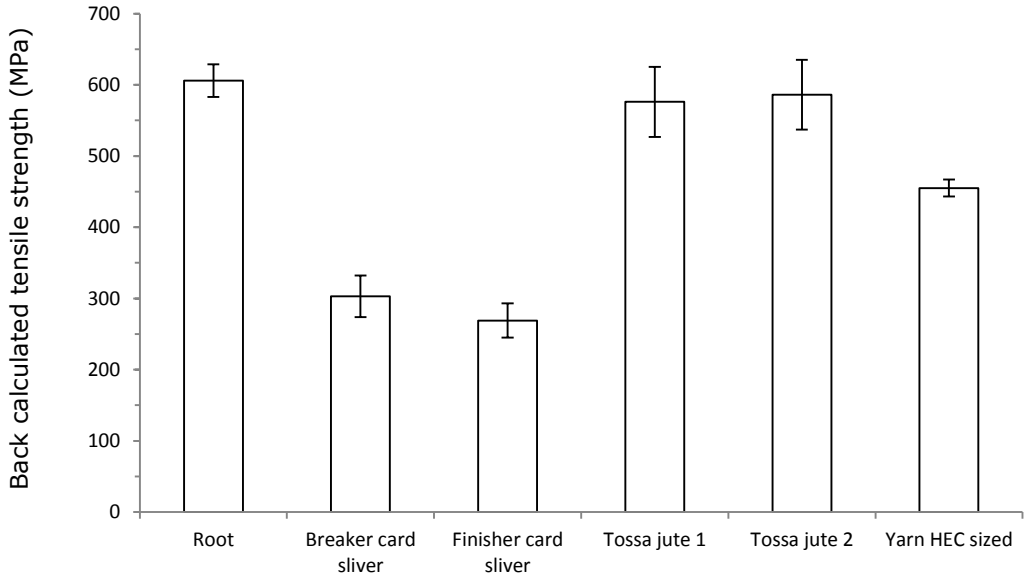


Fig. 7.25 Back calculated values of tensile strength of fibres (error bars indicate standard deviation)

UD jute composites manufactured with untreated raw fibres present high tensile properties 30.8 GPa tensile modulus (60% fibre weight fraction and polybutylene succinate matrix [193]) and 225 MPa tensile strength (50% fibre volume fraction and polyester matrix [196]). However in this work composites manufactured with raw fibre (49% fibre volume fraction) show impressively high tensile properties (33.3 GPa tensile modulus and 315 MPa tensile strength)

Strain at failure of the composite plaques studied in this chapter is presented in Table 7.2 and Fig. 7.26. Strains at failure of the composites made with slivers and yarn are 27-40% higher with respect to that of composite plaques

manufactured with raw fibres and these differences are statistically significant ($P<0.05$). A possible cause of these differences is the alignment of fibres in composites. Application of load in composites in the longitudinal direction of yarn or sliver may cause small straightening on reinforcement fibres due to the helical or wavy arrangement which possibly cause higher extension resulting higher strain at failure of composites made with slivers and yarn. From Fig. 7.26 it can be seen that the fibre volume fraction does not affect the strain at failure, as also found by other researchers [12]. Madsen et al. [12] also studied the influence of off-axis loading (0°, 10°, 20°, 30°, 45°, 60° and 90°) on the strain at failure, of composites. They found the highest strain

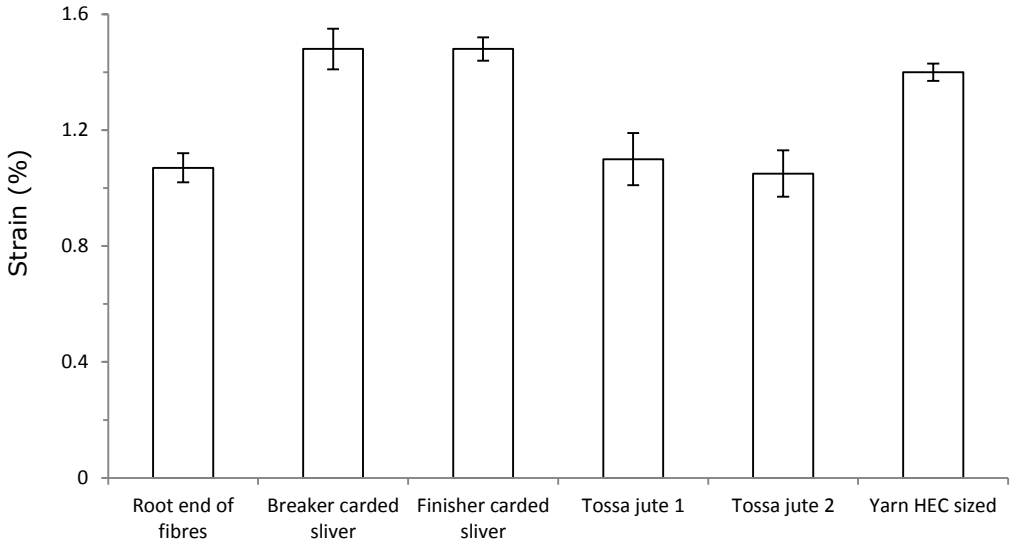


Fig. 7.26 Strain at failure of composites (error bars indicate standard deviation)

at failure for 0° loading and it decreases with increasing obliquity and was found to be lowest for 90°. However they used twisted hemp yarns as reinforcement material and the findings may not be comparable for the composite plaques made with aligned jute fibres.

The back calculated values of fibre tensile modulus and tensile strength from composites made with raw jute fibres are found to be between 61.9 – 64.3 GPa and 576– 606 MPa (Table 7.3) respectively. In this work tensile modulus and tensile strength of raw fibres are found to be 19.4-25.3 GPa and 292-468 MPa (Table 7.1) respectively where the width of the fibre is measured at three places and the mean value is considered as the effective fibre diameter assuming a circular cross-section. The values of the same parameters for fibres are found to be 45.4 GPa and 1108 MPa (Table 5.1) respectively when the thinnest places of fibres are considered as the fibre diameter. The fibre diameter correction factor is calculated in this work and it is found to be 1.19. Using the fibre diameter correction factor tensile modulus and tensile strength of fibres are found to be 28-36 GPa and 415-665 MPa respectively when mean diameter is considered as the effective diameter. Tensile modulus and tensile strength of fibres are found to be 64.5 GPa and 1.5 GPa respectively using the fibre diameter correction factor when the thinnest parts of fibres are considered as effective fibre diameter. The tensile moduli of fibres obtained considering the thinnest width of the fibre as the effective fibre diameter and correcting it using the correction factor is found to be very similar to the back calculated fibre tensile modulus achieved from the composites made with fibres. However the tensile strength of fibres obtained considering the mean value of width of three places as effective fibre diameter and correcting it using the correction factor is found to be very similar to the back calculated value of tensile strength achieved from the composites made with fibres. Apparently it seems that tensile modulus of jute fibres depends on the minimum diameter of fibres and tensile strength of jute fibre depends on the average diameter of fibres. It also be the case that the strength of jute fibres

in composites can still not be utilised fully due to poor bonding between hydrophilic fibre and hydrophobic resin. Thus composites show poor tensile strength and possibly 1.5 GPa is the real strength of jute fibre. As tensile strength depends on failure of composites hence it is necessary to achieve the highest possible bonding strength between fibre and matrix for proper utilisation of the tensile strength of fibres in composites. A number of research studies have been performed to overcome this limitation as discussed in Section 2.5.3.(II).

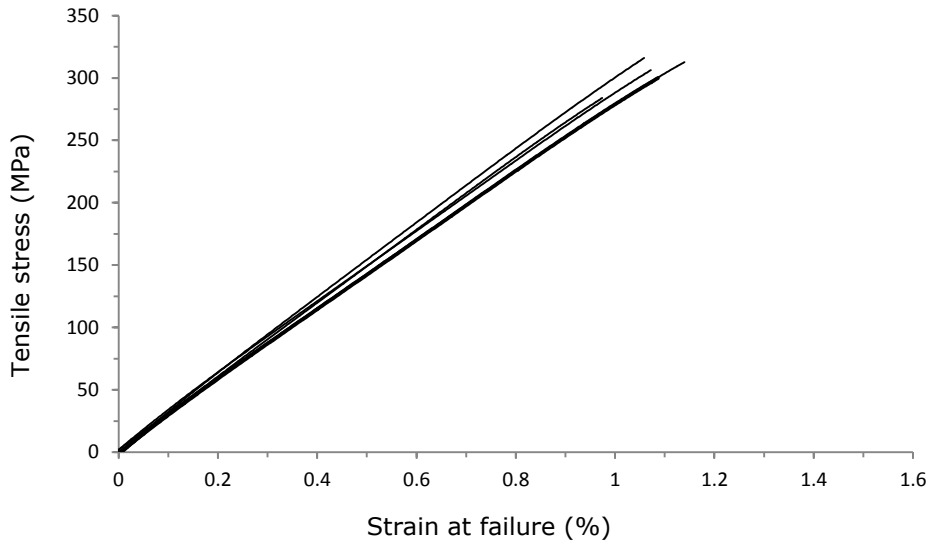


Fig. 7.27 Stress-strain curves of composites manufactured with the root end of fibres

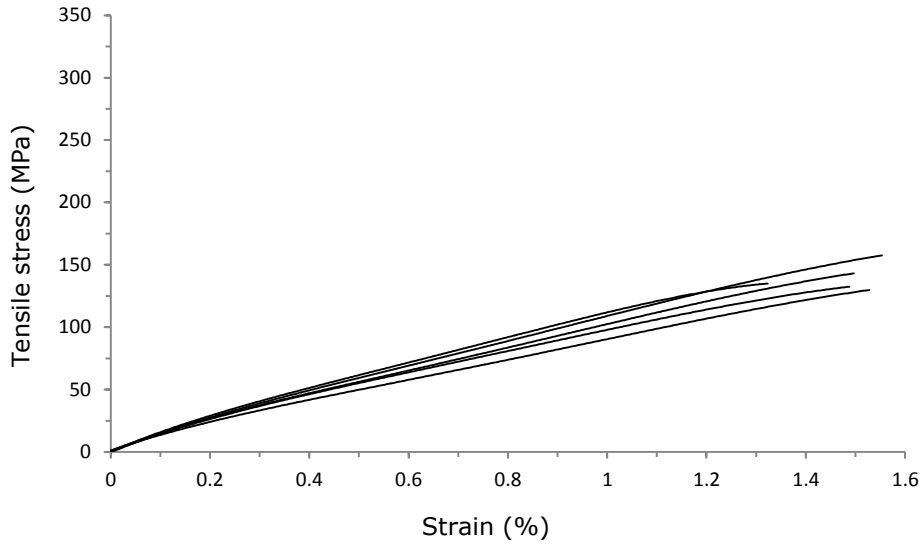


Fig. 7.28 Stress-strain curves of composites manufactured with breaker carded sliver

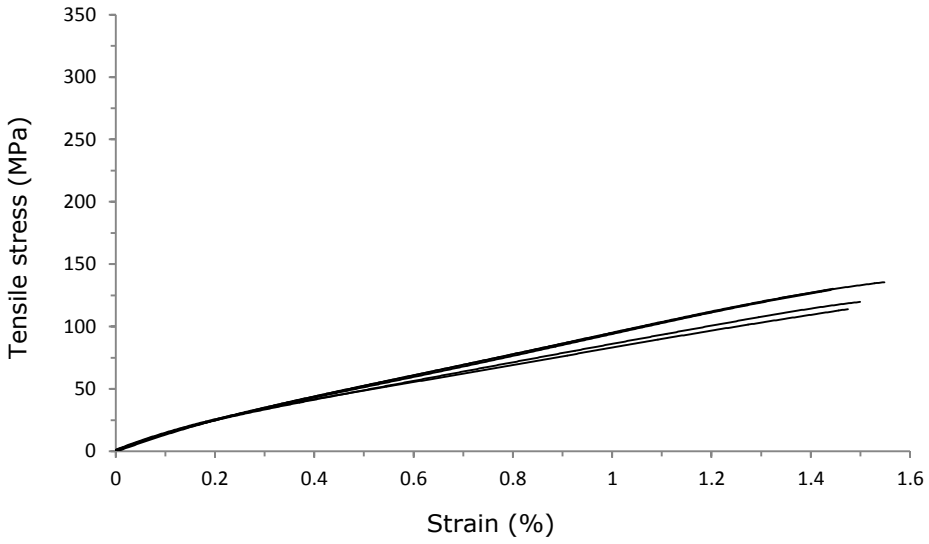


Fig. 7.29 Stress-strain curves of composites manufactured with finisher carded sliver

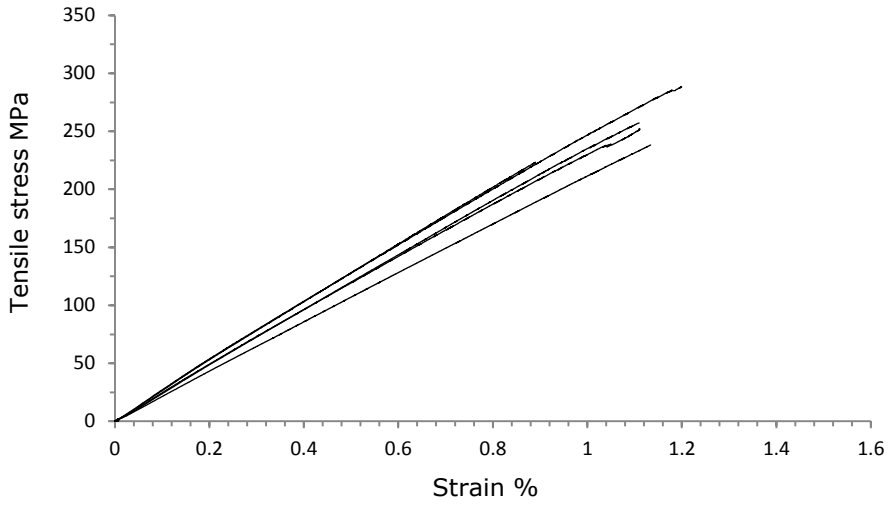


Fig. 7.30 Stress-strain curves of composites manufactured with tossa jute 1

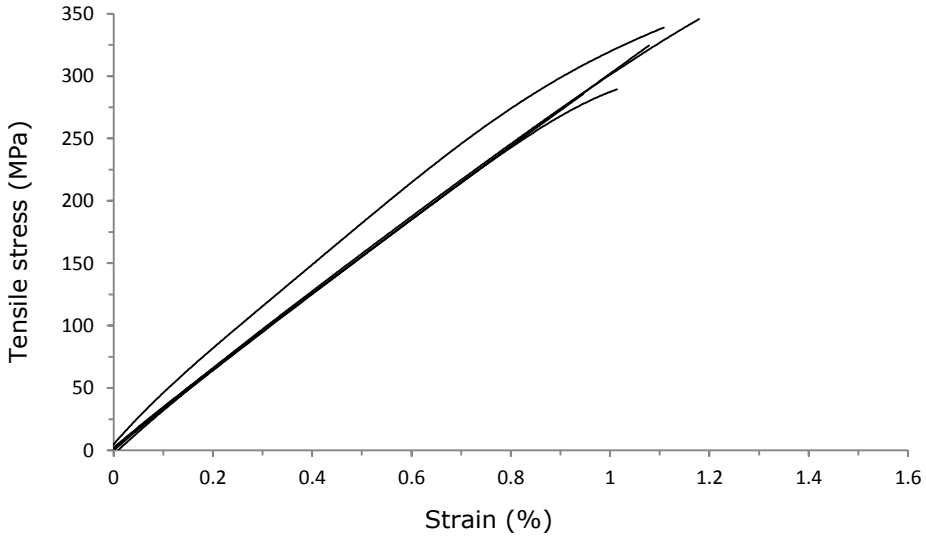


Fig. 7.31 Stress-strain curves of composites manufactured with tossa jute 2

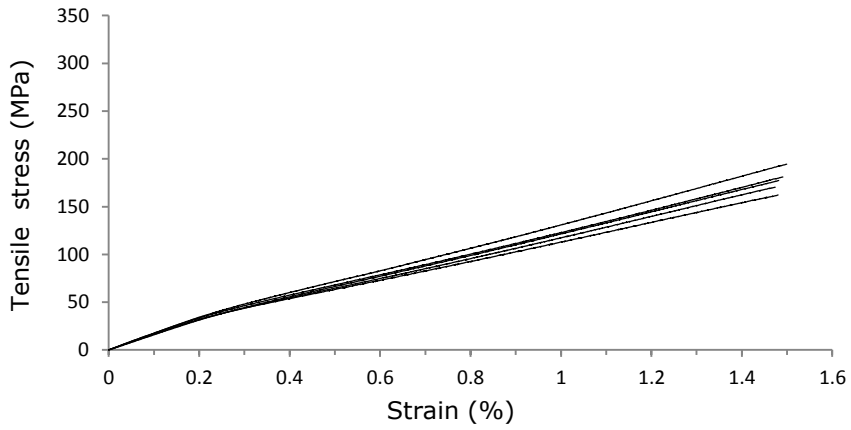


Fig. 7.32 Stress-strain curves of composites manufactured with raw yarn

Stress-strain curves of composites manufactured with root end of fibre, breaker carded sliver, finisher carded sliver, tossa jute 1, tossa jute 2 and yarn are presented in Fig. 7.27 to Fig. 7.32. The behaviour of stress-strain curves of composites made with raw fibre and of composites made with sliver and yarn are not similar. Quasi linear stress-strain curves are found for composites made with raw fibres and the stress-strain curves of composites made with slivers and yarn possess an initial linear portion, an extended curved region and are again linear up to failure which is similar to the characteristics of stress-strain curves of composites obtained in previous chapters as discussed in Section 3.5.6.(I). When micro-cracking of matrix starts possibly composites extend due to minor straightening of fibres for composites manufactured with yarns or slivers due to helical misalignments of fibres which ultimately presents a higher strain at failure with respect to composites manufactured with raw fibre. Nearly straight orientation of fibres in the longitudinal direction for composites manufactured with fibres prohibited any straightening of fibres that may have occured for yarn reinforced composites, resulting in no bending region as well as lower strain

to failure for composites manufactured with fibres with respect to composites manufactured with slivers and yarns.

7.6 Conclusions

Tensile properties of single jute fibres obtained from raw jute and yarn do not show any statistically significant differences ($P<0.05$) revealing that mechanical processing due to spinning does not deteriorate the tensile properties. Tensile moduli of single jute fibres tested from different positions of the plant are found to be identical. However tensile strength and strain at failure of fibre from the tip are significantly lower ($P<0.05$) than those of fibres from the root and the middle reveals localised variation of fibres. Tensile modulus of fibres from cultivator is significantly higher ($P<0.05$) than that of fibres extracted from breaker carded sliver, finisher carded sliver, 1st draw frame sliver, 3rd draw frame sliver and yarn. Possible cause of these differences is different origin of fibres. Similarly to other chapters large standard deviations of single fibre mechanical properties are also observed here.

UD composites made with raw jute fibres show impressively high values of tensile modulus and tensile strength with respect to composites made with yarns. Several detrimental factors such as off-axis loading, poor fibre distribution in composites and the compact structure of twisted yarn may be responsible for reducing the tensile properties of yarn reinforced composites. Due to better alignment and more even distribution of fibres, fibre reinforced composites show higher mechanical performance with respect to yarn reinforced composites. However composites made with yarn and sliver show higher strain at failure and less standard deviation in mechanical properties

with respect to those of composites manufactured with fibres. Linear fibrous mass is more even for mechanically produced slivers and yarn than fibrous mat produced manually by wrapping on glass fibre composites board.

It is noticed that back calculated tensile moduli of jute fibre computed from the tensile modulus of fibre reinforced composite plaques are comparable to the tensile modulus found when the thinnest part of fibre is used as the effective fibre diameter and is corrected with the fibre diameter correction factor determined in this work. However back calculated tensile strengths of fibre determined from fibre reinforced composite plaques are found more comparable with those obtained when the mean value of fibre width is considered as the effective fibre diameter.

Application of raw fibre for manufacturing of composites can eliminate some processing steps that are necessary to manufacture yarn as well as fabric, which may reduce the fibre processing expense.

CHAPTER 8

DISCUSSION AND CONCLUSIONS

8.1 Introduction

This chapter includes an overall discussion of the thesis. The main points of each chapter are summarised here including the conclusions. Some recommendations for future work are also given at the end of this chapter.

8.2 General Discussion

The influence of jute yarn hairiness on the mechanical properties of UD composites was studied in Chapter 3. To understand the effect of burning yarn hairs on the mechanical properties of yarns, both hairy and hairless yarns were tested for tensile properties, thermal analysis (DSC), identification of functional groups (FTIR), and surface chemical analysis (XPS). Results of these experiments showed no marked differences in properties between hairy and hairless yarns. UD composite plaques were manufactured with hairy and hairless yarns and subsequently tensile tests of composites were performed in both longitudinal and transverse directions with respect to the fibres. The results revealed a significant increase in tensile modulus and a significant decrease in strain at failure of composites manufactured with unsized hairy yarn in the longitudinal direction with respect to those of composites manufactured with unsized hairless yarn. Bonds between yarn hairs and the matrix were the possible cause to increase the tensile modulus, however the cause of decreasing strain at failure of composites manufactured with hairy yarn is unknown. A significant increase in tensile strength was found for composites manufactured with sized hairless yarns than composites

manufactured with sized hairy yarns in the transverse direction. The cause for this effect is unknown. Strain at failure of composites manufactured with unsized hairy yarn was significantly higher than that of composites manufactured with unsized hairless yarn in the transverse direction. Hairs of unsized hairy yarn created bonds with the matrix that increased the strain at failure of composites in the transverse direction. SEM EDX spectra obtained in this work confirmed that lumens of jute fibres were filled with resin, thus they did not contribute to the void content of composites.

Before investigating the influence of surface treatment in aqueous solution, it was important to understand if there was any change in tensile properties of jute due to water treatment. This phenomenon was studied in Chapter 4. To understand the effect of water treatment on jute, yarns with and without tension were dipped in water and then dried. Single fibre tensile test results showed that water treatment (with or without tension) did not affect the tensile properties. However water treatment of jute yarn with tension increased failure stress by 6% and water treatment of jute yarn without tension decreased failure stress by 23% with respect to raw yarns. The rise or fall in tensile strength of water treated yarn (with and without tension) was due to reordering of fibres in the yarn as an effect of shrinkage or the tendency to shrink due to dipping yarn in water [146]. Water treatment of jute yarn in a loose condition caused fibre as well as yarn shrinkage resulting in re-arrangement of fibres in yarns. This can bring higher obliquity of fibres to the longitudinal axis of yarn and higher off-axis loading, eventually contributing to lower ultimate stress. Holding yarn under tension when dipping in water prevented shrinkage which enabled fibres to be more aligned to the yarn axis resulting in higher tensile strength [146]. Composites

manufactured with water treated yarn with tension showed higher tensile properties and composites manufactured with water treated yarn without tension showed lower tensile properties with respect to those of composites manufactured with raw yarn. Rearrangement of fibres in water treated yarn with and without tension played a major role for increasing or decreasing the tensile properties of composites. Composites manufactured with HEC sized yarn also presented higher tensile properties with respect to tensile properties of composites manufactured with a similar type of unsized yarn. Reactive hydroxyl groups (-OH) of HEC created bonds between fibre and matrix which helped to increase the tensile properties of composites manufactured with HEC sized yarn.

Highlighting the tensile properties of fibres as well as yarns it was observed that around 20% tensile modulus and around 45% tensile strength of fibre were utilised in yarn and tensile properties of composites were closer to the tensile properties of fibres than yarn.

Yarns were treated in various concentrated aqueous solution of NaOH with and without tension in Chapter 5. Tensile test results affirmed that the alkali treatments studied in this work did not affect the tensile properties of single fibres. Composites manufactured with alkali treated yarn without tension showed lower back calculated values of fibre tensile strength (determined to remove the influence of fibre volume fraction on properties) with respect to composites manufactured with yarn treated in similar concentrated alkali solution with tension in all cases. These differences were entirely due to treatment of yarn in aqueous solution with and without tension. Composites manufactured with 1% and 3% alkali treated yarn without tension showed

lower (27% and 17% respectively) and composites manufactured with 5% alkali treated yarn with tension show higher (20%) back calculated values of fibre tensile strength with respect to the tensile strength of composites manufactured with raw yarn. A possible cause of inferior back calculated fibre tensile strength of composites manufactured with yarn treated in aqueous solution without tension is mentioned in earlier paragraph. A possible cause of the increase in back calculated fibre tensile strength of composites manufactured with alkali treated yarn with tension is also mentioned earlier paragraph. Some other unknown cause(s) may also be responsible for the increase in back calculated fibre tensile strength of composites manufactured with 5% alkali treated yarn with tension. Alkali treatment did not change the nature of force-displacement curves of fibres and similar behaviour in terms of stress-strain curves was found for composites manufactured with raw and alkali treated yarns.

Cellulose nano-particles (CNP) derived from cotton using an acid hydrolysis technique were used in this work (Chapter 6) to modify the jute yarn surface to understand the influence on the tensile properties of composites. CNP was collected at several steps of the manufacturing stage such as freeze dried CNP, amberlite resin treated non-dried CNP, non-amberlite resin treated non-dried CNP, and these were used to treat the jute yarn in an aqueous medium (amberlite resin treatment was performed to remove non H_3O^+ cations from the CNP suspension). In addition freeze dried CNP was mixed with resin prior to infusion to investigate it's influence on the tensile properties of jute/polyester UD composites. Composites manufactured with freeze dried CNP treated yarn showed inferior back calculated fibre tensile properties in most cases with respect to that of composites manufactured with HEC sized

raw yarn. Huge agglomerations of CNP on the yarn surface are possibly responsible for reduced tensile properties. Composites manufactured with CNP mixed in resin prior to infusion also showed inferior back calculated fibre tensile properties with respect to those of composites manufactured with HEC sized raw yarn. Resin became viscous due to mixing CNP which generated voids resulting in inferior tensile properties of composites. A composite plaque made with 1% CNP mixed in resin showed the lowest strain at failure among the composite plaques manufactured in Chapter 6. Composites manufactured with yarn treated by non-dried CNP showed better back calculated fibre tensile properties with respect to composites manufactured with other CNP treated yarn as well as CNP mixed in resin. Better distribution of non-dried CNP (i.e. reduction of agglomerations which helped to create bonds between fibres and matrix) as well as treatment of yarn in an aqueous CNP suspension with tension (discussed earlier) are the probable causes of enhanced tensile properties of composites. CNP treatment of yarn did not affect the stress-strain curves of composites.

Single jute fibres extracted from raw jute (from random position, from root end, from tip end and from the middle portion of the plant) collected from the cultivator, from different intermediate products during spinning (such as breaker carded sliver, finisher carded sliver, 1st draw frame sliver, 2nd draw frame sliver and 3rd draw frame sliver) and from yarn were tested for tensile properties. Raw fibres obtained from Janata Jute Mills Ltd and fibres extracted from yarn did not show any significant difference in tensile properties revealing that mechanical processing during spinning did not deteriorate the tensile properties of jute fibre. Fibres from different parts of the plant and random position showed similar tensile modulus however significantly lower

tensile strength ($P<0.05$) was found for fibres from the tip with respect to that of fibres from the root, middle or random position. Strain at failure decreased gradually from the root to tip and the difference between root and tip was statistically significant ($P<0.05$). Fibres from the cultivator presented significantly higher ($P<0.05$) tensile modulus than those of fibres extracted from breaker carded sliver, finisher carded sliver, 1st draw frame sliver, 3rd draw frame sliver and yarn. A difference in origin of fibres was a possible cause of these variations (raw fibre from cultivator and others are from Janat Jute Mills Ltd).

Tensile properties of composites manufactured with raw jute fibre, root end of the jute plant, intermediate products of jute spinning (breaker carded sliver and finisher carded sliver) were compared with tensile properties of composites manufactured with jute yarn to understand the influence of fibre alignment, fibre distribution and mechanical processing of fibres due to spinning on the tensile properties of composites. Possessing very similar fibre volume fraction, composites manufactured with raw fibres showed 28% higher tensile modulus and 24% higher tensile strength with respect to those of composites manufactured with jute yarn. A further increase in tensile properties of composites manufactured with jute fibre (69% increase in tensile modulus and 52.37% increase in tensile strength) was observed at increased fibre volume fraction (49%) compared to composites manufactured with yarn. Avoiding off-axis loading and better distribution of fibres in composites were the key factors for better tensile properties of composites manufactured with raw jute fibres and comparatively higher fibre volume fraction was also possible in this case due to better distribution of fibres with respect to yarns, as the yarn diameter was 10-12 times higher than the fibre

diameter. Thus raw jute presented to be a better reinforcement material than yarn. Improved mechanical properties of composites were also observed for composites manufactured with the root end of jute fibres. However poor mechanical properties were found for composites manufactured with breaker carded and finisher carded slivers. The misalignment of fibres, low fibre volume fraction and not using HEC as a binding material were the probable causes of poor mechanical properties of these composite plaques.

Back calculated values of tensile modulus and tensile strength of fibres from composite plaques manufactured with tossa jute 1, tossa jute 2 and fibres from the root end of the plant were very alike, revealing that the utilisation of tensile properties of fibres in composites was very similar for composites manufactured with fibres. Back calculated values of fibre tensile properties of composites manufactured with breaker carded and finisher carded sliver were very poor, revealing that these were not suitable fibre assemblies to manufacture composites with good mechanical properties.

Composites manufactured with raw jute fibres showed inferior strain at failure with respect to composites manufactured with slivers or yarns and all composite plaques manufactured with raw fibres showed very similar strain at failure. On the other hand composites manufactured with slivers and yarns presented very similar strain at failure. Thus it can be commented that helical or wavy alignment of fibres in composites increased the strain at failure which was possibly due to a minor ability of fibres to straighten in the longitudinal direction during application of load.

Tensile properties of fibres are determined considering the mean fibre width from three readings of each specimen (25 mm specimen length) as well as

the thinnest part of the fibre as the effective diameter assuming a circular cross-section. At the same time a fibre diameter correction factor was determined to minimise the error incurred due to assuming a circular cross-section. Back calculated tensile properties of fibres (calculated using the rule of mixtures) were used to compare tensile properties of fibres obtained from tensile testing. The back calculated tensile modulus of fibres obtained from composites manufactured with raw fibres was found comparable with the tensile modulus of fibres when the thinnest part is considered as the effective diameter and corrected with the fibre diameter correction factor. However the back calculated tensile strength of fibres obtained from composites manufactured with raw fibres was found comparable with the tensile strength of fibres when the mean fibre width was considered as the effective diameter and corrected with the fibre diameter correction factor.

8.3 Major Conclusions

- Removing hairs did not affect the tensile properties of yarns.
- Composites manufactured with unsized hairy yarns showed 5.5% higher tensile modulus in the longitudinal direction with respect to composites manufactured with unsized hairless yarn and composites manufactured with unsized hairy yarn showed 86% higher strain at failure in the transverse direction with respect to composites manufactured with unsized hairless yarn
- Water treatment of yarn with tension increased the tensile modulus by 18% over that of raw yarn. Composites manufactured with unsized water treated yarn with tension showed an 8% increase in tensile

strength over that of composites manufactured with unsized raw yarn. However water treated yarn without tension showed a 20% decrease in tensile modulus and 23% decrease in tenacity over those of raw yarn. Composites manufactured with unsized water treated yarn without tension showed 11% decrease in tensile modulus and 10% decrease in tensile strength over those of composites manufactured with unsized raw yarn.

- Tensile modulus of HEC sized water treated yarn without tension increased by 28% compared to unsized water treated yarn without tension. Tensile moduli and tensile strength of composites manufactured with HEC sized yarns (raw, water treated without tension and water treated with tension) increased 5-12% and 9-17% respectively compared to composites made with respective unsized yarns.

- Although composites manufactured with alkali treated yarn with tension showed 22-32% higher back calculated fibre tensile strength with respect to that of composites manufactured with alkali treated yarn without tension, this may be entirely due to water treatment.

- Composites manufactured with non-dried CNP treated yarn showed a 8-16% increase in back calculated fibre tensile modulus and a 14-43% increase in back calculated fibre tensile strength of composites with respect to those of composites manufactured with unsized raw yarn.

- Composites manufactured with raw yarn showed a 28% increase in tensile modulus, a 24% increase in tensile strength and a 21%

decrease in strain at failure with respect to those of composites manufactured with yarn.

- The spinning process did not deteriorate the tensile properties of jute fibre and fibres from the tip end of the plant showed lower tensile strength and lower strain at failure than those of fibres from root.

- The force-displacement curves of single jute fibres showed an initial small linear portion and then an upward curvature up to failure, however the force-displacement curves of single jute yarns show a small initial linear portion, a yield point and again a linear region up to failure in most cases.

- Composites manufactured with raw fibres showed a quasi-linear stress-strain curve and composites manufactured with yarn as well as slivers showed an initial linear portion, an extended curved region and then a linear portion up to failure.

8.4 Summary of original contribution

- Yarn hairiness increased tensile modulus of UD composites in longitudinal direction and strain at failure in transverse direction.
- HEC treatment of yarn increased the tensile properties of composites.
- Water treatment of yarn with tension increased the tensile properties of composites however water treatment of yarn without tension decreased the tensile properties of composites.
- Yarn treated with non-dried CNP increased the tensile properties of composites.

- Composites manufactured with raw fibres showed impressively high tensile properties.

8.5 Future Work

- Related to the causes mentioned that influence the tensile properties of composites manufactured with water treated yarn with and without tension a further study can be performed to find any other causes which influence the tensile properties of composites manufactured with water treated yarn with and without tension. This work was performed at room temperature and the influence of varying the temperature of water can be investigated. Variation of treatment time also can be studied.

- Investigate the influence of soap water treatment at different temperatures and varying treatment time. Soap water treatment may reduce added oil that may adhere due to application of emulsion (mentioned in Section 3.3.1) during spinning and any adhesion of oil from processing machineries. As emulsion contains jute batching oil (mineral based oil) and some lubricants such as gear oil and grease are used for smooth running of textile machinery. There is a possibility of these lubricants adhering to the yarn that may hinder bond formation between fibre and matrix.

- In this work alkali treatment is performed at 1%, 3% and 5% concentrated solution. A study could be performed to treat jute yarn in higher concentrated alkali solutions with and without tension varying

the treatment time and temperature to understand the influence on the mechanical properties of composites.

- As non-dried CNP treatment of yarns improved the tensile properties of composites thus raw fibre can be treated with non-dried CNP to understand the effect on tensile properties of composites.

- In this work yarn and raw fibres were used to manufacture UD composite plaques. Commercially available yarn which was used here is manufactured for textile use and is not suitable for composite manufacturing due to high twist per unit length (4.65 TPI was used for the yarn used in this work). Thus a suitable fibrous product is required to manufacture UD composites to reduce off-axis loading. This may be low twisted yarn or any other suitable product similar to roving or sliver (these are intermediate products of jute spinning). A study could be performed to design a suitable machine to meet this purpose.

- Sizing by HEC is an expensive method as after application of water based HEC, the material needs to be dried. Thus a study can be performed to produce a UD mat of yarn using a mechanical process such as sewing which is used to produce non-crimp fabric and non-woven fabric.

- Investigate the cause as to why back calculated tensile modulus of jute fibre is comparable with tensile modulus of fibre when the thinnest diameter is considered and back calculated tensile strength is comparable with tensile strength of fibre when the mean diameter is considered.

- As tensile strength and strain at failure of fibres from the tip are lower with respect to those of fibres from the root, hence investigate the mechanical properties of composite plaques manufactured with tip and root fibres to understand the localised variation of jute fibre properties on the mechanical properties of composites.

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9. Appendices

9.1 Appendix A: Raw tested data of Chapter 3

Tensile properties of yarn

Serial number	Tensile modulus (GPa)		Tenacity (cN/Tex)		Strain at failure (%)	
	Hairy yarn	Hairless yarn	Hairy yarn	Hairless yarn	Hairy yarn	Hairless yarn
1	5.2	5.2	12.7	10.8	5.0	12.2
2	3.8	4.4	13.3	12.5	8.9	9.2
3	5.5	5.2	11.1	14.0	3.9	7.7
4	4.0	5.4	14.0	12.4	6.3	3.4
5	3.7	5.5	9.9	11.5	4.1	3.1
6	5.7	5.1	13.9	11.9	4.1	4.6
7	4.1	6.1	9.9	11.3	4.2	3.7
8	3.7	5.1	16.0	8.9	7.7	3.1
9	4.2	5.8	14.4	8.6	5.9	2.5
10	4.8	5.4	14.0	9.2	4.7	2.8
11	4.8	5.7	10.1	12.8	4.1	4.8
12	4.5	5.9	11.6	12.2	4.3	4.0
13	4.2	5.6	11.7	13.9	4.2	5.6
14	3.8	7.4	11.8	10.3	4.6	3.1
Mean	**4.4**	**5.6**	**12.5**	**11.4**	**5.1**	**5.0**
Standard deviation	**0.7**	**0.7**	**1.8**	**1.7**	**1.5**	**2.7**

Tensile modulus (GPa) of composites (longitudinal direction of yarn)

Serial number	Hairy yarn (unsized)	Hairless yarn (unsized)	Hairy yarn (sized)	Hairless yarn (sized)
1	18.0	17.0	18.9	18.3
2	17.0	15.9	18.4	17.3
3	17.2	15.7	17.2	16.0
4	17.5	17.1	18.2	19.0
5	17.2	16.7	18.6	18.0
Mean	**17.4**	**16.5**	**18.3**	**17.7**
Standard deviation	**0.34**	**0.58**	**0.57**	**1.02**

Back calculated tensile modulus (GPa) of fibres (longitudinal direction)

Serial number	Hairy yarn (unsized)	Hairless yarn (unsized)	Hairy yarn (sized)	Hairless yarn (sized)
1	41.3	38.7	47.1	44.3
2	38.7	35.8	45.7	41.5
3	39.2	35.3	42.3	37.9
4	40.0	39.0	42.1	46.2
5	39.2	37.9	46.3	43.4
Mean	**39.7**	**37.3**	**44.7**	**42.7**
Standard deviation	**0.91**	**1.51**	**2.09**	**2.82**

Tensile strength (MPa) of composites (longitudinal direction of yarn)

Serial number	Hairy yarn (unsized)	Hairless yarn (unsized)	Hairy yarn (sized)	Hairless yarn (sized)
1	168	186	192	188
2	180	170	185	190
3	157	177	182	158
4	182	194	188	162
5	156	181	189	188
Mean	**169**	**182**	**187**	**177**
Standard deviation	**12**	**8**	**4**	**14**

Back calculated tensile strength (MPa) of fibres (longitudinal direction)

Serial number	Hairy yarn (unsized)	Hairless yarn (unsized)	Hairy yarn (sized)	Hairless yarn (sized)
1	328	375	419	398
2	359	333	399	403
3	299	352	390	314
4	365	396	407	326
5	296	362	410	398
Mean	**329**	**364**	**405**	**368**
Standard deviation	**29**	**21**	**10**	**39**

Strain at failure (%) of composites (longitudinal direction of yarn)

Serial number	Hairy yarn (unsized)	Hairless yarn (unsized)	Hairy yarn (sized)	Hairless yarn (sized)
1	1.31	1.41	1.44	1.37
2	1.44	1.47	1.25	1.36
3	1.29	1.48	1.33	1.36
4	1.31	1.50	1.40	1.48
5	1.43	1.49	1.26	1.36
Mean	**1.36**	**1.47**	**1.34**	**1.39**
Standard deviation	**0.06**	**0.03**	**0.07**	**0.05**

Tensile modulus (GPa) of composites (transverse direction of yarn)

Serial number	Hairy yarn (unsized)	Hairless yarn (unsized)	Hairy yarn (sized)	Hairless yarn (sized)
1	2.1	2.5	3.1	3.6
2	2.0	2.8	2.7	3.5
3	2.4	3.1	2.7	4.1
4	2.6	2.9	5.2	4.9
5	2.7	4.7	4.5	3.5
Mean	**2.4**	**3.2**	**3.6**	**3.9**
Standard deviation	**0.27**	**0.79**	**1.03**	**0.52**

Tensile strength (MPa) of composites (transverse direction of yarn)

Serial number	Hairy yarn (unsized)	Hairless yarn (unsized)	Hairy yarn (sized)	Hairless yarn (sized)
1	6.8	8.3	8.0	9.4
2	5.9	8.0	5.3	8.7
3	6.9	8.6	8.2	9.4
4	8.1	6.1	7.9	9.2
5	7.4	6.7	7.3	9.5
Mean	**7.0**	**7.6**	**7.3**	**9.2**
Standard deviation	**0.73**	**0.96**	**1.06**	**0.30**

Strain at failure (%)of composites (transverse direction of yarn)

Serial number	Hairy yarn (unsized)	Hairless yarn (unsized)	Hairy yarn (sized)	Hairless yarn (sized)
1	0.84	0.59	0.28	0.27
2	0.71	0.52	0.29	0.25
3	0.84	0.48	0.32	0.25
4	0.90	0.27	0.26	0.22
5	0.69	0.30	0.30	0.28
Mean	**0.80**	**0.43**	**0.29**	**0.25**
Standard deviation	**0.08**	**0.13**	**0.02**	**0.02**

9.2 Appendix B: Raw tested data of Chapter 4

Tensile test data of single jute fibre extracted from unsized raw yarn

Fibre / test number	Fibre diameter (μm)	Tensile modulus (GPa)	Failure load (N)	Tensile strength (MPa)	Strain at failure (%)
1	37	36.0	0.76	700	1.84
2	57	12.7	0.30	115	0.92
3	39	31.9	0.54	446	1.44
4	44	30.0	0.28	190	0.65
5	63	15.8	0.90	284	1.69
6	59	24.1	1.13	414	1.71
7	53	19.0	0.51	236	1.23
8	47	35.1	1.98	1160	2.67
9	52	23.9	0.35	166	0.74
10	52	23.0	0.37	175	0.74
11	45	23.7	1.02	653	2.36
12	46	29.7	1.23	738	2.05
13	52	17.5	0.30	140	0.81
14	57	17.4	0.40	156	0.89
15	55	24.3	1.00	414	1.58
16	77	10.0	1.21	257	2.04
17	54	22.6	0.23	99	0.45
18	49	23.0	0.76	410	1.64
19	43	33.0	0.88	614	1.82
20	55	20.3	0.80	341	1.55
21	49	24.3	0.19	97	0.41
22	35	28.8	0.26	270	0.91
23	45	27.8	0.64	410	1.48
24	41	33.8	0.44	339	1.08
25	45	30.4	1.13	702	2.07

26	77	14.2	1.35	292	1.97
27	52	24.6	1.16	545	2.00
28	42	23.8	0.62	447	1.56
29	67	15.4	0.91	254	1.48
30	89	12.8	2.20	356	2.44
31	47	24.8	0.22	126	0.53
32	48	17.5	0.65	357	1.68
33	39	32.7	1.17	964	2.09
34	53	20.5	0.53	238	1.34
Mean	**52**	**23.7**	**0.78**	**385**	**1.47**
Standard deviation	**12**	**6.9**	**0.48**	**247**	**0.59**

Tensile test data of single jute fibre extracted from HEC sized raw yarn

Fibre / test number	Fibre diameter (µm)	Tensile modulus (GPa)	Failure load (N)	Tensile strength (MPa)	Strain at failure (%)
1	44	20.8	0.84	552	2.40
2	43	21.4	0.79	553	2.52
3	51	20.9	0.76	368	1.92
4	39	22.1	0.43	360	1.46
5	41	12.8	0.57	443	2.54
6	35	24.1	0.38	403	1.68
7	35	30.7	0.89	950	2.26
8	37	28.5	0.50	480	1.70
9	35	21.2	0.32	334	1.66
10	43	20.5	0.49	338	1.61
11	53	19.9	0.47	214	1.08
12	44	28.2	0.64	433	1.43
13	55	28.2	0.99	416	1.54

14	80	13.1	1.52	306	2.05
15	86	9.9	0.44	76	0.85
16	75	10.0	0.45	102	1.03
17	61	15.4	1.20	416	2.22
18	63	7.0	0.39	123	1.38
19	63	16.4	1.91	619	2.87
20	79	9.6	1.34	273	2.48
21	52	18.2	0.97	461	2.30
22	45	21.7	0.51	323	1.81
23	69	15.6	1.09	294	1.84
24	64	20.5	1.99	620	2.94
25	77	13.6	1.50	324	2.18
26	67	15.3	0.85	244	1.52
27	52	10.7	0.36	168	1.60
28	37	20.2	0.32	295	1.50
Mean	**54**	**18.4**	**0.82**	**374**	**1.87**
Standard deviation	**15**	**6.2**	**0.47**	**179**	**0.53**

Tensile test data of single jute fibre extracted from unsized water treated yarn without tension

Fibre /test number	Fibre diameter (µm)	Tensile modulus (GPa)	Failure load (N)	Tensile strength (MPa)	Strain at failure (%)
1	36	30.6	0.18	175	0.61
2	66	18.1	1.43	417	2.16
3	29	34.7	0.25	398	1.13
4	48	19.6	0.68	374	1.86
5	47	17.9	0.42	241	1.31
6	44	33.2	1.31	845	2.24

7	42	27.7	0.91	669	1.91
8	43	28.8	0.48	329	1.22
9	28	44.1	0.17	281	0.64
10	53	19.1	0.73	327	1.72
11	52	14.5	0.24	110	0.82
12	81	13.5	1.69	329	2.11
13	51	30.8	0.39	192	0.71
14	54	14.5	0.77	337	1.89
15	46	31.9	1.49	914	2.18
16	87	4.4	0.18	31	0.72
17	59	13.2	0.77	279	1.82
18	64	18.7	1.45	452	2.12
19	68	15.1	1.76	485	2.58
20	55	23.0	0.67	280	1.32
21	76	12.4	0.55	120	1.00
22	65	10.8	1.04	309	2.48
23	52	21.5	1.04	481	2.08
24	48	27.5	1.34	749	2.56
25	53	19.7	1.34	604	2.56
26	63	15.5	0.64	202	1.59
27	46	25.5	1.37	814	2.50
Mean	**54**	**21.7**	**0.86**	**398**	**1.70**
Standard deviation	**14**	**8.8**	**0.49**	**229**	**0.64**

Tensile test data of single jute fibre extracted from HEC sized water treated yarn without tension

Fibre /test number	Fibre diameter (µm)	Tensile modulus (GPa)	Failure load (N)	Tensile strength (MPa)	Strain at failure (%)
1	63	16.5	0.83	270	1.37
2	87	7.7	0.78	131	1.87
3	70	11.0	0.59	152	1.43
4	70	10.8	0.29	75	0.70
5	58	19.9	0.52	199	1.09
6	65	21.6	0.99	294	1.63
7	62	26.0	1.31	437	2.13
8	44	25.8	0.49	320	1.34
9	54	25.0	0.41	177	0.72
10	63	17.7	0.65	211	1.60
11	44	27.8	0.65	427	1.48
12	52	26.0	0.81	383	1.48
13	49	29.2	0.92	486	1.66
14	94	14.8	2.72	389	2.54
15	58	20.0	1.17	440	1.78
16	67	16.1	0.46	131	0.86
17	57	17.6	0.97	377	1.96
18	57	19.1	1.01	392	1.94
19	50	22.2	0.98	494	2.02
20	65	18.1	0.56	170	0.97
21	57	16.7	0.94	365	2.04
22	49	19.9	0.29	154	0.80
23	39	23.0	0.46	376	1.70
24	52	23.5	1.28	595	2.40
25	85	9.7	1.04	184	1.80

26	44	27.1	0.94	613	2.04
27	54	25.1	1.40	615	2.17
28	57	17.4	0.94	368	1.90
Mean	**60**	**19.8**	**0.87**	**329**	**1.62**
Standard deviation	**13**	**5.6**	**0.46**	**152**	**0.49**

Tensile test data of single jute fibre extracted from unsized water treated yarn with tension

Fibre /test number	Fibre diameter (µm)	Tensile modulus (GPa)	Failure load (N)	Tensile strength (MPa)	Strain at failure (%)
1	34	65.3	1.06	1154	1.74
2	67	16.9	0.97	272	1.61
3	51	26.2	0.81	399	1.46
4	64	18.3	1.39	438	2.09
5	47	26.4	0.47	272	1.04
6	43	24.3	0.39	272	1.13
7	41	29.1	0.66	495	1.70
8	35	16.2	0.15	158	0.98
9	51	26.4	1.43	688	2.34
10	42	32.5	0.37	273	0.85
11	45	21.2	0.30	188	0.98
12	37	24.2	0.36	324	1.46
13	52	19.5	0.78	365	1.75
14	52	21.6	1.28	605	2.67
15	45	24.4	0.44	275	1.09
16	50	25.9	0.92	468	1.73
17	47	33.5	0.63	371	1.13
18	59	17.4	0.52	191	1.12

19	42	27.0	0.61	442	1.82
20	48	25.3	0.29	161	0.69
21	45	20.5	0.60	370	1.89
22	52	17.3	0.58	272	1.47
23	49	22.2	0.90	470	1.91
24	48	28.6	0.83	458	1.61
25	48	28.2	1.08	595	2.02
26	45	21.6	0.61	389	1.88
27	44	15.9	0.52	344	1.96
28	51	15.3	0.40	194	1.27
29	49	20.1	0.50	260	1.25
30	52	17.8	0.52	243	1.37
31	51	20.8	0.83	399	1.77
32	46	27.8	0.84	498	1.81
33	53	24.6	0.97	435	1.67
Mean	**48**	**24.3**	**0.70**	**386**	**1.55**
Standard deviation	**7**	**8.7**	**0.31**	**188**	**0.44**

Tensile test data of single jute fibre extracted from HEC sized water treated yarn with tension

Fibre/test number	Fibre diameter (µm)	Tensile modulus (GPa)	Failure load (N)	Tensile strength (MPa)	Strain at failure (%)
1	51	24.3	0.50	252	1.11
2	37	40.3	0.59	540	1.46
3	75	11.4	0.98	225	1.86
4	61	17.6	1.01	350	1.80
5	33	44.7	0.29	333	0.78
6	66	15.3	0.68	200	1.36
7	46	20.1	0.31	180	0.96
8	45	18.0	0.33	203	1.13
9	63	18.0	0.52	163	0.94
10	33	34.4	0.52	616	1.80
11	46	19.5	0.90	554	2.46
12	46	21.3	0.73	437	1.84
13	41	27.1	0.46	346	1.29
14	64	11.7	0.31	95	0.93
15	69	16.3	0.86	229	1.42
16	67	11.7	1.21	344	2.21
17	54	19.1	1.23	533	2.45
18	45	26.5	0.78	496	2.49
19	63	17.7	0.71	228	1.37
20	74	7.5	0.41	96	1.14
21	78	13.4	2.00	421	2.46
22	59	13.3	0.70	260	1.68
23	56	14.6	0.68	276	1.69
24	84	9.9	1.53	273	2.14
25	66	20.3	1.47	426	1.86

26	65	16.0	1.56	465	2.32
27	52	15.2	0.84	389	2.22
28	62	17.7	1.04	351	1.88
29	45	15.5	0.83	524	2.30
30	37	19.3	0.27	250	1.25
31	58	16.3	0.45	170	1.05
32	54	15.9	1.03	448	2.11
33	41	22.5	0.49	362	1.56
34	43	29.0	0.72	504	1.63
35	43	13.5	0.26	182	1.51
36	51	24.1	1.36	654	2.33
Mean	**56**	**19.4**	**0.79**	**344**	**1.69**
Standard deviation	**12**	**7.8**	**0.42**	**147**	**0.51**

Tensile modulus (GPa) of yarn

Test number	Raw	Raw HEC	Wet without tension unsized	Wet without tension HEC sized	Wet with tension unsized	Wet with tension HEC sized
1	5.3	5.0	3.6	3.0	4.4	3.9
2	3.8	4.5	4.1	4.1	6.0	5.3
3	5.5	4.3	3.1	4.8	4.2	5.6
4	4.1	5.0	2.9	4.6	5.3	5.2
5	3.7	4.0	4.6	4.7	4.8	4.9
6	5.8	4.3	4.5	4.2	4.2	6.5
7	4.2	3.9	2.8	4.1	6.3	5.6
8	3.7	3.7	3.8	5.1	5.9	5.6
9	4.2	4.1	3.9	5.2	5.7	5.9
10	4.9	4.4	3.4	5.0	5.8	6.0

11	4.9	4.9				
12	4.5					
13	4.3					
14	3.9					
Mean	**4.5**	**4.4**	**3.7**	**4.5**	**5.3**	**5.5**
Standard deviation	**0.7**	**0.4**	**0.6**	**0.6**	**0.8**	**0.7**

Yarn failure load (N)

Test number	Raw	Raw HEC	Wet without tension unsized	Wet without tension HEC sized	Wet with tension unsized	Wet with tension HEC sized
1	35.5	39.1	34.5	33.7	29.4	29.3
2	37.3	27.5	31.7	39.7	43.5	43.9
3	31.1	41.1	23.0	23.0	36.1	37.2
4	39.2	27.9	28.4	44.0	43.3	46.6
5	27.7	34.2	33.0	26.9	28.9	38.0
6	38.8	28.1	19.8	27.7	45.4	55.3
7	27.6	39.6	25.7	25.1	35.9	38.5
8	44.8	31.8	21.9	28.6	33.9	37.7
9	40.4	33.9	25.6	29.5	38.6	38.7
10	39.3	29.8	25.8	24.6	35.5	42.5
11	28.2	35.7				
12	32.5					
13	32.9					
14	33.0					
Mean	**34.9**	**33.5**	**27.0**	**30.3**	**37.0**	**40.7**
Standard deviation	**5.1**	**4.9**	**4.9**	**6.9**	**5.4**	**6.9**

Tenacity (cN/Tex) of yarn

Test number	Raw	Raw HEC	Wet without tension unsized	Wet without tension HEC sized	Wet with tension unsized	Wet with tension HEC sized
1	12.7	14.0	12.3	12.0	10.5	10.5
2	13.3	9.8	11.3	14.2	15.5	15.7
3	11.1	14.7	8.2	8.2	12.9	13.3
4	14.0	10.0	10.1	15.7	15.5	16.6
5	9.9	12.2	11.8	9.6	10.3	13.6
6	13.9	10.0	7.1	9.9	16.2	19.7
7	9.9	14.1	9.2	9.0	12.8	13.7
8	16.0	11.4	7.8	10.2	12.1	13.5
9	14.4	12.1	9.2	10.5	13.8	13.8
10	14.0	10.6	9.2	8.8	12.7	15.2
11	10.1	12.7				
12	11.6					
13	11.7					
14	11.8					
Mean	**12.5**	**12.0**	**9.6**	**10.8**	**13.2**	**14.6**
Standard deviation	**1.8**	**1.7**	**1.7**	**2.3**	**1.9**	**2.3**

Tensile strength (MPa) of yarn

Test number	Raw	Raw HEC	Wet without tension unsized	Wet without tension HEC sized	Wet with tension unsized	Wet with tension HEC sized
1	181	199	176	172	150	149
2	190	140	162	203	222	224
3	159	210	117	117	184	190
4	200	142	145	224	221	238
5	141	174	168	137	147	194
6	198	143	101	141	232	282
7	141	202	131	128	183	196
8	229	162	112	146	173	192
9	206	173	131	150	197	197
10	200	152	132	125	181	217
11	144	182				
12	166					
13	168					
14	168					
Mean	**178**	**171**	**137**	**154**	**189**	**208**
Standard deviation	**26**	**24**	**24**	**33**	**28**	**33**

Strain at failure (%) of yarn

Test number	Raw	Raw HEC	Wet without tension unsized	Wet without tension HEC sized	Wet with tension unsized	Wet with tension HEC sized
1	5.0	4.8	6.2	6.6	5.8	5.5
2	8.9	3.6	4.8	7.1	14.0	7.9
3	3.9	6.3	3.6	3.9	6.2	5.2
4	6.3	3.2	5.8	8.3	7.7	8.9
5	4.1	5.4	6.0	4.1	4.1	6.1
6	4.1	4.7	3.4	6.1	8.4	13.7
7	4.2	5.4	16.2	5.4	4.5	5.6
8	7.7	4.6	3.7	4.7	4.1	5.1
9	5.9	5.0	5.1	5.0	5.6	5.3
10	4.7	4.0	5.9	3.7	4.8	5.5
11	4.1	4.3				
12	4.3					
13	4.2					
14	4.6					
Mean	**5.1**	**4.7**	**6.1**	**5.5**	**6.5**	**6.9**
Standard deviation	**1.5**	**0.8**	**3.5**	**1.4**	**2.8**	**2.6**

Tensile modulus (GPa) of composites

Test number	Raw unsized yarn	Raw HEC sized yarn	Wet yarn without tension and unsized	Wet yarn without tension and HEC sized	Wet yarn with tension and unsized	Wet yarn with tension and HEC sized
1	18.0	18.9	15.8	19.6	18.0	18.5
2	17.0	18.4	15.7	17.0	17.0	19.4
3	17.2	17.2	15.6	16.6	18.0	20.3
4	17.5	18.2	15.6	16.3	16.7	19.5
5	17.2	18.6	15.1	17.6	18.1	19.0
Mean	**17.4**	**18.3**	**15.5**	**17.4**	**17.6**	**19.3**
Standard deviation	**0.3**	**0.6**	**0.2**	**1.2**	**0.6**	**0.6**

Tensile strength (MPa) of composites

Test number	Raw unsized yarn	Raw HEC sized yarn	Wet yarn without tension and unsized	Wet yarn without tension and HEC sized	Wet yarn with tension and unsized	Wet yarn with tension and HEC sized
1	168	192	160	161	193	217
2	180	185	151	196	186	212
3	157	182	159	187	183	209
4	182	188	157	173	182	221
5	156	189	144	185	182	213
Mean	**169**	**187**	**154**	**180**	**185**	**215**
Standard deviation	**12**	**4**	**6**	**12**	**4**	**4**

Back calculated tensile modulus (GPa) of fibres

Test number	Raw unsized yarn	Raw HEC sized yarn	Wet yarn without tension and unsized	Wet yarn without tension and HEC sized	Wet yarn with tension and unsized	Wet yarn with tension and HEC sized
1	41.3	47.1	37.3	47.9	43.4	43.7
2	38.7	45.7	37.0	40.6	40.6	46.1
3	39.2	42.3	36.8	39.5	43.4	48.6
4	40.0	45.1	36.8	38.7	39.8	46.4
5	39.2	46.3	35.4	42.3	43.7	45.1
Mean	**39.7**	**45.3**	**36.7**	**41.8**	**42.2**	**46.0**
Standard deviation	**0.91**	**1.64**	**0.66**	**3.28**	**1.64**	**1.61**

Back calculated tensile strength (MPa) of fibres

Test number	Raw unsized yarn	Raw HEC sized yarn	Wet yarn without tension and unsized	Wet yarn without tension and HEC sized	Wet yarn with tension and unsized	Wet yarn with tension and HEC sized
1	328	419	320	323	412	467
2	359	399	295	420	392	454
3	299	390	317	395	384	446
4	365	407	312	356	381	478
5	296	410	276	389	381	456
Mean	**329**	**405**	**304**	**377**	**390**	**460**
Standard deviation	**29**	**10**	**16**	**34**	**12**	**11**

Strain at failure (%) of composites

Test number	Raw unsized yarn	Raw HEC sized yarn	Wet yarn without tension and unsized	Wet yarn without tension and HEC sized	Wet yarn with tension and unsized	Wet yarn with tension and HEC sized
1	1.31	1.44	3.47	1.16	3.12	1.44
2	1.44	1.25	3.45	1.46	3.58	1.39
3	1.29	1.33	3.52	1.45	3.61	1.33
4	1.31	1.40	3.36	1.35	3.75	1.44
5	1.43	1.26	3.57	1.39	3.41	1.42
Mean	**1.36**	**1.34**	**3.48**	**1.36**	**3.49**	**1.40**
Standard deviation	**0.06**	**0.07**	**0.07**	**0.11**	**0.22**	**0.04**

9.3 Appendix C: Raw tested data of Chapter 5

Tensile test data of single jute fibre extracted from unsized raw yarn

Fibre or test number	Fibre diameter (µm)	Tensile modulus (GPa)	Tensile strength (MPa)	Strain at failure (%)
1	41	26.7	1409	2.72
2	28	64.3	1071	1.57
3	34	41.1	884	1.68
4	28	64.2	1415	2.17
5	30	43.1	1155	2.03
6	47	19.4	557	1.97
7	48	29.6	1098	2.51
8	30	44.0	1160	2.00
9	26	41.3	478	1.07
10	34	32.7	779	2.78
11	22	68.1	1005	1.63
12	38	14.5	1176	2.36
13	27	48.1	413	0.86
14	33	29.0	1089	2.26
15	31	42.8	811	1.66
16	29	56.1	1602	2.76
17	30	56.1	2159	2.72
18	55	13.0	374	2.04
19	35	34.7	661	1.85
20	23	58.8	1678	2.14
21	26	61.9	1385	2.10
22	26	65.2	1222	1.67
23	25	50.4	1451	2.31
24	25	75.1	1453	1.86
25	30	45.0	1065	2.11

26	39	47.9	1212	2.02
27	30	52.6	1164	2.06
Mean	**32**	**45.4**	**1108**	**2.03**
Standard deviation	**8**	**16.3**	**403**	**0.46**

Tensile test data of single jute fibre extracted from unsized and 1% NaOH treated yarn without tension

Fibre or test number	Fibre diameter (µm)	Tensile modulus (GPa)	Tensile strength (MPa)	Strain at failure (%)
1	29	26.7	304	1.16
2	36	58.0	603	0.98
3	29	46.8	1005	2.04
4	30	49.0	1292	2.08
5	40	29.2	1093	2.75
6	45	33.4	1074	2.47
7	33	46.2	1020	2.01
8	44	25.3	593	1.81
9	26	66.2	1774	2.38
10	28	51.3	1424	2.26
11	33	39.6	1217	2.42
12	34	35.1	1315	2.60
13	45	18.1	263	1.32
14	53	21.1	453	1.68
15	38	24.7	857	2.21
16	30	56.9	2184	2.89
17	19	92.2	1101	1.16
18	26	75.6	1423	1.75
19	47	20.2	568	2.26

20	32	41.8	1053	2.17
21	43	26.0	1284	2.48
22	32	55.7	1322	2.01
23	45	28.2	556	1.80
24	36	45.4	1179	2.14
25	34	34.2	864	2.36
26	38	35.9	1489	3.06
Mean	**35**	**41.6**	**1050**	**2.09**
Standard deviation	**8**	**17.8**	**442**	**0.52**

Tensile test data of single jute fibre extracted from unsized and 1% NaOH treated yarn with tension

Fibre or test number	Fibre diameter (μm)	Tensile modulus (GPa)	Tensile strength (MPa)	Strain at failure (%)
1	15	66.9	653	0.96
2	33	54.7	1425	2.48
3	25	42.0	976	1.85
4	30	50.9	1085	1.97
5	23	43.3	810	1.68
6	36	29.8	642	1.85
7	35	49.9	610	1.28
8	46	23.9	651	2.54
9	23	95.3	1787	1.75
10	50	31.4	468	1.29
11	23	51.3	1107	1.64
12	26	59.9	1359	2.07
13	52	24.6	339	1.18
14	33	50.6	1620	2.80

15	32	44.4	1474	2.73
16	32	33.4	524	1.40
17	32	33.9	620	1.67
18	35	41.3	1320	2.61
19	34	59.3	1712	2.62
20	30	41.9	1353	2.22
21	42	31.0	1086	3.07
22	33	45.3	1459	2.90
23	32	37.0	391	1.05
24	40	34.2	288	0.92
25	40	37.8	678	1.85
26	22	46.8	868	1.65
Mean	**33**	**44.6**	**973**	**1.92**
Standard deviation	**8.6**	**14.8**	**445**	**0.62**

Tensile test data of single jute fibre extracted from unsized and 5% NaOH treated yarn without tension

Fibre or test number	Fibre diameter (µm)	Tensile modulus (GPa)	Tensile strength (MPa)	Strain at failure (%)
1	58	39.1	1024	2.49
2	43	45.1	961	2.10
3	41	61.6	749	1.37
4	36	16.8	528	2.13
5	15	76.5	843	1.09
6	23	58.3	1088	1.79
7	25	29.7	801	1.96
8	34	73.6	995	1.40
9	27	32.2	1535	2.93

10	31	50.1	880	1.87
11	30	39.8	611	1.47
12	44	40.5	382	1.04
13	21	69.3	1048	1.25
14	23	71.3	1629	2.04
15	41	23.5	631	2.42
16	42	50.1	1252	2.36
17	25	30.9	475	1.68
18	39	26.3	773	2.15
19	26	34.6	1167	2.52
20	42	18.4	375	1.66
21	28	56.0	1739	2.46
22	35	33.0	319	1.04
23	26	39.7	759	1.86
24	26	46.9	1049	2.18
25	29	43.7	1220	2.34
26	27	48.6	1558	2.72
27	29	30.4	1094	2.52
28	32	30.9	608	1.78
Mean	**32**	**43.5**	**932**	**1.95**
Standard deviation	**9**	**16.2**	**378**	**0.52**

Tensile test data of single jute fibre extracted from unsized and 5% NaOH treated yarn with tension

Fibre or test number	Fibre diameter (µm)	Tensile modulus (GPa)	Tensile strength (MPa)	Strain at failure (%)
1	49	16.1	210	1.40
2	29	49.8	1008	2.48
3	30	46.4	983	2.76
4	21	56.4	1015	1.86
5	18	62.6	1285	2.32
6	30	39.4	925	2.88
7	24	64.0	1720	3.58
8	27	41.1	1109	3.02
9	24	41.3	1127	2.73
10	37	24.3	226	1.08
11	25	40.2	831	3.14
12	42	18.1	261	1.56
13	29	31.3	848	2.40
14	37	38.4	640	1.96
15	18	92.6	1493	1.61
16	29	39.3	717	1.76
17	22	98.7	1712	1.88
18	21	50.0	1304	3.11
19	25	43.0	1042	2.68
20	34	34.9	563	1.72
21	28	37.2	1049	3.44
22	36	23.6	679	2.31
23	25	72.7	1376	1.82
24	24	48.5	757	1.42
Mean	**29**	**46.2**	**953**	**2.29**
Standard deviation	**7**	**20.1**	**406**	**0.68**

Tensile modulus (GPa) of composite plaques

Name of the composite plaque ↓ / Test number →	1	2	3	4	5	Mean	Standard deviation
Raw yarn HEC sized	18.9	18.4	17.2	18.2	18.6	**18.3**	**0.6**
1% NaOH no tension HEC sized	17.4	18.0	18.8	17.0	19.2	**18.1**	**0.8**
1% NaOH with tension HEC sized	19.7	20.7	19.3	18.3	21.5	**19.9**	**1.1**
3% NaOH no tension HEC sized	17.5	19.0	19.6	19.6	19.5	**19.0**	**0.8**
3% NaOH with tension HEC sized	18.7	20.5	20.2	20.2	21.8	**20.3**	**1.0**
5% NaOH no tension HEC sized	23.7	21.4	24.7	21.5	22.2	**22.7**	**1.3**
5% NaOH with tension HEC sized	25.4	21.1	24.6	22.3	22.2	**23.1**	**1.6**

Tensile strength (MPa) of composite plaques

Name of the composite plaque ↓ / Test number →	1	2	3	4	5	Mean	Standard deviation
Raw yarn HEC sized	192	185	182	188	189	**187**	**4**
1% NaOH no tension HEC sized	138	169	161	155	175	**160**	**13**
1% NaOH with tension HEC sized	206	199	190	183	203	**196**	**8**
3% NaOH no tension HEC sized	167	160	172	162	172	**167**	**5**
3% NaOH with tension HEC sized	191	204	214	204	220	**207**	**10**
5% NaOH no tension HEC sized	222	213	203	212	220	**214**	**7**
5% NaOH with tension HEC sized	256	255	234	226	237	**242**	**11**

Strain at failure (%) of composite plaques

Name of the composite plaque ↓ / Test number →	1	2	3	4	5	Mean	Standard deviation
Raw yarn HEC sized	1.44	1.25	1.33	1.40	1.26	**1.34**	**0.07**
1% NaOH no tension HEC sized	1.23	1.33	1.23	1.35	1.37	**1.30**	**0.06**
1% NaOH with tension HEC sized	1.38	1.35	1.31	1.35	1.30	**1.34**	**0.03**
3% NaOH no tension HEC sized	1.37	1.25	1.31	1.21	1.31	**1.29**	**0.08**
3% NaOH with tension HEC sized	1.27	1.39	1.41	1.30	1.29	**1.33**	**0.06**
5% NaOH no tension HEC sized	1.51	1.49	1.29	1.47	1.46	**1.44**	**0.08**
5% NaOH with tension HEC sized	1.36	1.48	1.34	1.28	1.34	**1.36**	**0.07**

Back calculated values of tensile modulus (GPa) of composite plaques

Name of the composite plaque ↓ / Test number →	1	2	3	4	5	Mean	Standard deviation
Raw yarn HEC sized	47.1	45.7	42.3	45.1	46.3	**45.3**	**1.64**
1% NaOH no tension HEC sized	41.6	43.3	45.5	40.4	46.5	**43.5**	**2.3**
1% NaOH with tension HEC sized	47.4	50.3	46.6	43.7	52.4	**48.1**	**3.0**
3% NaOH no tension HEC sized	41.2	45.4	47.1	47.1	46.7	**45.5**	**2.2**
3% NaOH with tension HEC sized	43.1	48.1	47.1	47.2	51.2	**47.3**	**2.6**
5% NaOH no tension HEC sized	47.1	41.9	49.1	42.3	43.7	**44.8**	**2.8**
5% NaOH with tension HEC sized	53.7	43.9	51.9	46.6	46.3	**48.5**	**3.7**

Back calculated values of tensile strength (GPa) of composite plaques

Name of the composite plaque ↓ Test number →	1	2	3	4	5	Mean	Standard deviation
Raw yarn HEC sized	419	398	390	407	410	**405**	**10**
1% NaOH no tension HEC sized	258	345	323	306	362	**319**	**36**
1% NaOH with tension HEC sized	448	428	403	384	439	**420**	**24**
3% NaOH no tension HEC sized	332	313	346	318	346	**331**	**14**
3% NaOH with tension HEC sized	388	423	449	423	464	**429**	**26**
5% NaOH no tension HEC sized	400	381	359	378	396	**383**	**15**
5% NaOH with tension HEC sized	503	500	451	433	458	**469**	**28**

9.4 Appendix D: Raw tested data of Chapter 6

Tensile modulus (GPa) of composites

Sample name	1	2	3	4	5	Mean	Standard deviation
Raw yarn	18.0	17.0	17.2	17.2	17.2	**17.4**	**0.3**
Raw yarn HEC sized	18.9	18.4	17.2	18.2	18.6	**18.3**	**0.6**
0.5% CNP sized (FD)	17.2	16.8	17.9			17.3	0.4
1% CNP sized (FD)	14.6	17.4	17.5	16.9		16.6	1.2
0.5%CNP sized (RTND)	17.2	20.4	17.7	17.8	19.9	18.6	1.3
1%CNP sized (RTND)	19.5	20.2	19.7	19.4	19.9	19.7	0.3
1%CNP sized (NRTND)	19.4	21.3	21.4	21.2	20.2	20.7	0.8
0.5%CNP mixed in resin	17.6	17.9	16.4	18.7	18.3	17.8	0.8
1%CNP mixed in resin	15.9	16.7	16.2	17.2	13.9	16.0	1.1

Tensile strength (MPa) of composites

Sample name	1	2	3	4	5	Mean	Standard deviation
Raw yarn	168	180	157	182	156	**169**	**12**
Raw yarn HEC sized	192	185	182	188	189	**187**	**4**
0.5% CNP sized (FD)	209	183	206			199	12
1% CNP sized (FD)	153	153	178	161		162	10
0.5%CNP sized (RTND)	230	219	222	218	194	216	12
1%CNP sized (RTND)	220	233	218	219	224	223	6
1%CNP sized (NRTND)	210	208	250	223	233	225	16
0.5%CNP mixed in resin	190	186	157	190	192	183	13
1%CNP mixed in resin	166	173	160	182	150	166	11

Back calculated tensile modulus (GPa) of composites

Sample name	1	2	3	4	5	Mean	Standard deviation
Raw yarn	41.3	38.7	39.2	39.2	39.2	**39.5**	**0.9**
Raw yarn HEC sized	46.9	45.4	42.2	45.1	46.2	**45.2**	**1.5**
0.5% CNP sized (FD)	38.3	37.3	40.1			**38.6**	**1.2**
1% CNP sized (FD)	34.8	42.8	43.1	41.4		**40.6**	**3.4**
0.5%CNP sized (RTND)	37.6	45.6	38.9	39.1	44.4	**41.1**	**3.2**
1%CNP sized (RTND)	45.3	47.1	45.8	45.0	46.3	**45.9**	**0.8**
1%CNP sized (NRTND)	39.5	43.8	44.1	43.6	41.3	**42.5**	**1.8**
0.5%CNP mixed in resin	41.3	42.1	38.0	44.2	43.2	**41.8**	**2.1**
1%CNP mixed in resin	36.7	38.8	37.5	40.2	32.1	**37.1**	**2.7**

Back calculated tensile strength (MPa) of composites

Sample name	1	2	3	4	5	Mean	Standard deviation
Raw yarn	328	359	299	365	296	**329**	**29**
Raw yarn HEC sized	419	398	390	405	411	**404**	**10**
0.5% CNP sized (FD)	426	360	419			**402**	**30**
1% CNP sized (FD)	307	307	379	330		**331**	**29**
0.5%CNP sized (RTND)	493	465	473	463	403	**459**	**30**
1%CNP sized (RTND)	465	499	459	462	475	**472**	**14**
1%CNP sized (NRTND)	407	403	498	437	460	**441**	**35**
0.5%CNP mixed in resin	394	384	305	394	400	**375**	**36**
1%CNP mixed in resin	329	348	313	373	297	**332**	**27**

Strain at failure (%) of composites

Sample name	1	2	3	4	5	Mean	Standard deviation
Raw yarn	1.31	1.44	1.29	1.31	1.43	**1.36**	**0.06**
Raw yarn HEC sized	1.44	1.25	1.33	1.40	1.26	**1.34**	**0.07**
0.5% CNP sized (FD)	1.60	1.54	1.54		1.56	**0.03**	
1% CNP sized (FD)	1.39	1.18	1.39	1.33		**1.32**	**0.09**
0.5%CNP sized (RTND)	1.55	1.40	1.55	1.54	1.33	**1.47**	**0.09**
1%CNP sized (RTND)	1.40	1.44	1.42	1.41	1.42	**1.42**	**0.01**
1%CNP sized (NRTND)	1.29	1.32	1.55	1.36	1.46	**1.39**	**0.10**
0.5%CNP mixed in resin	1.36	1.37	1.30	1.36	1.38	**1.35**	**0.03**
1%CNP mixed in resin	1.39	1.38	1.30	1.44	1.1	**1.3**	**0.12**

9.5 Appendix E: Raw tested data of Chapter 7

Tensile test data of single jute fibre extracted from raw jute received from cultivator

Fibre or test number	Fibre diameter (μm)	Tensile modulus (GPa)	Tensile strength (MPa)	Strain at failure (%)
1	87	14.6	256	1.72
2	53	22.0	395	1.87
3	64	25.7	301	1.19
4	57	31.0	649	2.08
5	62	19.1	470	2.13
6	78	21.9	386	1.77
7	75	19.3	591	2.85
8	73	22.7	625	2.85
9	63	20.9	331	1.58
10	95	17.1	563	3.12
11	75	20.0	478	2.44
12	39	28.6	549	1.94
13	63	26.0	503	1.88
14	61	23.6	400	1.72
15	63	20.6	158	0.76
16	63	25.9	545	2.21
17	67	24.1	522	2.37
18	59	31.5	794	2.61
19	89	17.3	446	2.60
20	82	14.6	307	2.60
21	73	25.7	527	2.06
22	102	15.8	464	3.26
23	52	25.2	506	2.05
24	73	20.7	504	2.58
25	71	19.9	420	2.16
Mean	**70**	**22.2**	**468**	**2.18**
Standard deviation	**14**	**4.5**	**133**	**0.57**

Tensile test data of single jute fibre extracted from root end of the plant

Fibre or test number	Fibre diameter (μm)	Tensile modulus (GPa)	Tensile strength (MPa)	Strain at failure (%)
1	63	23.1	366	1.48
2	73	19.1	527	2.41
3	89	13.9	454	2.86
4	82	17.0	595	3.06
5	100	12.4	409	3.24
6	57	29.2	678	2.08
7	79	12.8	267	1.83
8	54	22.0	214	1.08
9	62	20.2	525	2.25
10	50	24.4	621	2.17
Mean	**71**	**19.4**	**466**	**2.25**
Standard deviation	**15**	**5.2**	**145**	**0.65**

10.1.5 Tensile test data of single jute fibre extracted from middle part of the plant

Fibre or test number	Fibre diameter (μm)	Tensile modulus (GPa)	Tensile strength (MPa)	Strain at failure (%)
1	40	32.1	267	0.84
2	40	40.8	833	1.69
3	65	20.1	546	2.56
4	52	24.5	634	2.18
5	57	15.5	95	0.51
6	52	23.5	400	1.62
7	53	24.8	476	1.68
8	51	32.4	184	0.58
9	69	21.0	688	2.96
10	71	18.5	442	2.17
Mean	**55**	**25.3**	**457**	**1.68**
Standard deviation	**10**	**7.3**	**219**	**0.79**

Tensile test data of single jute fibre extracted from tip end of the plant

Fibre or test number	Fibre diameter (μm)	Tensile modulus (GPa)	Tensile strength (MPa)	Strain at failure (%)
1	36	32.1	310	1.27
2	50	24.3	220	0.92
3	69	8.6	166	0.96
4	46	25.2	362	0.56
5	81	11.1	136	1.19
6	80	11.6	173	1.50
7	38	51.4	769	1.50
8	51	24.8	423	1.63
9	45	18.8	141	0.62
10	64	22.3	235	1.05
11	38	28.1	280	1.01
Mean	**54**	**23.5**	**292**	**1.11**
Standard deviation	**16**	**11.4**	**175**	**0.33**

Tensile test data of single jute fibre extracted from raw jute received from Janata Jute Mills Ltd.

Fibre or test number	Fibre diameter (μm)	Tensile modulus (GPa)	Tensile strength (MPa)	Strain at failure (%)
1	58	30.0	198	0.67
2	101	15.4	297	2.32
3	83	21.0	514	2.32
4	86	15.8	160	1.06
5	73	25.7	421	1.60
6	77	23.6	512	2.11
7	97	13.6	255	1.91
8	64	26.3	483	1.88
9	79	21.1	317	1.50
10	63	23.9	470	1.92
11	93	14.0	145	1.02
12	72	19.5	412	2.10
13	81	19.2	377	1.86
14	99	13.6	231	1.66
15	60	29.3	558	1.89
16	67	24.2	421	1.70
17	81	14.1	156	1.12
18	87	18.0	365	2.12
19	83	12.4	197	1.62
20	84	15.8	289	1.82
21	87	18.9	514	2.66
22	69	24.3	353	1.46
23	70	28.0	682	2.39
24	67	19.7	405	2.06
25	67	31.6	641	2.01
26	39	36.8	514	1.39
Mean	**76**	**21**	**380**	**1.78**
Standard deviation	**14**	**6**	**147**	**0.46**

Tensile test data of single jute fibre extracted from breaker carded sliver

Fibre or test number	Fibre diameter (μm)	Tensile modulus (GPa)	Tensile strength (MPa)	Strain at failure (%)
1	76	18.9	195	1.05
2	64	11.6	238	1.87
3	71	11.4	254	2.02
4	52	25.0	491	1.86
5	75	13.3	113	0.88
6	54	15.3	361	1.94
7	40	27.4	390	1.28
8	61	15.4	288	1.70
9	72	18.2	253	1.37
10	51	16.9	563	2.69
11	72	13.4	482	2.66
12	52	21.5	488	2.12
13	63	16.5	381	2.08
14	50	17.8	251	1.35
15	60	18.2	569	2.36
16	37	24.1	255	1.02
17	49	17.2	370	1.86
18	63	14.3	582	2.80
19	70	17.8	252	1.41
20	37	30.9	851	2.46
21	68	12.3	336	2.16
22	69	11.5	145	1.37
23	49	22.6	531	2.02
24	45	21.0	386	1.45
Mean	**58**	**18.0**	**376**	**1.82**
Standard deviation	**12**	**5.1**	**166**	**0.53**

Tensile test data of single jute fibre extracted from finisher carded sliver

Fibre or test number	Fibre diameter (μm)	Tensile modulus (GPa)	Tensile strength (MPa)	Strain at failure (%)
1	75	7.7	155	1.70
2	55	10.0	189	1.70
3	63	5.3	41	0.84
4	59	10.8	244	1.87
5	36	21.7	269	1.36
6	39	25.0	699	2.23
7	61	21.9	584	2.32
8	81	13.3	297	1.98
9	99	9.4	244	2.22
10	117	5.5	144	2.07
11	37	41.2	620	1.58
12	39	2.2	405	1.36
13	48	13.9	402	2.08
14	54	14.2	194	1.26
15	47	19.3	377	1.70
16	39	21.2	108	0.54
17	59	10.8	60	0.61
18	37	21.5	191	0.92
19	61	15.4	221	1.40
20	47	20.6	590	2.44
21	50	15.9	593	2.65
22	63	12.9	401	2.37
23	42	17.9	588	2.58
24	42	18.9	343	1.95
25	59	14.2	353	2.24
Mean	**56**	**15.6**	**332**	**1.76**
Standard deviation	**19**	**7.8**	**186**	**0.59**

Tensile test data of single jute fibre extracted from 1st draw frame sliver

Fibre or test number	Fibre diameter (μm)	Tensile modulus (GPa)	Tensile strength (MPa)	Strain at failure (%)
1	48	17.8	521	2.10
2	78	11.6	161	1.42
3	36	19.5	172	0.98
4	56	12.9	482	2.57
5	45	26.5	564	1.86
6	41	18.4	348	1.63
7	44	19.5	438	1.79
8	42	14.7	129	0.85
9	35	23.4	389	1.68
10	44	21.1	520	2.28
11	44	15.9	414	2.08
12	37	23.0	100	0.42
13	39	15.7	633	2.58
14	33	36.5	974	2.20
15	40	15.8	521	2.23
16	70	13.2	477	2.85
17	37	22.8	348	1.50
18	66	11.1	239	1.71
19	81	6.0	182	2.40
20	49	13.6	424	2.30
21	42	17.2	340	1.67
22	41	21.4	669	2.34
23	45	18.4	487	2.14
24	50	27.4	388	1.31
25	52	19.2	350	1.66
26	40	28.18	420	1.31
Mean	**48**	**18.9**	**411**	**1.84**
Standard deviation	**13**	**6.3**	**186**	**0.57**

Tensile test data of single jute fibre extracted from 2nd draw frame sliver

Fibre or test number	Fibre diameter (μm)	Tensile modulus (GPa)	Tensile strength (MPa)	Strain at failure (%)
1	36	26.6	816	2.20
2	44	24.5	626	2.26
3	79	11.1	178	1.51
4	40	36.9	842	2.00
5	34	24.3	265	1.02
6	47	20.7	523	2.38
7	61	19.8	683	2.76
8	40	23.2	423	1.76
9	43	31.7	595	1.89
10	73	12.9	320	2.01
11	45	18.9	320	1.62
12	46	23.1	504	2.02
13	37	46.4	817	1.60
14	78	13.4	240	1.55
15	35	38.0	807	2.03
16	68	14.6	308	1.79
17	73	16.1	257	1.32
18	57	15.4	494	2.43
19	57	15.9	315	1.68
20	60	18.1	664	2.74
21	41	19.1	596	2.29
22	52	15.2	468	2.18
23	50	20.3	692	2.59
24	40	25.8	1096	2.99
Mean	**52**	**22.2**	**535**	**2.03**
Standard deviation	**14**	**8.5**	**232**	**0.47**

Tensile test data of single jute fibre extracted from 3rd draw frame sliver

Fibre or test number	Fibre diameter (μm)	Tensile modulus (GPa)	Tensile strength (MPa)	Strain at failure (%)
1	73	9.6	95	0.98
2	46	18.1	484	2.19
3	54	12.1	172	1.37
4	60	16.9	395	2.00
5	46	17.1	328	1.76
6	79	8.5	239	2.36
7	63	12.9	271	1.76
8	58	12.4	230	1.54
9	32	27.5	468	1.59
10	55	17.1	414	2.15
11	69	11.8	171	1.53
12	54	14.9	352	1.90
13	50	16.4	547	2.90
14	35	29.9	182	0.59
15	37	25.3	434	1.69
16	61	7.8	92	1.27
17	41	59.2	1172	2.62
18	43	22.2	752	2.54
19	47	21.9	355	1.54
20	42	7.7	518	2.78
21	55	14.3	307	1.97
22	43	23.1	631	2.12
23	43	18.7	407	2.10
24	48	16.4	71	0.52
25	72	10.3	149	1.39
Mean	**52**	**18.1**	**369**	**1.81**
Standard deviation	**12**	**10.3**	**237**	**0.60**

Tensile test data of single jute fibre extracted from raw yarn

Fibre or test number	Fibre diameter (μm)	Tensile modulus (GPa)	Tensile strength (MPa)	Strain at failure (%)
1	47	33.5	910	2.88
2	38	23.9	880	2.43
3	41	20.7	209	1.28
4	60	11.1	87	0.81
5	70	10.3	221	1.88
6	53	12.2	302	1.82
7	39	19.1	282	1.27
8	51	25.5	769	2.66
9	56	13.9	542	3.08
10	73	12.5	433	2.48
11	42	20.4	229	1.21
12	60	15.4	137	0.91
13	47	26.0	884	3.08
14	63	9.7	99	1.32
15	37	32.9	296	0.96
16	50	21.0	722	2.74
17	39	22.2	455	2.25
18	61	11.5	331	2.39
19	101	10.0	279	2.45
20	45	20.1	439	2.22
21	58	9.4	174	2.04
22	51	16.0	485	2.28
23	64	20.7	444	2.05
24	69	8.4	81	1.14
25	43	23.7	590	2.32
26	57	15.6	436	2.39
27	67	19.3	473	2.19
28	53	18.07	267	1.63
Mean	**55**	**18**	**409**	**2.01**
Standard deviation	**14**	**7**	**241**	**0.66**

Tensile modulus (GPa) of composites

Composites manufactured with	1	2	3	4	5	Mean	Standard deviation
Root	31.6	28.7	29.2	32.6	31.8	**30.8**	**1.5**
Breaker card sliver	13.0	13.9	13.2	14.5	11.3	**13.2**	**1.1**
Finisher card sliver	12.2	12.3	12.2	12.2	12.8	**12.3**	**0.2**
Tossa jute 1	21.8	27.2	25.0	24.9	27.3	**25.2**	**2.0**
Tossa jute 2	31.5	38.6	33.4	33.1	30.1	**33.3**	**2.9**
Yarn HEC sized	20.0	21.1	19.3	18.8	19.2	**19.7**	**0.8**

Back calculated tensile modulus (GPa) of composites

Composites manufactured with	1	2	3	4	5	Mean	Standard deviation
Root	65.8	59.4	60.5	68.0	66.3	**64.0**	**3.4**
Breaker card sliver	31.2	33.9	31.8	35.7	26.2	**31.8**	**3.2**
Finisher card sliver	29.7	30.0	29.7	29.7	31.5	**30.1**	**0.7**
Tossa jute 1	52.6	67.2	61.3	61.0	67.5	**61.9**	**5.4**
Tossa jute 2	60.5	75.0	64.4	63.8	57.7	**64.3**	**5.9**
Yarn HEC sized	46.8	49.7	44.9	43.6	44.7	**46**	**2.1**

Tensile strength (MPa) of composites

Composites manufactured with	1	2	3	4	5	Mean	Standard deviation
Root	284	301	312	313	304	**303**	**10**
Breaker card sliver	132	157	143	134	130	**139**	**10**
Finisher card sliver	136	130	129	119	114	**126**	**8**
Tossa jute 1	243	239	253	261	290	**257**	**18**
Tossa jute 2	285	334	322	346	290	**315**	**24**
Yarn HEC sized	211	211	202	201	210	**207**	**5**

Back calculated tensile strength (MPa) of composites

Composites manufactured with	1	2	3	4	5	Mean	Standard deviation
Root	564	602	626	628	608	**606**	**23**
Breaker card sliver	281	355	314	287	276	**303**	**29**
Finisher card sliver	300	282	279	249	234	**269**	**24**
Tossa jute 1	538	527	565	586	665	**576**	**49**
Tossa jute 2	524	624	600	649	535	**586**	**49**
Yarn HEC sized	466	466	442	439	463	**455**	**12**

Strain at failure (%) of composites

Composites manufactured with	1	2	3	4	5	Mean	Standard deviation
Root	0.97	1.09	1.14	1.06	1.07	**1.07**	**0.05**
Breaker card sliver	1.49	1.55	1.49	1.32	1.53	**1.48**	**0.07**
Finisher card sliver	1.55	1.44	1.44	1.49	1.47	**1.48**	**0.04**
Tossa jute 1	1.13	0.94	1.12	1.11	1.21	**1.10**	**0.09**
Tossa jute 2	0.93	1.08	1.07	1.17	1.00	**1.05**	**0.08**
Yarn HEC sized	1.36	1.41	1.39	1.43	1.44	**1.40**	**0.03**

9.5 Appendix F: Publication

Nag R.K., Long A.C., & Clifford M.J., 'Influence of Hydroxyethyl Cellulose Treatment on the Mechanical Properties of Jute Fibres, Yarns, and Composites'. Proc. International Conference on Natural Fibers—Sustainable Materials for Advanced Applications 2013, University of Minho, 9 to 11 June 2013