

Tensile Properties of As-received and Surface-treated Coir Fibres and Composites



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Abstract

The primary aim of this research project was to characterise and to determine the tensile properties of as-received and surface-treated coir fibres prior to use in composites. The term “as-received” is defined here as where the coir fibres that were received from the supplier was cleaned using a vacuum device, washed thoroughly in tap water for 30 minutes and dries at 80 °C for two hours. The surface treatment of the as-received coir fibres in sodium hydroxide involved immersion at specified concentrations of 1, 5 and 20 % for 24, 48 and 72 hours at 30, 50 and 80 °C. The silanes used were (3-aminopropyl) trimethoxy silane and (3-glycidyloxopropyl) trimethoxy silane, where the NaOH concentrations studied were 0.5, 1 and 1.5 % in distilled water.

The heat treatment temperature was of particular interest because it is known that lignin can cross-link above 140 °C: it was anticipated that this would lead to an increase in the Young’s modulus. The heat treatment was carried out at 40, 60, 80, 120, 160, 180 and 200 °C in a vacuum oven and tube furnace in air. The vacuum oven was used to investigate the effect of temperature in the absence of air. A brief study was undertaken to investigate the effect of lateral compaction of the fibres on the tensile properties. This was undertaken to determine if the compaction of the intrinsic porosity in the fibres would influence the apparent tensile properties.

Variability in the diameter of coir fibre along the length of randomly selected fibres was observed. The tensile strength was found to decrease as a function of the coir fibre diameter. Similar to the findings on the influence of diameter on the tensile properties, a steady decrease in the tensile strength but an increase in the Young’s modulus of coir as the gauge length increased was observed.

However, heat treatment of coir fibre in the absence of air led to a reduction in the tensile strength of the fibres especially above heat treatment temperature of 160 °C. A reduction of 12 to 27% in strength was observed for the vacuum oven heat-treated coir fibres and a reduction of 28 to 88% observed in the tube furnace (with air) treated fibres. Unidirectional arrays of coir fibres were hybridized with spread E-glass to manufacture a new class of hybrid composites. An improved tensile property was recorded for the E-glass hybridized coir fibres such as has not been reported in the literature.

Dedications

This thesis is dedicated to:

The one and only true God.

“So then, it is not of him who wills nor of him who runs but of God who shows mercy,”
Romans 9, verse 16.

My late father

An inventor and my mentor. “Do not say you do not know it, all you need is to read it and then you will know it”-Honourable John. C. Udebuani. You were overjoyed when I was considered for the scholarship for my PhD in the UK in 2016. When I came home to see you in 2019, the way you wept at my departure still pierces my soul, I never knew it was the last time I would see you alive. Continue to rest in the bosom of the Lord.

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Table of Contents

Contents

1 Introduction	1
1.1 Background to the Research	1
1.2 Aims and objectives	2
1.3 Structure of the Thesis	2
2 Literature Review	3
2.1 Introduction	3
2.2 Natural fibres	3
2.2.1 Properties and applications of natural fibres and their composites	7
2.2.2 Issues associated with natural fibres and their composites	11
2.2.2.1 Relatively poor tensile strength of natural fibres	13
2.2.2.1.1 Strength as a function of the diameter of the fibre	13
2.2.2.1.2 Strength as a function of the gauge length of the fibre	14
2.2.2.1.3 Strength as a function of the strain rate	16
2.2.2.1.4 Strength as a function of the density of defects present in the fibre	16
2.2.2.1.5 Strength as a function of the porosity and microfibril angle of the fibre	16
2.2.2.1.6 Strength as a function of age, specie, origin and method of extraction of fibre	16
2.2.2.2 Moisture ingress in natural fibres	17
2.2.2.3 Wettability of Natural Fibres	20
2.2.3 Coir fibre	23
2.2.3.1 The unique features of coir fibre	26
2.2.3.2 Microstructure and morphology of coir fibre	27
2.3 Treatment for coir fibres	32
2.3.1 NaOH treatment	32
2.3.1.1 Reports on the NaOH treatment of coir fibres	33
2.3.2 Silane treatment of coir fibres	35
2.3.3 Heat Treatment of coir fibres	36
2.3.4 Hot compaction of fibres	37
2.4 Moisture absorption kinetics of natural fibres	38
2.5 Tensile testing of coir fibre	39
2.5.1 Weibull Statistics for the analysis of the tensile strength of reinforcing fibres	41
2.6 Density relationships in natural fibres	42

2.7 Thermogravimetric analysis of natural fibres	43
2.8 Manufacturing methods for Composites	44
2.9 Hybrid composites.....	47
2.10 Summary of literature review.....	49
3 Experimental Techniques and Methodology	50
3.1 Materials.....	50
3.1.1 Reinforcements.....	50
3.1.2 Resin Systems.....	51
3.1.3 Chemicals	52
3.2 Preparations and Methods	53
3.2.1 Sodium hydroxide treatment of coir fibres	53
3.2.1.1 Factorial-based design of experiments.....	53
3.2.1.2 Preparation of NaOH solutions at specified concentrations (1, 5 and 20 %).	54
3.2.1.3 Treatment of coir fibre using specified concentrations of NaOH.....	55
3.2.2 Treatment of coir fibres using 3-(aminopropyl) trimethoxysilane and 3-(glycidyloxypropyl) trimethoxy silane.	56
3.2.3 Hot-compaction of coir fibre.....	58
3.2.4 Heat treatment of coir fibres in vacuum oven and in a tube furnace	60
3.3 Water absorption of coir fibres	62
3.4 Production of unidirectional coir fibre arrays	62
3.5 Spreading of E-glass fibre bundles.....	63
3.5.1 Mechanically-induced fibre spreading.....	65
3.5.2 Post-processing of spread E- glass fibre.....	66
3.6 Single fibre tensile testing.....	69
3.6.1 Preparation and end-tabbing of tensile test specimen.	69
3.6.2 Tensile Testing of as-received coir fibre	70
3.6.3 Tensile testing of spread E-glass and T700 carbon fibres.....	71
3.7 Manufacture of composites.....	71
3.7.1 Manufacture of coir fibre composites using hand-lay-up	71
3.7.2 Manufacture of Spread E-glass composites.....	72
3.7.3 Manufacture of coir-glass hybrid composites	73
3.7.4 Manufacture of coir composites using vacuum bagging	73
3.7.5 Tensile testing of composites	76
3.8 Optical microscopy.....	76

3.9 Scanning Electron Microscopy	76
3.10 Thermogravimetric analysis.....	77
3.11 Differential Scanning Calorimetry	77
4 Results and Discussions.....	78
4.1 Introduction.....	78
4.2 Variability in the diameter of coir fibre	78
4.2.1 Possible reasons for the observed variability in the fibre diameter	81
4.3 Tensile properties of as-received coir fibres	86
4.3.1 Effects of the fibre diameter on the tensile properties of as-received coir fibres	86
4.3.2 Effect of gauge lengths on the tensile strength of as-received coir fibres	90
4.3.3 Effects of the cross-head displacement rates of the mechanical test machine on the tensile properties of coir fibres	93
4.3.4 Failure analysis of as-received coir fibres	96
4.3.5 Weibull distribution of as-received coir fibres.....	97
4.3.6 Microstructural Characterisation of coir fibres	99
4.3.6.1 Image analysis of as-received coir fibres	106
4.3.6.1.1 Determination of the volume fraction of pores in as-received coir fibre via ImageJ ..	107
4.4 Treatment of coir fibres using sodium hydroxide (NaOH).....	109
4.4.1 Visual and microstructural observations of NaOH-treated coir fibres	109
4.4.1.1 Image Analysis of NaOH-treated coir fibres.....	114
4.4.2 Effects of NaOH treatments on the tensile properties of as-received coir fibres	114
4.4.2.1 Factorial analysis results for Strength, Stiffness and Elongation for NaOH treated coir fibres	121
4.4.2.2 Weibull modulus of the NaOH-treated coir fibres.....	123
4.4.3 Failure analysis of NaOH-treated coir fibres.....	127
4.4.4 Weight losses of coir fibres after NaOH treatment	130
4.5 Hot-compaction of coir fibre.....	131
4.5.1 Tensile properties of hot-compacted coir fibres	132
4.5.1.1 Factorial regression models for the tensile properties of hot compacted coir fibres	134
4.5.1.2 Weibull Distribution of hot compacted coir fibres	136
4.5.1.3 Microstructure of the as-received hot-compacted coir fibres	137
4.5.1.4 Failure analysis of AR hot-compacted coir fibres	139
4.5.2 Hot-compaction of water-treated coir fibres	140
4.5.2.1 Tensile properties of water-treated hot-compacted coir fibres.....	140

4.5.2.1.1 Factorial regression models for the water-treated hot compacted coir fibres for diameter, strength and stiffness.....	142
4.5.2.1.1.1 Diameter versus Pressure, Temperature and Duration at 85% and 95% confidence levels for the water-treated hot-compacted coir fibre.....	143
4.5.2.1.1.2 Strength versus Pressure, Temperature and Duration at 75% Confidence level	144
4.5.2.1.1.3 Stiffness versus Pressure, Temperature and Duration at 70% confidence level	145
4.5.2.2 Weibull analysis of water-treated hot-compacted coir fibres.....	146
4.5.2.4 Tensile test graphs of water-treated hot-compacted coir fibres.....	148
4.6 Heat treatments of coir fibres	150
4.6.2 Weibull Analysis of heat-treated coir fibres	153
4.6.3 Failure analysis of the heat-treated coir fibres.....	154
4.6.4 Scanning electron microscopy analysis of coir fibres before and after heat treatment ...	156
4.7 Treatment of coir fibres using silane	158
4.7.1 Tensile properties of the silane treated coir fibres.....	159
4.7.2 Weibull characterization of silane treated coir fibres	162
4.7.3 Failure traces of amino and epoxy silane-treated coir fibres	163
4.7.4 Scanning Electron Microscopy of silane treated coir fibres.....	165
4.8 Water absorption of coir fibres	166
4.9 Thermal properties of coir fibre	168
4.10 Synthetic fibre reinforcements and matrices.....	175
4.10.1 Spread E-glass fibre.....	175
4.10.1.1 Tensile and Weibull properties of the spread E-glass fibre	175
4.10.2 T700 Spread carbon fibre reinforcement	178
4.10.2.1 Tensile and Weibull properties of T700 Spread carbon fibre	178
4.10.3 Matrices for composites manufacture	179
4.10.3.1 Synthetic epoxy/amine thermosetting resin	179
4.10.3.1.1 Differential Scanning Calorimetry (DSC) traces of cast synthetic epoxy/amine thermosetting resin (Araldite LY3505 and XB 3403 hardener)	180
4.11 Manufacture of composites.....	183
4.11.1 Manufacture of coir composites.....	183
4.11.1.1 Tensile test results of the manufactured coir composites	183
4.11.1.2 Correlation between the thickness and the tensile properties of coir composites	185
4.11.2 Tensile properties and statistical analysis of coir sandwich composites	186
4.11.2.1 Coir sandwich composites: Correlation between the specimen thickness and the tensile properties.....	188

4.11.3 Failure modes of the manufactured composites.....	189
4.11.3.1 Failure mode of composites manufactured using Vacuum pump.....	189
4.11.3.2 Failure mode of composites manufactured using Hand layup (HLU)	190
4.11.3.3 Failure mode of amino silane treated coir composites manufactured using hand layup	192
4.11.3. 4 Failure mode of single layer Spread E-glass composites manufactured using hand layup	192
4.11.3. 5 Failure modes of GCG in comparison with CGC composites manufactured using hand layup.....	193
4.11.3. 6 Failure mode comparisons of sandwich composites manufactured using hand layup	194
4.11.4 Microstructures of the manufactured coir composites and the fibre volume fraction in the composites.....	194
4.11.5 Microstructure of spread E-glass and coir sandwich composites.....	196
5 Conclusions	200
6 Recommendations for further work	201
7 References	202

List of Figures

Figure 2.1 A classifications of natural fibres adapted from Saxena <i>et al.</i> , (2011)	3
Figure 2. 2 (a) World productions of Natural fibres 2012-2017 adapted from; (Food and Agriculture Organization Corporate Statistical Database, 2018) (b) World productions of Natural Fibres 2019 adapted from; (Discover Natural Fiber Initiative, 2020).....	4
Figure 2. 3 Prices of natural fibres adapted from; (Food and Agriculture Organization Corporate Statistical Database, 2018).....	5
Figure 2. 4 Non-renewable energy requirements for production of glass fibres in comparison with natural fibres.....	6
Figure 2. 5 CO ₂ emissions from the production of glass and natural fibres: adapted from Joshi <i>et al.</i> , (2004).....	7
Figure 2. 6 Comparison between the cost of synthetic and natural fibres adapted from: Peças <i>et al.</i> , (2018).....	7
Figure 2. 7 Different areas of applications of natural fibre composites adapted from: Adekomaya, (2020).....	9
Figure 2. 8 Global motor vehicle production showing continuous growth in the automobile industry adapted from: (International Organization of Motor Vehicle Manufacturers (OICA), 2019).....	10
Figure 2. 9 Level of production of natural fibres in comparison with synthetic fibres adapted from: (Discover Natural Fibre Initiative (DNFI) Rijavec, (2019)	12
Figure 2. 10 Effect of fibre diameter on the tensile strength of coir fibre at constant test length (Tomczak <i>et al.</i> , 2007).	14
Figure 2. 11 Tensile strength of coir fibre a function of the test length (Tomczak <i>et al.</i> , 2007)	15
Figure 2. 12 Demonstrations of contact angles formed by sessile liquid on a uniform surface (Yuan, Yuehua and Lee, 2013).....	21
Figure 2. 13 Basic processes of production of coir fibre [courtesy: G Fernando, University of Birmingham].	24
Figure 2. 14 Hayleys Sri-Lanka 3-Tie Coir fibre bundle	24
Figure 2. 15 Leading producers and global production of coir fibre adapted from: (Food and Agriculture Organization Corporate Statistical Database, 2018).....	25
Figure 2.16 Prices of coir fibre for seven consecutive years adapted from; (Food and Agriculture Organization Corporate Statistical Database, 2018).....	25
Figure 2. 17 Annual export of coir fibres adapted from: (Food and Agriculture Organization Corporate Statistical Database, 2018).....	26
Figure 2. 18 Structure of natural fibre adapted from Cordeiro, (2016) and Latif <i>et al.</i> , (2019)	28

Figure 2. 19 Longitudinal image of coir fibre showing globular protrusions referred to as tyloses (Karthikeyan, Balamurugan and Kalpana, 2014).	28
Figure 2. 20 Micrographs of coir fibre cross-section (a) showing lacuna and elementary fibres and (b) showing microfibrils (Tran <i>et al.</i> , 2015; Yan <i>et al.</i> , 2016).	29
Figure 2. 21 Variations in the concentrations of NaOH and treatment temperature as applied by different authors for surface treatments of coir fibre.....	34
Figure 2. 22 General structure of an organo-silane.	35
Figure 2. 23 Illustration of single fibre tensile test specimen mounted on a window paper frame with dashed line showing the cutting of the two sides of the paper prior to tensile testing (Buana <i>et al.</i> , 2013).	40
Figure 2. 24 Schematic representaton of a typical hot press adapted from Nam <i>et al.</i> , (2011b)	45
Figure 2 25 manually aligned coir fibre preform for composites production showing gaps in between the filaments (Kim, 2014).....	47
Figure 3.1 As-received 3-tie coir fibre bundle.....	50
Figure 3.2 Photograph of the experimental setup and equipment used for the treatment of coir fibres with NaOH.....	56
Figure 3.3 Dimensions of the coir sample for hot-compaction and the section of the coir fibre that was tensile tested.	59
Figure 3.4 Experimental set-up for hot-compaction of coir fibre.....	60
Figure 3.5 Schematic illustration of hot-press setup for hot-compaction of coir fibre.	60
Figure 3.6 Photograph of the experimental setup for drying the coir fibres in a vacuum oven: (a) Control temperature knob (b) liquid nitrogen flask (c) safety temperature knob (d) vacuum gauge (f) silica gel canister.	61
Figure 3.7 Schematic diagram of the tube furnace for the heat treatment of coir fibres.....	62
Figure 3.8 Stages of production of unidirectional coir fibre arrays.....	63
Figure 3.9 Schematic illustration of a creel of glass fibre bundle showing the relative positions of the creel and the positions where the test specimens were obtained	64
Figure 3.10 Key components of the fibre spreading rig.....	66
Figure 3.11 Schematic illustration of the fibre spreading rig used in this research Figure 3.11 Schematic illustration of the fibre spreading rig used in this research	66
Figure 3.12 Photograph of a section of Spread E-glass using the fibre-spreading rig prior to post-processing.	67
Figure 3.13 A photograph of the longitudinal view of the micro-fibre fabric-covered roller.....	67

Figure 3.14 Schematic illustration of the E-glass post-processed spread fibre using the micro-fibre fabric-covered roller indicating a V-shaped end.....	68
Figure 3.15 Schematic of the final thin section of post-processed E-glass fibre.	68
Figure 3.16 Schematic illustration of as-received coir fibre showing the section from which tensile sample was obtained	69
Figure 3.17 Schematic illustration of specimen preparation for the single fibre tensile test.	70
Figure 3.18 Experimental set up for tensile testing of coir fibres.....	70
Figure 3.19 Dimensions of the tensile test specimen for spread E-glass fibre and T700 Carbon fibre.	71
Figure 3.20 Schematic illustration of hand-layup used in composite manufacture.	72
Figure 3.21 Initial experimental set up for composite manufacture using vacuum pump.	74
Figure 3.22 Complete experimental set up for composites manufacture using vacuum pump.	75
Figure 3.23 Schematics of the composites manufactured for testing.	75
 Figure 4.1 Variability in the coir fibre diameter: a-j represent 1cm mark each along the fibre length.	79
Figure 4.2 A photograph of coir fibres before washing showing debris-rich regions.....	80
Figure 4.3 Coir fibres after washing showing traces of residual components.....	80
Figure 4.4 Histogram showing the diameters of coir fibres with debris.....	81
Figure 4.5 A photograph showing coconut tree (Courtesy: C Madueke).	81
Figure 4.6 A Photograph of coconut husks before they were sent to the coconut mill (see Section 2.2.3 for reference).	82
Figure 4.7 A Photograph of coconut husks in the waterpool (see Section 2.2.3 for reference).	82
Figure 4.8 A photograph of the drum with spikes used to separate coir fibres from the husk (see Section 2.2.3 for reference).	83
Figure 4.9 A Photograph of coir fibers from the husk (see Section 2.2.3 for reference).	83
Figure 4.10 A photograph of the drying of de-husked fibers (see Section 2.2.3 for reference).	84
Figure 4.11 photograph of coir fibres showing the combing of coir fibres after dehusking (see Section 2.2.3 for reference).	84
Figure 4.12 Photograph showing the zoning of coir fibre to determine the diameter distribution. ...	85
Figure 4.13 Average diameters of coir fibres using the zones defined in Figure 4.12.....	86
Figure 4.14 Effects of fibre diameter on the tensile strength of as-received coir fibres. The dotted line represents the linear regression for the dataset.	87

Figure 4.15 Effect of fibre diameter on the Young's modulus of as-received coir fibres. The dotted line represents the linear regression for the dataset.	88
Figure 4.16 Effect of fibre diameter on the elongation at break of as received coir fibres.	88
Figure 4.17 Effects of gauge lengths on the tensile strength of as-received coir fibre.	92
Figure 4.18 Effects of gauge lengths on the Young's modulus of as-received coir fibre.	93
Figure 4.19 Effects of cross-head displacement rate on the tensile strength of as-received coir fibres.	95
Figure 4.20 Effects of cross-head displacement rate on the Young's modulus of as received coir fibres.	95
Figure 4.21 The load/extension traces for as-received coir fibres at a cross-head displacement rate of 1mm/minute and gauge lengths of 20, 60 and 100 mm.	96
Figure 4.22 Stress/strain curves for as-received coir fibres at a cross-head displacement rate of 1mm/minute and gauge lengths of 20, 60 and 100 mm.	97
Figure 4.23 Stress/strain curves for as-received coir fibres at a cross-head displacement rate of 1mm/minute and gauge lengths of 20, 60 and 100 mm.	98
Figure 4.24 Weibull Survival probability of as-received coir fibre at 20 mm gauge length and crosshead speed of 1mm/minute.	99
Figure 4.25 Micrograph of unwashed coir fibre showing a furfuraceous (flaky) surface.	100
Figure 4.26 Micrographs of as-received coir fibre showing globular protrusions referred to as tyloses.	101
Figure 4.27 Micrograph of as-received coir fibre showing helical spirals arranged in parallels manner.	101
Figure 4.28 Figure 4.28 Optical longitudinal micrographs of as-received coir	102
Figure 4.29 Transverse section of an optical micrograph of as-received coir fibre.	103
Figure 4.30 A SEM of the cross-section of as-received coir fibre.	103
Figure 4.31 SEM of the cross-section of as-received coir fibre showing deep-rooted pores.	105
Figure 4.32 SEM of the cross-section of as-received coir fibre showing thick primary cell.	106
Figure 4.33 Micrographs showing different sizes of lacuna from different sections of as-received coir fibres (a) middle (b) tail.	106
Figure 4.34 4.34 Relationship between the fibre diameter and the porosity of the fibre	108
Figure 4.35 Relationship between the fibre diameter and the pore count/size.	108
Figure 4.36 Photographs of NaOH-treated fibres showing change in colour at (i) 20% NaOH concentration, 30 oC and for 24 hours duration and (ii) 20% NaOH concentration, 50 oC and for 24 hours duration	110

Figure 4.37 An optical micrograph of NaOH-treated coir fibre showing decrease in the size of lumens. This batch was treated at 20% NaOH concentration, 80 oC and for 24 hours.....	110
Figure 4.38 (a-c) SEM micrographs of the surfaces of NaOH-treated coir fibres at varying concentrations, temperatures and durations.....	112
Figure 4.39 (a-d) SEM images of the cross sections of NaOH-treated coir fibres at different NaOH treatment conditions.	114
Figure 4.40 showing the effects of NaOH treatment conditions on the tensile properties of coir fibre. The NaOH treatment conditions are as stated in Tables 4.6(a-c).....	119
Figure 4.41 showing the effects of NaOH treatment conditions on the Young's modulus properties of coir fibre. The NaOH treatment conditions are as stated in Tables 4.6(a-c).	120
Figure 4.42 showing the effects of NaOH treatment conditions on the elongation at failure of coir fibre. The NaOH treatment conditions are as stated in Tables 4.6(a-c).	120
Figure 4.43 Effects of NaOH concentrations on the tensile strength of as-received coir fibres at 50% NaOH concentration	121
Figure 4.44 NaOH concentration exerted the highest influence on the strength properties of coir fibre.....	122
Figure 4.45 NaOH concentration exerted the highest influence on the Young's modulus properties of coir fibres.	122
Figure 4.46 NaOH concentration exerted the highest influence on the elongation at break of coir fibres.	123
Figure 4.47 Weibull distributions of NaOH-treated coir fibres at 20% NaOH concentration, 30 oC and for duration of 24 hours.....	126
Figure 4.48 Weibull distributions of NaOH-treated coir fibres at 20% NaOH concentration, 30 oC and for duration of 24 hours.....	127
Figure 4.49 (a-e) Failed surfaces of NaOH treated coir fibres at different NaOH treatment conditions.	130
Figure 4.50 Bar charts showing the weight losses of as-received coir fibres after NaOH treatment.	131
Figure 4.51 Effect of hot compaction on the tensile properties of coir fibres (a) strength and (b) Young's modulus.....	134
Figure 4.52 Pareto Charts for the hot compacted coir fibres (a) for strength at $\alpha=0.15$ and (b) stiffness at $\alpha=0.05$	135
Figure 4.53 Microstructures of as-received hot-compacted coir fibre.....	138
Figure 4.54 Relationship between (a) the tensile strength, (b) the stiffness and the porosity of hot compacted as-received coir fibre	139

Figure 4.55 Failure traces of as-received hot compacted coir fibres.....	140
Figure 4.56 Tensile properties of water-treated hot compacted coir fibres for (a) Strength (b) Stiffness.....	142
Figure 4.57 Pareto Chart for Diameter versus Pressure Temperature and Duration at (a) 85% and (b) 95% Confidence levels	144
Figure 4.58 Pareto Chart for the strength of water treated hot compacted coir fibre at 75% confidence level.	145
Figure 4.59 Pareto Chart for the stiffness of water-treated hot compacted coir fibre at 70% confidence level.	146
Figure 4.60 Microstructure of water-treated hot-compacted coir fibre at a compaction pressure of 6 MPa, temperature of 140 oC and duration of 15 minutes.	148
Figure 4.61 Failure traces of water-treated hot compacted coir fibres.	149
Figure 4.62 Stress-strain curves for AR and water treated samples at optimum compaction conditions.....	150
Figure 4.63 Effect of heat treatment temperature on the (a) tensile strength (b) Young's modulus and (c) elongation at break of coir fibres.....	153
Figure 4.64 Stress-Strain curves of (a) vacuum oven and (b) furnace oven heat treated coir fibres at optimum strength heat treatment temperature (40 oC).	155
Figure 4.65 shows the SEM micrographs of (a) untreated (b) heat-treated coir fibres.	156
Figure 4.66 SEM micrographs of heat-treated coir fibres at 40 oC in the (a) vacuum and (b) tube furnace	156
Figure 4.67 SEM of fractured heat-treated coir fibre samples.	158
Figure 4.68 Effects of concentrations of silane on the tensile strength of coir fibres.....	160
Figure 4.69 Effects of Amino and Epoxy silane on (a) tensile strength (b) stiffness of coir fibres.	162
Figure 4.70 Failure traces of (a) amino and (b) epoxy treated coir fibres.	164
Figure 4.71 Micrographs of amino silane-treated coir fibre at 1.5% concentration of amino silane.	165
Figure 4.72 SEM of fractured (a) amino and (b) epoxy silane treated coir fibres	166
Figure 4.73 Moisture absorption of as received coir fibres as a function of the square root of time at different temperatures.	167
Figure 4.74 TGA and DTG of untreated and treated coir fibres (a) As received (b) NaOH- treated (c) vacuum oven dried (d) amino-treated (e) epoxy-treated and (f) AR hot compaction at 140 oC.	172
Figure 4.75 Tensile strengths of AR coir fibre in comparisons with the treated coir fibres.	173
Figure 4.76 Young's modulus of AR coir fibre in comparisons with the treated fibres.	173

Figure 4.77 Values of the Weibull modulus obtained from AR coir fibre in comparisons with the treated fibres.	174
Figure 4.78 Failure curves of AR coir fibre in comparisons with the treated fibres.	175
Figure 4.79 Stress-strain curves of spread E-glass fibre	176
Figure 4.80 Weibull distribution of spread E-glass fibres	177
Figure 4.81 Stress-Strain curves of T700 Spread Carbon fibre.	178
Figure 4.82 Weibull distribution of T700 Spread Carbon fibre	179
Figure 4.83 Stress-Strain curve of Araldite LY 3505 (epoxy resin) and Hardener XB 3403 (amine hardener)	180
Figure 4.84 (a) first heating scan (b) second heating scan and (c) third heating scan of epoxy/hardener.	182
Figure 4.85 Comparisons of the tensile properties of coir composites with their manufacturing methods (a) strength (b) Young's modulus.	184
Figure 4.86 Comparisons of the tensile properties of coir sandwich composites (a) strength and (b) Young's modulus	187
Figure 4.87 Comparison of stress-strain curves of single, double and triple layer coir composites manufactured using vacuum pump.	190
Figure 4.88 Comparison of stress-strain curves of single, double and triple layer coir composites manufactured using hand layup technique.	191
Figure 4.89 Comparison of stress-strain curves of coir composites manufactured using hand layup, vacuum pump and autoclave techniques.	191
Figure 4.90 stress-strain curves of amino silane treated coir composites	192
Figure 4.91 Stress-Strain curves of single layer spread E-glass composites.	193
Figure 4.92 Comparison of Stress-Strain curves of GCG and CGC.	194
Figure 4.93 (a) Single (b) double (c) tripple layer coir fibre composites. Error! Bookmark not defined.	
Figure 4.94 Micrograph showing a monolayer of post-spread 2400 Tex E-glass fibre reinforced epoxy resin composite; magnification x10.	197
Figure 4.95 (a-c) shows micrographs of coir-spread-glass-coir (CGC) composite	198

List of Tables

Table 2.1 Properties of coir and some selected natural fibres in comparison with synthetic fibres	9
Table 2.2 Chemical Composition of natural fibres.....	11
Table 2.3 Surface properties of natural fibres	22
Table 2.4 Methods and matrices used in the manufacture of coir composites	46
Table 2.5 Properties of coir fibre hybrid composites.....	48
Table 3.1 Selected properties for E-glass and T700 Carbon fibres	51
Table 3.2 Design of experiments summary for the treatment of coir fibres with NaOH using Minitab-18 Software	53
Table 3.3 Complete experimental design for the treatment of coir fibres with NaOH	54
Table 3.4 Design of experiments for the treatment of coir fibre using epoxy and amino silanes.....	57
Table 3.5 Experimental design for hot compaction of coir fibres.....	59
Table 3.6 Parameters used for operating the fibre spreading rig.....	65
Table 4.1 Compilation of coir fibre diameters and the resulting tensile properties. The data from the current study have been included for comparison purposes.	89
Table 4.2 Tensile properties of as-received coir fibres at varying gauge lengths and at constant cross-head displacement rates of 1mm/minute	92
Table 4.3 Tensile properties of as-received coir fibres at strain rates of 1, 10 and 100 mm with at a gauge length of 20 mm	94
Table 4.4 Weibull Parameters on the tensile strength of as-received coir fibres at varying gauge lengths and crosshead displacement rates.....	99
Table 4.5 Porosity of coir fibre determined from image analysis.....	107
Tables 4.6 (a-c) Tensile test results of as-received coir fibres treated with NaOH.....	117
Tables 4.7 (a-c) Weibull analysis for NaOH-treated coir fibres.....	124
Table 4.8 Tensile test result hot-compacted coir fibres	133
Table 4.9 Weibull Distribution of the hot-compacted coir fibres	137
Table 4.10 Tensile properties of water-treated hot compacted coir fibre	141
Table 4.11 Weibull parameters of water-treated hot-compacted coir fibres	147
Table 4.12 Tensile tests results of the vacuum oven heat treated coir fibres from 40 to 200 oC.....	151
Table 4.13 Tensile test results of the tube furnace heat treated coir fibres from 40 to 200 °C.....	152

Table 4.14 Summary of the Weibull findings on the heat-treated coir fibres	154
Table 4.15 Concentrations of amino and epoxy silane and the treatment durations.....	159
Table 4.16 Tensile properties of coir fibres treated using (3-Aminopropyl) trimethoxy and (3-Aminopropyl) triethoxy silane	161
Table 4.17 Weibull distributions of silane treated coir fibres.....	163
Table 4.18 Weight gain after water absorption and the diffusion coefficient of as received coir fibre at different temperatures.	168
Table 4.19 TGA of treated and untreated coir fibres.....	169
Table 4.20 Fisher Pairwise Comparisons of the tensile properties of treated and untreated coir fibres at 95% Confidence level (all treatments).....	174
Table 4.21 Tensile and Weibull properties of the spread E-glass fibre	177
Table 4.22 Glass transition temperatures for the LY3505 epoxy resin and XB3403 amine hardener	182
Table 4.23 Tensile properties of the manufactured coir composites.....	183
Table 4.24 Fisher Pairwise comparisons of coir composites	185
Table 4.25 Relationship between the thickness and the tensile properties of the monolayer coir composites manufactured using HLU and VP techniques	186
Table 4.26 Properties of coir sandwich composites	186
Table 4.27 Fisher Pairwise comparison for difference of means for coir sandwich composites.....	188
Table 4.28 Correlation between the specimen thickness and the tensile properties for coir fibre sandwich composites.....	189

1 Introduction

1.1 Background to the Research

Coir, a typical lignocellulosic fibre continues to be used in a range of applications including: durable ropes, rugs, doormats, mattresses and upholsteries. Moreover, the technical applications of coir are seen in erosion-control blankets owing to its slow rate of degradation. Other applications of coir are in automotive, building and construction (Arancon, 2008; Nguyen and Indraratna, 2017; Iman *et al.*, 2018; Dhaliwal, 2019). Natural fibres such as coir are cost-effective (see Section 2.2), readily available, non-toxic, sustainable and environmentally friendly when compared to synthetic fibres. However, they have a number of limitations. For example, the mechanical properties of coir fibres are significantly lower than their synthetic counterparts (see Table 2.1). Other limitations of coir fibres include the intrinsic variability in the fibre length and diameter (Satyanarayana *et al.*, 1986; Tomczak, Sydenstricker and Satyanarayana, 2007; Defoirdt, Biswas, Vriese, Quan, *et al.*, 2010; Mathura and Cree, 2016), high porosity (Tran *et al.*, 2015; da Luz *et al.*, 2017), low degradation temperature in the range of 170- 200 °C (Van Dam *et al.*, 2004; Ezekiel *et al.*, 2011; Dicker *et al.*, 2014) and poor resistance to moisture ingress which is typical of all natural fibres (Khalil *et al.*, 2012; Masoodi and Pillai, 2012). Therefore the commercial viability of coir fibres for use as reinforcement in fibre reinforced organic matrix composites remains uncompetitive with synthetic fibre reinforcements for structural applications.

The microstructure of coir plays a significant role in the tensile strength and Young's modulus of the fibre (see Table 2.1). The microstructure consists of globular protrusions known as tyloses, pores, elementary fibres and microfibrils within the elementary fibres and a cylindrical channel called lacuna (Chand and Satyanarayana, 1986; Bismarck *et al.*, 2001; Tran *et al.*, 2015; Yun *et al.*, 2016). Since coir has a multi-cellular microstructure composed of elementary fibrils that are packed in a helical manner (Tomczak, Sydenstricker and Satyanarayana, 2007; Tran *et al.*, 2015; Yu, Xie and Du, 2016), tensile-loading a macro fibre results in the helices uncoiling (Martinschitz *et al.*, 2008) at a relatively low stress (Yu, Xie and Du, 2016). Moreover, the presence of the lacuna, intrinsic porosity and the tyloses as mentioned earlier means that the tensile stress that can be sustained will be significantly lower than a solid fibre with an equivalent cross-section.

Surface treatments of coir fibres such as treatments using sodium hydroxide (NaOH) have been utilized to improve the tensile properties of coir fibre (Nayak, *et al.*, 2001; Nam *et al.*, 2011a; Yan *et al.*, 2016). The effects of compacting coir fibres to minimize the intrinsic pore volume fraction have scarcely been reported. Unidirectional coir mono-fibre composites were manufactured in single, double and triple layers using hand layup processing techniques in order to compare their properties.

1.2 Aims and objectives

The issue is that the mechanical properties of natural fibres such as coir are too low to compete effectively with synthetic fibres hence the aims and objectives of this research:

- (i) To evaluate the tensile properties of as-received, chemically-treated, heat-treated and compacted single-coir fibres in order to find out the effects of these treatments on their mechanical strength.
- (ii) To analyse the coir fibre strength distribution using Weibull statistical analyses as a function of chemical, thermal and physical treatment.
- (iii) To hybridise as-received coir fibres with spread E-glass and manufacture a hybrid sandwich composites to improve the mechanical strength.

1.3 Structure of the Thesis

The current chapter gives the motivational background for this research.

Chapter 2 presents detailed literature review on the compositions, microstructure and morphology, surface treatments and an analysis of the tensile properties of coir fibres.

Chapter 3 presents the experimental techniques and methods adopted in the investigations on the effect of various thermal, chemical and physical treatments of coir fibres.

Chapter 4 highlights the results obtained with the experiments carried out and the obtained results.

Chapter 5 presents the conclusions of the research with recommendations and future research respectively.

2 Literature Review

2.1 Introduction

This chapter presents a literature review that is focused on coir fibres. The first section covers a general classification for natural fibres and their mechanical, thermal, chemical and physical properties. The advantages and limitations of natural fibres are reviewed along with selected synthetic fibres. The second part of this review is focused on the microstructure of coir fibres. The third part is concerned with previous research on the techniques that have been used to improve the overall mechanical properties of coir including thermal, chemical and physical treatments. Other relevant topics that are covered in this chapter include the sorption and desorption of water in and out of coir, and the production and evaluation of coir fibre composites.

2.2 Natural fibres

A classification for natural fibres is shown in Figure 2.1 where it is seen that such fibres are derived from minerals, plants and animals. Mineral-based fibres are often associated with extractive and manufacturing industries whilst plants and animal-based fibres are associated with agriculture. Natural fibres possess desirable attributes for use as reinforcements in composites.

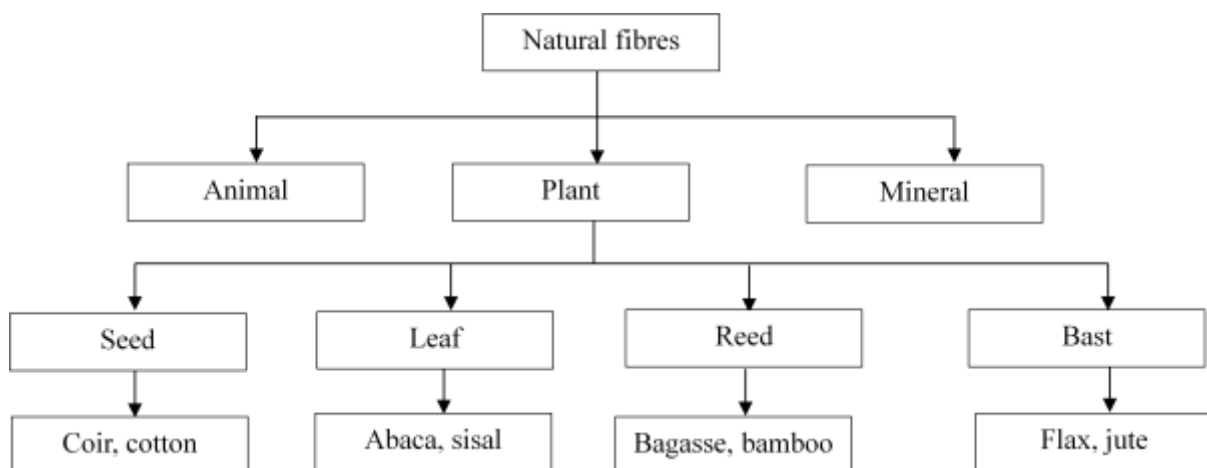
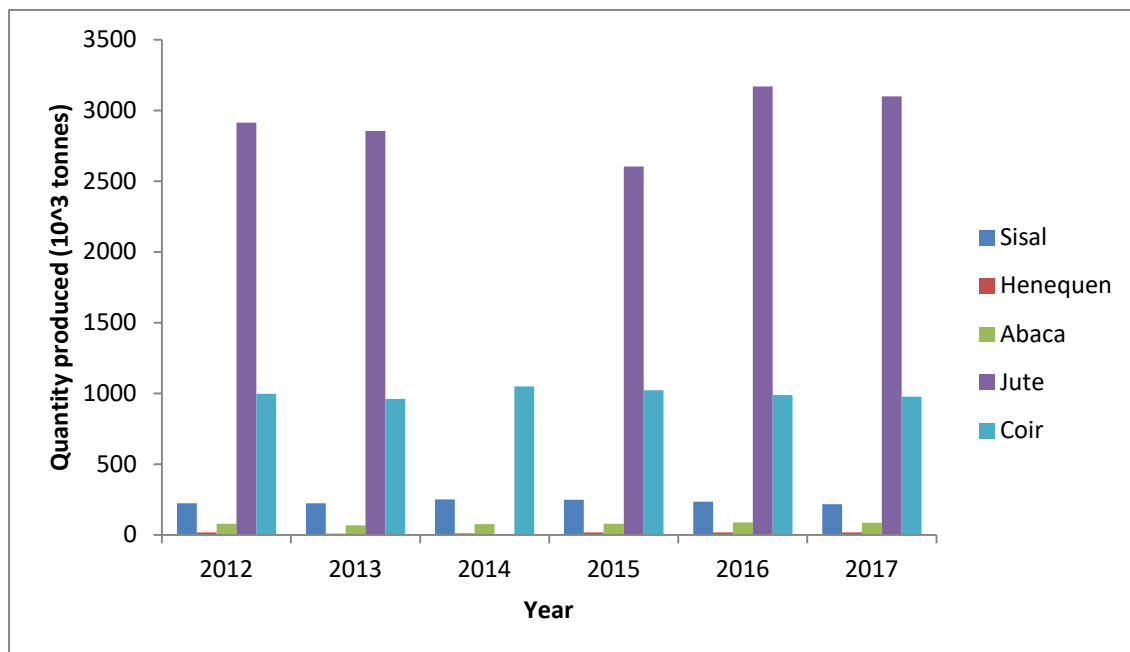


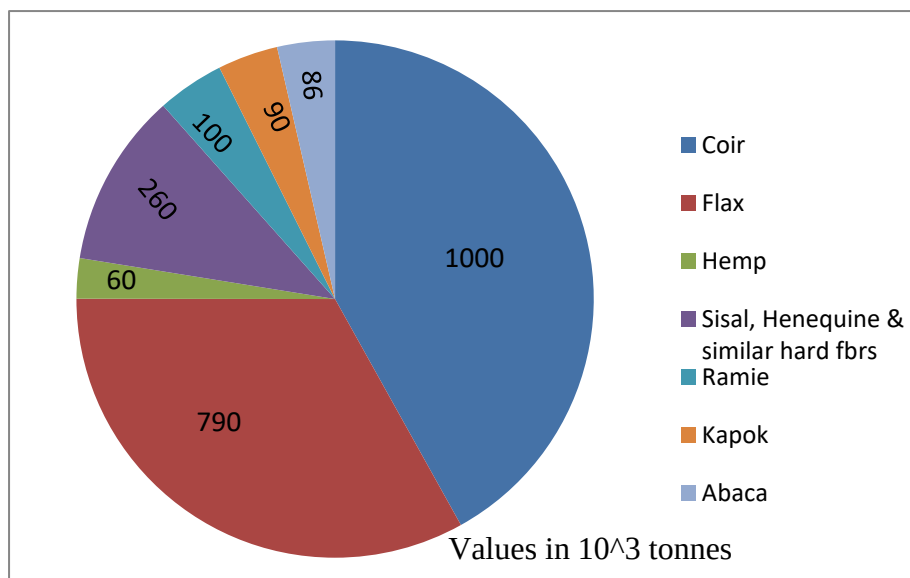
Figure 2.1 A classifications of natural fibres adapted from Saxena *et al.*, (2011)

With reference to Figure 2.2 (a) and (b), natural fibres are in abundance with yearly production of about 22.6×10^6 tonnes. Jute fibre maintained the lead for five consecutive

years. Within this period, the quantity of jute fibre produced stood at 77.78% of the total yearly average production followed by coir with 26.53% and sisal with 6.18%. The compound annual growth rate (CAGR) of natural fibre within this period was 0.64%. In 2019, coir fibre topped the chart with a total volume of production of 1,000,000 tonnes.



(a)



(b)

Figure 2.2 (a) World productions of Natural fibres 2012-2017 adapted from; (Food and Agriculture Organization Corporate Statistical Database, 2018) (b) World productions of Natural Fibres 2019 adapted from; (Discover Natural Fiber Initiative, 2020).

From Figure 2.3 abaca fibre appeared to be most expensive of all the fibres listed and coir the least. The price of abaca was initially making an average gradual yearly increase of 8.8% but stabilized from 2017 with average price of \$2475/tonne. The price of jute was fairly stable with yearly average price of \$736/tonne. Sisal recorded an all-time high in 2016 and 2017 and a drop in price by 27% in 2018 which saw the price fall from \$2025/tonne to \$1750 /tonne. The price of coir was fairly stable between 2014 and 2017 and then hit a record high in 2018, this saw the price jump from \$401/tonne to \$1772 /tonne. The reasons for this jump in price of coir are discussed in Section 2.2.3. The average yearly price ratio for abaca, sisal, jute and coir within this period was 5:4:2:1 respectively.

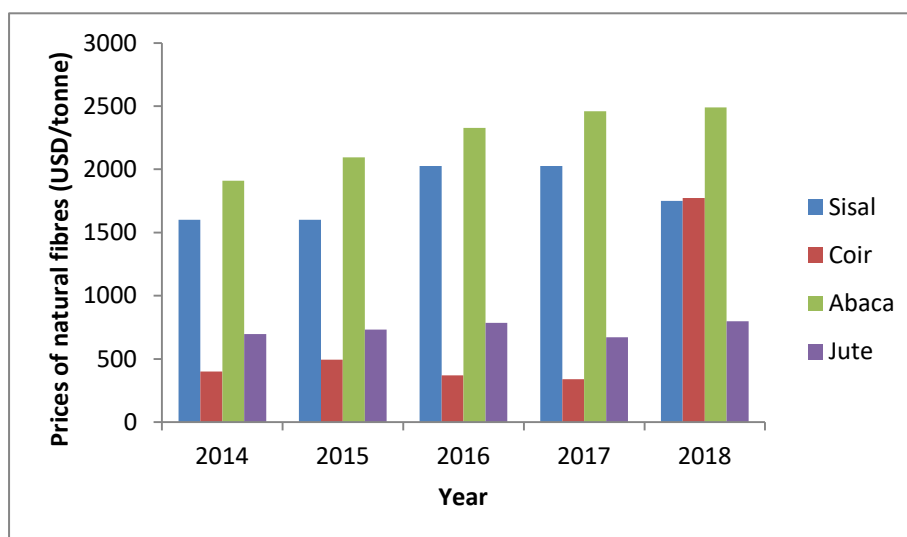


Figure 2.3 Prices of natural fibres adapted from; (Food and Agriculture Organization Corporate Statistical Database, 2018).

Previously, renewable resources such as natural fibres or lubricating oil obtained from agricultural produce were neglected because of fossil resources available as raw materials for chemical industries for production of precursors. For example polyacrylonitrile (PAN), which is a synthetic precursor for the production of carbon fibres (Tobergte and Curtis, 2013). This led to consumption of only the agricultural food products leaving the by-products such as fibres from animals and plants sources inadequately used. Thereby the main interest in agriculture was efficient food production for human consumption, leaving non-edible products for engineering applications grossly underdeveloped.

Currently, natural fibres have gained significant recognition in various spheres of life. The non-renewable nature of fossil resources, environmental hazards associated with their

extractions and the inevitable increase in CO₂ release to the atmosphere has led to awareness of potential uses of agricultural wastes to partially if not completely replace fossil resources (Jarukumjorn and Suppakarn, 2009; Xie *et al.*, 2010; Tobergte and Curtis, 2013). In the case of non-renewable reinforcements, the energy consumption arise from operations such as the production of glass fibre and some synthetic fibres are in the areas of melting, spinning, transport, etc. while for natural fibres, their energy requirement are mostly in cultivation, transport, and fibre separation (Dissanayake *et al.*, 2009; Schmitz *et al.*, 2011). The total non-renewable energy requirements for the production of glass fibre, flax fibre and China reed fibre are compared as shown in Figure 2.4, thus indicating that more energy is used (in total) in the production of synthetic fibres such as glass fibre than natural fibre. It follows that the total pollutant emissions such as CO₂ generated by the production of synthetic fibre are much more than those generated by the production of natural fibre (see Figure 2.5). Biomass of natural fibres are a good source of bio-ethanol and lignin production (Hemmati, Seradj and Mehrabi, 2017; Peças *et al.*, 2018; Dhaliwal, 2019). The differences in the prices of natural fibres and synthetic fibres are shown in Figure 2.6. Besides, the extensive usage of synthetic fibres may lead to acute ecological damage, it can also contribute to complete depletion of natural resources such as crude oil (Chauhan, Kärki and Varis, 2019).

Though the use of man-made fibres may be growing in leaps and bounds, the need for natural fibres can never be overemphasised. One of the key drivers of such need is the increasing consumer awareness of environmental concerns.

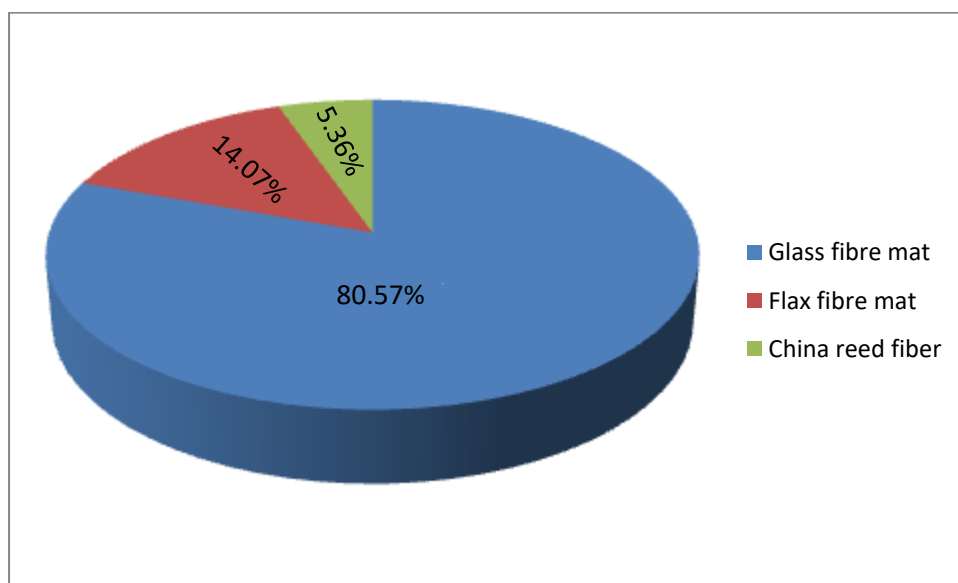


Figure 2.4 Non-renewable energy requirements for production of glass fibres in comparison with natural fibres.

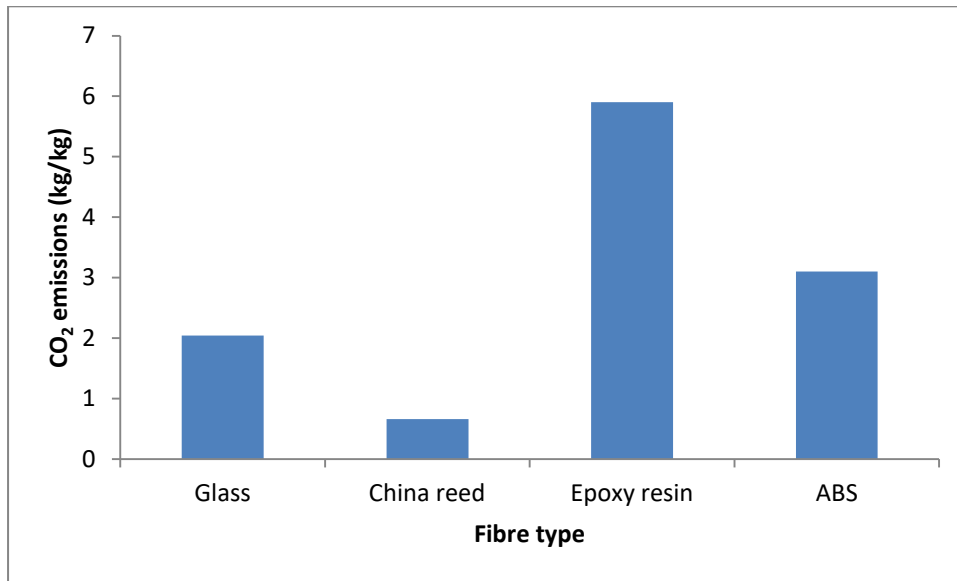


Figure 2.5 CO₂ emissions from the production of glass and natural fibres: adapted from Joshi *et al.*, (2004).

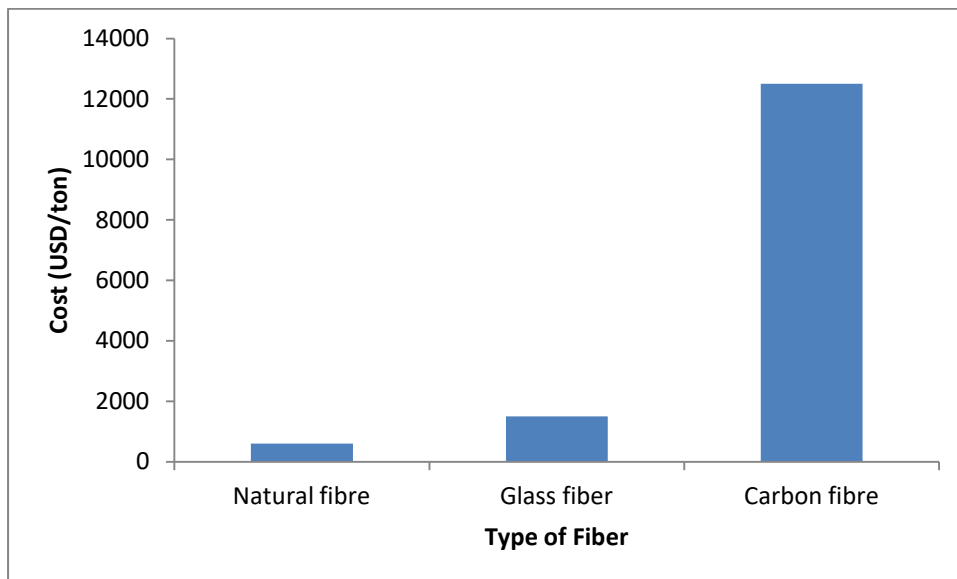


Figure 2.6 Comparison between the cost of synthetic and natural fibres adapted from: Peças *et al.*, (2018).

2.2.1 Properties and applications of natural fibres and their composites

Though the properties of natural fibres such as the tensile strength and stiffness have been reported to be too low (see Table 2.1) to compete effectively with synthetic fibres, yet they are making significant inroads in various industrial sectors. Figure 2.7 shows the use of natural fibres as reinforcements in composites for various applications. They have a lower

density than glass and most synthetic fibres and therefore offer significant weight savings and fuel efficiency when compared with synthetic fibres (Peças *et al.*, 2018; Chauhan, Kärki and Varis, 2019). This light weight advantage has propelled their adoption in the automotive sector such that natural fibre reinforced composites are currently used in vehicle interior parts such as seat back, interior door panels, spare tyre covers, package trays, map boxes, seat liners, and sun roof frames which weighs 50% less than a metal sun roof frame (Suddell, 2008; Chauhan, Kärki and Varis, 2019). With the continuous growth in motor vehicle production as shown in Figure 2.8, it is expected that production and demand for natural fibres will increase. The building, construction and sporting sectors are not left out in the use of natural fibre composites. Audio components, Cellucomp fishing rods found in the leisure industry are based on natural fibres (Suddell, 2008). Sustainability and availability of raw materials have been counted as part of the drivers in the use of natural fibre composites (Rohit and Dixit, 2016; Khan, Sultan and Ariffin, 2018; Chauhan, Kärki and Varis, 2019). Besides low density, natural fibres possess high specific properties (see Table 2.1). Furthermore, natural fibres in composites have displayed excellent acoustic and thermal properties when compared with synthetic fibres such as carbon and glass, low tool wear performance, no off-gassing of toxic compounds and positive effects on agriculture (Taylor, Karus and Kaup, 2002; Chauhan, Kärki and Varis, 2019).

Table 2.1 Properties of coir and some selected natural fibres in comparison with synthetic fibres.

Fibre	Tensile Strength (MPa)	Tensile Modulus (GPa)	Specific strength (KNm/kg)	Weibull Modulus	Elongation at break (%)	Density (kg/m ³)	Onset Degradation Temperature (°C)
Jute	393-1316	13- 91.9	302-615	2.74 - 3.0	1.3-1.8	1300	240
Sisal	468-640	9.4-22	323-441	3.7	2.0-2.5	1450	230
Coir	131-175	4-6	114-152	2.74	30.0-45.0	1150	230
Hemp	514 - 2140	24.8 - 143.2	347-1446	4.6	1.6 -1.8	1480	205
E-Glass	2000-3500	70	130-211	-	2.5	1900-2500	N/A
T700 Carbon	4000	230-240	2105	-	1.4-1.8	1800-2000	N/A

Compiled from the following references: (Carl and Brook, 1985; Ticoalu, Aravinthan and Cardona, 1997; Gassan and Bledzki, 1999; Brígida et al., 2010; Alves Fidelis et al., 2013; Saheb and Jog, 2015; Fiore et al., 2016; Latif et al., 2019; Park 2006; Dam 2004;. Bechermann; Li and Tabi; Ferreira, Cruz and Figueiro, 2018; Brígida et al., 2010b; Sanjay, Arpitha and Yogesha, 2015).

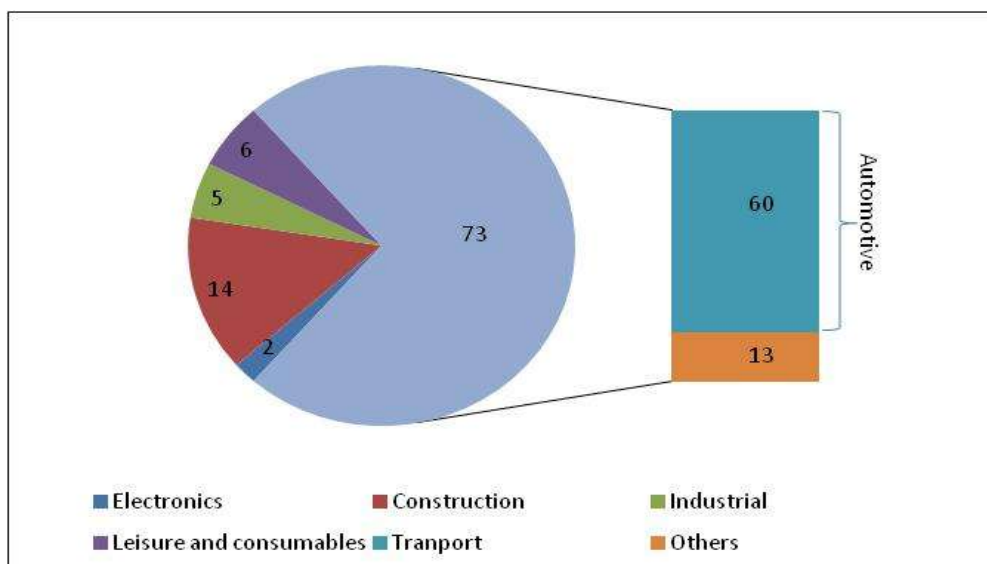


Figure 2.7 Different areas of applications of natural fibre composites adapted from: Adekomaya, (2020).

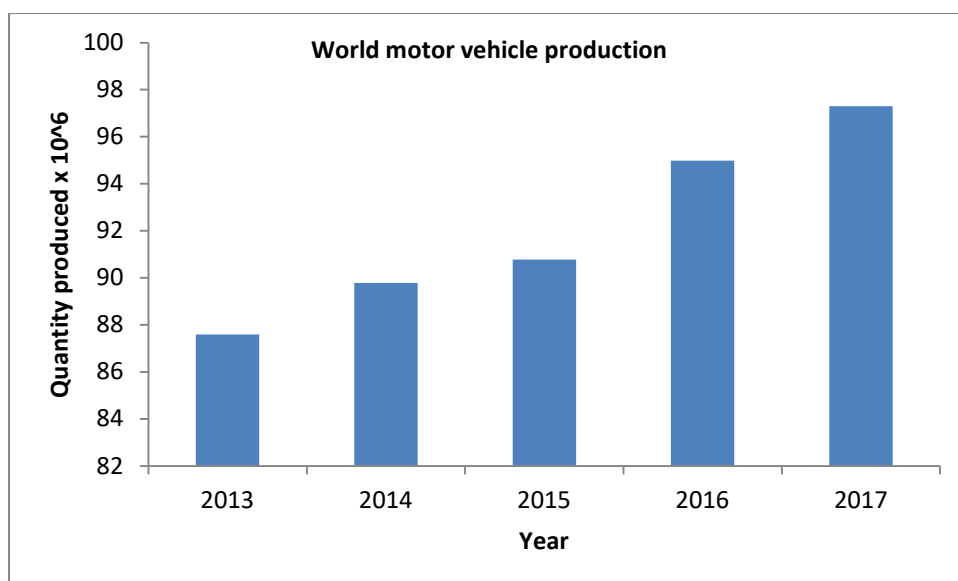


Figure 2.8 Global motor vehicle production showing continuous growth in the automobile industry adapted from: (International Organization of Motor Vehicle Manufacturers (OICA), 2019).

The basic chemical components of natural fibres are hemicellulose, cellulose, lignin and pectin as displayed in Table 2.2. The nature of these chemical compositions varies from fibre to fibre. The properties of each of the chemical components influence the overall properties of the fibre. The cellulose, hemicellulose and lignin are interconnected. Cellulose content has been associated with the strength of the fibre. The crystalline region of the cellulose is not affected by moisture absorption. Hemicellulose has been associated with an affinity for moisture, thermal degradation and biodegradation as a result of its low crystallinity and low degree of polymerisation, lignin is rather a complex molecule with a three-dimensional structure confirmed to be amorphous and as a result displays moisture-dependent properties (Persson, 2000a). The cellulose content of the fibres are in the range; 43-74.4%, the lignin content 3.7-35.8%, whereas the hemicellulose, pectin and moisture contents are in the ranges: 10-13%, 2-10% and 8-13% respectively.

Table 2.2 Chemical Composition of natural fibres.

Fibre	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Pectin (%)	Wax (%)	Ash (%)	MFA (°)	Moisture content (%)
Jute	61-71.5	13.6-20.4	12-13	0.2	0.5	-	8.0	12.6
Sisal	67-78	10.0-14.2	8.0- 11.0	10.0	2.0	-	20.0	11.0
Coir	43-46	17.6-21	31-35.8	1.8-4	-	2.6- 3.4	45	8.0
Hemp	70.2- 74.4	17.9-22.4	3.7-5.7	0.9	0.8	-	6.2	10.0 - 10.8

Compiled using the following sources: (Bledzki and Gassan, 1999; Khalil, Alwani and Omar, 2006; Wu et al., 2006; Dhakal, Zhang and Richardson, 2007; Kabir, Wang, Lau and Cardona, 2012; Shahzad, 2013; Bakri, Chandrabakty and Soe, 2016; Mathura and Cree, 2016; Latif et al., 2019).

2.2.2 Issues associated with natural fibres and their composites

Although natural fibres possess certain advantages over synthetic fibres, their limitations have to be overcome for them to compete favourably with synthetic fibres. The following section presents a brief review on the limitations associated with natural fibres such as strength substituting to moisture ingress and wettability.

The average yearly production of synthetic fibres from 2015 to 2019 was 72 million metric tonnes as shown in Figure 2.9. A CAGR of 2.55% was recorded. This yielded 42 million metric tonnes higher than the average yearly production of natural fibres.

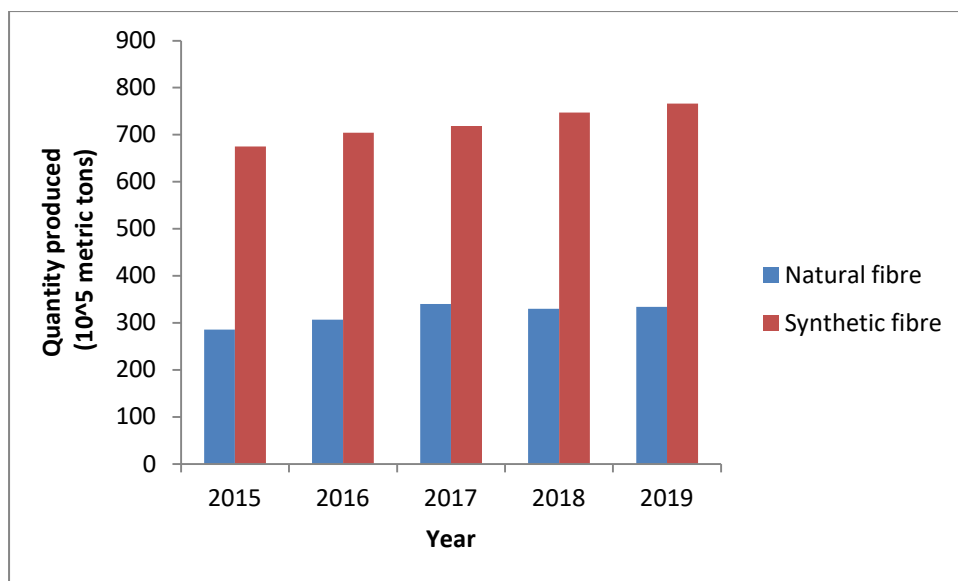


Figure 2.9 Level of production of natural fibres in comparison with synthetic fibres adapted from: (Discover Natural Fibre Initiative (DNFI) Rijavec, (2019).

The annual average production of synthetic fibres far outweighs that of natural fibres. The reason is that synthetic fibres such as carbon and glass are still in very high demand owing to their superior performance properties such as high specific tensile strength and stiffness, uniform diameter, as well as established manufacturing techniques. Natural fibres are still far behind synthetic fibre composites both in production and in application owing to a number of reasons. The reasons include: lack of technical advancements in the raw materials manufacturing process, non-availability of enhanced equipment in the processing of natural fibre which reduces efficiency and rate of production, incompatibility of the natural fibre surface functional group with the functional group of the existing hydrophobic resins systems in different end-use sectors resulting to poor fibre-matrix interfacial adhesion with poor load-transfer efficiency (Rohit and Dixit, 2016; Chauhan, Kärki and Varis, 2019). Natural fibres can begin to degrade at temperatures within the range of 170-200 °C (Van Dam *et al.*, 2004; Ezekiel *et al.*, 2011; Dicker *et al.*, 2014). As a consequence there exists a drawback in the use of natural fibres as reinforcement in some thermoplastic matrices of high melting temperature. Variability in their mechanical properties is influenced by several factors such as lack of standardization in processing and surface treatments. Their susceptibility to moisture absorption and poor wettability have been mentioned as the downsides in the use and application of natural fibres (Azwa *et al.*, 2013; Hariprasad, Dharmalingam and Praveen Raj, 2013; Peças *et al.*, 2018; Dhaliwal, 2019). An often-overlooked but significant hindrance to the wide use of natural fibres in composites application is restricted access due to geographical barriers between the countries where natural fibres are grown and countries

where they are used as reinforcements in composites production such as North America, the United Kingdom, France, Germany and Spain.

2.2.2.1 Relatively poor tensile strength of natural fibres

Investigations into the tensile strengths of natural fibres have been carried out and reported by several researchers. A number of factors have been identified to exert influence on the tensile strength of these fibres.

2.2.2.1.1 Strength as a function of the diameter of the fibre

Natural fibres possess a common property of non-uniformity along their length and as a result have variable diameter and variable cross-sectional area. The mechanical properties of natural fibres such as the tensile strength have been reported to be a function of their equivalent diameter. The diameter of the fibre has also been linked to the interfacial shear strength τ as stated in equation [2.1] (Jawaid, Khalil and Bakar, 2010; Fiore *et al.*, 2016).

$$\tau = \frac{F_{max}}{\pi d l_e} \quad [2.1]$$

where d is the diameter of the fibre, F_{max} is the debonding force and l_e is the embedded length of the fibre in the matrix.

The inverse relationship between the tensile strength and the fibre diameter has been reported consistently for most lignocellulosic fibres (Mukherjee and Satyanarayana, 1984; Tomczak, Sydenstricker and Satyanarayana, 2007; Fiore *et al.*, 2016). In this respect, (Monteiro *et al.*, 2011) suggested a hyperbolic inverse correlation of strength and diameter with evidences from nine different types of natural fibres as;

$$\sigma = \frac{A}{d} - B \quad [2.2]$$

where σ is the variation of strength, d is the diameter of the fibre and A and B are constants.

Griffith's model displays an indirect proportionality between the Young's modulus and the fibre diameter as shown in Equation 2.3;

$$E_f(d_f) = A + \frac{B}{d_f} \quad [2.3]$$

where A and B are constants, d_f = diameter of the fibre and $E_f(d_f)$ = Young's modulus or property analysed.

Mathura and Cree, (2016) reported an increase in the tensile strength from 48.45 to 134.41 MPa as the fibre diameter decreased. Studies carried out by Tomczak, Sydenstricker and

Satyanarayana, (2007) on the lignocellulosic fibres from Brazil revealed similar trend as shown in Figure 2.10.

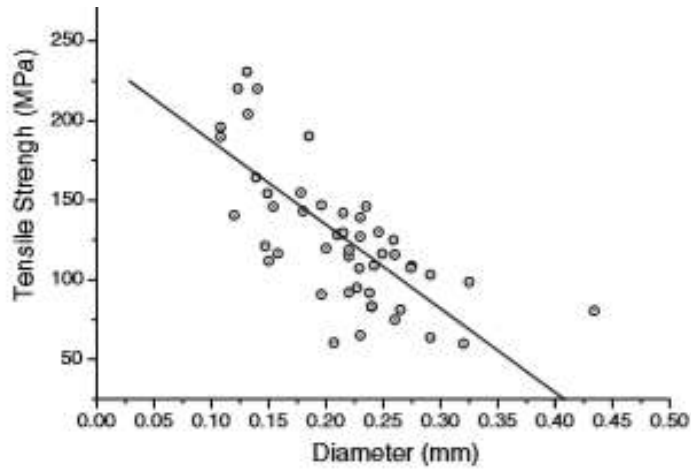


Figure 2.10 Effect of fibre diameter on the tensile strength of coir fibre at constant test length (Tomczak *et al.*, 2007).

The explanation is that natural fibres are non-uniform with intrinsic diameters. The thicker the fibre the more flaws and irregularities it contains and the thinner the fibre the fewer flaws and irregularities or defects present (Satyanarayana and Sukumaran, 1986; Monteiro *et al.*, 2011). Therefore as the diameter of the fiber increases, the probability of the number of defects present will increase.

2.2.2.1.2 Strength as a function of the gauge length of the fibre

The tensile strength of fibres has been associated with its gauge length. Several researchers have reported the influence of gauge length on the tensile properties of reinforcing fibres. The tensile strength has been observed to increase with decrease in the gauge length (Mukherjee and Satyanarayana, 1984; Defoirdt *et al.*, 2010; Fidelis *et al.*, 2013; Mathura and Cree, 2016). It follows similar trends as with the fibre diameter: the longer the gauge length the higher the probability of the presence of flaws and as a result a decrease in tensile strength as shown in Figure 2.11.

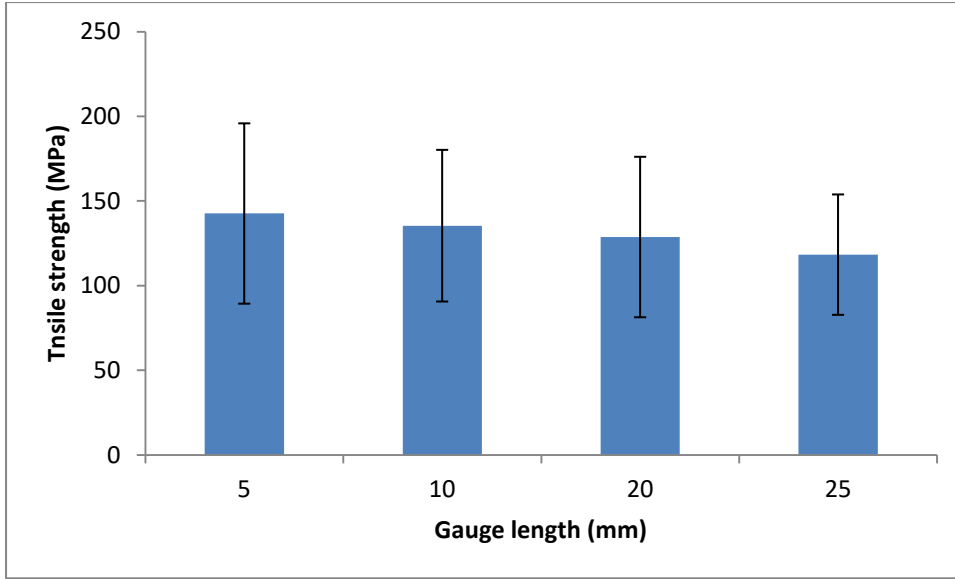


Figure 2.11 Tensile strength of coir fibre a function of the test length (Tomczak *et al.*, 2007).

In Figure 2.11, a linear relationship exists between the strength and the gauge length. As the sample gauge length gets longer, the strength of the sample decreases and vice versa. Several authors have reported that the reason for this relationship is as a result of the number of defects and so the weak links present in the fibre. The incremental change in strength $d\sigma$ of the fibre with a corresponding change in gauge length dL according to Mukherjee (1984) is represented by Equation [2.4];

$$d\sigma = \alpha \frac{dL}{L} \quad [2.4]$$

where σ is the strength of the fibre, α is a measure of the frequency of occurrence of weak links; and L is the gauge length.

The general nature for change in strength with change in gauge length is thus given by integrating Equation [2.4]; where the strength of the fibre of length L is represented by σ_o

$$\sigma_L = \sigma_o + \alpha \ln \frac{L}{L_o} \quad [2.5]$$

The slope of the tensile strength versus gauge length yields is equal to the defect density (Satyanarayana, Guimara and Wypych, 2007; Tomczak, Sydenstricker and Satyanarayana, 2007).

2.2.2.1.3 Strength as a function of the strain rate

Different authors have reported the influence on the tensile properties of coir fibre as a result of the strain rate (Tomczak *et al.*, 2007; Fidelis *et al.*, 2013; Mathura and Cree, 2016) and observed that the speed of testing had the predominant effect on the ultimate tensile strength of fibre. But it has been previously noted that the diameter as well as the gauge length are also contributory factors to the tensile strength variation. A strain rate dependency of the fibre reinforcement with respect to the fibre diameter was observed by Welsh and Harding, (1985) and (Kim, Oshima and Kawada, 2013).

2.2.2.1.4 Strength as a function of the density of defects present in the fibre

Defects present in natural fibres have been identified as a major detrimental influence on the tensile strength of natural fibres. The extent of defect in natural fibres varies according to the diameter of the fibre. Fibres with higher cross-sections display higher density of defects or irregularities both internal and external than fibres with lower cross-sections. Larger diameter fibres therefore possess weaker points than thinner-diameter fibre. Fibres with a lower cross-sectional area have been found to be of more uniformity with lower porosity and hence fewer defects and therefore higher strength (Monteiro *et al.*, 2012). The slope of the plot of tensile strength against test length gives an indication of the defect density (Defoirdt *et al.*, 2010). Defoirdt *et al.*, (2010) investigated the defect density of white coir, brown coir, bamboo and jute and found out that their defect densities are in the order; bamboo>brown coir>jute>white coir. This was attributed to the extraction method adopted, which suggested that the steam explosion extraction of the bamboo fibres introduced significant defects, whereas the mechanical extraction of white coir caused least defects.

2.2.2.1.5 Strength as a function of the porosity and microfibril angle of the fibre

The poor tensile strength of natural fibres has been reported by several authors to originate from its microstructure such as the porosity and pore size distribution (Burgert, 2006; Ozturk, 2007) as well as the microfibril angle, cellulose and lignin content. For better clarity, these will be discussed in Section 2.2.3.2.

2.2.2.1.6 Strength as a function of age, specie, origin and method of extraction of fibre

Investigations into the properties of natural fibres of the same type but different origin, specie and maturity have been carried out. Observations revealed that the properties of natural fibres are largely as a result of the soil type in which they are grown, plant species, age, origin and

extraction method (van Dam *et al.*, 2006; Tomczak, Sydenstricker and Satyanarayana, 2007; Mathura and Cree, 2016; Dhaliwal, 2019). Coir fibres of different varieties and different ages but of Philippine origin were investigated by van Dam *et al.*, (2006). These fibres were extracted manually from the husk. The varieties were compared with retted coir fibres from India. It was observed that the tensile strength of the retted India coir fibre was 248 MPA while the strongest fibre of all the species of the manually extracted Philippine coir fibre was 114 MPa. The different species also showed significant variations in strength.

2.2.2.2 Moisture ingress in natural fibres

Several reports have confirmed the susceptibility of natural fibres to moisture ingress. The poor resistance to moisture ingress of natural fibres has been reported to reduce the potentials of natural fibres to be used as reinforcements in composites. Moisture ingress reduces the crystallinity of the fibre, weakens the interfacial bond strength, gives rise to swelling and promotes microcracks in the composites (Sreekala *et al.*, 2002; Khalil *et al.*, 2012; Muñoz and García-Manrique, 2015; Londhe, Mache and Kulkarni, 2016).

Moisture ingress in cellulosic fibres has been reported (Khalil *et al.*, 2012; Masoodi and Pillai, 2012). The moisture ingress is accelerated via capillary action through the pores present in natural fibres (Ashori and Sheshmani, 2010). Masoodi and Pillai, (2012) and Yahaya *et al.*, (2016) determined the changes in the thickness and overall volume caused by moisture ingress using the following equation;

$$T_{re}(t) = 100 \times \left(\frac{T_i - T_o}{T_o} \right) \quad [2.6]$$

where the change in the relative thickness or thickness swelling percentage, thickness at time t , and the initial thickness are represented by T_{re} , T_i and T_o respectively.

$$V_{re}(t) = 100 \times \left(\frac{V_i - V_o}{V_o} \right) \quad [2.7]$$

where the relative change in volume or volume swelling, volume at time t and the initial volume are represented by V_{re} , V_i and V_o respectively.

Water ingress was calculated as a percentage using the difference in weight as follows

$$W_e(t) = 100 \times \left(\frac{W_i - W_o}{W_o} \right) \quad [2.8]$$

The relative change in weight (water absorption in percentage), the weight at time t , the initial weight at $t = 0$, and the soaking time are represented by W_e , W_i , W_o , and t respectively.

Masoodi and Pillai, (2012) and Khalil *et al.*, 2012; observed that changes in the thickness recorded as a result of moisture ingress is affected by time and type of epoxy specimens matrix as well as the type of fibre.

Ashori and Sheshmani, (2010), Masoodi and Pillai, (2012) and Ashik, Sharma and Guptha, (2018) noted that the degree of changes in the thickness varies from fibre to fibre and the ratio of fibre to matrix in the composite ascertaining that as the fibres get swollen, stress is exerted on the matrix which can lead to the composite undergoing micro-cracking and subsequent failure.

However, moisture absorption of natural fibres can be minimised by several processes such as: coating (Munirah Abdullah and Ahmad, 2012; Londhe, Mache and Kulkarni, 2016), treatment with chemicals such as NaOH, acetylation, toluene diisocyanate, silane on alkali (Sreekala *et al.*, 2002; Haameem *et al.*, 2016; Yan *et al.*, 2016), use of coupling agent such as Maleated anhydride grafted polypropylene (MAPP), maleic anhydride polyethylene (MAPE), silane (Ashori and Sheshmani, 2010; Sawpan, Pickering and Fernyhough, 2011; Robertson *et al.*, 2013). Abdullah and Ahmad, (2012) reported a decrease in water absorption in coir fibres after surface treatments using NaOH, silane and alkalized silane.

Coupling agents improve the adhesion between fibre and matrix, thus aiding in reacting with the hydroxyl group and decrease in the rate of the diffusion process and hence reduction in moisture ingress (Ashori and Sheshmani, 2010; Munirah Abdullah and Ahmad, 2012; Robertson *et al.*, 2013). Coatings such as latex coatings acts in a similar way as coupling agents by partially shielding the pores on the surface of the fibre and thus reducing the penetration of moisture into the fibre. Hence a decrease in moisture ingress has been recorded for latex-coated fibres. Decrease in moisture ingress has been observed after treatment with gamma ray irradiation which has been noted decreases the capillary action resulting in reduced sorption. Chemical treatments such as NaOH and acetylation have been reported to decrease moisture ingress thus reducing the absorption of moisture through the capillary (Sreekala and Thomas, 2003; Sawpan, Pickering and Fernyhough, 2011; Kabir, Wang, Lau and Cardona, 2012).

Moisture ingress increases as the fibre content increases because the cellulose content becomes higher and subsequently there are more fibre-to-fibre interactions. Fibre type,

geometry and size distribution influence moisture intake: smaller diameters and more uniformity across the size of the fibre lead to reduction in moisture uptake by narrowing transport pathways between contacting fibres (Robertson *et al.*, 2013; Muñoz and García-Manrique, 2015).

Considering the structure of coir, with numerous pores and high level of porosity in its untreated form, it may therefore be expected that coir fibres will be more prone to moisture absorption than sisal and jute that are more compacted with less porosity but this is not the case. The cellulose content of coir as shown in Table 2.2 is much less than that of several natural fibres such as sisal and jute, since there are fewer free OH groups. It is expected that coir fibres within a specific diameters or geometry will be much less prone to moisture ingress than most natural fibres upon treatments with chemicals and coupling agents.

Even the advanced high-profile synthetic fibres such as aromatic polyamide or aramid fibres are also susceptible to moisture. An increase in volume fraction of aramid fibre results in an increase in water absorption (Gopalan, Rao, and Murthy, 1986; Fink 2014). Nevertheless, aramid fibre is still a material of choice for high performance applications. However what needs to be ascertained as pointed out by Giridhar, Kishore and Rao, (1986) is to what degree does the moisture influence the mechanical properties of composites manufactured with aramid fibre? This question can as well be applied to coir fibre composites to achieve optimum properties.

There exist several conflicting reports on the effects of moisture ingress on the mechanical properties of natural fibres. These reports are based on the fact that mechanical performance of natural fibres are grossly dependent on the fibre type and are therefore a function of their chemical structure, microfibrillar angle and cellulose content. Upon absorption of water, the tensile strength decreases. An approximate 30% decrease was observed in salt water sorbed fibres, desorbed fibres display similar effects. An increase in elongation to failure of fibre consequent upon sorption has been reported. This is so because the amorphous phase becomes favoured in the swollen stage, the lignin-cellulose network is disrupted by the presence of water molecules (Sreekala *et al.*, 2002; Munirah Abdullah and Ahmad, 2012; Haameem *et al.*, 2016; Venkatachalam *et al.*, 2016). On the contrary, Muñoz and García-Manrique, (2015) and Robertson *et al.*, (2013) reported an increase in tensile strength of water-absorbed flax fibre and hemp unsaturated polyester composites samples respectively with higher increase as the fibre content increases when compared to dry samples. This was attributed to the swelling of the fibre owing to moisture absorption and subsequent closure of

gaps possibly created during processing. Similar reports were published for jute fibre reinforced unsaturated polyester composites (Dhakal, Zhang and Richardson, 2007). The increase in strength was connected to the filling up of the gaps achieved during manufacturing processes as a result of the swelling of the fibre. The swollen fibre fills the gaps exerting pressure on the matrix, and hence there is an improved adhesion with more efficient stress transfers. An increase in elongation was also reported by the authors. Robertson *et al.*, (2013) reported an increase in strength, modulus and elongation after treatment of wood pulp composites with maleated polyethylene (MAPE) coupling agent. MAPE increased the interfacial bond strength leading to increase in the load transfer efficiency and hence increase in the mechanical properties. However, Haameem *et al.*, (2016) reported a sharp decrease in the tensile strength of both alkali treated and untreated Napier grass fibre composites. This was attributed to poor stress transfer owing to poor interfaces between the fibre and the matrix. Poor interfaces have been associated with the wettability of the reinforcing fibre.

2.2.2.3 Wettability of Natural Fibres

In the manufacture of natural fibre composites, a fibre-resin interface is created. The mechanical properties of the final product are subject to how well the resin wets the fibre. It is therefore ideal to determine the wettability of fibre in readiness for manufacturing composites. Poor wetting has been reported as one of the drawbacks in the use of natural fibres as reinforcements (Väisänen, Das and Tomppo, 2017). Investigations into wetting coupled with surface chemistry of natural fibres enhances the characterisation of the fibre surface, this contributes to the selection of the best-suited treatments for the fibre and modification of the surface energy of the fibre matrix giving rise to improved fibre-resin interface (Fuentes *et al.*, 2011). Wetting is relevant to the fibre-matrix adhesion. The level of adhesion controls the efficiency of stress transfer from the resin to the fibre at the interface region (Tran *et al.*, 2013). The wettability of the fibre surface can be measured by the contact angle. The contact angle is the angle formed at the intersection of the liquid-solid interface or liquid-vapour interface (Chini and Amirfazli, 2011; Yuan, Yuehua; Lee, 2013; Akindoyo *et al.*, 2016). The lower the contact angle the higher the wettability and vice versa as in the case of coir (Table 2.3) and the higher the adhesion ability of the fibre onto the matrix. Better wettability is achieved as the contact angle approaches zero (Park *et al.*, 2006; Yuan, Yuehua and Lee, 2013; Orue *et al.*, 2015; Akindoyo *et al.*, 2016; Bakri, Chandrabakty and Soe,

2016). This is shown in Figure 2.12 where a high contact angle is observed when the liquid forms a bead-like substance on the surface. Contact angle is influenced by fibre treatment.

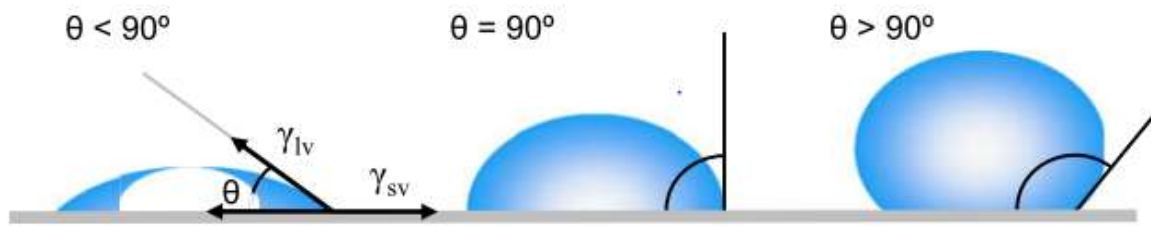


Figure 2.12 Demonstrations of contact angles formed by sessile liquid on a uniform surface (Yuan, Yuehua and Lee, 2013).

Reports have indicated that a high concentration of lignin or the presence of hemicelluloses and extractives on the fibre surface contribute to a large extent their wettability (Young, 1976; Barsberg and Thygesen, 2001; Fuentes *et al.*, 2011). Another interfacial characteristic of lignin is that it lowers the adhesion of water to the surface. Lignin forms granules in water, these granules are so strong that they do not spread out to form a film on the substrates but rather bind to the substrates through electrostatic interactions. Therefore the wetting properties are subject to the morphology of adsorbed layer and the location of bare lignin in the adsorbed layer (Laschimke, 1989; Maximova *et al.*, 2004).

Techniques best suited for the measurement of wetting of fibres include the Wilhelmy's technique and fluid geometry (Barsberg and Thygesen, 2001; Khalil *et al.*, 2001; Fuentes *et al.*, 2011; Tran *et al.*, 2013). This permits the determination of the dynamic contact angle of the different test liquids on the fibre. The fibre surface energy can be obtained using the static equilibrium contact angle. The contact angles formed via the expanding and contracting the liquid are called the advancing contact angle θ_{adv} and the receding contact angle θ_{rec} respectively. The difference between these two angles is named hysteresis. This hysteresis has been proved to be as a result of the surface roughness (Yuan, Yuehua and Lee, 2013). It has been reported that the wetting properties can be affected by the surface irregularities such as fibre geometry, sorption and diffusion and by treatment of fibre (Young, 1976; Barsberg and Thygesen, 2001; Fuentes *et al.*, 2011).

Table 2.3 Selected surface properties of natural fibres.

Fibre	SR (nm)	Crystallinity (%)	CA (H ₂ O) (°)	Surface Energy (mN/m)	IFSS (MPa)	Surface Composition				
						C (%)	O (%)	N (%)	Si (%)	O/C
Coir i, ii, iii, vi	53.84	30.0	71.4- 82.9	38.4	7.95 (in epoxy matrix)	74.9	21.8	1.7	0.9	0.29
Sisal v	-	-	70.6	34.7	2.4 (PLA)	NA				
Hemp iv	-	-	67.05	35.2	5.9					
Jute iv	-	-	78.46	29.5	17.9					

SR=Surface Roughness; CA=Contact angle

Compiled using the following sources: (Sawpan, Pickering and Fernyhough, 2011ⁱ; Yu, Xie and Du, 2016ⁱⁱ; Bakri et al., 2019ⁱⁱⁱ; Park et al., 2006^{iv}; Orue et al., 2015^v; Tran et al., 2011^{vi}).

The adhesion at the fibre-matrix interface can be explained through an interaction of physical adhesion through a good wettability between the fibre and the matrix, chemical bonding with mechanical interlocking also essential for good interfacial adhesion. However these interactions are dependent on the functional groups on the surface of the fibre and matrix at the interfacial contact area (Tran *et al.*, 2011).

The level of adhesion of fibre and matrix is determined by their interfacial shear strength (IFSS). The IFSS is measured by using the fibre pull-out test (Akindoyo *et al.*, 2016; Fiore *et al.*, 2016; Bakri *et al.*, 2019).

The IFSS is determined using Equation [2.1] as previously stated.

The IFSS can be improved by subjecting the fibre to chemical treatment such as NaOH treatment. The interfacial shear strength for sisal, jute, hemp, have been noted to increase upon treatments with NaOH, sodium bicarbonate (NaHCO₃) and maleic anhydride polypropylene (MAPP) (Park *et al.*, 2006; Orue *et al.*, 2015; Bakri, Chandrabakty and Soe, 2016). Though several studies have been carried out on the use of natural fibres as reinforcement, not as much work has been carried out on coir as reinforcement in composites.

2.2.3 Coir fibre

This section gives a general overview of coir fibre, countries where coir fibres are produced, average annual production, export rate, economic growth, the compound annual growth rate and market values of coir. The section further provides a wider coverage of the properties, chemical composition and microstructure of coir.

Several studies have been carried out on the tensile properties, physical properties and chemical treatments of coir fibres (Tomczak, Sydenstricker and Satyanarayana, 2007; Satyanarayana, Arizaga and Wypych, 2009; Sumi, Unnikrishnan and Mathew, 2016; Yan *et al.*, 2016; Chandrasekar *et al.*, 2017; Kiruthika and Veluraja, 2017).

Coir fibres can be obtained from mature (Tran *et al.*, 2015; Nguyen and Indraratna, 2017) coconut (11 months and above with brown husks) or premature (Brígida *et al.*, 2010b; de Farias *et al.*, 2017) coconut (less than 11 months with green husks) as brown and white fibres respectively. The more commonly exploited is the brown coir fibre.

With reference to Figure 2.13: (a) represents; the raw material for the production of coir fibres as used in this study, obtained from the coconut husks of matured and dry coconut. The fibre must be separated from the coconut husk through mechanical process known as decortications (Ezekiel *et al.*, 2011; Bakri *et al.*, 2018) or by low technology method known as retting (Prasad, Pavithran and Rohatgi, 1983; Gu, 2009; Mathura and Cree, 2016). (b) represents the retting process whereby the husk is soaked in water for a period of 4 to 6 months; (c) represents the sun-drying of the retted fibres. The decorticated or retted fibres contain both coir fibre and the soft moss-like coir pith largely interwoven. The ratio of coir fibre to coir pith is about 30% to 70% by weight (Dam *et al.*, 2004). The pith is separated from the fibre through the revolving screener as represented in (e). The screened fibres are combed, sorted and baled for export as represented in (f), (g) and (h) respectively. Out of the sorted coir fibres is the 3-tie coir fibre bundle shown in Figure 2.14. The bundle was obtained by selecting sorted coir fibres of length within the range of 100 to 350 mm. The average weight of each bundle was 0.25kg.



Figure 2.13 Basic processes of production of coir fibre [courtesy: G Fernando, University of Birmingham].



Figure 2.14 Hayleys Sri-Lanka 3-Tie Coir fibre bundle.

Coconut from which coir fibres are obtained are grown in tropical countries such as India, Sri Lanka, Thailand, Indonesia, Brazil, Bangladesh, Vietnam, Jamaica and Nigeria (Rout *et al.*, 2001; Dam *et al.*, 2004; Tomczak, Sydenstricker and Satyanarayana, 2007; Jústiz-Smith, Virgo and Buchanan, 2008; Njoku *et al.*, 2012; Yan *et al.*, 2016) with a predominance in India, Sri Lanka, Vietnam and Thailand as shown in Figure 2.15. The global production and trade volumes of coir fibre are measured by the United Nations Food and Agriculture Organisation UN (FAO) and other accredited organizations. The global average annual production of coir stood at 998.3×10^3 tonnes between 2012 and 2017 as shown in Figure 2.16. An increase of 0.17% was observed in 2019 (see Figure 2.2(b) leading to 1,000,000 tonnes being produced. India maintained the lead with 60.87% of the total production followed by Sri Lanka with 12.51%, then Vietnam with 12.01%.

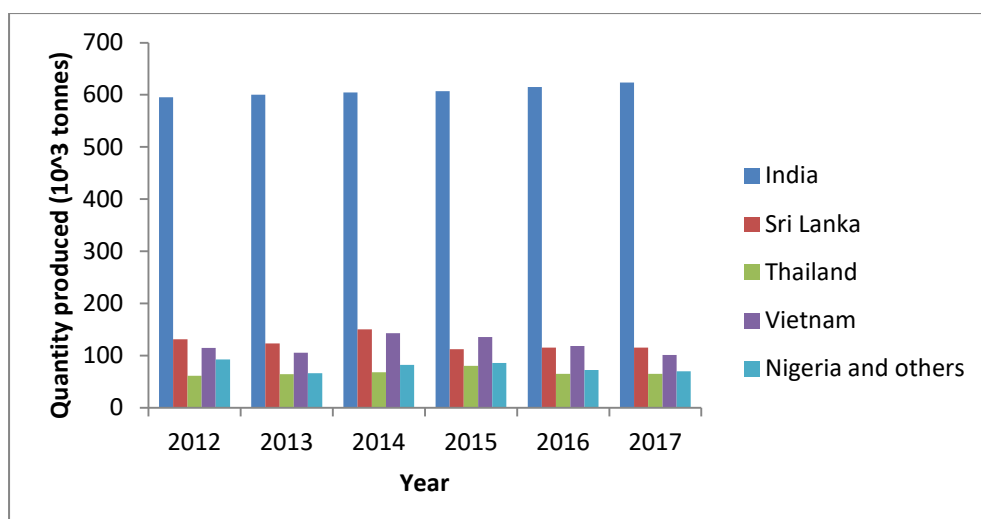


Figure 2.15 Leading producers and global production of coir fibre adapted from: (Food and Agriculture Organization Corporate Statistical Database, 2018).

Coir had maintained its position as the cheapest of the listed natural fibres as shown in Figure 2.16). The price of a tonne of coir fibre had been on the decline from \$625/tonne in 2012 to \$340/tonne in 2017. In 2018, a sudden leap in price occurred where bristle coir fibre sold at \$1772/tonne. This sudden leap has been attributed to increasing research on the treatments of coir fibre leading to enhanced properties and thus increases in applications of the fibre (Dhaliwal, 2019).

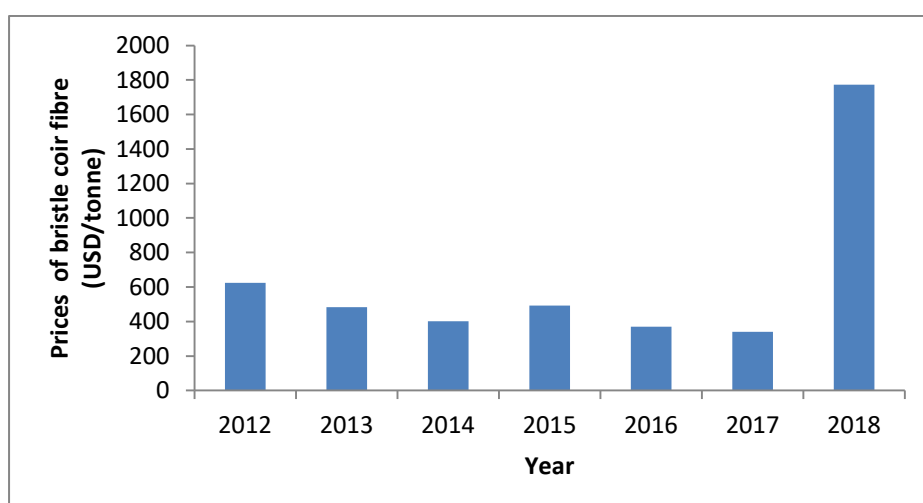


Figure 2.16 Prices of coir fibre for seven consecutive years adapted from; (Food and Agriculture Organization Corporate Statistical Database, 2018).

Export of coir fibre from Sri Lanka, China, Thailand, Malaysia, Indonesia, Vietnam and Africa has been on a steady increase as shown in Figure 2.17 with a Compound Annual

Growth Rate (CAGR) of 9.76% from 2012 to 2017, an average of about 962.02×10^3 tonnes of coir fibres was annually exported to countries such as China, Russia, Europe, North America. The global average annual production of coir stood at 998.3×10^3 tonnes within this period. About 96% of the overall produced coir fibres were exported for use in various industries.

Results from Figure 2.17 of the production and export of coir fibre show that there is a productivity growth and export growth. There exists a relationship between productivity and export. According to Bernard and Jensen, (2001), an improvement in productivity can be observed as a result of expertise achieved through interactions with the export market. The expertise could be via better management practices and enhanced research and development.

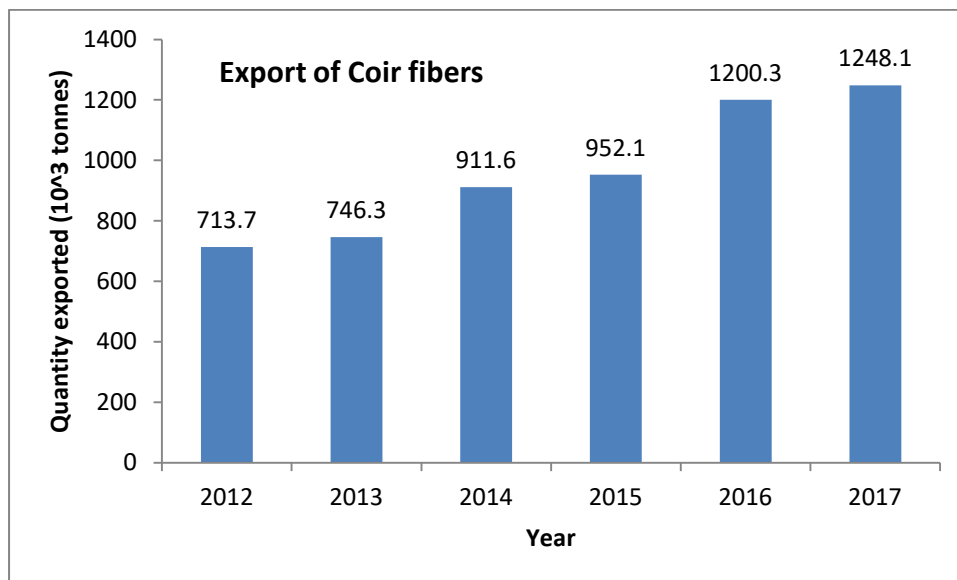


Figure 2. 17 Annual export of coir fibres adapted from: (Food and Agriculture Organization Corporate Statistical Database, 2018).

From the reports above, there is an apparent linear growth in the production rate and a corresponding growth in export rate of coir fibres. Possibly their eco-friendly values and significant weight reduction values have prompted several manufacturers to come up with innovations of various coir composites parts. But still, even much more applications of coir out to be sought considering the growth rate of the production of the fibre.

2.2.3.1 The unique features of coir fibre

Coir is a typical but versatile lignocellulosic fibre possessing the lowest density of all the natural fibres considered for technical applications as shown in Table 2.1. The lignin content

and failure strain of coir fibre is significantly higher than that of any other commercial fibre as displayed in Tables 1 and 2. Lignin in the sclerenchyma cells contributes to the stiffness of the cell wall of the fibres protecting the carbohydrate from chemical damage (Carl and Brook, 1985; Khalil, Alwani and Omar, 2006; Tian *et al.*, 2013). Lignin has been reported to resist biodegradation and exhibit increased toughness. Outdoor applications of coir are as a result of its high lignin content (Haque *et al.*, 2009). Lignin extracted from coir has several applications. Coir has been reported to be the only natural fibre that is resistant to a salt water environment (Arancon, 2008; Yan *et al.*, 2016; Pillai *et al.*, 2018). These basic properties of coir have resulted in its long-standing applications with limited competition in a choice range of designs of durable ropes, rugs, doormats, mattresses and upholsteries while the technical applications of coir are seen in erosion control blankets owing to its slow degradation properties. Other applications of coir are in automotive, building and construction (Arancon, 2008; Nguyen and Indraratna, 2017; Iman *et al.*, 2018; Dhaliwal, 2019).

Coir possesses other advantages as with natural fibres as mentioned in Section 2.2.1. However the technical performance of coir as reinforcement in polymer composites has been considered unsatisfactory to a certain degree (Rout, *et al.*, 2001). This unsatisfactory performance of coir is not unconnected with several factors as earlier mentioned but significantly as a result of its microstructure as explained further in the next section.

2.2.3.2 Microstructure and morphology of coir fibre

Plant fibre has a rather complex structure as with the basic features shown in Figure 2.18. The cell wall comprises the middle lamella, primary and secondary cell walls. The primary issue in the use of coir fibres in polymer composites is their relatively low mechanical properties as shown in Table 2.1. This has been traced to their microstructure. Scanning electron microscopy of the surface of coir as shown in Figures 2.19 reveals the presence of globular protrusions known as tyloses. A cross-section of coir fibre reveals the presence of pores, a large number of elementary fibres, microfibrils within the elementary fibres and a cylindrical channel called lacuna located at the centre (Chand and Satyanarayana 1986; Bismarck *et al.*, 2001; Geethamma *et al.*, 2005; Gu, 2009; Carvalho *et al.*, 2010; Nam *et al.*, 2011a; Tran *et al.*, 2015; Yun *et al.*, 2016). Coir is a typical multicellular fibre made up of xylem, parenchyma, phloem and xylem-parenchyma. The arrangement of these cells, their number and shape, distinguish coir from other natural fibres by giving it its unique properties. The elementary fibres within the coir fibre bundle have a large number of microfibrils, these

elementary fibres are bonded together with the help of the middle lamella (Martinschitz *et al.*, 2008; Tran *et al.*, 2015). Figure 2.20 (a) and (b) display images of elementary fibres with varying sizes of lumen and microfibrils in both the primary and secondary cell walls.

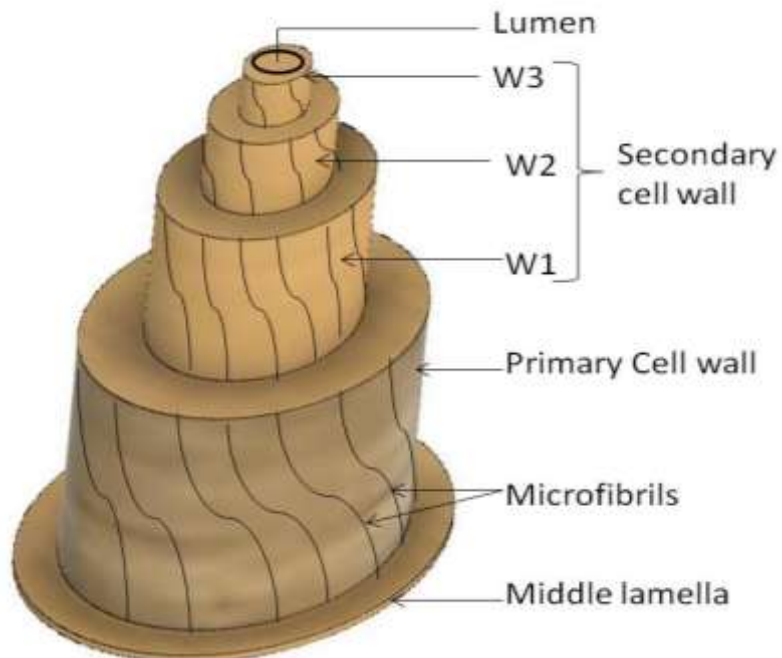


Figure 2.18 Structure of natural fibre adapted from Cordeiro, (2016) and Latif *et al.*, (2019).

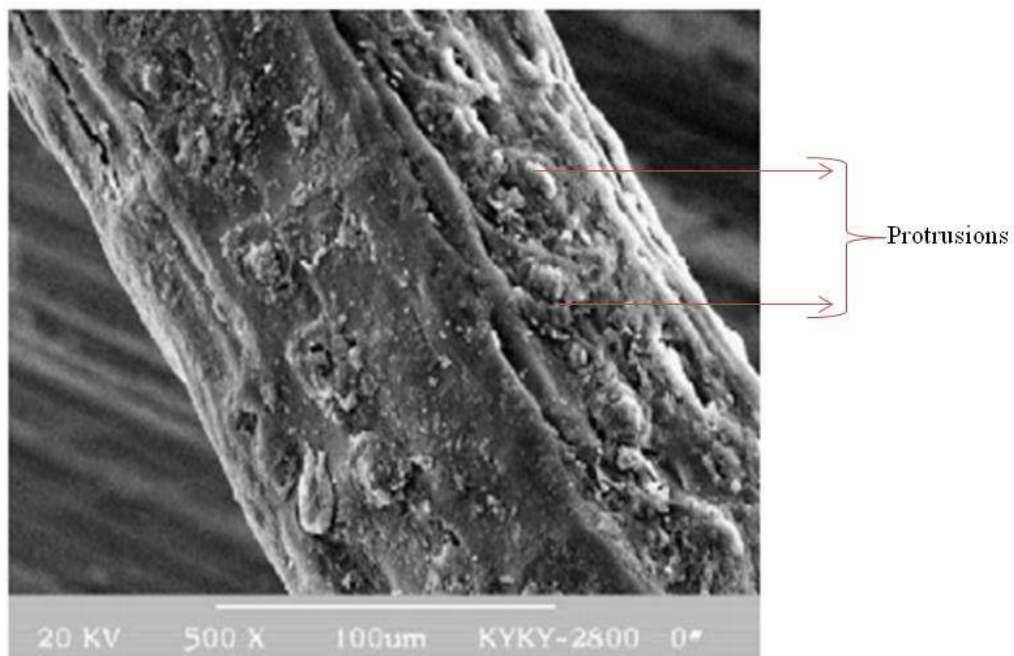


Figure 2.19 Longitudinal image of coir fibre showing globular protrusions referred to as tyloses (Karthikeyan, Balamurugan and Kalpana, 2014).

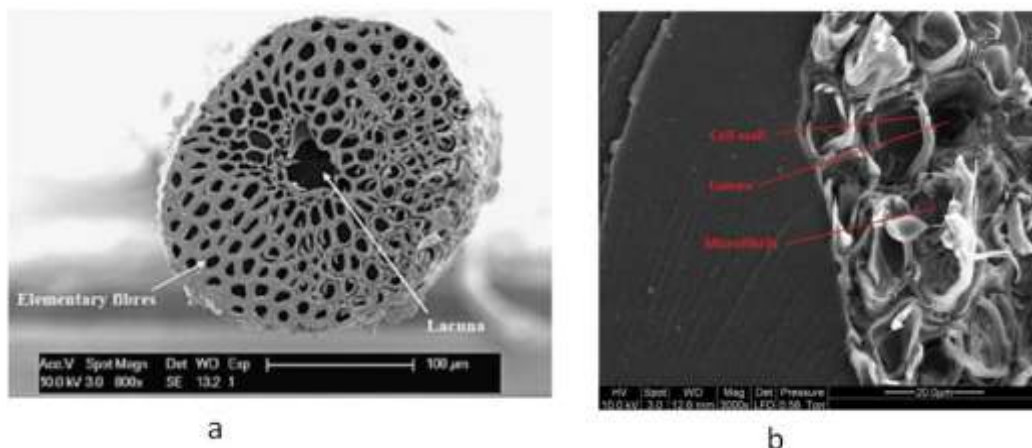


Figure 2.20 Micrographs of coir fibre cross-section (a) showing lacuna and elementary fibres and (b) showing microfibrils (Tran *et al.*, 2015; Yan *et al.*, 2016).

Tyloses could be described as balloon-like protrusions of the xylem of parenchyma cells that bulge out of the xylem vessels. According to Pallardy, (2008) the protrusion is as a result of pressure within the plant cell wall. Tyloses possess cellulosic cell wall and can exist in large numbers and varying sizes (Agrios, 2019), the number and distribution of tyloses can aid in determining their importance. They can completely block the xylem vessel and block water transport. They have been noted to ward off pathogens. Hence plants with many tyloses are less susceptible to pathogen attack (Pallardy, 2008; Agrios, 2019). Hence coir fibre exhibits high resistance to attack by diseases and displays slower degradation in comparison with other natural fibres. The reduced moisture content when compared with jute, sisal and hemp fibres as shown in Table 2.2 may be linked to its porosity. Significant variation in the development of tyloses may be experienced based on plant origin and lignifications of these tyloses will amount to an increase in lignin content of coir fibre. This will subsequently result in a reduction in tensile strength as the tensile strength of single fibres has been reported to decrease with an increase in the amount of lignin (Zhang *et al.*, 2013).

Microfibrills are located on all the cell wall layers but absent in the middle lamella. The anisotropic deformation and expansion of plant cells are determined by the cellulose microfibrils. However, they differ in the degree of organisation, thickness and in the quantity of chemical constituents and, as a result, the microfibril orientation and dimensions exert considerable influence on the stiffness of the wall (Persson, 2000b; Ptashnyk and Seguin, 2016) The orientation of these microfibrils within the cell wall layers affects the dimensional stability of the fibre. Microfibrils have been reported to initially lie parallel to the sides of

primary cell wall but perpendicular to the growth or extension direction. The reorientation of these microfibrils towards the growth extension in the course of elongation of the cell wall has been reported (Joffe, Andersons and Wallström, 2003; Sultana, 2014; Ptashnyk and Seguin, 2016; Sun *et al.*, 2016) and microfibrillar angle (MFA) has been defined as the angle between the microfibrils located in the secondary cell wall of the fibre and the fibre axis stating that MFA is subject to the type of fibre, location of fibre and origin of fibre. The mechanical properties of natural fibre such as modulus, strength and failure strain have been associated with the microfibril angle. MFA is responsible for the non-linear portion of the stress-strain graph of natural fibres; it was further observed that for higher MFA, the elastic portion is smaller and vice versa. Low stiffness of fibres are as a result of high MFA. It has been reported that the microfibril angle in the secondary wall layer exerts an influence on the Young's modulus and the extent of the influence exerted is a function of the radius of the fibre (Satyanarayana *et al.*, 1982; Persson, 2000b; Martinschitz *et al.*, 2008; Sultana, 2014; Tran *et al.*, 2015). Mechanical properties have earlier been stated to be a function of the density and microfibril angle of the secondary cell wall layer (Persson, 2000). Yu, Xie and Du, (2016) observed the length/diameter ratio of coir microfibrils to be in the range of 25-150. From the observed result, the packing model of the coir helix crystal ribbon and multilevel fibrils was derived. Coir fibre is made up of many tube-like fibres consisting of right-handed helix crystal ribbons (Yu, Xie and Du, 2016).

The Elementary fibres: Coir fibre consists of elementary fibres that are held in place together by a lignin-cellulose matrix. Each elementary fibre is basically an assembly of the cell wall (primary and secondary), lumen and the microfibrils. The bulk fibre is composed of cellulose, hemicellulose, lignin, waxes as shown in Table 2.2. About 70% of this cellulose is found in the elementary fibre as amorphous or crystalline cellulose (Martinschitz, 2008; Yan *et al.*, 2016).

The Lumen: The pores present in coir fibre have been described as lumens. The shape of different cells represent the size of the lumen in the tissue (Chen, 2015; Shugang *et al.*, 2016). The lumen functions to maintain the pressure around the interior of the cell wall, offering shape and support to the cell by acting as the fluid reservoir and capillary system of the cell (Fu and Yuan, 2017; Brischke, 2019). The free-water which occurs as the moisture content approaches 25-30% is contained in the cell lumens. This free water which attracts biological attack can be harnessed to create conditions that will suppress or limit moisture. The permeability of these pores to water or fluids is reduced via fibre treatment which leads

to improved mechanical properties. Filling of lumen with substances other than resin may result in an increase in strength and decrease in moisture uptake. Certain methods to fill the lumen and modifying treatments are geared towards improved strength (Fu and Yuan, 2017; Lin, Fu and Qin, 2017; Morrelli, 2018).

Pore and the porosity of coir fibre: The microstructure of coir consists of numerous pores. Porosity comprises the, lumen, central cavity and pores situated between the fibrils and lamellae. The pores have been described as secondary lumens and the central cavity that runs along the length of the fibre, as lacuna. Porosity has been reported to have a substantial impact on strength. Treatment of fibre leads to decrease in the fibre porosity (see Section 2.3). The correlation between the diameter, tensile strength, Young's modulus, test length have been investigated (Prasad, Pavithran and Rohatgi, 1983; Ozturk, 2007; Tran *et al.*, 2015). However the porosity of fibre has not been correlated with the fibre diameter and subsequently the strength of the fibre. On this note, Luz *et al.*, (2017) investigated coir fibres of different diameter range and proposed a relationship between porosity and the diameter of fibre. According to Luz *et al.*, (2017), coir fibres consists of open (P_o) and closed (P_c) porosity. The total porosity P_T , is the sum of the open and closed porosities. These porosities were calculated using Equations [2.9-2.11]:

$$P_T = \left(1 - \frac{\rho_g}{\rho_{ab}}\right) \quad [2.9]$$

$$P_o = \left(1 - \frac{\rho_g}{\rho_{ap}}\right) \quad [2.10]$$

$$P_c = P_T - P_o \quad [2.11]$$

where ρ_g , ρ_{ab} and ρ_{ap} are the geometric, absolute and apparent densities respectively.

Hence the diameters of coir fibres could be increased by increasing both total and closed porosities. The larger the diameter or the thicker the fibre is, the more the total pores and the higher the porosity and the lower the diameter, the fewer the pores was said to lead to lower porosity.

$$\text{Hence } d = CP \quad [2.12]$$

where the diameter and the porosity of the fibre are represented by d and P respectively and C is a constant (see Section 2.2.2.1).

2.3 Treatment of coir fibres

A large number of studies have been carried out to improve the performance of natural fibres in terms of their mechanical properties for their use as the reinforcement in composites. This includes the treatment of natural fibres. The properties of coir fibres have been deemed as unsatisfactory for use as reinforcement in composites as shown in Table 2.1 where the mechanical properties of coir such as strength and stiffness are poorer than those of other natural fibres. These relatively poor mechanical properties have been associated with the concentration of cellulose, lignin and the microfibril angle present in the fibre as displayed in Table 2.2. Surface modifications are therefore carried out on natural fibres to improve their performance in composites via improved fibre-matrix adhesion. Surface modification of natural fibres via surface treatments include alkali treatment (Rahman and Khan, 2007; Nam *et al.*, 2011b; Yan *et al.*, 2016), acetylation (Khalil *et al.*, 2001), coupling agents (Asumani, Reid and Paskaramoorthy, 2012; Orue *et al.*, 2015; Asim *et al.*, 2016), oxidation (Saw, Sarkhel and Choudhury, 2011), graft copolymerization (Rout, *et al.*, 2001; Khan *et al.*, 2003).

2.3.1 NaOH treatment

Alkali treatment is an economically viable treatment method geared towards the modification of the surface of natural fibre to improve the wettability and the fibre-matrix interfacial bond strength. It is an age-old process utilized in the cotton industry to improve the lustre and the tensile strength of cotton (Prasad, Pavithran and Rohatgi, 1983). Several studies have been carried out on the effects of sodium hydroxide as a surface treatment for coir fibres. The treatments usually consist of soaking coir fibre in different concentrations of NaOH for specific durations and at certain temperatures. Alkali treatment enhances the fibre-resin adhesion due to the removal of components such as wax and reduction or removal of chemical components such as lignin and hemicellulose from the fibre. This brings about changes in the arrangement of units in the cellulose macromolecule (Alexander Bismarck *et al.*, 2001; Wang and Huang, 2009a). The removal of these substances leads to disruption of hydrogen bonding thereby increasing surface roughness. This treatment is said to lead to improved adhesion with the matrix (Rout *et al.*, 2001; Yan *et al.*, 2016). Scanning electron microscopy of coir reveals globular protrusions on the fibre surface (see Figure 2.19). NaOH removes these protrusions and thereby creates voids or rougher surfaces. These rougher surfaces increase mechanical interlocking between the fibre and the resin and hence improves the interfacial bond strength (Prasad, Pavithran and Rohatgi, 1983; Rout, Misra and Tufupathy, 2001; Nam *et al.*, 2011b; Saw, Sarkhel and Choudhury, 2011; Rait, 2014; Yu, Xie and Du,

2016). Positive effects of NaOH treatment on the tensile strength of coir fibres have been reported. Such effects include; closure of lacuna, reduction in fibre diameter, reduction in the porosity of fibre, thickening of cell wall and changes in the structure of tyloses (Prasad, Pavithran and Rohatgi, 1983; Hai, Kim and Lee, 2012; Karthikeyan and Balamurugan, 2012; Tran *et al.*, 2015). Negative effects such as rupture of cells and damage to the fibrils via the removal of lignin and hemicellulose which bind the cells in the fibre have been reported (Prasad, Pavithran and Rohatgi, 1983; Saw, Sarkhel and Choudhury, 2011).

2.3.1.1 Reports on the NaOH treatment of coir fibres

Coir fibres were soaked in 2-10% NaOH concentrations between 26-28 °C for 28 days. Post-processing was carried out by soaking the fibres in distilled water for 24 hours and drying was carried out in an air-circulating oven. An improvement in the tensile strength was observed at 6% concentration (Gu, 2009). Njoku *et al.*, (2012) obtained a similar result under the same conditions but at room temperature with a treatment time of 48 hours and NaOH concentration of 5%. Rout, Misra and Tfupathy, (2001) also obtained the same result on dewaxed coir fibre but at a NaOH concentration of 2%.

Prasad, Pavithran and Rohatgi, (1983) observed an increase in tensile strength at 5% NaOH concentration and soaking time of 72 hours but the alkali solution was changed every 24 hours. At 5% NaOH concentrations and at varying conditions of temperature and duration; improved tensile strength was observed by Bismarck *et al.*, (2001); Narendar and Dasan, (2014); and Yan *et al.*, (2016). Andic-Cakir *et al.*, (2014) had a similar treatment to that of Bismarck *et al.*, (2001); Narendar and Dasan, (2014); and Yan *et al.*, (2016) but carried out a post-processing step with distilled water and acetic acid and recorded improvement in flexural strength and toughness. Room temperature treatments were also carried out by Dixit and Verma, (2012) and Manjula *et al.*, (2018); both ended up with reduced moisture absorption and increased tensile strength owing to the significant differences in the soaking time. But Rahman and Khan, (2007) observed a decrease in the tensile properties with NaOH concentrations between 5-50%, treatment temperature of 100 °C and a duration of 30 minutes. Nam *et al.*, (2011) reported increased ultimate tensile strength at 5wt% NaOH at room temperature as a result of closure of lacuna and decrease in fibre diameter but decrease in the ultimate tensile strength was reported owing to cell rupture and fibril exposure after a 72-hour treatment. Bledzki, Reihmane and Gassan, (1996) reported an increase in crystallinity index of NaOH-treated coir. Defoirdt *et al.*, (2010) reported poor adhesion leading to micro cracking of composites as a result of swollen fibres after NaOH treatment.

Arsyad, (2017) investigated the degree of roughness, bonding ability and tensile strength of coir fibre at alkali treatment concentrations of 5%, 10%, 15% and 20% for 3 hours at a temperature of 90 °C and reported improvement in strength through lignin reduction. Variations in NaOH treatment durations and concentrations by several researchers are represented in Figure 2.21.

With these conflicting reports, it is clear that NaOH concentration, temperature and duration have not yet been optimised for effective result in the treatment of natural fibres for composite manufacture.

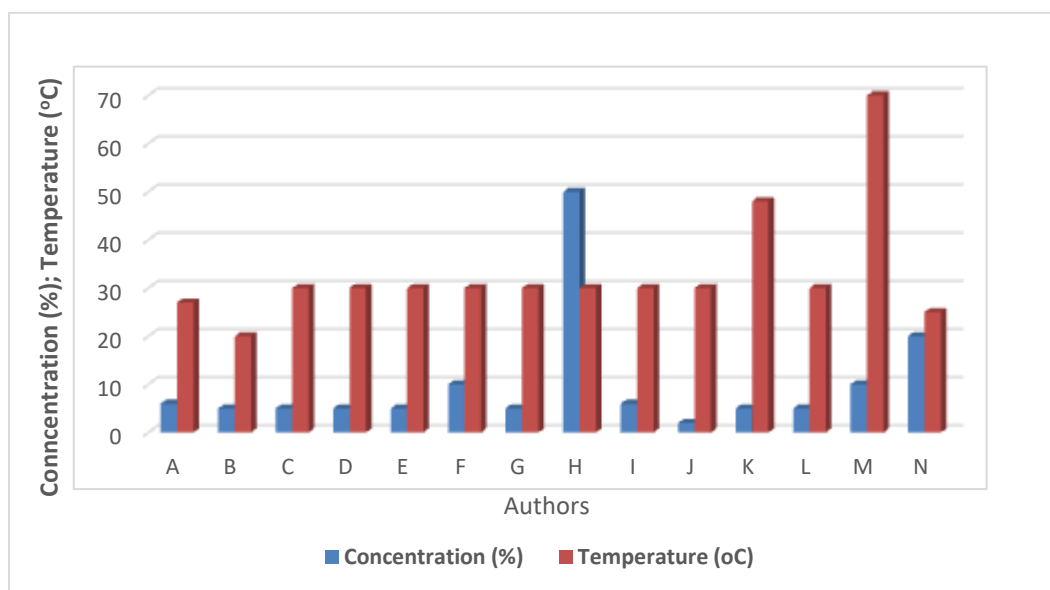


Figure 2.21 Variations in the concentrations of NaOH and treatment temperature as applied by different authors for surface treatments of coir fibre.

Compiled using the following sources: (A) Wang and Huang, 2009a; (B) Yn et al., 2016; (C) Nam et al., 2011a; (D) Bismarck et al., 2001; (E) Narendar and Dasan, 2014; (F) Varma,D,S 1984; (G) Dixit and Verma, 2012; (H) Manjula et al., 2017 (I) Karthikeyan,A, 2012; (J) Khanam et al.,2010; (K) Njoku et al., 2012;(L) Silva et al., 1999; (L) Siva et al., 1999; (M) Hariprasad, Dharmalingam and Praveen, 2013; (N) Rahman and Khan, 2007.

Many researchers have limited the treatment temperature of coir fibre to room temperature and temperatures below 30 °C (Bismarck et al., 2001; Nam et al., 2011b; Dixit and Verma, 2012; Karthikeyan and Balamurugan, 2012; Andic-Cakir et al., 2014; Narendar, Dasan and Jayachandran, 2016; Yan et al., 2016). Post-processing of treated fibres was mostly carried

out at undisclosed temperatures. 90% of the previous researchers adopted the same NaOH treatment concentration of 5% and used similar test durations. Studies revealed that many researchers employed the same NaOH treatment conditions used for other natural fibres with coir. The NaOH concentration used by Sawpan, Pickering and Fernyhough, (2011) concerning jute fibre surface treatments was adopted by Yan *et al.*, (2016) in their research. Intensive and thorough studies on the three key conditions of NaOH treatment (concentration, temperature and duration) have not been adequately investigated. Most of the treatments were by soaking. Temperature, concentration and duration were not adequately varied. A few pre-processing treatments were carried out in an air-circulating oven. The number of samples treated was not reported. The effects of NaOH treatment on coir fibres were under-reported with no clear experimental design apparent prior to undertaking the experiments.

Therefore more work needs to be done to identify:

- (i) The optimum temperature, duration and concentration for enhanced properties.
- (ii) The degree of dependence of each variable on one another for improved performance.
- (iii) The variable that exerts the highest and the least contribution on the properties.
- (iv) All the possible effects of NaOH treatment on natural fibres and their impacts on the mechanical properties.

2.3.2 Silane treatment of coir fibres

Silane coupling agent has been reported to be an effective treatment in the surface characteristics modification of natural fibres (Bisanda and Ansell, 1991; Xie *et al.*, 2010). One of the common coupling agents used is the organo-silane. The general chemical structure of organo silane is as shown in Figure 2.22 (Xie *et al.*, 2010).



Figure 2.22 General structure of an organo-silane.

‘OR’ hydrolyse and react with OH on the fibre. X is a functional group that can react with a specified functional group in the resin.

Coir fibres were treated with 5% γ -methacryloxy propyl trimethoxy silane in methanol at room temperature for 24 hours without pre-treatment; 8.4% decrease in tensile strength, 15.8% increase in modulus and 13% decrease in failure strain were observed (Hill and Khalil, 2000). A reduction in strength with an increase in Young’s modulus was recorded after 2-

0.25% ethoxy silane treatment of coir at room temperature for 60 minutes preceded by pre-treatment with 10% NaOH at 30 °C for 4 hours (Varma, 1984) while Munirah and Ahmad, (2012) observed 25% increase in strength with NaOH pre-treated coir fibres.

Silane treatment carried out on coir prior to manufacture of composites revealed 8.33% increase in the Young's modulus and a decrease in strength by 13.79% (Arrakhiz *et al.*, 2012). When coir fibres were pre-treated with 5% NaOH at room temp for 4 hours followed by 0.1% ethoxy silane in water at room temperature for 120 minutes, a significant increase in strength of the composites by 57.52% was observed (Hemsri *et al.*, 2012). A reduction in weight by 4.3% and 3.9% for untreated and NaOH pre-treated coir fibres respectively have been observed (Javadi *et al.*, 2010). Treatment temperatures were not effectively reported.

2.3.3 Heat Treatment of coir fibres

Natural fibres are made up of different chemical constituents; as a result, heat treatment at increased temperature can give rise to varying degrees of chemical and physical changes. Such physical changes include change in colour and appearance, weight, fibre orientation while the chemical changes have been associated with the decomposition of the chemicals that make up the fibres. Natural fibres have showed different decomposition temperatures and different levels of changes in physical properties. Investigations from many researchers confirm that natural fibres undergo thermal degradation in two stages; (a) at lower temperatures (150-300 °C) and (b) at higher temperatures (300-400 °C). (a) has been linked with the degradation of cellulose and includes hydrolysis, oxidation, dehydration among other mechanisms while (b) is mainly associated with degradation of both cellulose and lignin with formation of charred product (Varma, Varma and Varma, 1986; Silva *et al.*, 1999; Dam *et al.*, 2006).

In one study, coir fibres were subjected to heat treatment in an air-circulating oven at a temperature of 150 °C for 10 minutes; a drop in tensile strength by 0.58% and increase in Young's modulus by 20% were recorded, however, at 200 °C for the same duration, a drop in the tensile strength and increase in modulus by 40% and 20% respectively were recorded (Ezekiel *et al.*, 2011). In a similar way, at heat treatment temperatures of 100, 140 and 180 °C, a drop in the tensile strength by 14, 24 and 29% and a drop in the value of the degree of polymerization by 5, 12 and 19% respectively were observed (Khan, Alam and Terano, 2012). An improvement in dimensional stability has been recorded for heat treated fibres. At 170 °C and duration of 2 hours for Eucalypt wood, a 60% anti shrinkage efficiency in radial

direction was recorded (Esteves, Domingos and Pereira, 2007). After heat treatment of hemp and bamboo fibres, a 60% decrease in the tensile strength was recorded for both fibres (Ochi, Takagi and Niki, 2002). Kenaf and bamboo fibres were subjected to an oven heat treatment temperature of 140 °C, an increase in tensile strength, fracture strain, crystallinity index and in the length to width ratio were observed but at temperature above 140 °C, the tensile strength of kenaf fibres decreased (Cao, Sakamoto and Goda, 2007; Yun *et al.*, 2016). Dixit and Verma, (2012) recorded a reduction in moisture content, increase in crystallinity index, increase in elastic modulus and increase in fracture strain at 140-150 °C for 4 hours of oven heat treatment.

2.3.4 Hot compaction of fibres

Compaction is a term usually used in soil science. Soil structure is modified via compaction. Compaction reduces the porosity of soil and hence modifies the pore system (Pagliai, 1992). Reduction of soil porosity via pressure application has been associated with increase of moisture penetration resistance (Pagliai, 1992). The properties of coir are largely dependent on its structure, functional group present, its moisture absorption capacity, level of porosity, and its load-bearing capacity. The inter-connected porosity of coir lignocellulosic fibre is one of the factors leading to the poor tensile strength of its composites (Luz *et al.*, 2017). These pores result in increased porosity in coir composites and hence reduced fatigue resistance, higher susceptibility to water penetration and increased scatter in Weibull strength (Hai, Kim and Lee, 2012). Detailed insight into the porosity in coir fibres and pore size distribution can be obtained via ImageJ software or equivalents - which accounts for pore diameters of various sizes. Recent technological advances with respect to sample preparation, potting, polishing and image analysis have led to better quantification of coir pore volume fraction.

In one study, the porosity of calcium phosphate cement composite was decreased from 50 to 30% via compaction pressures of between 18 and 106 MPa and as a result, the Weibull modulus increased, hence suggesting a decrease in flaw size with increase in compaction pressure (Barral *et al.*, 2001). Swolfs *et al.*, (2014) observed that process parameters for hot press compaction such as pressure, temperature and dwell time exert influences on the resulting tensile properties and impact resistance of materials. They reported a decrease in penetration impact resistance and tensile properties with increasing compaction temperature of self-reinforced polypropylene (PP) composites. For natural fibre composites such as flax and jute manufactured via hot pressing, an increase in tensile strength, flexural strength and modulus were observed with optimal parameters determined by compaction temperature,

duration and fibre volume fraction (Nam *et al.*, 2011a.; Yan, Chouw and Yuan, 2012). Little work has been published on hot press compaction of lignocellulosic natural fibres in the context of manufacturing composites.

2.4 Moisture absorption kinetics of natural fibres

Moisture absorption of natural fibres and natural fibre composites presents a strong drawback in their outdoor applications as has been previously discussed. Moisture ingress leads to reduction in the mechanical performance of natural fibre composites, therefore a good understanding of moisture permeability, diffusivity and solubility is of paramount importance for optimum performance of these fibres in composites. Several attempts have been made in studying moisture absorption of natural fibres such as oil palm fibre (Sreekala and Thomas, 2003), jute (Giridhar, Kishore and Rao, 1986), hemp (Saikia, 2010), flax (Muñoz and García-Manrique, 2015) and sisal (Giridhar, Kishore and Rao, 1986) fibres as well as their composites at varying temperatures and certain measures such as coating and treatment of fibres have been employed.

Mechanisms of water absorption in natural fibres occur mainly by diffusion. Diffusion could be Fickian (Saikia, 2010; Muñoz and García-Manrique, 2015) or non-Fickian (Sreekala and Thomas, 2003) or both Fickian and non-Fickian (Sreekala and Thomas, 2003). Water absorption of natural fibres and natural fibre composites occur at room temperature and at elevated temperature (Giridhar, Kishore and Rao, 1986; Dhakal, Zhang and Richardson, 2007; Muñoz and García-Manrique, 2015). For single natural fibres, two stage absorption behaviour has been observed with the first stage occurring rapidly with a linear uptake of about 30-60% sorption while the second stage proceeds more slowly until the saturation point (Alix *et al.*, 2009; Saikia, 2010; Abdullah and Ahmad, 2012).

Studies on moisture sorption behaviour of single fibres such as okra and bowstring hemp fibres at different temperatures by (Saikia, 2010) showed that moisture uptake of natural fibres is a function of temperature and diffusion coefficient. The coefficient of diffusion and water uptake increase with an increase in the fibre volume fraction as a result of increase in cellulose and void content with more compact fibres displaying a lower level water uptake (Giridhar, Kishore and Rao, 1986; Dhakal, Zhang and Richardson, 2007; Ho *et al.*, 2012; Muñoz and García-Manrique, 2015; Haameem *et al.*, 2016). Tensile and flexural properties of natural fibres and their composites decrease with increase in water sorption (Sreekala and Thomas, 2003; Dhakal, Zhang and Richardson, 2007; Chaudhary, Bajpai and Maheshwari,

2018). Treated fibres are less prone to water absorption than untreated, their failure mode displays interface effects of debonding, dislocation and fracture (Alix *et al.*, 2009; Abdullah and Ahmad, 2012; Chaudhary, Bajpai and Maheshwari, 2018). Water content W_a is calculated using the following equation:

$$W_a = \left[\frac{M_w - M_d}{M_d} \times 100 \right] \% \quad [2.13]$$

M_d = constant mass (g) of dry coir fibre sample

M_w = mass (g) of wet coir fibre sample after time t

The kinetics of absorption of water by fibre at specified temperature as has been stated by Saikia, (2007) and Gridhar, Kishore and Rao, (1986) is given as:

$$W_a = M_m \left[1 - \frac{8}{\pi^2} \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} \exp \left\{ -\frac{D(t)}{h^2} \pi^2 (2n+1)^2 \right\} \right] \quad [2.14]$$

M_m = maximum absorbed water content at equilibrium

h = initial diameter of the sample

D = diffusion coefficient

n = summation index

From the slope of the linear portion of weight gain curve versus the square root of time, the diffusion coefficient D of the coir fibre samples for the specified temperatures can be determined using:

$$D = D_0 \exp \left(-\frac{E_D}{RT} \right) \quad [2.15]$$

where

D_0 = pre-exponential factor of the diffusion, R = universal gas constant, T = absolute temperature, E_D = activation energy of diffusion

Plotting $\ln D$ versus $1/T$, E_D can be determined from the slope of the linear region.

2.5 Tensile testing of coir fibre

The tensile strength of natural fibres is usually determined through the single-fibre tensile test. Single-fibre tensile testing of coir fibres has been carried out by several authors (Defoirdt *et al.*, 2010; Muensri *et al.*, 2011; Alves *et al.*, 2013; Mathura and Cree, 2016). This starts with careful selection of the fibres with fairly uniform diameters. A single-fibre is mounted on a paper frame as shown in Figure 2.23. The function of the paper frame is to hold

the fibre in position and protect it from being crushed during tensile testing. It also serves to define the gauge length for tensile testing. The diameter of the fibre is taken at different points along the gauge length prior to mounting it on the paper frame (Defoirdt *et al.*, 2010; Muensri *et al.*, 2011). The frame assembly is secured in the clamping fixture on the tensile test machine and the frame is cut (as shown by the dotted line in Figure 2.23).

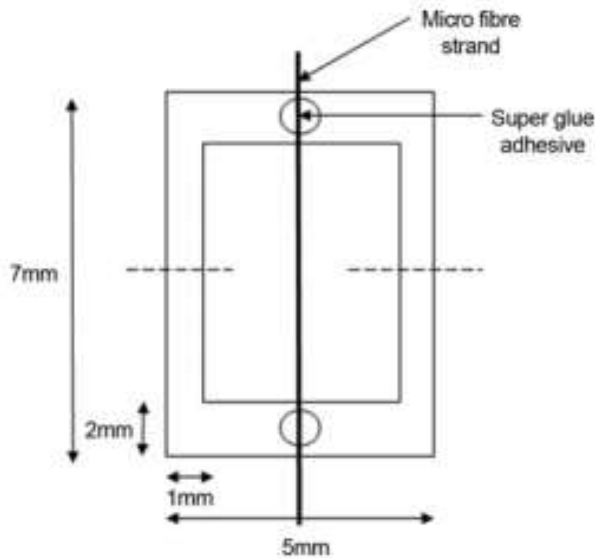


Figure 2.23 Illustration of single fibre tensile test specimen mounted on a window paper frame with dashed line showing the cutting of the two sides of the paper prior to tensile testing (Buana *et al.*, 2013).

Tensile test is usually carried out in accordance with ASTM 3822-07 which recommends a gauge length of 10 mm and above. Natural fibres have been identified as fibres with non-uniform properties as a result of their intrinsic nature, so a minimum sample size requirement of approximately 30 must be used for meaningful statistical analysis. The literature on this topic has been integrated in the Results and Discussion chapter.

Microstructural parameters of plant fibre have been reported to contribute immensely to their mode of failure (Satyanarayana *et al.*, 1986; Gassan and Bledzki, 1999; Martinschitz *et al.*, 2008); these parameters include microfibrillar angle, cellulose content and cell dimensions. The microfibrils located in the cell wall form a helical angle with the fibre axis bringing about a spiral arrangement of the crystallites with pitch differing for different fibres. Pullout of the microfibrils prior to breaking with an intracellular fracture mode could be as a result of large failure strain such as found in coir. For fibres such as sisal with lower microfibrillar angle ($10\text{--}22^\circ$) and higher (67%) percentage of cellulose, the fracture does not originate from the microfibrils but the fracture mode is rather intercellular (Satyanarayana *et al.*, 1986). Fracture has also been reported to take place at the defective portion of the crystalline fibrils

in pineapple/sisal fibres (Fa'bio *et al.*, 2007). Since thicker fibres contain a greater number of weaker points in terms of defects, they are also more prone to premature failure (Monteiro *et al.*, 2011). On the application of stress during tensile testing, the weaker microfibrils break leading to a flaw or microcracks in the fibre structure and subsequent brittle failure of the fibre.

2.5.1 Weibull Statistics for analysing the tensile strength of reinforcing fibres

The Weibull distribution is a continuous probability distribution used to assess the strength distribution exhibited by fibres (Kulkarni, Satyanarayana and Rohatgi, 1983; Xia *et al.*, 2009). The assumptions in the Weibull distribution are as follows:

- (i) The material must be brittle which means that it does not exhibit a significant plastic deformation before failure (Xia *et al.*, 2009).
- (ii) The strength of the material must be dependent on the presence of flaws within the material (Xia *et al.*, 2009; Defoirdt *et al.*, 2010).
- (iii) The Weibull modulus and characteristic strength of the fibres must be positive numbers. If the Weibull modulus becomes negative then the mean strength of the fibre increases with increase of fibre length which is not observed in brittle fibres (Zafeiropoulos and Baillie, 2007).

If a chain is made up of several links, n , then P is the probability of failure at any load L applied to a single link, then the probability of failure of a whole chain made up of n links is Pn , the chain fails if anyone of its links fails (Weibull, 1951).

Probability of non-failure of the chain = $1-Pn$, which is equal to the probability of simultaneous non-failure of all the links $(1-P)^n$. Therefore;

$$1 - P_n = (1 - P)^n \quad [2.16]$$

Any distribution function may be written in the form

$$F(x) = 1 - e^{-\varphi(x)} \quad [2.17]$$

$$P_{(n)} = 1 - e^{-\varphi(x)} \quad [2.18]$$

Specifying $\varphi(x)$; the simple function satisfying this condition is;

$$\frac{(x-x_u)^m}{x_0} \quad [2.19]$$

Therefore,

$$F_x = 1 - e^{-\frac{(x-x_u)^m}{x_0}} \quad [2.20]$$

(General Weibull equation)

This can be written as:

$$F_{(\sigma)} = 1 - e^{-\left(\frac{\sigma}{\sigma_0}\right)^m} \quad [2.21]$$

Equation [2.21] is the two-parameter Weibull distribution.

F_{σ} = Strength distribution

σ_0 = Scale parameter

m = Shape parameter. The bigger the value of the shape parameter the smaller the variation in the data, in synthetic fibres, m value ranges between 5 and 15 while in natural fibres, it is between 1 and 6 (Defoirdt, *et al.*, 2010; Weyenberg *et al.*, 2000).

n = Sample size assessed

Weibull strength varies from natural fibre to natural fibre (see Table 2.1). Weibull parameters are influenced by the degree and distribution of flaws in the fibre and hence scatter in the fracture strength. Weibull parameters increase with increase in fibre diameter (Kulkarni, Satyanarayana and Rohatgi, 1983). It has been observed that fibre treatment offers no significant effect on the Weibull parameters but fibre alignment and uniform tensile loading are critical factors affecting the consistency of the Weibull parameters (Zafeiropoulos and Baillie, 2007; Trujillo *et al.*, 2014).

2.6 Density relationships in natural fibres

The density of natural fibres has been a major attraction for its use in the manufacture of composites especially for light-weight applications. Several research studies have been carried out to determine the bulk (Dam *et al.*, 2006; Kandemir *et al.*, 2020) and absolute (Truong *et al.*, 2009) density of natural fibres. The absolute density is a measure of the solid matter, it therefore does not take into account the pores and lumen as opposed to the bulk density which includes the pores and lumen. The absolute density of most plant fibres is in the range of 1400-1500 kg/m³, the absolute density of pure cellulose and regenerated cellulose have been given as 1592 kg/m³ and 1583 kg/m³ (Mwaikambo and Ansell, 2001). The low density of natural fibres in comparison with synthetic fibres such as carbon and glass fibres has been attributed to the presence of porosity in natural fibre. The absolute density of

natural fibres is carried out using gas pycnometer. Measurement of bulk density is usually carried out using solvent such as benzene and the bulk density calculated using Equation [2.22].

$$\rho_b = \frac{\rho_s W_f}{W_f - W_{fs}} \quad [2.22]$$

where w_f is the weight of the fibre, ρ_b is the bulk density, ρ_s is the density of the solvent, W_{fs} is the weight of the immersed fibre.

2.7 Thermogravimetric analysis of natural fibres

One of the drawbacks of natural fibres is their thermal degradation at elevated temperatures. Thermogravimetric analysis has been performed on natural fibres to ascertain their weight loss as a result of thermal degradation at increasing temperature. The derivatives (DTG) of the thermogravimetric analysis have also been reported. Natural fibres contain organic materials which are influenced by heat treatments at certain temperature levels. Heat treatments at elevated temperatures have yielded changes in chemical and physical properties of natural fibres. These changes are not limited to weight loss, strength, modulus, crystallinity, enthalpy and orientation of microfibril angle and colour; the changes have been linked to their structural characteristics, morphology, impurities and defects (Yang and Kokot, 1996; Shahzad, 2013). Thermal degradation of natural fibres especially at temperatures above 200 °C has led to reduced mechanical properties of the fibre (Saheb and Jog, 1999).

However, thermal stability of natural fibres can be improved by fibre treatment such as NaOH treatment, coating the fibre, grafting the fibre with monomers and by polymerisation. The explanation is that untreated fibres contain thermally unstable constituents such as hemicelluloses and pectin but through alkali treatment, these constituents are reduced (Beckermann and Pickering, 2008).

Thermal degradation of natural fibres depends on the environment: air or inert gas. Thermal degradation of natural fibres occurs in stages (i): the first stage results in weight loss at a temperature less or equal to 100 °C: this has been linked to evaporation of water absorbed by the natural fibre, (ii) the second stage usually sets in at about 260 °C resulting in a gradual but steady decrease in weight; this stage is the initial stage of thermal degradation (onset temperature), (iii) the third stage results in a rapid weight loss resulting to a significant thermal degradation with the liberation of volatiles and char. The derivative of the TGA trace

(DTG) curve usually features the onset of weight loss and maximum weight loss with their corresponding temperatures. Two decomposition peaks had been displayed by treated and untreated coir fibre at 200-250 °C and greater than 350 °C (Saw, Sarkhel and Choudhury, 2011). A two-stage thermal degradation of hemp fibre with the first stage associated with the degradation of cellulose while the second stage was associated with the degradation of cellulose and lignin was recorded. This was followed up by heating the hemp fibres up to 260 °C resulting in the softening of lignin with the fibre bundles being opened into single fibres. This was more pronounced in fibres heated in air than those heated in nitrogen atmosphere (Saheb and Jog, 1999; Prasad, Sain and Roy, 2005; Beckermann and Pickering, 2008; Shahzad, 2013).

A systematic investigations of coir fibre sample by Rosa *et al.*, (2009) via thermogravimetry and DTG analysis in nitrogen atmosphere revealed decomposition profiles of coir characterized by three peaks: the first peak was between room temperature and 150 °C, the second occurred at about 190 °C and the third occurred between 290 and 360 °C. These decompositions have been attributed to several factors such as the evaporation of water, degradation of hemicellulose and degradation of cellulose respectively. Similar observations were made by Ezekiel *et al.*, (2011). Tomczak, Sydenstricker and Satyanarayana, (2007) observed mass losses of 5.4%, 19.6% and 40.1% at three transition temperatures of 67.2 °C, 259.2 °C and 313.4 °C respectively in an inert atmosphere by thermogravimetry (DTG) of coir fibres. The degradation of coir fibre has been reported from temperatures ranging from 230 °C to 268.8 °C this has been associated with vaporization of volatile components and the weight loss experienced above 200 °C is as a result of oxidation, followed by the onset of lignin decomposition at 380 °C (Dam *et al.*, 2004; Brígida *et al.*, 2010). Different decomposition onset temperatures have been recorded for different fibres as shown in Table 2.1.

2.8 Manufacturing methods for Composites

Natural fibres possess peculiar properties and as a result they are affected by their manufacturing techniques. They possess limited temperature exposure with maximum limit of 200 °C above which leads to fibre degradation and loss of properties. For good quality composites with enhanced properties and performance, the interface and manufacturing routes as well as processing conditions such as temperature and relative humidity are important considerations. The most widely used manufacturing techniques for composites

include hand lay-up, resin transfer moulding, compression moulding, hot-pressing (Figure 2.24), extrusion, autoclave (Joseph *et al.*, 2002; Cheng *et al.*, 2009; Rao, Rao and Prasad, 2010; Ho *et al.*, 2012; Thakur, Thakur and Gupta, 2014; Pickering, Efendy and Le, 2016). In a study, coir/PBS composite was fabricated via hot pressing technique at a temperature of 150 °C and pressure of 10 MPa for 10 minutes, and cooling was achieved by quenching with ice water without water absorption by the composite (Nam *et al.*, 2011a). The main parameters to monitor in hot-pressing are the compaction pressure, dwell time, sample thickness and size, temperature and the viscosity of matrix, residual stresses, fibre breakage, sink marks, scorching and warpage (Ho *et al.*, 2012).

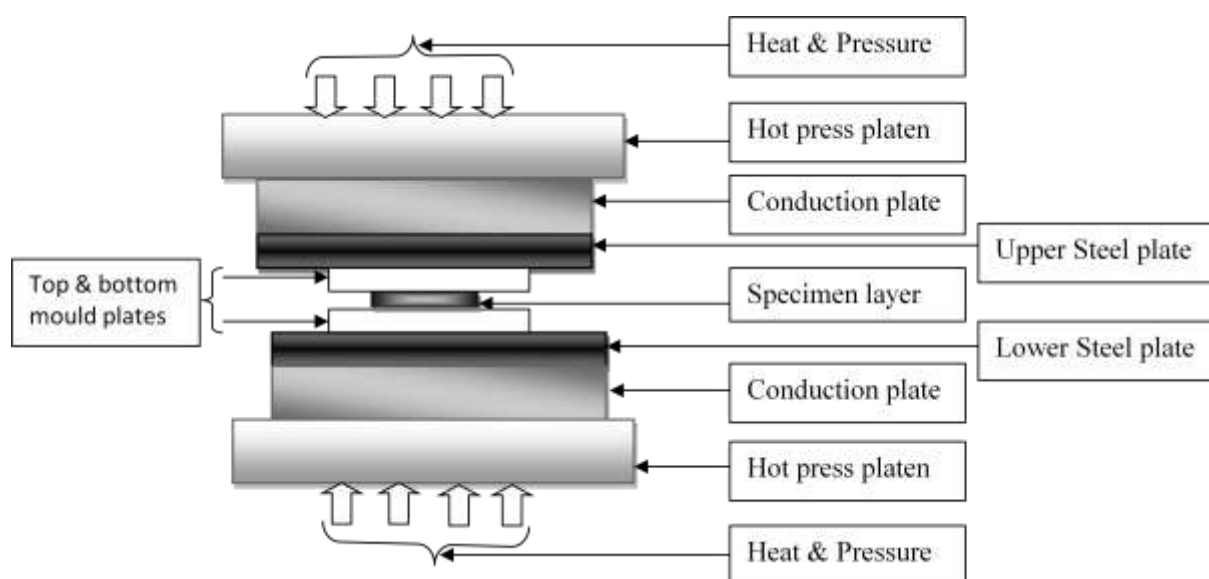


Figure 2.24 Schematic representation of a typical hot press adapted from Nam *et al.*, (2011b).

The manufacturing methods that have been used for coir fibre composites are as shown in Table 2.4. The Table shows that hand layup has been predominantly used in the manufacture of coir fibre composites where the fibres or fabrics are manually impregnated by the resin using injectors, rollers, and brushes. The uniformity of the composite in terms of thickness, fibre to matrix ratio and void content throughout the sample depends on the skills of the laminator (Cheung *et al.*, 2009; Elkington 2015). From Table 2.4, the highest tensile strength and Young's modulus of 39.80 MPa and 3.60 respectively were achieved using vacuum assisted resin transfer moulding. Apart from the manufacturing technique, other factors that have been noted to contribute to the properties of the manufactured composites include; fibre orientation (Geethamma *et al.*, 1998), fibre length (Joseph *et al.*, 1999), fibre volume fraction (Arib, Sapuan and Ahmad, 2006), chemical treatment (Rout *et al.*, 2001; Nam *et al.*, 2011b)

and processing temperature (Bodros *et al.*, 2007). The presence of void has been noted to lead to reduction in the tensile properties (Hill and Khalil, 2000). From Table 2.4, Rait, (2014) obtained a tensile strength of 30.44 MPa. Using manually-prepared coir fibre preform as shown in Figure 2.25. The preform constitutes several gaps in between the filaments hence the reduced tensile strength could be attributed to these gaps. It is not easy to compare the tensile properties of coir composites manufactured by different authors because these properties depend on several factors as mentioned earlier other than the manufacturing method used.

Table 2.4 Methods and matrices used in the manufacture of coir composites

Production method	Resin used	Tensile strength (MPa)	Young's modulus (GPa)	Elongation at break (%)	Author
Hand layup	Epoxy LY 3505 and Hardener XB 3403	30.44	2.82	1.00	(Rait, 2014)
Hand layup	Epoxy CY 205 and Hardener HY 951	17.86	NA	NA	(Harish <i>et al.</i> , 2009)
Hand layup	Epoxy LY 556 Hardener HY 951	13.05	2.06	NA	(Biswas, Kindo and Patnaik, 2011)
Vacuum assisted resin transfer moulding	Polyester (Crystic 471 PALV, Scott Bader) and catalyst (Methylethyl ketone peroxide)	39.80	3.60	5.20	(Hill and Khalil, 2000)
Resin Infusion	Epoxy LY 3505 and Hardener XB 3403	28.3	2.9	NA	(Kim, 2014)
Hot press	Poly(butylene Succinate)	NA	NA	NA	(Nam <i>et al.</i> , 2011b)

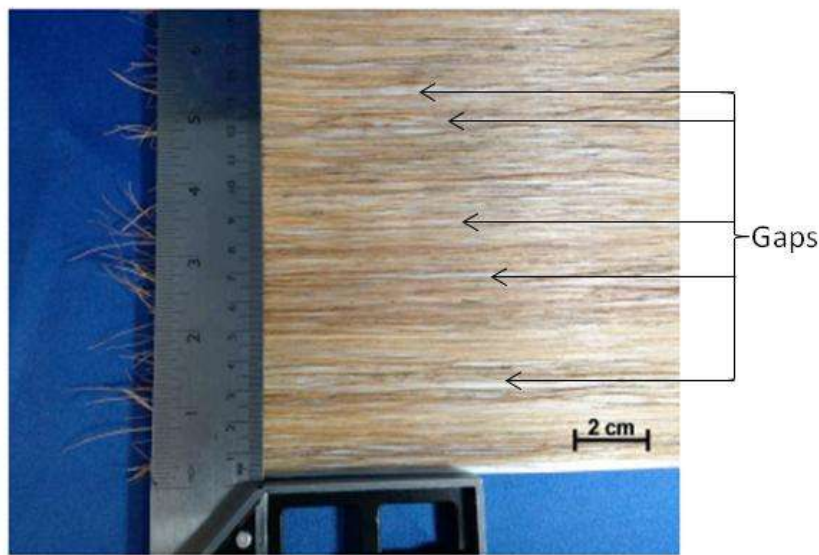


Figure 2 25 manually aligned coir fibre preform for composites production showing gaps in between the filaments (Kim, 2014).

2.9 Hybrid composites

The concept of fibre hybridization is to achieve cost effective, light weight and improved performance properties of composites which are not obtainable in non-hybridized composites for engineering application. Many researchers have reported on coir fibre hybrid composites as shown in Table 2.5, though none has been done with spread glass. Pavithran, Mukherjee and Brahmakumar, (1991) examined coir-glass polyester intermingled fibre hybrid composites, and found lower moisture absorption, better resistance to weathering, better flexural and interlaminar shear strength than that of non-hybrid coir-polyester composite. Sanjay, Arpitha and Yogesha, (2015) studied the mechanical properties of natural fibre reinforced woven jute glass hybrid epoxy composites; it was found that the tensile and flexural properties of the composites improved. Water uptake of hybridized composites increases with increasing fibre content (Akash *et al.*, 2016). The mechanical properties of natural fibre composites have been significantly improved by glass fibre addition (Sreekala *et al.*, 2002; Pandita *et al.*, 2014; Bakri, Chandrabakty and Soe, 2016). The effects of hybridization of natural fibre with synthetic fibres differ in the literature. George, Sreekala and Thomas, (2001) reported a positive hybrid effect as a result of increase in fibre loading. Thwe and Liao, (2000) reported a positive hybrid effect as a result of fibre length, fibre loading and level of adhesion.

Table 2.5 Properties of coir fibre hybrid composites.

Hybrid	Tensile strength (MPa)	Young's modulus (GPa)	Flexural strength (MPa)	References
Coir-Glass (40:60)	50.56	0.2	34.76	(Chaithanyan <i>et al.</i> , 2013)
Coir-Glass (50:50)	71.16	0.4	45.10	
Coir-Glass (10:20)	110.4	0.90	287.4	(Bakri, Chandrabakty and Soe, 2016)
Coir-Glass (15:15)	79.4	0.64	206.7	
Coir-Banana (untreated)	13.63	0.94	36.00	(Hariprasad, Dharmalingam and Raj, 2013)
Coir-Banana (treated)	16.43	1.13	20.52	
Coir-Glass-Coir	47	1.45	65	(Jayabal, Natarajan and Sathiyamurthy, 2011)
Coir-Glass-Glass	52	1.34	71	
Coir-Glass	34	1.15	64	
Coir-Sisal (20:20)	48	N/A	76.68	(Akash <i>et.,al.</i> , 2016)

Processing effects such as shrinkage during curing of resin, cooling rate, and volatiles from resin curing have since been reported to lead to increase in void content. Fewer voids result in

improved mechanical properties of hybrid composites. This can be achieved through close packing or arrangement of fibres (Sreekala *et al.*, 2002).

2.10 Summary of literature review

From the above literature review, several conclusions can be drawn:

- (i) Natural fibres possess variable diameters and lengths even within the same species.
- (ii) Surface treatments such as alkali treatment and hot compaction bring about changes in the surface properties of natural fibres promoting increase in the tensile properties. However the optimum treatment conditions and the variable(s) with significant contribution to the improved tensile properties remain to be ascertained. Heat treatment often brought about a decrease in the tensile properties.
- (iii) Manufacture of natural fibre composites and hybridisation of natural fibres still require a significant amount of research work. Better manufacturing and processing techniques are necessary to improve the quality and the properties of the manufactured composites.

3 Experimental Techniques and Methodology

3.1 Materials

The development of the fibre into composites involved: fibre selection, pre-treatments of the fibres by washing and drying, chemical and physical treatments of the fibres and finally, composites manufacture. This section therefore gives a detailed account of all materials used in this research project to achieve this.

3.1.1 Reinforcements

(i) Coir Fibres: the retted (a process used to separate coir fibres from coconut husk) coir fibres were supplied by Hayleys Fibre Plc, Sri Lanka. The fibres came in several 3-tie bundles of non-uniform lengths. The term 3-tie is used because the bundle is secured by three coir fibre strands. This class of bundle represents the longest untwisted coir fibres sold commercially by Hayleys and it is considered to be the premium quality. The fibres were fumigated prior to dispatch to the UK. A photograph of the 3-tie coir fibre bundle is shown in Figure 3.1. The average weight of each bundle after measurement was found to be 0.25 kg. The fibre lengths were in the range 100 mm to 350 mm.



Figure 3.1 As-received 3-tie coir fibre bundle.

(ii) E-glass fibres: Creels of E-glass of 2400 TEX Owens-Corning Advantex R25H were supplied by Halyard Precision Composites, UK. The electrically-graded glass fibres contained approximately 2400 individual filaments in each bundle, designed for use in several applications including filament winding and pultrusion. These fibres were supplied (with an epoxy compatible “size”) in the form of a creel or bobbin. The E-glass fibres were used in the fibre-spreading experiments (see Section 3.6) and in the manufacture of composites (covered in Section 3.7) for improved strength. Selected properties of the E-glass fibre bundle are as given in Table 3.1.

(iii) T700 Carbon fibres: These were Carbon T700SC-12K-60E. They were supplied by Toray carbon fibre Europe. The fibre bundle was spread using an in-house fibre spreading

rig; this is discussed in Section 3.6. The spread fibres were used in the production of composites with and without coir fibres. The properties of the fibre according to the manufacturers are given in Table 3.1.

Table 3.1 Fibre Suppliers and selected properties for E-glass and T700 Carbon fibres.

Fibre type	MFR	TEX (g/1000) (m)	AF diameter (μm)	Size content (%)	Tensile strength (MPa)	Elongation at break (%)	Density (kg/m^3)
E-glass	Owens Corning	2400	24	0.5	2750	3.0	2620
Carbon	Toray Carbon Fibre Europe	800	7	0.2	4900	2.1	1800

*MFR=Manufacturer; AF=Average fibre; TEX=number of filaments in a bundle;
size=coating*

3.1.2 Resin Systems

Resin systems used were as follows:

(i) Synthetic epoxy/amine thermosetting resin: Araldite LY 3505 (epoxy resin) and hardener XB 3403 (amine hardener) are commercially available laminating resin system used for manufacturing composites. They were supplied by Huntsman Advanced Materials, UK. The manufacturer's recommended stoichiometric ratio for the epoxy and amine is 100:35 parts by weight respectively. The required quantities of the epoxy resin and the amine hardener were weighed out carefully using an Ohaus Analytical Plus model AP110S, 5-digit balance. The mixture was stirred continuously for about 10 minutes with a wooden spatula before degassing in a vacuum chamber using a Gallenkamp vacuum unit (model OVL570 supplied by Island Scientific Ltd, UK,) for about 15 minutes. The weighing and mixing of the resin and hardener were carried out under a fixed air-extraction system. Appropriate personal protection equipment was worn during this operation.

(ii) Bio epoxy resin: Super sap ® epoxy systems [Epoxy (Part A) and hardener (Part B)] are low-viscosity bio-derived epoxy resin compatible with glass, carbon, aramid and natural fibres. They are used in the production of composites via hand layup, vacuum resin infusion and resin transfer moulding. The resin and hardener was manufactured by Entropy resins USA, and supplied by Ferrer Dalmau, UK. The stoichiometric ratio of resin to hardener is 100:47 parts by weight respectively. Mixing of the two components was carried out with a wooden spatula until a consistent and homogenous blend was achieved.

(iii) End-tab resin for single-coir fibre tensile test:

A number of adhesives were evaluated prior to selecting Loctite Super Glue-a general purpose cyanoacrylate adhesive supplied by Loctite Super Glue UK. This was used in bonding the single coir fibre specimen to the “window frame” (see Section 3.7 for details).

(iv) End-tab resin for composites: Sand-blasted aluminium end-tabs were bonded to the ends of the specimens using Scotch weld 9323-2 UK. The stoichiometric ratio for the resin and hardener was 100:27 parts by weight respectively. The mixing procedure used was as described previously.

(v) Potting resin for metallographic and optical microscopy: EpofixTM supplied by Struers Ltd UK was used for mounting specimen for optical microscopy. Samples were sectioned to have a flat edge and these were mounted on moulds supplied by Struers Ltd. UK. The mix ratio of the Epofix resin to hardener as 15:2 parts by weight respectively. The components were mixed and degassed as mentioned previously.

3.1.3 Chemicals

(i) NaOH: Sodium hydroxide pellets (Honeywell, Sigma-Aldrich Company Ltd, UK). The appropriate quantity was weighed and dissolved with the required volume of deionised water and used in the treatment of coir fibres. A magnetic stirrer was used to aid the dissolution. Due to the exothermic nature of the dissolution, approximately 0.1g of the NaOH was introduced until the required mass was dissolved. This operation was carried out in a fume cupboard.

(ii) Amino-silane: (3-Aminopropyl) trimethoxysilane used in the fibre treatment was supplied by Sigma-Aldrich Company, Ltd. UK. A septum was used to stopper the bottle and a syringe and needle were used to withdraw the liquid from its storage container. The amino silane was prepared at specified concentrations of 0.5%, 1%, and 1.5% in distilled water. The coir fibres were treated with the prepared solutions.

(iii) Epoxy-silane: (3-Glycidyloxypropyl)-trimethoxysilane is an epoxy-terminated silane and it was supplied by Sigma-Aldrich Company, Ltd. UK. It was prepared in concentrations of 0.5, 1 and 1.5% and it was used to carry out surface treatment on coir fibres.

(iv) Sulphuric acid: Sulphuric acid (H_2SO_4) was supplied by the Sigma-Aldrich Company Ltd. UK. It was used to neutralise the NaOH waste solution.

(v) Lignin: supplied by Sigma-Aldrich Company, Ltd. UK.

(vi) DMSO: Dimethyl sulphoxide was supplied by the Sigma Aldrich Company Ltd, UK. It was used in the dissolution of lignin for the infill experiments.

3.2 Preparations and Methods

The as-received coir fibre bundle was vacuum-cleaned to extract loose dust from the fibres. The fibres were washed with warm tap water and this procedure was repeated five times and then dried in an air-circulating Memmert supplied by Fisher Scientific, UK oven at 80 °C for two hours. The fibres were inspected and sorted where the debris-free and those with diameters ranging between 0.21 to 0.22 mm about the middle and a minimum length of 120 mm were selected. The fibres were stored in a moisture-free container at room temperature until required.

3.2.1 Sodium hydroxide treatment of coir fibres

3.2.1.1 Factorial-based design of experiments

These experiments were designed to derive the optimal conditions for treating coir fibres with sodium hydroxide (NaOH). Multilevel factorial design of experiments using Minitab-18 Software was adopted. The design summary is displayed in Table 3.2 and the complete experimental design is shown in Table 3.3. Three factors were chosen: temperature; concentration of NaOH; and treatment time. The experiments were carried out based on temperature levels at; 30 °C, 50 °C and 80 °C.

Table 3.2 Design of experiments summary for the treatment of coir fibres with NaOH using Minitab-18 Software.

Factors:	3	Replicates:	1
Base number of experiments:	27	Total number of experiments:	27
Base blocks:	1	Total blocks:	1

Table 3.3 Complete experimental design for the treatment of coir fibres with NaOH

Factors: Minitab Symbols			Factors: Experimental Conditions		
NaOH Concentration	Temperature	Duration	NaOH Concentration (%)	Temperature (°C)	Duration (Hours)
1	1	1	1	30	24
1	1	2	1	30	48
1	1	3	1	30	72
1	2	1	1	50	24
1	2	2	1	50	48
1	2	3	1	50	72
1	3	1	1	80	24
1	3	2	1	80	48
1	3	3	1	80	72
2	1	1	5	30	24
2	1	2	5	30	48
2	1	3	5	30	72
2	2	1	5	50	24
2	2	2	5	50	48
2	2	3	5	50	72
2	3	1	5	80	24
2	3	2	5	80	48
2	3	3	5	80	72
3	1	1	20	30	24
3	1	2	20	30	48
3	1	3	20	30	72
3	2	1	20	50	24
3	2	2	20	50	48
3	2	3	20	50	72
3	3	1	20	80	24
3	3	2	20	80	48
3	3	3	20	80	72

3.2.1.2 Preparation of NaOH solutions at specified concentrations

This was carried out using 1000 ml beaker, magnetic stirrer and weighing balance. The required quantity of deionised water (DI) was measured using measuring cylinder and transferred to 1000 ml glass beaker. The required quantity of NaOH pellets was weighed and

introduced gradually into the deionised water and stirred using magnetic stirrer until dissolution was achieved. The concentrations used were 1, 5 and 20 % of NaOH.

3.2.1.3 Treatment of coir fibre using specified concentrations of NaOH

Debris-free, washed and dried coir fibres of minimum length of 120 mm and of fairly uniform diameter about the mid-length were selected. The required quantities of these fibres were placed in the glass jar (see Figure 3.2). The prepared NaOH solution was introduced into the custom-made glass jar until the fibres were submerged completely. The glass jar was placed in a water bath and secured with clamps. The surface of the water bath was covered with aluminium foil to minimise heat loss and the evaporation of water. A condenser was attached along with the water inlet and outlet tubes as shown in Figure 3.2. The start of the treatment time was recorded when the water bath attained the set temperature (30, 50 or 80 °C). The power to the water bath was turned off when the desired treatment time was reached. After the treatment solution in the glass jar in the water bath attained room temperature, it was decanted and neutralized with H_2SO_4 before disposal. 600 ml DI water was transferred into the glass jar containing the treated fibres and bubbled with nitrogen for 10 minutes. A fresh solution of deionised water introduced and the procedure repeated until a neutral pH was achieved. The treated fibres were dried in an air-circulating Memmert laboratory oven at 80 °C for 2 hours. The fibres were stored in a moisture-free weather-resistant container until required.

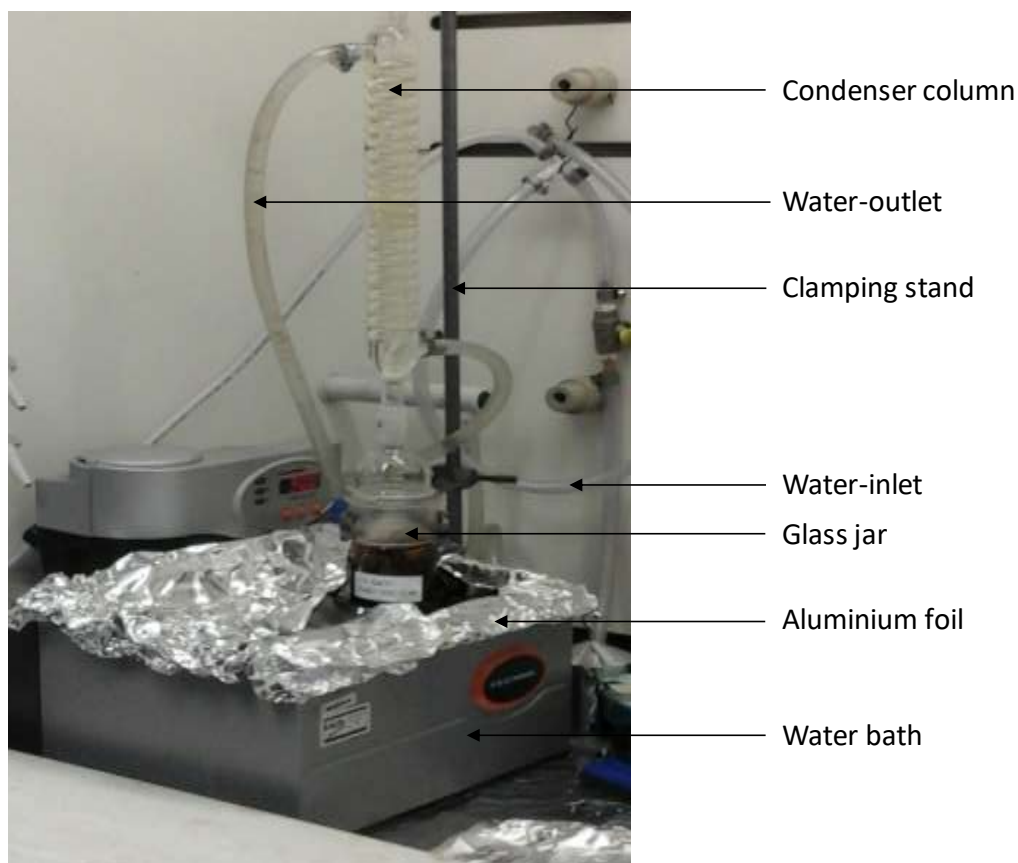


Figure 3.2 Photograph of the experimental setup and equipment used for the treatment of coir fibres with NaOH.

3.2.2 Treatment of coir fibres using 3-(aminopropyl) trimethoxysilane and 3-(glycidyloxypropyl) trimethoxy silane.

The experiments were carried out at different concentrations of silane. The treatment durations were varied. The complete set of experiments with nine experimental conditions is displayed in Table 3.4.

Table 3.4 Design of experiments for the treatment of coir fibre using epoxy and amino silanes.

Experiment order	Factors: Experimental Conditions	
	Concentration of silane (%)	Duration of Treatment (minutes)
1	0.5	15
2	0.5	30
3	0.5	60
4	1.0	30
5	1.0	60
6	1.0	15
7	1.5	60
8	1.5	15
9	1.5	30

The required quantity of deionised water was measured using a measuring cylinder and the corresponding quantity of ethanol was weighed out on a four-digit analytical balance. The ethanol and deionised water ratio was 4:1 respectively. The mixture of ethanol and water and 3-(aminopropyl) trimethoxysilane were labeled as “A” and “B” respectively.

The required amount of 3-(aminopropyl) trimethoxysilane (B) was added to the mixture of ethanol and deionized water (A). 2.5 g of selected fibres were submerged in this solution at room temperature and the specified time as stated in Table 4. The fibers were washed and dried in the air-circulating oven at 50 °C for 12 hours. The silane-treated coir fibres were stored in a moisture-free air-tight container until required.

The above-mentioned procedure was used on the coir fibres with 3-(glycidyloxypropyl) trimethoxy silane.

3.2.3 Hot-compaction of coir fibre

The compaction of coir fibres was carried out using Moore Max 25 Ton hydraulic press, UK. Several factors were involved (temperature, duration and pressure) and Minitab-19 Multilevel Factorial Design of Experiments was used as outlined in Table 3.5. Thirty five debris-free as-received coir fibres were selected and the diameters were measured at their mid-length positions. The minimum length of the selected fibres was 230 mm as shown in Figure 3.3. The end-sections of the fibres were secured on a metal plate with double-sided adhesive tape and the opposite ends of the fibres were fixed in position using an adhesive tape. The fibres were kept as straight as practically possible under low-tension and each fibre was numbered. The experimental set up is as shown in Figure 3.4. Up to 35 fibres were compacted per experiment. The individually numbered, aligned and secured samples were introduced to the Moore Max 25 Ton hydraulic hot-press (see Figure 3.5) with the platens barely touching the steel plates. The temperature was turned on and a thermocouple was used to ensure that the desired temperature had been reached. Once the desired temperature was reached, the required pressure was applied and maintained for the set time. The electrical power to the platens was turned off on reaching the set time and the pressure was maintained whilst the platens cooled to 40 °C. At this stage, the pressure was released and the steel plates removed using temperature-resistant gloves.

The above-mentioned procedure was carried out using as-received coir fibres and samples that have been treated previously in water at 80 °C for twenty four hours.

Table 3.5 Experimental design for hot compaction of coir fibres.

Run order	Factors (Minitab):Multi Factorial Design			Factors: Experimental Conditions		
	Pressure	Temperature	Duration	Pressure (MPa)	Temperature (°C)	Duration (minutes)
1	4	70	5	4	70	5
2	4	70	15	4	70	15
3	4	70	30	4	70	30
4	4	140	5	4	140	5
5	4	140	15	4	140	15
6	4	140	30	4	140	30
7	6	70	5	6	70	5
8	6	70	15	6	70	15
9	6	70	30	6	70	30
10	6	140	5	6	140	5
11	6	140	15	6	140	15
12	6	140	30	6	140	30

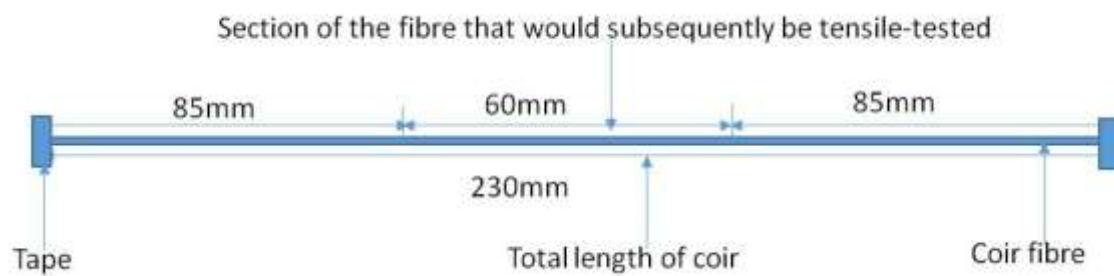


Figure 3.3 Dimensions of the coir sample for hot-compaction and the section of the coir fibre that was tensile tested.

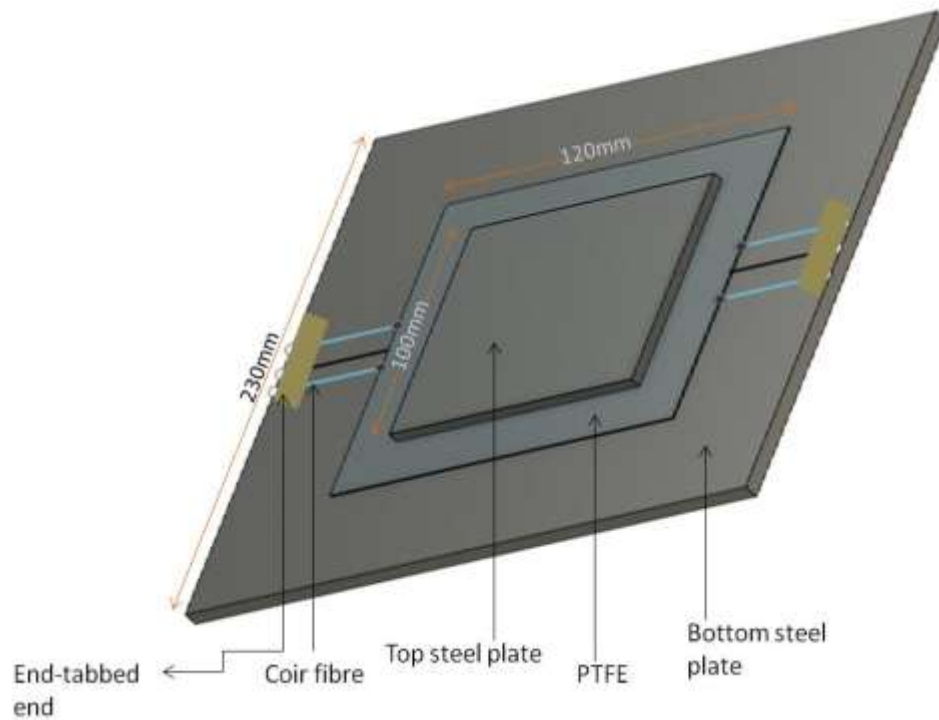


Figure 3.4 Experimental set-up for hot-compaction of coir fibre.

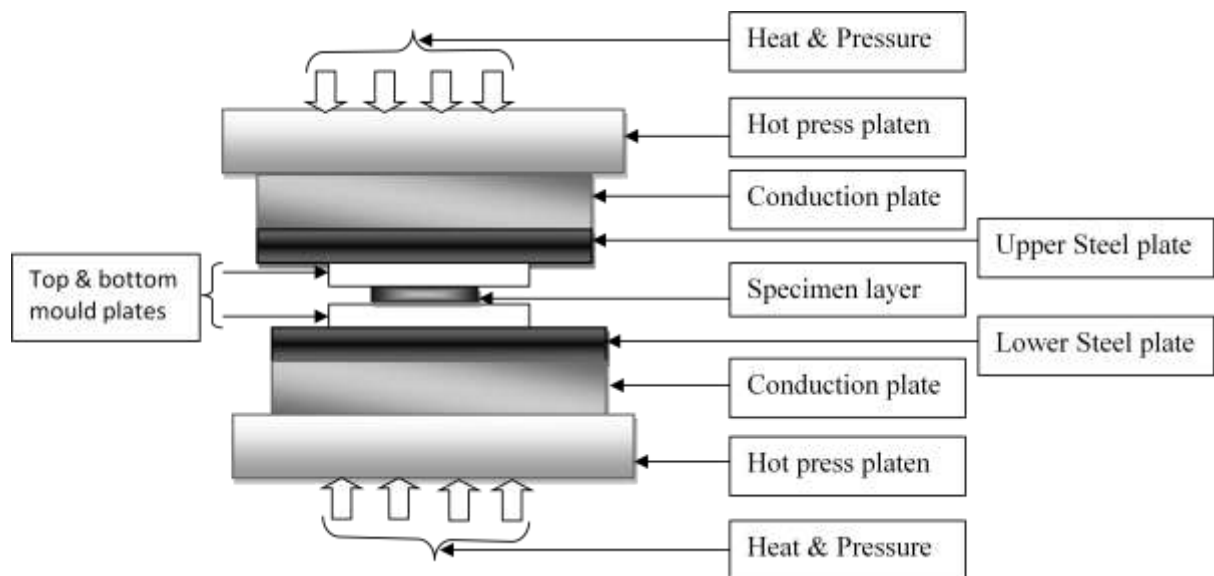


Figure 3.5 Schematic illustration of hot-press setup for hot-compaction of coir fibre.

3.2.4 Heat treatment of coir fibres in vacuum oven and in a tube furnace

Washed and dried coir fibres were heated in a Gallenkamp vacuum oven model OVL570, UK. The air intake nozzle was connected to a silica gel canister to minimize atmospheric

moisture entering the chamber when air was let into the oven to equalize the pressure. Thirty individual coir fibres per batch were subjected to heat treatment at 40, 60, 80, 120, 160, 180 and 200 °C for 6 hours. After this period, the power to the oven was turned off and it was permitted to cool naturally to room temperature, after which the samples were removed and stored in a moisture-free air-tight container until required. Figure 3.6 shows the experimental setup for heat-treating the coir fibres in the vacuum oven.

The above-mentioned experiments were also carried out in air using a 240 V tube furnace supplied by Severn Furnaces Ltd. UK with model number: TF5021ZF and serial number 911211. The furnace uses silicon carbide element, enabling temperature of up to 1200 °C to be achieved. The elements are located evenly around the hot zone to ensure an optimal level of temperature uniformity. The fibres were placed longitudinally in a glass tube and the glass tube placed in the tube furnace. The schematic representation of the tube furnace used in the heat-treatment of coir fibre is shown in Figure 3.7. Liquid nitrogen was used to trap the output gas from the output nozzle.

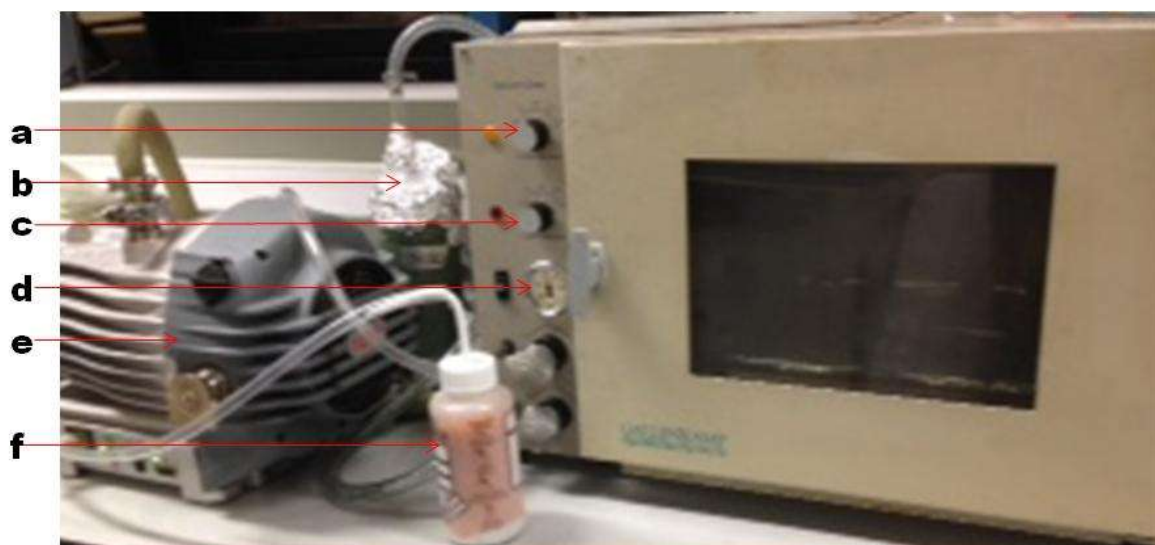


Figure 3.6 Photograph of the experimental setup for drying the coir fibres in a vacuum oven: (a) Control temperature knob (b) liquid nitrogen flask (c) safety temperature knob (d) vacuum gauge (f) silica gel canister.

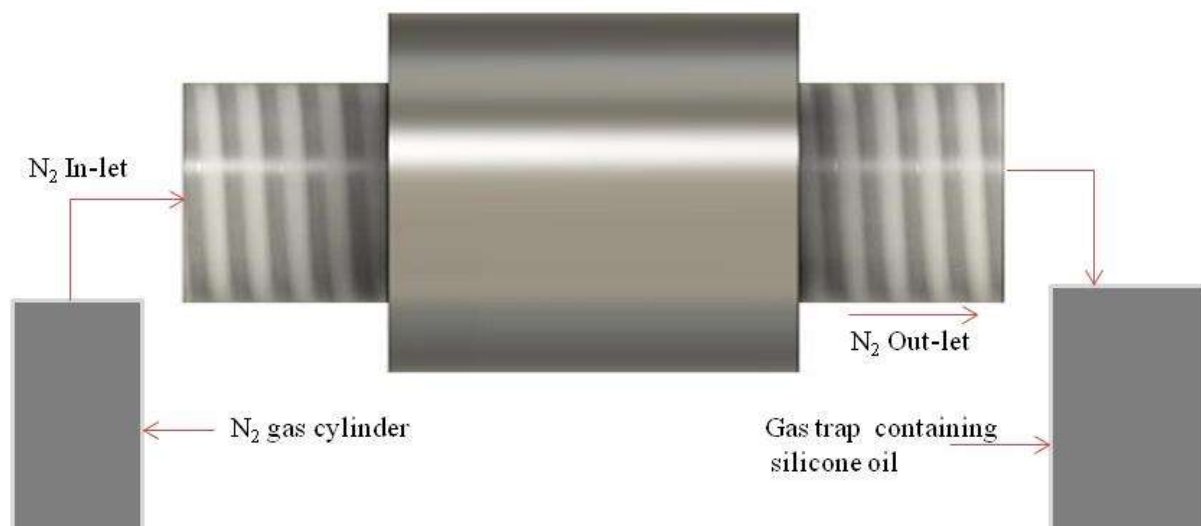


Figure 3.7 Schematic diagram of the tube furnace for the heat treatment of coir fibres.

3.3 Water absorption of coir fibres

Each glass vial of length 7 cm with known mass of pre-dried coir fibres was filled with distilled water and the immersion time of the fibre was noted. Six vials were placed in a 500 ml beaker and filled with distilled water. This assembly was placed in a temperature-regulated water bath that was set at 40 °C. Each vial was taken out and fibre was removed from the water-filled glass vial and dried with lint-free tissue. The fibre was weighed after it had attained room temperature and returned to the vial filled with distilled water and placed in the glass beaker and returned to the water bath.

The weight of the individual fibres was measured every 60 minutes between normal working hours. Tensile test was carried out on the samples when an equilibrium weight was achieved at 40 °C in water. These measurements were carried out over 5 days. These experiments were repeated at 50, 60 and 80 °C.

3.4 Production of unidirectional coir fibre arrays

Previously-washed and dried as-received coir fibres of diameter ranging between 0.21 to 0.22 mm about the middle and minimum length of 120 mm were selected. The production of a unidirectional aligned array of coir fibres was carried out as follows:

5 cm length of polyimide adhesive tape was attached on each end of a PTFE-coated aluminium substrate. The selected single fibres were manually and individually attached to the adhesives starting at one end of the substrate. Light tension was applied to each fibre and combed with a lint-remover cloth before it was secured on the adhesive tape.

Figure 3.8 (a-f) represents photograph showing the sequence of events, from the as-received fibre bundle to the production of the aligned coir fibre array.

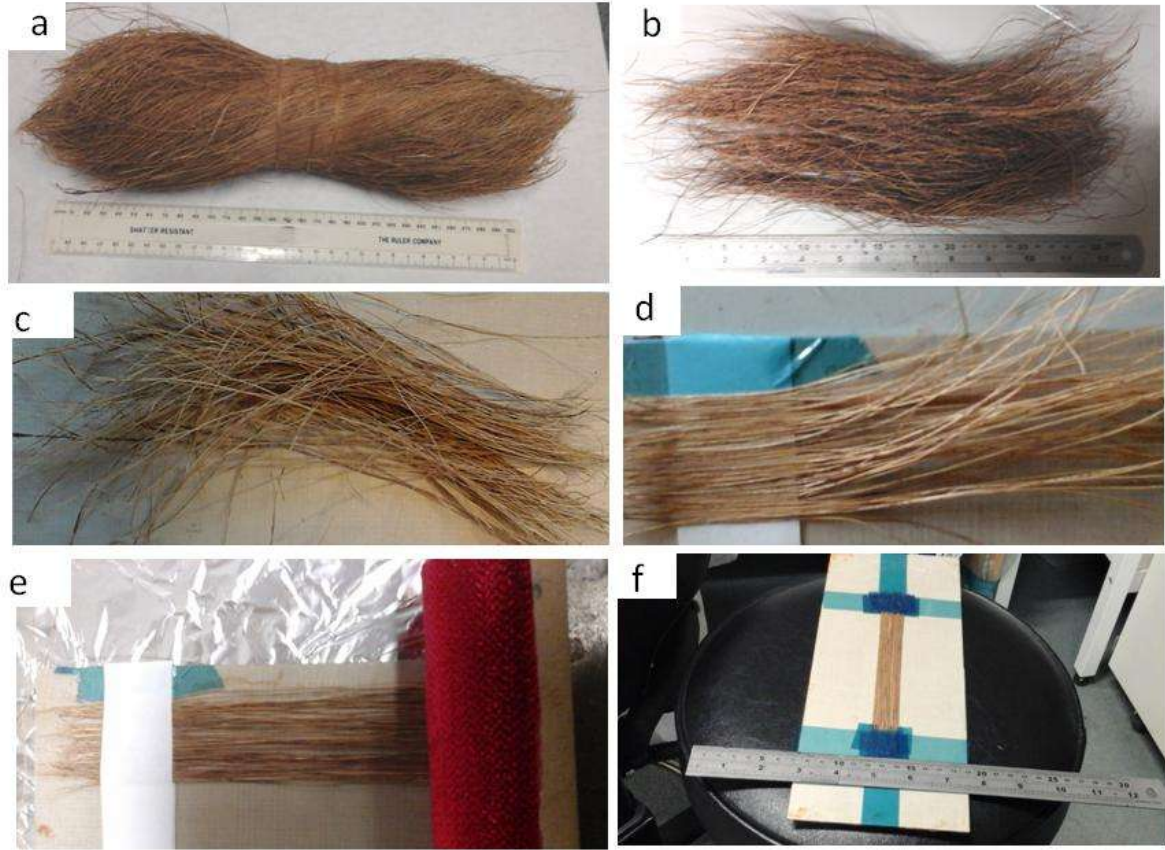


Figure 3.8 Stages of production of unidirectional coir fibre arrays.

(a) As-received coir fibre bundle. (b) Vacuum-cleaned and washed fibres. (c) Fibres dried at 80 °C for two hours. (d) Array of aligned fibres arranged manually and secured on the adhesive tape at one end.

(e) Combing of the fibres from the secured-end to the unsecured section with a cloth for removing lint from clothing. (f) An array of unidirectional coir fibres.

3.5 Spreading of E-glass fibre bundles

Fibre spreading was carried out in order to spread the fibre bundles to improve impregnation by the resin. As a result, it is important to note the position at which the fibres were obtained from the fibre creel to determine if the strength and stiffness of the spread fibre are a function of its relative position on the creel. Figure 3.9 shows a schematic diagram of the glass fibre creel and the positions from which the fibres were obtained. The height of the creel was segmented into top, middle and bottom represented by h_1 , h_2 and h_3 respectively. The radius

of the external layer, central layer and internal layer of the creel are represented by r_1 , r_2 and r_3 respectively. The symbol representations are as follows:

h_1 = top = 30% of the height of the glass fibre creel.

h_2 = middle= 60% of the height of the creel.

$h_3 = h_1$ =30% of the height of the glass fibre creel.

The radius of the external layer $=r_1= 110$ mm.

The radius of the central layer, r_2 , was from 50 mm of the external layer radius to 90 mm towards the internal radius of the creel. The radius of the internal layer, r_3 , was from 90 mm of the central layer to the internal radius of the creel.

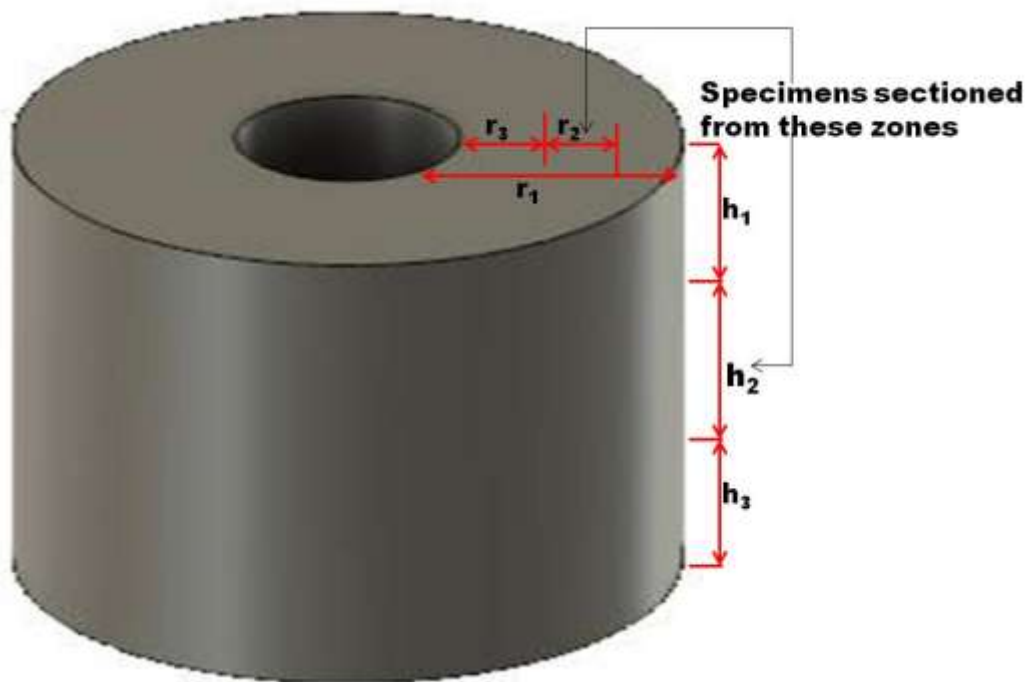


Figure 3.9 Schematic illustration of a creel of glass fibre bundle showing the relative positions on the creel and the positions where the test specimens were obtained.

3.5.1 Mechanically-induced fibre spreading

The spreading of as received E-glass fibre bundle and T700 Carbon fibre bundle were carried out using custom-built mechanical fibre spreading rig (Irfan *et al.*, 2012). The primary objectives of this mechanical spreading were:

- (i) To produce spread fibres without inducing fibre damage and
- (ii) To produce fibres with an optimum spread width without segmentation.

A schematic diagram of the spreading rig is shown in Figure 3.11. The as-received E-glass fibre bundle from the fibre payout creel through a pinch rollers were fed onto the pre-tension rollers which regulate the fibre payout by controlling the tension and thus minimising the risk of fibre damage. From the pre tension roller, the bundle makes way into the spreading rig that bears three roller carrier hubs with microfiber-coated acetal rods accommodating axial displacement as the rotation breaks up the binder on the bundle ensuring even spread of the fibre. The independent control of the three hubs was achieved by an individual 12-Volts DC direct-drive motor and gearbox within the control panel. At the end of the spreading rig is an exit roller. A photograph of the key components of the spreading rig is shown in Figure 3.10. The spread fibre bundle spooled on the cylindrical cardboard mandrel was unspooled and manually sectioned for tensile tests and preforms for manufacturing composites. Table 3.6 shows the parameters that were used for the fibre spreading set up. A photograph of the spread E-glass fibre is shown in Figure 3.7.

Table 3.6 Parameters used for operating the fibre spreading rig.

Operating parameter for the fibre spreading rig	Value
Pre-tension (N)	12
Winding speed (m/min)	1
Rotating hub speed (rpm)	100



Figure 3.10 Key components of the fibre spreading rig.

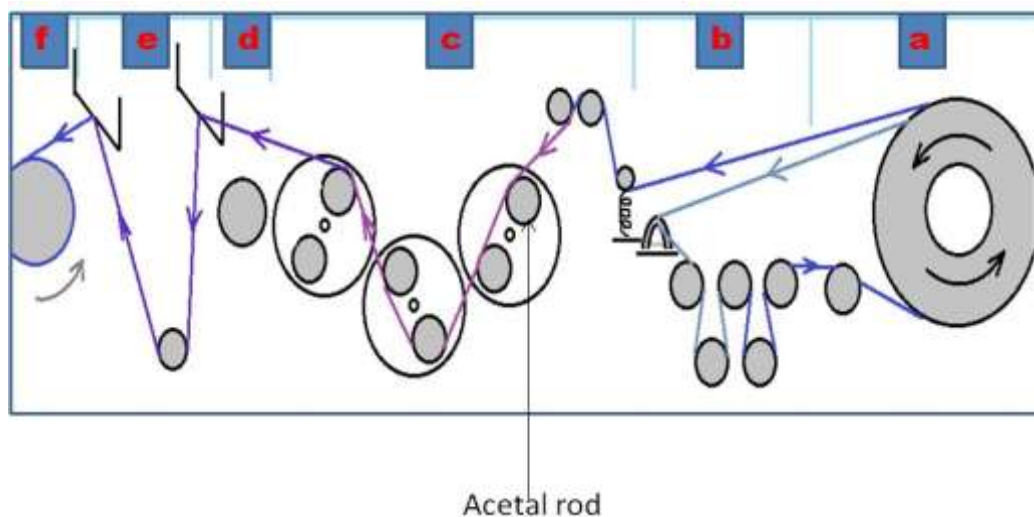


Figure 3.11 Schematic illustration of the fibre spreading rig used in this research

(a) Motorized (12V) fibre payout creel (b) 5 Pre-tension rollers coated with PTFE (c) 3 roller carrier hubs each housing 2 acetal rods with a micro-fibre coating (d) An exit roller (e) Tensioning setup (f) motorized haul-off unit.

3.5.2 Post-processing of spread E- glass fibre

Post-processing of the spread bundle was necessary to control the width of the spread fibres and to minimise the gap between the spread filaments prior to the production of composite. Hand layup and manual impregnation was used. The following section gives a description of the procedures used.

Aluminium plates of size 300 mm x 120 mm x 1.5 mm were sectioned from aluminium sheet. These plates were coated with PTFE adhesive-backed sheet. The pre-spread E-glass

was unspooled in uniform lengths of 250 mm from the mandrel directly onto the PTFE-coated aluminium plate. One end (A) of the fibre was secured onto the PTFE-coated-plate with adhesive tape while the opposite end (B) was left unsecured. This was the first stage (Stage 1) of the E-glass fibre processing. A photograph of the spread E-glass fibre using the fibre spreading rig is shown in Figure 3.12.

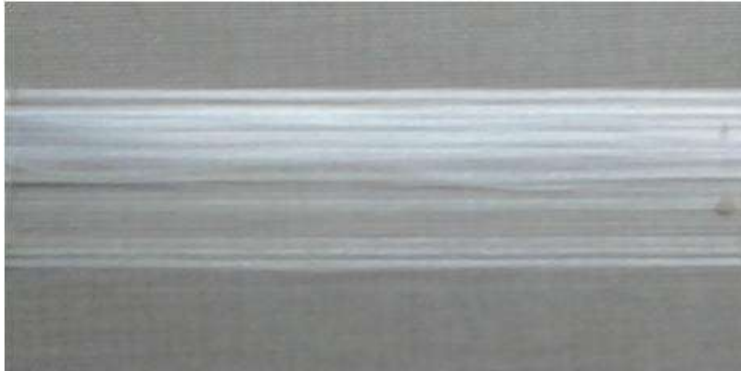


Figure 3.12 Photograph of a section of Spread E-glass using the fibre-spreading rig prior to post-processing.

Stage 2 involved the use of a micro fibre fabric-covered roller to manually brush the fibre along its length in one direction as gently as possible. A photograph of the micro fibre fabric is shown in Figure 3.13. Similar rollers can be seen on the motorized carrier hubs shown in Figure 3.10. The brushing of the fibre was carried out starting from the loose end (B), this was done in small lengths movement until the entire length was brushed. A flat-edged bamboo wooden spatula was deployed to even the spread. At the end of this processing by manual manipulation, the loose end of the fibre was much wider than the secured end. A schematic illustration of the processed fibre in stage two is shown in Figure 3.14.



Figure 3.13 A photograph of the longitudinal view of the micro-fibre fabric-covered roller.

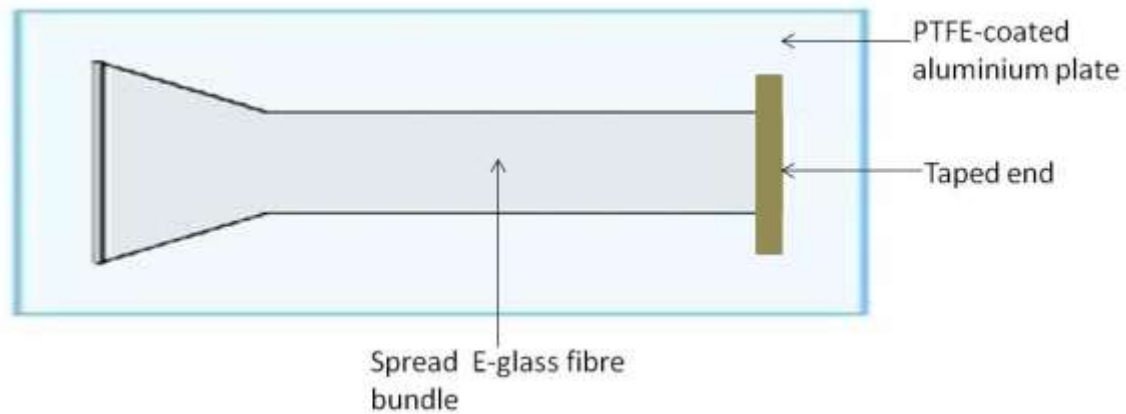


Figure 3.14 Schematic illustration of the E-glass post-processed spread fibre using the micro-fibre fabric-covered roller indicating a V-shaped end.

In the stage three, the wider end (B) of the spread fibre was secured using adhesive tape. The adhesive tape at the opposite end (A) was carefully detached leaving the filaments free. The micro fibre fabric was again used to brush the filaments at the thinner end which has been freed. The brushing continued until equilibrium was attained. The width of the bundle was uniformly spread along the length. Again a flat-edged bamboo spatula was used to flatten the fibres and this aided in closing any gaps between the filaments. The fibres on this end were securely taped to ensure no slips in the production of composites.

In stage four, end B was released, the fibre bundle was brushed using the micro-fibre fabric and adjusted using the wooden spatula for an acceptable finish, leaving the fibre width uniform along the length. A schematic of this final stage of post-processing is shown in Figure 3.15.

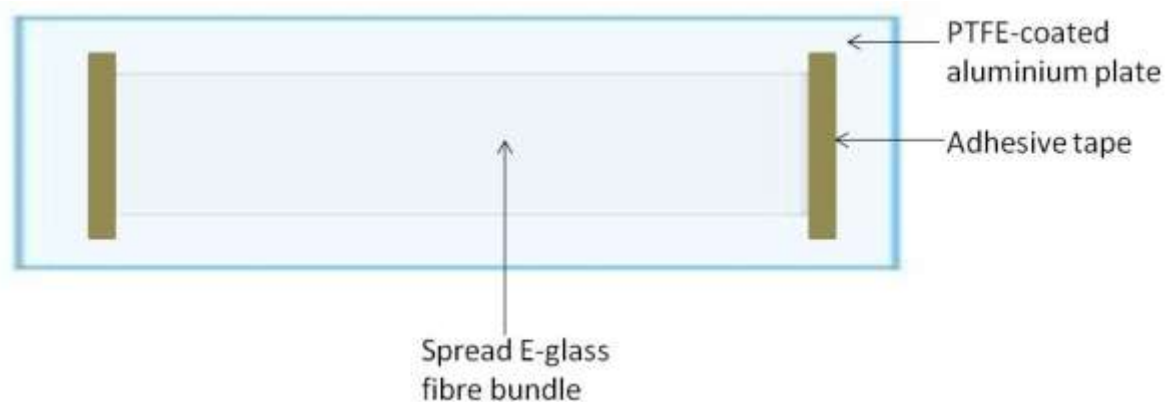


Figure 3.15 Schematic of the final thin section of post-processed E-glass fibre.

3.6 Single fibre tensile testing

Single fibre tensile tests were conducted on as-received coir fibres, to determine their tensile properties. Fibres were selected from the previously cleaned and washed fibre (Detailed in Section 3.2). The minimum sample size was thirty fibres per batch.

3.6.1 Preparation and end-tabbing of tensile test specimen.

With reference to Figure 3.16, as-received coir fibre tensile length was taken from the mid-section of the fibre length.

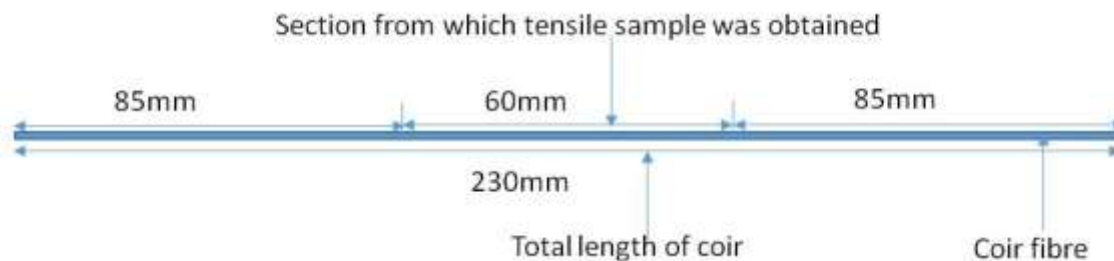
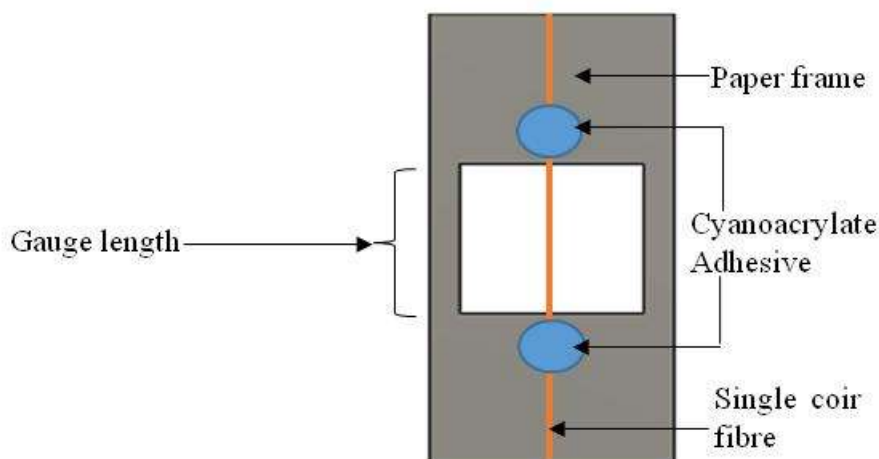


Figure 3.16 Schematic illustration of as-received coir fibre showing the section from which tensile sample was obtained.

The paper frame for mounting the tensile test sample in the Instron was prepared from a graph sheet. The graph paper was sectioned into frames of 60 mm x 40 mm to cover the test length. A slot of length equal to the gauge length was cut out in the middle of the frame. A test specimen of length 60 mm was placed in the middle of the slot in a longitudinal manner and secured by means of cyanoacrylate adhesive. A duplicate window frame was used to secure the fibre by placing it directly above the first window frame. The schematics of the end tabbing set up are shown in Figures 3.21 and 3.22.



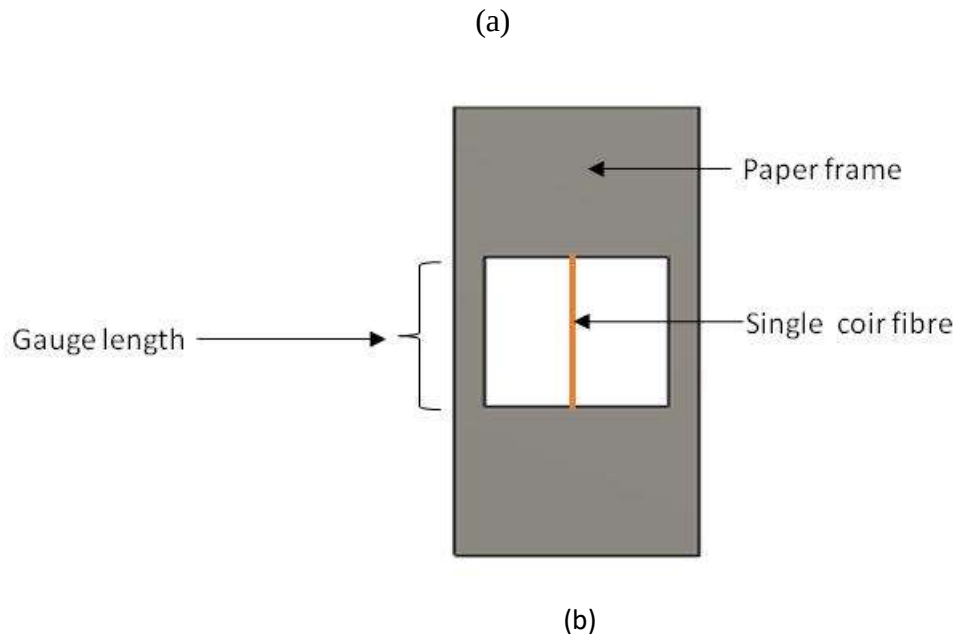


Figure 3.17 Schematic illustration of specimen preparation for the single fibre tensile test.

(a) Fibre held in place using cyanoacrylate adhesive and (b) second paper frame placed on the first to secure the fibre.

3.6.2 Tensile Testing of as-received coir fibre

Previously washed and dried as-received single coir fibres were tested on Instron 5566 tensile testing machine. A load cell of 100N and a cross-head displacement rate of 1mm/minute were used. Gauge lengths of 20, 60 and 100 mm were explored. The experiment was carried out at 24 °C and at a relative humidity of 50%. The tensile test set-up is shown in Figure 3.18.

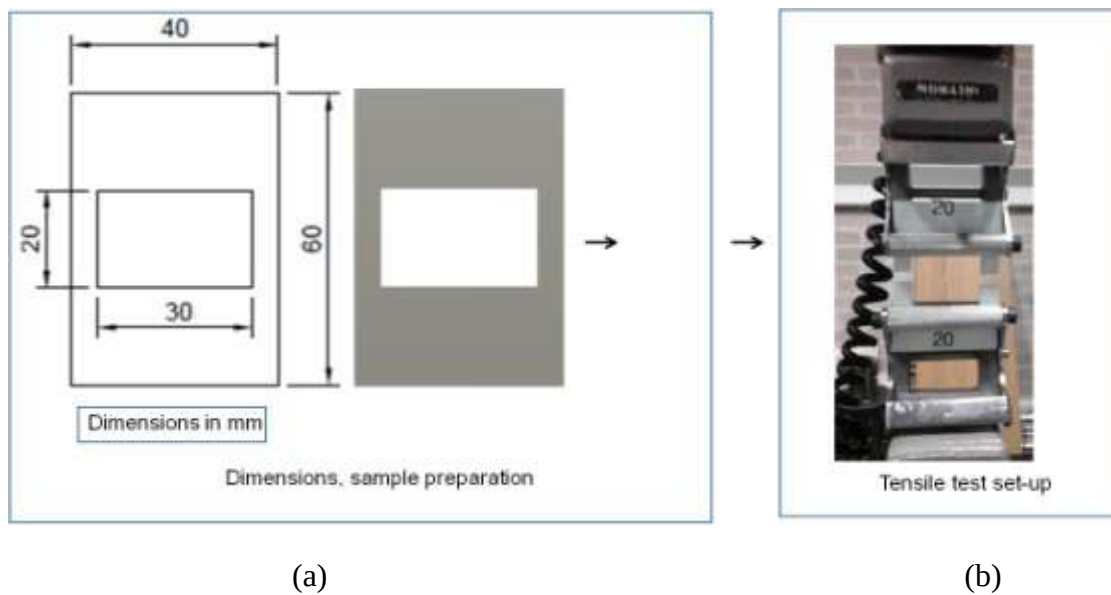


Figure 3.18 Experimental set up for tensile testing of coir fibres.

(a) Dimensions (b) tensile testing using Instron 5566.

3.6.3 Tensile testing of spread E-glass and T700 carbon fibres

Sections were taken directly from the mandrel and end-tabbed with the dimensions given as in Figure 3.19. The tensile tests were carried out on the spread E-glass and spread T700 carbon fibres using Instron 5566 at a crosshead speed of 1mm/minute and a gauge length of 100 mm. The load-cell capacity was 100 N.

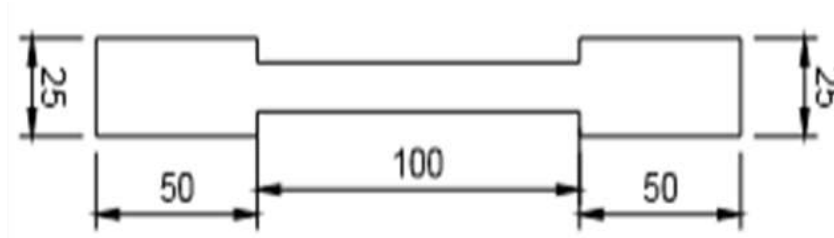


Figure 3.19 Dimensions of the tensile test specimen for spread E-glass fibre and T700 Carbon fibre.

3.7 Manufacture of composites

Composites samples were manufactured using as-received coir fibre and spread E-glass fibre. The composites manufactured include monofibre and hybrid composites. The manufacture of the composites was carried out using the epoxy resin system: LY3505 and XB3403 mixed according to the stoichiometric ratio described in Section 3.1.2. The samples were manufactured using manual impregnation.

3.7.1 Manufacture of coir fibre composites using hand-lay-up

Aligned coir fibre arrays produced in Section 3.4 were used to manufacture coir composites. Epoxy LY3505 and hardener XB 3403 were weighed using the specified stoichiometric ratio as discussed in Section 3.1.3 and mixed manually. Degassing of the resin system was achieved by placing the mixed resin system in a vacuum desiccator for 30 minutes. A mould release agent (PAT-607PCM supplier, UK.) was applied on a rectangular aluminum plate coated with Polytetrafluoroethylene (PTFE) film. The prepared array was secured at two shorter opposite ends of this plate. Droplets of the mixed resin were dispensed onto the surface of the coir fibre array using thermoplastic pipette. Thirteen rows and three columns of the droplets were introduced to the coir surface and impregnation achieved in the transverse, length-wise, and through-thickness directions of the coir array. It was left for about three minutes to allow further permeation of the resin into the array. A polyethylene film was placed on the wetted sample. The resin was gently press-pushed over the plastic film using wooden spatula down to the opposite end of the coir fibre array only in one direction ensuring complete removal of any trapped bubbles.

For the two and three-layer composites, the same process was used but however, as soon as the manual manipulation of the resin had been achieved, the second unimpregnated layer was placed on the first impregnated layer in the same direction and the application of resin was carried out in single layer. The resin was applied layer by layer. Finally, a small amount of the impregnating resin was dropped on the last surface and a layer of PE placed on top and the excess resin spread out. A rectangular plate and a weight of 1kg were placed on the yet-to-cure composite. A schematic illustration of the setup is displayed in Figure 3.20. The composite was left to cure at room temperature for 24 hours after which the PE was removed and post curing was carried out in air-circulating oven at 80 °C for 6 hours. The samples were trimmed to the desired dimensions.

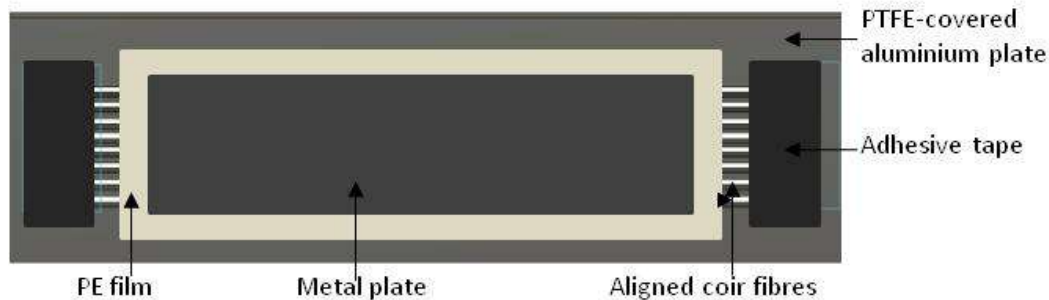


Figure 3.20 Schematic illustration of hand-layup used in composite manufacture.

3.7.2 Manufacture of Spread E-glass fibre composites

Spread E-glass fibre bundles were post-processed as previously described in Section 3.5.2. On the completion of the post-processing, cyanoacrylate adhesive was smeared on thin paper strips used to secure the two opposite ends of the fibre.

This was important to maintain the desired architecture and reduce the spatial in the orientation of the filament to the barest minimum prior to impregnation. It is left for two minutes for the fibres to be completely bonded to the adhesive. The same procedure was also carried out on the opposite sides. The post-processed fibres were prepared for impregnation. A mould release agent (PAT-607/PCM) was applied on a PTFE-covered aluminium plate (specification as in that of coir fibre) prior to use. The processed fibres were placed on this plate and impregnation was carried out manually. The resin and hardener were weighed,

mixed and degassed as previously described in Section 3.7.1 and complete impregnation was carried out as described in the same section.

3.7.3 Manufacture of coir/E-glass hybrid composites

For hybrids non-mono fibre composite manufacture, the glass fibres were impregnated first as discussed above and placed onto the manually impregnated coir fibre. After the last layer, a PE film was applied over the surface of the impregnated composites. A pressure in the form of a metal plate with an additional weight of 100 kg was applied and the entire setup left to cure at room temperature after which the PE film was removed. Post-curing was carried out at 80 °C for 6 hours. This procedure was employed to produce Coir-Glass-Coir, Glass-Coir-Glass and Glass-Glass-Coir-Coir-Glass-Glass composites.

3.7.4 Manufacture of coir composites using vacuum bagging

The epoxy resin system was mixed in the stoichiometric ratio previously specified. An aluminium PTFE-coated rectangular tooling plate of dimension 300 mm x 120 mm x 1.5 mm was cleaned with soap and water and left to dry. On drying, it was cleaned with acetone several times and left for 5 minutes to dry. PAT-656/B3R mould release agent was applied on the surface of the plate using polyester fabric and left for 10 minutes for the coating to dry. This was repeated for four times for enhanced demoulding after composite manufacture. The surface of the plate was taped round using butyl double-sided sealant adhesive tape (supplied by Easy Composites Ltd, UK) in a rectangular manner as shown in Figure 3.21. The coir fibre arrays were secured individually on the enclosed region at two ends at intervals of about 30 mm as shown in Figure 3.21b. For the production of double layer, the second layer was placed on top of the first and secured as in the first layer. The same process was applied for manufacturing three layers. The two ends of the attached arrays were covered with breather fabrics. The breather fabrics for the first end was cut in smaller rectangular shapes enough to cover each end of the array, these were dipped in the impregnating resin and placed on the ends and acted as the resin-inlet. The opposite end was covered with single length of two breather fabrics to absorb excess resin. The setup was subsequently covered with a vacuum bag (R250 and supplied by Easy Composites Ltd, UK) by attaching on the butyl double-sided sealant tape ensuring no leakage while creating a penetrating point for the vacuum pipe to be attached. After ensuring no leakage, a vacuum pressure of 10 mbar was applied for 20 minutes. The setup was left to cure at room temperature for 24 hours. Post-curing was

achieved at 80 °C for 6 hours. The initial and complete experimental setups are shown in Figures 3.21 and 3.22 respectively.

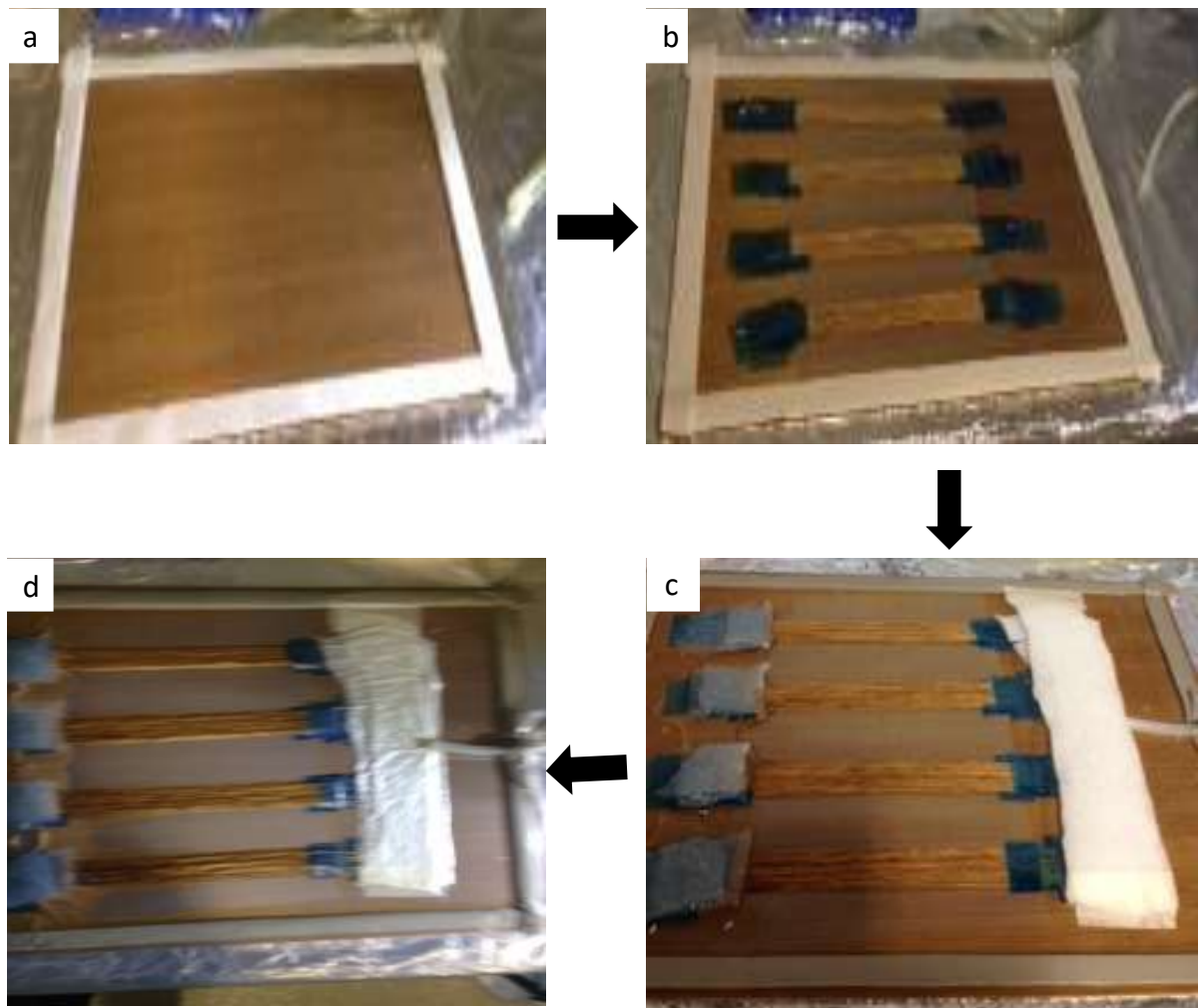


Figure 3.21 Initial experimental set up for composite manufacture using vacuum pump. (a) Taping of the PTFE-coated aluminium tool plate (b) The arrays secured on the plate (c) the two opposite ends of the arrays covered with breather fabric (d) The vacuum bag taped on the adhesive and impregnation carried out via vacuum pressure.

The experimental procedures that were developed and used in this section were progressed with assistance from Mr Tao Ma (PhD researcher, School of Metallurgy and Materials, University of Birmingham).

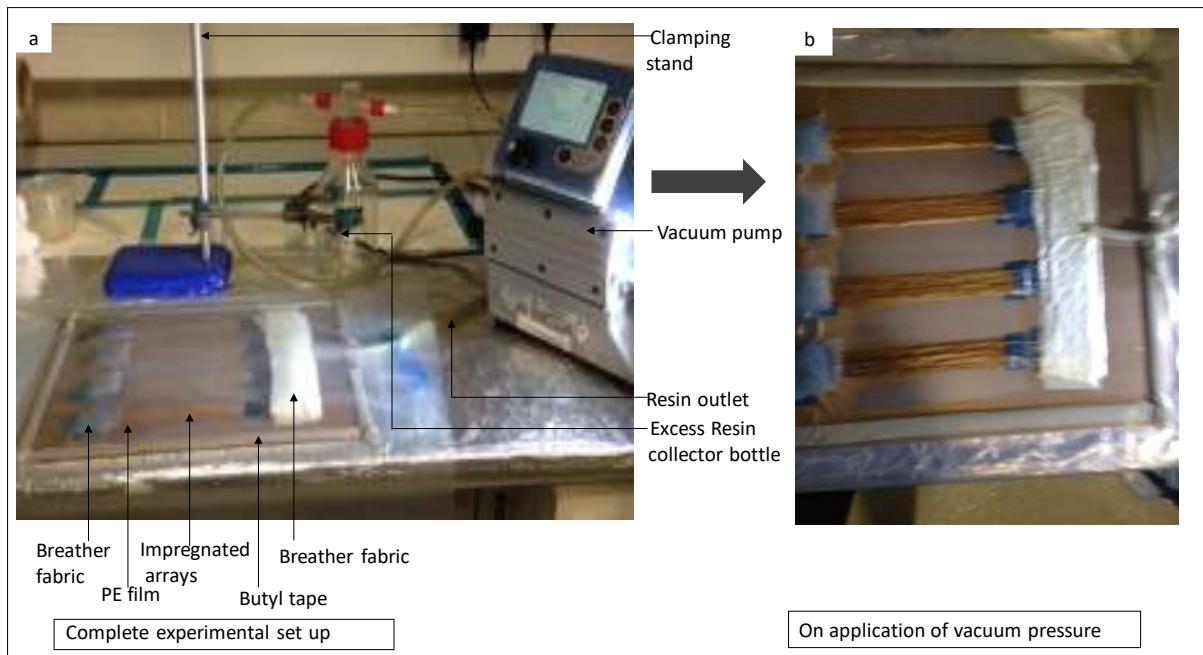


Figure 3.22 Complete experimental set up for composites manufacture using vacuum pump.

The schematics of the composites manufactured in this study for testing is shown in Figure 3.23

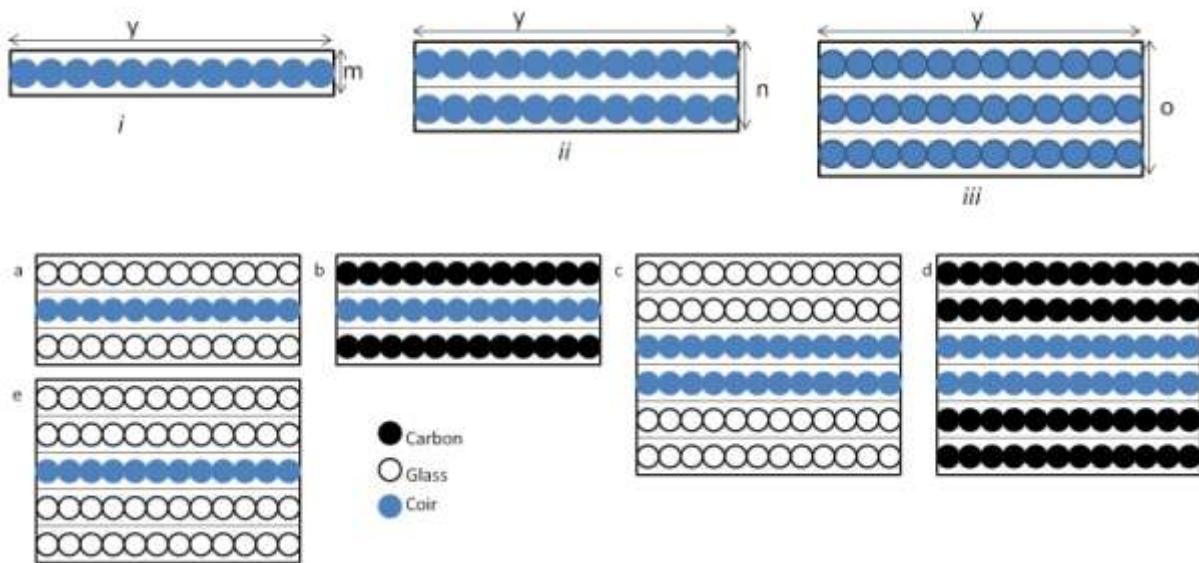


Figure 3.23 Schematics of the composites manufactured for tensile testing. (i) Single layer coir monofibre (ii) double-layer coir monofibre (iii) Triple-layer coir monofibre (a) Glass-Coir-Glass (b) Carbon-Coir-Carbon (c) 2Glass-2Coir-2Glass (d) 2Carbon-2Coir-2Carbon (e) 2Glass-Coir-2Glass.

3.7.5 Tensile testing of composites

In order to determine the tensile properties of the manufactured composites, tensile tests were carried out. The tensile tests were performed in accordance with BS-EN-ISO527-5:2009 on an Instron 5566 at a cross-head speed of 1mm/minute. The composites samples were end-tabbed using sand-blasted aluminium end tabs. The width and thickness of the composites were measured at three different positions using digital Vernier callipers and micrometer gauge respectively. In order to measure the standard applied strain, an extensometer was utilized.

3.8 Optical microscopy

Optical microscopy was carried out on the as-received, chemically-treated hot compacted coir fibres and coir fibre composites including their tensile fracture surfaces in order to determine their microstructures and failure modes.

Samples for optical microscopy were prepared according to standard metallographic methods for potting and polishing to achieve flatness and reproducibility at the shortest possible time for good quality images, crisp enough for volume fraction. Zeiss Axioskop-2 optical microscope was used to obtain the images. At this point, ImageJ software was introduced. By navigating through the extensive list of ImageJ commands, several functions such as area fractions and pixel value statistics of selections defined by the user can be performed. ImageJ was carried out on different gray levels such as 8-bit, 16-bit or 32-bit, providing 3D visualization to images when the images are stacked. ImageJ image analysis software was used to analyse the images in order to obtain the intrinsic porosity for as-received and treated coir fibres. The percentage porosity was computed using the software. A total of 100 images for as-received coir fibre were analysed and recorded. ImageJ image analysis software was also used to determine the fibre volume fraction of the coir monofibre and hybrid composites.

3.9 Scanning Electron Microscopy

Several approaches were carried out in preparing coir fibre samples for scanning electron microscopy (SEM) in order to ensure that no damage was done on the microstructure through sample preparation. The approaches include:

- (i) Drying of coir fibres at 80 °C for 6 hours in a vacuum oven and at 80 °C for 6 hours in air oven before sectioning with a fresh blade for microscopy examination.

(ii) Immersion of fibres into liquid Nitrogen and sectioning it with fresh surgical blade at an angle of 90 °C whilst inside the liquid nitrogen.

(iii) Immersion of as received coir fibres in liquid nitrogen for four minutes and fracturing the fibre using a pair of pliers when at low temperature.

The sectioned samples of length 10mm were mounted on the sample holder with the aid of carbon tapes. The sample surface was sputter-coated with thin layers of Au/Pd alloy in the coating chamber using 0.1 Torr vacuum and at a current of 25 mA for three minutes to improve image resolution. The morphology was characterized using Joel 6060 and Hitachi S4700 Table top SEM 3030 scanning electron microscope.

3.10 Thermogravimetric analysis

The thermogravimetric analysis was carried out on as-received and treated coir fibres. The TGA was studied using a Netzsch instrument for thermogravimetry under an argon atmosphere. The weight of the sample was 10 ± 2 mg at a temperature range of 25 and 1000 °C and a heating rate of 10° C/min. The TGA analysis software was used to obtain the derivative of TGA curves (DTG).

3.11 Differential Scanning Calorimetry

Differential Scanning Calorimetry (DSC) (Perkin Elmer DSC 7, UK) was carried out on the epoxy matrix. The epoxy samples were sectioned into 5 mm lengths and introduced into DSC pan. This was crimped using the Perkin Elmer press set up. The pressed samples were introduced into the sample chamber of Perkin Elmer DSC 7 with the software recording the heat flow against temperature.

4 Results and Discussions

4.1 Introduction

This section commences with an insight into the extent of variability in the diameters of coir fibres. It gives a summary of the results obtained from single-fibre tensile testing at specified gauge lengths and cross-head displacement rates as a function of specified thermal and chemical treatments. The mechanical properties of single-coir fibre after flat-wise compaction are also presented. The findings on the effects of hybridised coir/E-glass fibre composites are reported. A detailed discussion is presented on the microstructure of coir fibre in relation to its properties.

4.2 Variability in the diameter of coir fibre

Figure 4.1 shows the variability in the diameter along the length of randomly selected coir fibres. The measurement points cover 100 mm at 10 mm intervals as indicated in Figure 4.1. For each sample, the diameters were labelled a-j starting from one end of the fibre to the opposite end. The average diameter for the dataset presented in Figure 4.1 is 0.25 ± 0.03 mm. However it is seen that fibre number 4 has a large fibre diameter of 0.31 ± 0.05 mm and fibre number 8 has a small average diameter of 0.21 ± 0.03 mm. It is difficult to discern any specific trend to the diameter distributions along the length of the ten fibres. This is not surprising as a number of factors can affect the fibre diameter at each of the measurement points (G Fernando, University of Birmingham; Personal communication) as discussed in further in this section.

(i) Natural fibres are notorious for the variability in the fibre length distribution (Satyanarayana *et al.*, 1986; Mathura and Cree, 2016). In the current study, 100 fibres were selected at random from a bundle and their lengths were measured. The as-received fibre lengths varied from 80 mm to 320 mm. It was not possible to conclude if the length of the fibre has any correlation as a function around the circumference of the coconut and the thickness of the mesocarp (G Fernando, University of Birmingham; Personal communication). This is because the spatial positions of the fibres are not maintained during the de-husking and removal of the fibres (G Fernando, University of Birmingham; Personal communication); this is discussed in Section 4.2.1. In the current study, the fibres from the as-supplied 3-tie bundles (See Figure 2.1) were selected manually. However, as will be

discussed in Section 4.2.1, a selection method was used for selecting the fibres for the various experiments that were undertaken.

(ii) Due consideration needs to be given to the macrostructure of the coconut and the mode of extraction of the fibres, their packaging at the factory and the cleaning and drying operations in the laboratory. This is discussed in the following section.

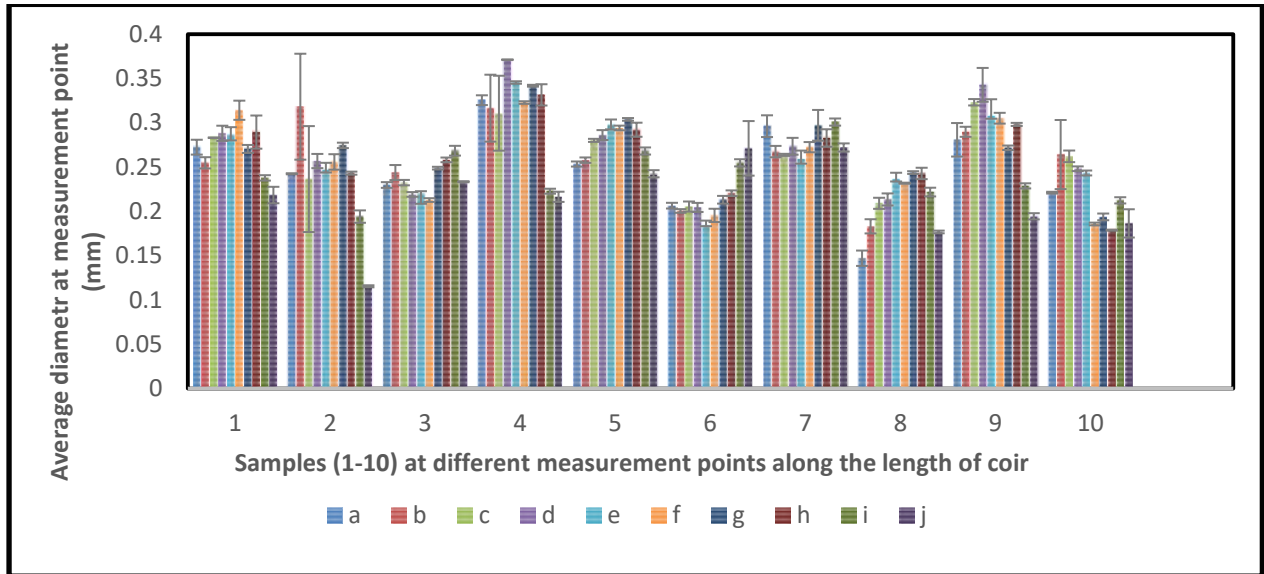


Figure 4.1 Variability in the coir fibre diameter: a-j represents 1cm mark each along the fibre length.

The diameters of these fibres were measured using micrometer. The large scatters in fibres 2, 3 and 10 can be attributed to the presence of debris or as a result of the residual components on the fibres as shown in Figures 4.2 and 4.3. The significance of having the debris on the surface when the diameter is measured is shown in Figure 4.4 where the average diameter of the fibres is 0.46 ± 0.09 mm with significant scatter in the diameters.

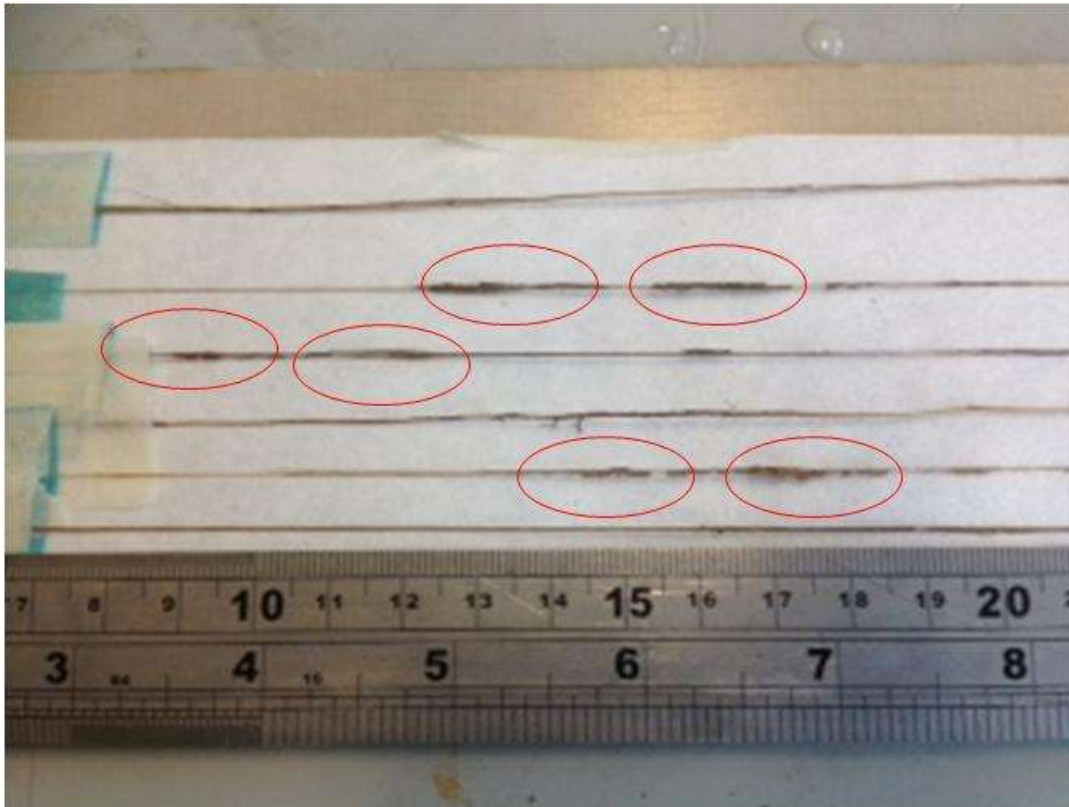


Figure 4.2 A photograph of coir fibres before washing showing debris-rich regions.

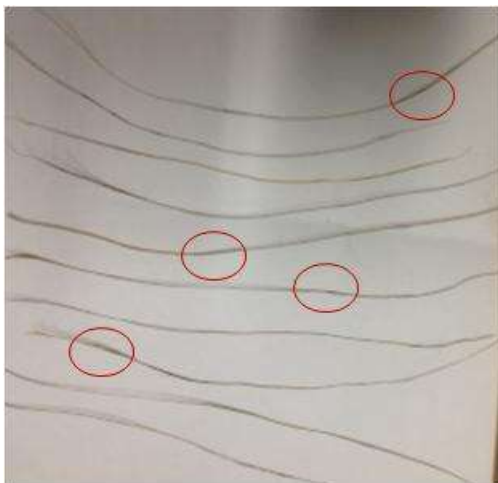


Figure 4.3 Coir fibres after washing showing traces of residual components.

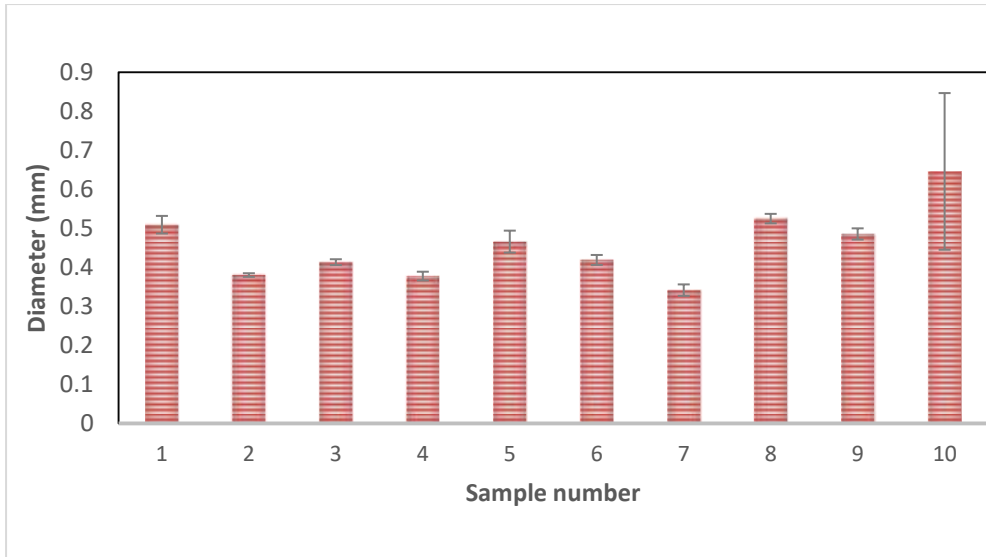


Figure 4.4 Histogram showing the diameters of coir fibres with debris.

4.2.1 Possible reasons for the observed variability in the fibre diameter

A photograph of a bunch of coconuts growing from a tree is shown in Figure 4.5.



Figure 4.5 A photograph showing coconut tree (Courtesy: C Madueke, PhD researcher, University of Birmingham).

With reference to Figure 4.5, it is necessary to establish if there is a distribution in the length of the fibres in the mesocarp when traversing from the surface of the endocarp to the exocarp of the coconut. Moreover it is necessary to establish if any correlation exists between specified points along the length of the coir fibre and the diameter. However, this is not straightforward to establish because of the various procedures that are used in the separation of the fibres from the mesocarp. In other words, the original position of the fibers is not maintained. A further explanation for this statement is given below.

After the coconut is harvested from the tree, the seed, nut or endocarp is separated from the mesocarp. This is generally achieved manually by droving the coconut into a spike and bending it; three to four such operations are carried out to extract the nut. The nut contains the endosperm and depending on the maturity, the copra.

The mesocarp with the thin exocarp layer can be stored for some time before it is sent to the coconut mill where it is kept until required; this is shown in Figure 4.6. From here on, the term “husk” will be used to describe the mesocarp and the exocarp. The husk is transferred to a water pool where they are immersed for up to three to four weeks (see Figure 4.7).



Figure 4. 6 A Photograph of coconut husks before they were sent to the coconut mill (Courtesy of G. Fernando, University of Birmingham).



Figure 4.7 A Photograph of coconut husks in the waterpool (Courtesy of G. Fernando, University of Birmingham).

A range of machines are used to separate the coir fibres that are contained in the husk, in some equipment designs, the operator clutches a segment of the husk and forces it between the rotating drum with the spikes and a counter rotating bar. The rotating spikes effectively shear the fibres apart. A photograph of the drum with spikes is shown in Figure 4.8. The husk

is then turned by 180° and the intact segment is subjected to the same procedure. This process can be repeated more than once depending on the decision of the operator on extent of perceived separation of the fibres. The term “defiberising” is used to describe this process. In some of the newer equipment designs the husk are placed on a rotating carrier that feeds a rotating drum with spikes.



Figure 4.8 A photograph of the drum with spikes used to separate coir fibres from the husk (Courtesy of G. Fernando, University of Birmingham).

The appearance of the fibers emanating from the semi-automatic de-fiberising machine is shown in Figure 4.9. In some instances, the de-fiberised husk is dried in natural sunlight to remove moisture (see Figure 4.10) and though this depends on the practices carried out by a particular mill.



Figure 4.9 A Photograph of coir fibers from the husk (Courtesy of G. Fernando, University of Birmingham).



Figure 4.10 A photograph of the drying of de-husked fibers (Courtesy of G. Fernando, University of Birmingham).

With reference to the coir fibres that were used in the current study, whilst the possibility of fibres from different husks mixing is low, it is possible. This is increased at the next stage of processing the coir fibres where they are combed manually on a comb consisting of vertical metal spikes as shown in Figure 4.11. In this process, two handfuls of the coir fibre bundles are combed repeatedly through the metal spikes. As with the manual defiberising process, here half the length is combed first. The number of combing actions carried out is determined by the operator and it is based on experience and the visual appearance of the coir fiber.



Figure 4.11 photograph of coir fibres showing the combing of coir fibres after dehusking (Courtesy of G. Fernando, University of Birmingham).

The photographs presented in Figures 4.5-4.11 raise a number of issues with regard to the variability in the physical and mechanical properties of coir fibres. Figure 4.5 showed a bunch of coconut on the tree. When the coconut is harvested, it is transported to the mill where the endocarp is extracted from the mesocarp. The issue here is that there is a

possibility that coconuts from the same plantation or different plantations can be mixed. The same applies to the husks shown in Figures 4.6 and 4.7. The issue needs to be considered because the age of the husks and their maturity may be different. Furthermore, the time the husks spend in sunlight prior to be transferred to the water pool and the duration they spend immersed will vary. Hence the degree of microbial degradation of the hemicellulose and the moisture content may be different. This in turn could affect the efficiency of the de-fiberising process. Residual hemi-cellulose and cellulose-based particulates on the surface of the coir fibres can have a significant influence on giving erroneous results on the diameter of the fibres. Therefore, if due care and attention is not given to the washing, drying and inspection of the as-received 3-tie coir fibre bundles, significant variability in diameter will be observed as was the case in the current study.

As a result of the variability in the diameters of the coir fibres presented in Figure 4.1, ten coir fibres of similar lengths were selected and zoned into A, B and C, each zone is 1/3 of the entire length as shown in Figure 4.12. In each zone, the diameter of the coir fibre was taken in three defined positions. Individual measurements were taken in each zone. Coir fibre diameters were found to be most uniform towards the mid-section (zone B) as shown in Figure 4.13.

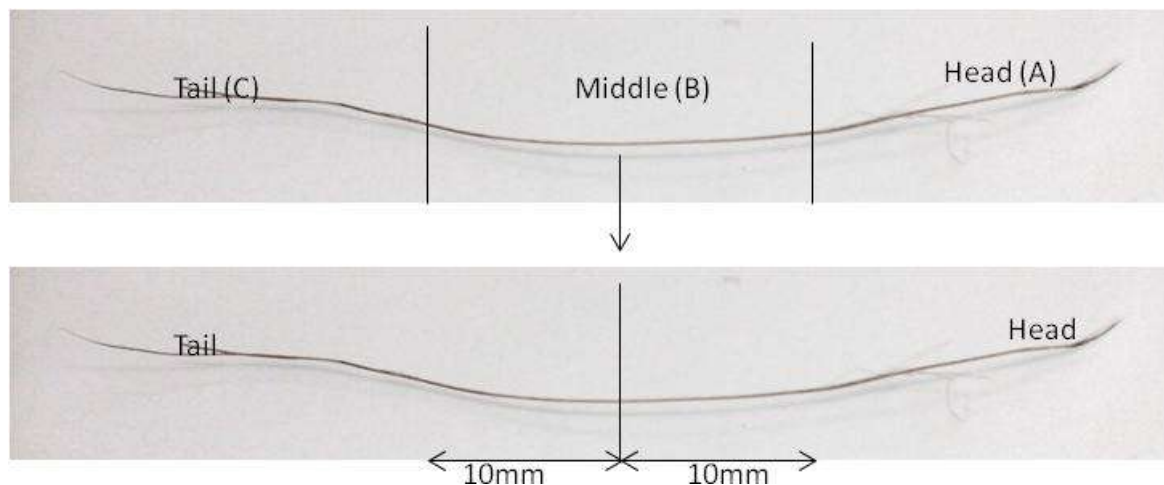


Figure 4.12 Photograph showing the zoning of coir fibre to determine the diameter distribution.

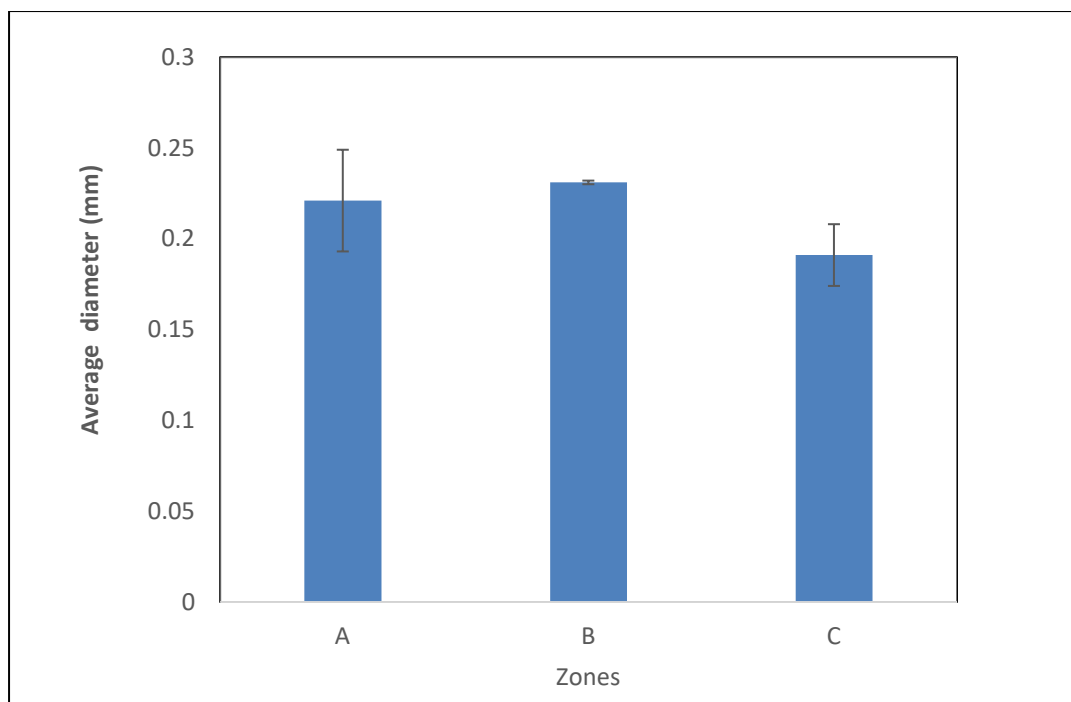


Figure 4.13 Average diameters of coir fibres using the zones defined in Figure 4.12.

It is interesting to note that the scatter in the datasets in Figure 4.13 is larger in zones A and C. The actual reason for this is not known but it may be due to the de-fiberising process. When the husk is fed into the rotating drum with the short spikes, it was observed that the centre section of the husk spends a greater time being sheared. However, the possibility of the hemi-cellulose and other cellulose derivatives being more densely packed at the ends needs further investigation.

4.3 Tensile properties of as-received coir fibres

The tensile properties of the as-received coir fibres were studied as functions of the gauge lengths and the cross-head displacement rates. The tensile strengths of thirty individual as-received coir fibres per batch at different gauge lengths and cross-head displacement rates were determined.

4.3.1 Effects of the fibre diameter on the tensile properties of as-received coir fibres

Several reports exist in the literature as to whether the tensile properties of natural fibres are affected by the diameter of the fibres. Tomczak, Sydenstricker and Satyanarayana, (2007), Defoirdt *et al.*, (2010) and Mathura and Cree (2016) observed a significant increase in strength with reduction in the fibre diameter.

In this study, stress-strain curves were obtained from coir fibres with diameters ranging between 0.03 to 0.45 mm at a gauge length of 20 mm and a cross-head displacement rate of 1mm/minute. The values for the Young's modulus were taken from the initial linear part of the stress-strain curve. Figures 4.14, 4.15 and 4.16 show the strength, stiffness and elongation as functions of the fibre diameter.

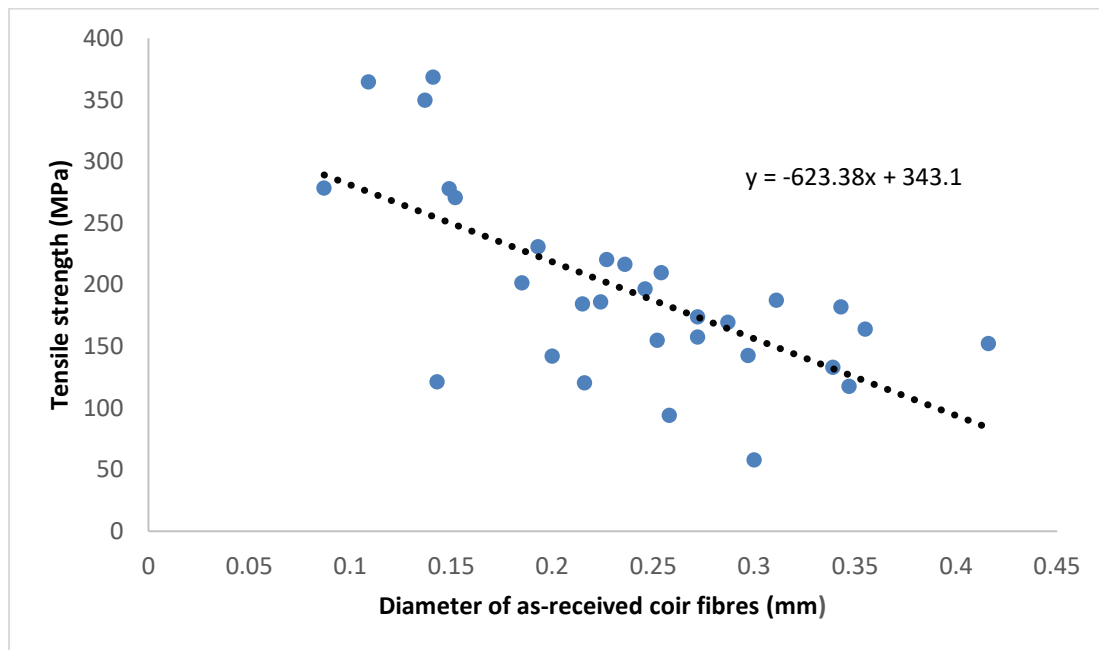


Figure 4.14 Effects of fibre diameter on the tensile strength of as-received coir fibres. The dotted line represents the linear regression for the dataset.

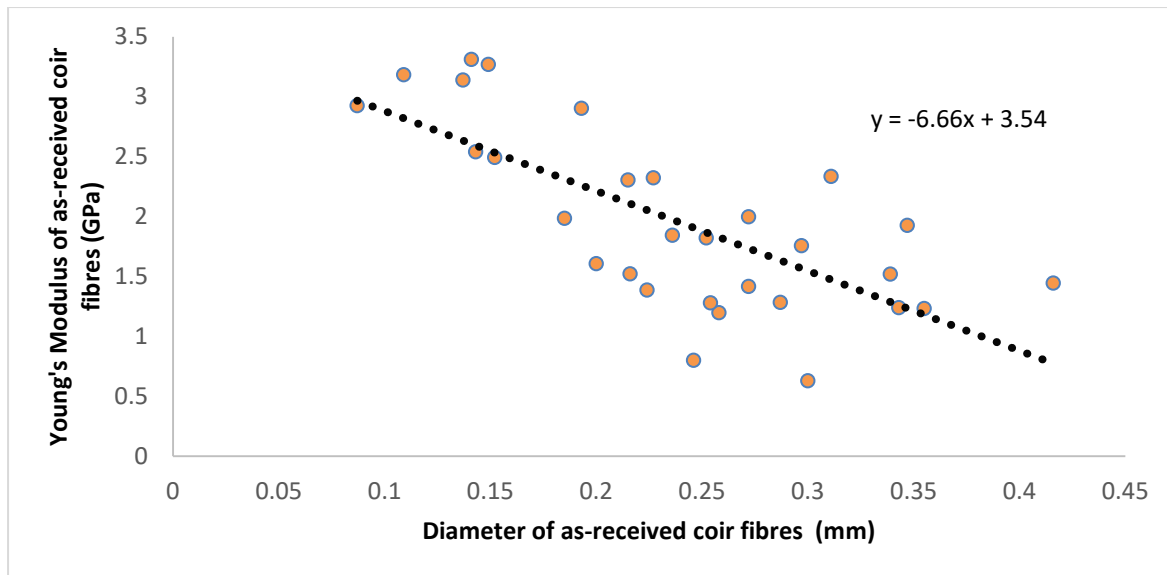


Figure 4. 15 Effect of fibre diameter on the Young's modulus of as-received coir fibres. The dotted line represents the linear regression for the dataset.

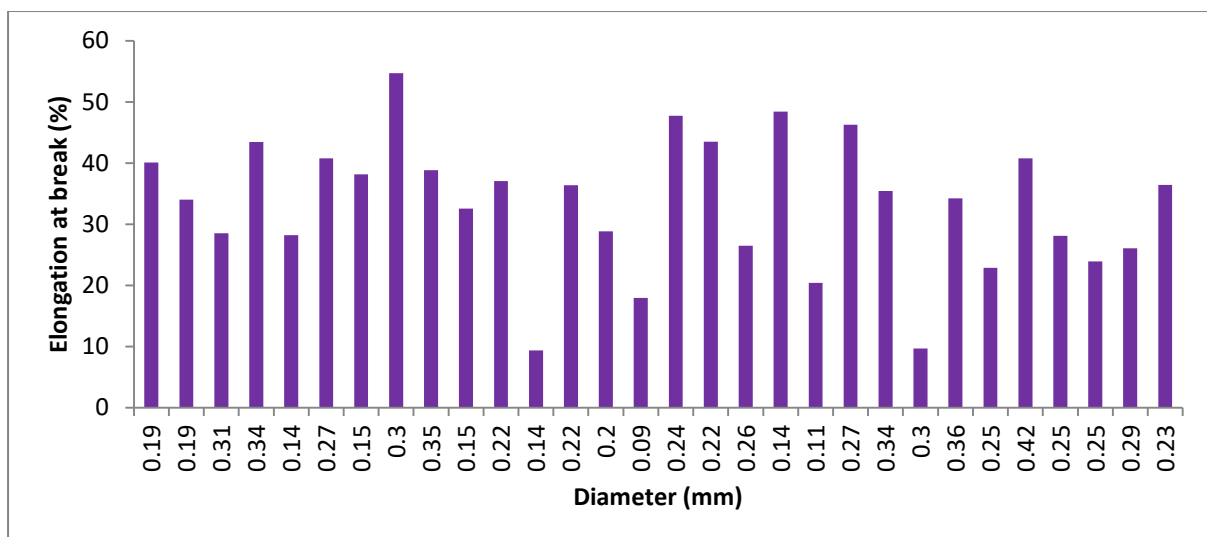


Figure 4. 16 Effect of fibre diameter on the elongation at break of as received coir fibres.

In Figure 4.14, it can be seen that the tensile strength decreases as a function of the coir fibre diameter. A multitude of reasons can be attributed to this observed trend. Firstly, it is reasonable to assume that the presence of any residual hemi-cellulose or other debris on the surface of the fibre will contribute to a larger apparent diameter but this will not contribute significantly to the load-bearing ability of the fibre as it is not present from one end of the fibre to the other. It was observed (as shown in Figure 4.2), that the debris was localised and in most instances, it could be removed manually. During the de-fiberising process, the exocarp of the coconut is not removed from the mesocarp as a separate process. Instead, it is shredded during de-fiberising and combing as shown in Figures 4.8 and 4.11 respectively.

Hence small sections can be retained on the coir fibres. The same applies to the region between the endocarp and the mesocarp where some debris can be retained on the surface of the coir fibres. Secondly, it was mentioned previously that it cannot be assumed that the fibres in a bundle of the 3-tie coir fibres belong to the same coconut. Hence, the maturity, storage conditions and subsequent defiberising method (manual or semi-automatic) can influence the physical properties of the fibres. It was observed that the residual debris on the coir fibres was higher, in general, for those where the defiberising was carried out manually. Thirdly, the moisture content in the fibres can have an influence on the diameter as coir fibres can absorb up to 8%. In the current study, the fibres were cleaned with a vacuum device to remove bulk of the debris, washed thoroughly with tap water and dried at 80 °C for 2 hours prior to storage in a desiccator containing silica gel at 40 °C. Residual hemicellulose and other forms of debris can enable moisture to be absorbed giving rise to erroneous data on the fibre diameter. Fourthly, the variability in the diameter of the coir fibres was seen in Figure 4.1. Therefore, in order to enable comparisons between data presented by different laboratories, it is necessary to standardise on the locations where the diameter is taken and the number of measurement points over the gauge length of the coir fibres.

Table 4.1 Compilation of coir fibre diameters and the resulting tensile properties. The data from the current study have been included for comparison purposes.

Diameter (μm)	Tensile strength (MPa)	Young's modulus GPa	Elongation at failure (%)	Gauge length (mm)	Crosshead displacement rate (mm/minute)	References
225.00	128.7	2.30	29.91	20	5	(Tomczak, <i>et al.</i> , 2007)
150.80	99.8	0.5	44	<5	NA	(Buana <i>et al.</i> , 2013)
234.00	90.81	NA	31.02	10	1	(Anggraini <i>et al.</i> , 2016)
252.96	81.40	2.69	28.97	20	2	(Rait, 2014)
240.00	194.26	1.95	33.3	20	1	Current study
244.00	110.37	0.94	61.45	60	1	Current study
270.00	95.44	0.63	62.45	100	1	Current study

Table 4.1 shows the variation in the diameter of coir fibres reported by different authors. Most of the authors reported multiple measurements along the gauge length however, none of the authors presented in the table reported from which part of the fibre the gauge length was obtained.

In this current report, the gauge length was obtained from the mid section of the middle part of the fibre (see Figure 4.12). The fibre diameter was taken at six different positions along this gauge length.

Fifthly, it is known that the intrinsic pore volume in coir fibres is of the order of 21.1 to 46.3% (Tran *et al.*, 2015; da Luz *et al.*, 2017). Since the pores do not contribute to the tensile strength, it is necessary to establish the relationship between the diameter and the effective cross sectional area of the cell walls in order to normalise the tensile strength data to the fibre diameter. Sixthly, the probability of the occurrence of flaws increases with the volume of the fibre, therefore, it is reasonable to expect the tensile strength to decrease as the diameter increases for a given gauge length. A similar argument can be used to account for the decrease in the tensile strength as a function of the fibre gauge length. Finally, the mode of defiberisation needs to be considered. The process is extremely harsh and significant surface-damage can be caused to the coir fibres. Therefore, seeking correlation between the tensile strength and other physical properties of coir fibres needs to be approached with caution. One partial solution is to use a large sample population to determine the tensile properties of natural fibres in general. As stated previously, in the current study, thirty individual coir fibres were used to deriving the average tensile strength of 194.26 ± 76.03 MPa. The corresponding value for the tensile modulus and the ultimate failure strain were 1.95 ± 0.75 GPa and 33.30 ± 10.90 % respectively. It can be seen that the tensile strength and Young's modulus increase with decreasing diameter contrary to what obtained by Satyanarayana *et al.*, (1986) but however inline with the observations made by Monteiro *et al.*, (2011) and corroborated by the Griffiths model as described in equations 2.2 and 2.3.

4.3.2 Effect of gauge lengths on the tensile strength of as-received coir fibres

Similar to the findings on the influence of diameter on the tensile properties, reports on the influence of gauge length on the tensile properties needed to be confirmed. Mir *et al.*, (2012) reported a steady decrease in the tensile strength but an increase in Young's modulus of both treated and untreated coir as the gauge length increases from 5 to 35 mm. A similar trend

was observed by Mukherjee and Satyanarayana, (1984), Tran, *et al.*, (2010), Fidelis *et al.*, (2013), Mathura and Cree, (2016). The effect of gauge length on the strength has been attributed to the number of defects present in the fibre.

Table 4.2 shows the tensile results of as-received coir fibres with gauge lengths of 20, 60, and 100 mm. The sample size was 30 per gauge length and the average diameter of the fibres were 0.24 ± 0.08 , 0.27 ± 0.04 and 0.25 ± 0.04 mm for gauge lengths of 20, 60 and 100 mm respectively. The average tensile strength for the 20, 60 and 100 mm gauge lengths of coir were 194.3, 110.4 and 95.4 MPa respectively (Madueke, Kolawole and Tile, 2021). As stated previously, the coir fibre samples were selected manually after careful visual inspection to ensure that the surface of the fibres was free of debris from the de-fiberising, washing and drying procedures. With reference to Figure 4.13, zone-B (mid-section of the coir fibre) was selected for the tensile tests. The crosshead displacement rate of the mechanical test machine was kept constant at 1mm/minute. The tests were conducted at ambient temperature of 45 °C and relative humidity of 45%. The load cell was rated to 100N. The results showed deviations in strength, modulus and elongation at all the gauge lengths. With reference to Table 4.2 also reported in Madueke, *et al.*, (2021), an increase in gauge length from 20 to 60 mm led to decrease in strength and stiffness by 43 and 51% respectively. When the gauge length was further increased to 100 mm, the tensile strength and stiffness were decreased by 52 and 68% respectively. The observed trends are in agreement with the tensile data for as-received coir fibres at similar gauge lengths reported by several authors in the literature (Defoirdt, *et al.*, 2010; Fidelis *et al.*, 2013; Trujillo *et al.*, 2014). However, they differ from the trends obtained by Mathura and Cree, (2016) whereby the tensile strength of untreated coir fibre from Trinidad for gauge length of 20 mm at average fibre diameter between 0.16 and 0.56 mm was 139 ± 15.53 MPa.

Comparing tensile data of natural fibres from different labs is notoriously difficult for the reasons discussed previously. The tensile strength obtained experimentally can also be influenced by the alignment of the sample in the grips of the tensile test machine. Fibre slippage from within the end-tabbed region can also have a bearing on the data obtained but this would be obvious in the load/displacement trace. Previous authors have attributed the variations in the tensile data or differences in the fibre diameter, defect density, specie, growing conditions, extraction method or level of maturity of the coir fibres (see Chapter 2). A detailed discussion on this topic was presented in the literature review (see Chapter 2).

The bar charts shown in Figures 4.17 and 4.18 show that strength and stiffness increase as the gauge length of the as-received coir fibres decreases. The abnormally high elongation to failure associated with the large gauge lengths could be as a result of the uncurling of the helical spirals as shown in Section 4.3.6, Figure 4.27. The higher the gauge length the more the spirals are being uncured and the higher the elongation.

Table 4.2 Tensile properties of as-received coir fibres at varying gauge lengths and at constant cross-head displacement rates of 1mm/minute.

Gauge length (mm)	Sample size	Tensile Strength (MPa)	Young's modulus (GPa)	Elongation at break (%)
20	30	194.26 ± 76.03	1.95 ± 0.75	33.3 ± 10.90
60	30	110.37 ± 37.17	0.94 ± 0.43	61.45 ± 24.29
100	30	95.44 ± 40.71	0.63 ± 0.18	62.45 ± 31.56

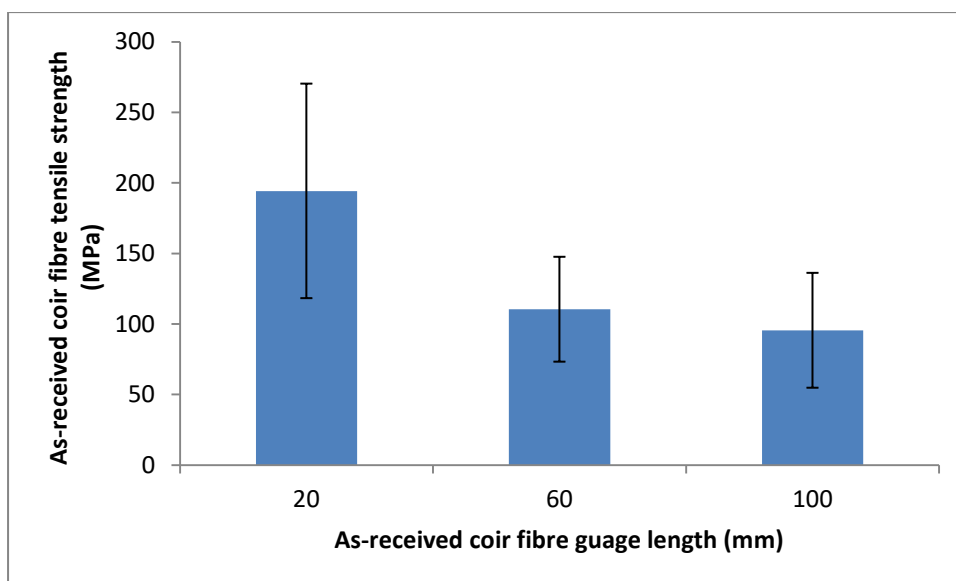


Figure 4.17 Effects of gauge lengths on the tensile strength of as-received coir fibre.

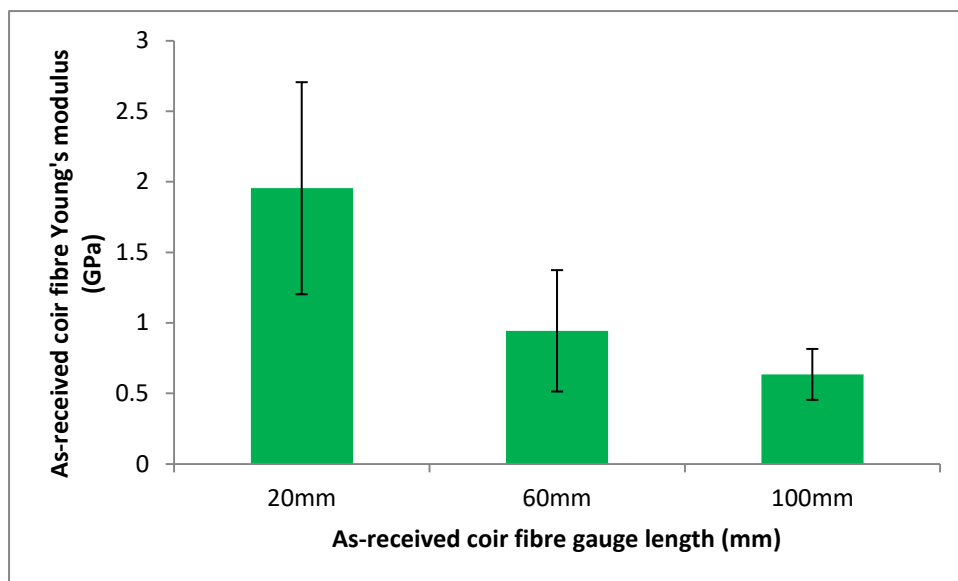


Figure 4.18 Effects of gauge lengths on the Young's modulus of as-received coir fibre.

4.3.3 Effects of the cross-head displacement rates of the mechanical test machine on the tensile properties of coir fibres

In order to gain some knowledge of the effect of the cross-head displacement rates on the tensile properties of coir fibres, three different cross-head displacement rates of 1, 10 and 100 mm/minute were evaluated. The selected coir fibres had a nominal outer diameter of 0.230 mm and the gauge length of 20 mm along with a sample size of 30 for each category.

A summary of the average tensile strength and Young's moduli as a function of the three cross-head displacement rates used is presented in Table 4.3. Figure 4.19 shows that the apparent tensile strength decreases as the cross-head displacement rate increases. This can be attributed to the high degree of intrinsic porosity, the presence of tyloses and the helical nature of the elementary fibres. The data presented in Table 4.3 suggests that the coir fibres become more sensitive to the intrinsic flaws and porosity as the rate of stress application is increased (Kulkarni et al 1983). With most classes of viscoelastic polymers, apparent stiffness will increase as the rate of stress application increases. The data presented in Table 4.3 shows a marginal increase in the Young's modulus when the rate of loading is increased from 1 to 10 mm/min but then it decreases between 10 to 100 mm/minute.

Failure mechanisms of natural fibres such as coir have since been linked to the behaviour of the microfibril and the microfibril angle when subjected to tensile loading. It has been reported that the microfibril angle in the secondary wall layer exerts an influence on the Young's modulus and the extent of the influence exerted is a function of the radius of the

fibre (Satyanarayana *et al.*, 1982; Persson, 2000b; Martinschitz *et al.*, 2008; Sultana, 2014; Tran *et al.*, 2015). The microfibril angle has been related to the Young's modulus using equation [4.1] (Satyanarayana *et al.*, 1986; Tomczak, Sydenstricker and Satyanarayana, 2007):

$$E_f = \frac{E \cdot \cos^2 \theta [k \cdot (1 - 2 \cot^2 \theta)^2]}{E \cdot \cos^2 \theta + k \cdot (1 - 2 \cot^2 \theta)^2} \quad [4.1]$$

where θ is the microfibril angle, K is the bulk modulus, E = calculated modulus and E_f observed modulus of fiber. Increase in tensile strength of coir fibres as the cross head displacement rate increases has been reported (Tomczak, Sydenstricker and Satyanarayana, 2007) while no change has been reported on the Young's modulus as the cross-head displacement rate increases (Fidelis *et al.*, 2013). As previously stated comparing tensile data of natural fibres from different labs is difficult for the reasons discussed earlier. The tensile strength obtained experimentally can also be affected by other factors as discussed in Section 4.3.2.

Table 4.3 Tensile properties of as-received coir fibres at strain rates of 1, 10 and 100 mm with at a gauge length of 20 mm.

Cross-head displacement rate (mm/minute)	Tensile strength (MPa)	Young's modulus (GPa)	Elongation at break (%)
1	194.26 ± 76.03	1.95 ± 0.75	33.30 ± 10.90
10	148.85 ± 51.65	2.29 ± 1.08	33.91 ± 10.86
100	135.24 ± 54.90	2.05 ± 0.67	35.32 ± 11.39

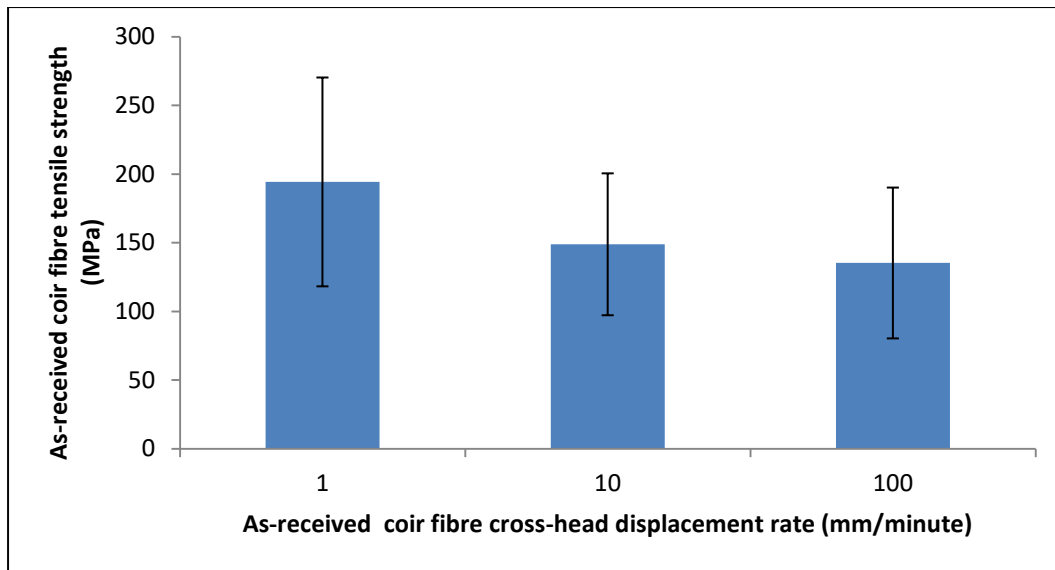


Figure 4.19 Effects of cross-head displacement rate on the tensile strength of as-received coir fibres.

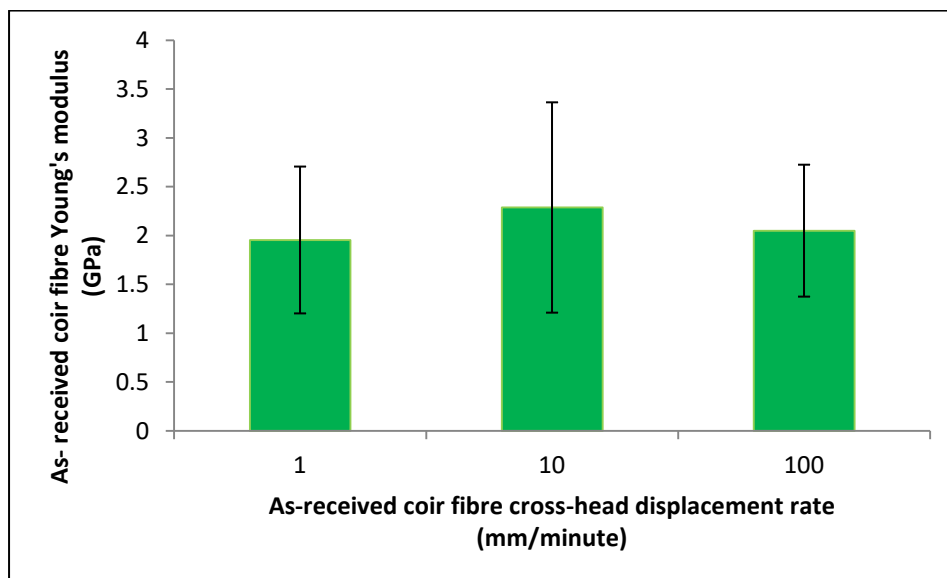


Figure 4.20 Effects of cross-head displacement rate on the Young's modulus of as received coir fibres.

With other classes of viscoelastic materials, the apparent Young's modulus is generally seen to increase as a function of the loading rate. In the current case, the Young's modulus is seen to increase from 1.95 to 2.29 GPa when the cross-head displacement rate is increased from 1 to 10 mm/minute but then it is seen to decrease when the loading rate was increased to 100 mm/minute as shown in Figure 4.20.

4.3.4 Failure analysis of as-received coir fibres

After loading the coir fibre to failure at a cross-head speed of 1 mm/minute and gauge lengths of 20, 60 and 100 mm respectively, the load/ extension and stress/strain graphs are shown in Figures 4.21 and 4.22 and the fracture surfaces are as shown in Figures 4.23, 4.24 and 4.25. The failure stresses were grouped into “high”, “mid” and “low” representing groups with the highest, mid and lowest failure stresses respectively. Over 65% of the failures occurred at about 55 , 65 and 75 % within the 20, 60 and 100 mm gauge lengths respectively. The initial linear region decreases as the gauge length increase for all the gauge lengths. The initial linear region was followed by a steep region and a non- linear region before fracture particularly for gauge lengths of 20 and 60 mm. This has been attributed to the differences in the gauge length, the length/diameter ratio of the fibers, the microfibril angle and the degree of flaws (Satyanarayana *et al.*, 1986; Mathura and Cree, 2016). Hence at a cross-head displacement rate of 1mm/minute, the average failure stress at 20 mm gauge length was 43 and 51% higher than the average failure stresses at 60 and 100 mm gauge lengths respectively. Similar explanations go for the failure of as received coir fibre at cross-head displacement rates of 1, 10 and 100 mm/minute. However, it is worthwhile to note the irregularities of the microstructure (discussed in Section 4.3.6) of the failure surfaces shown in Figures 4.23, 4.24 and 4.25 indicating that each fibre has its own intrinsic morphology which might have resulted in the differences in the failure pattern and failure stress.

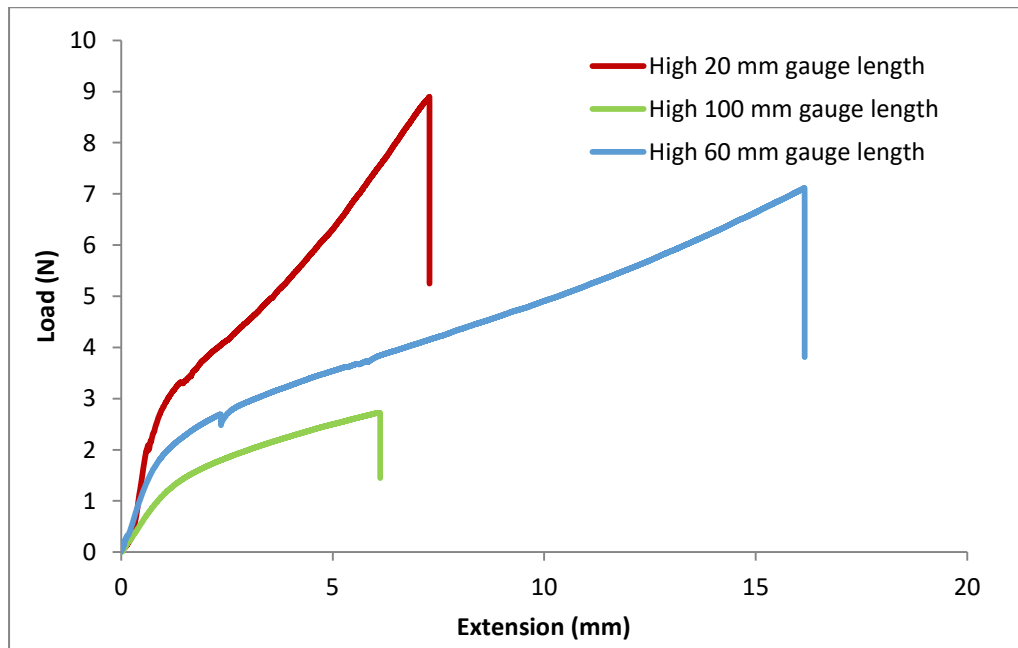


Figure 4.21 The load/extension traces for as-received coir fibres at a cross-head displacement rate of 1mm/minute and gauge lengths of 20, 60 and 100 mm.

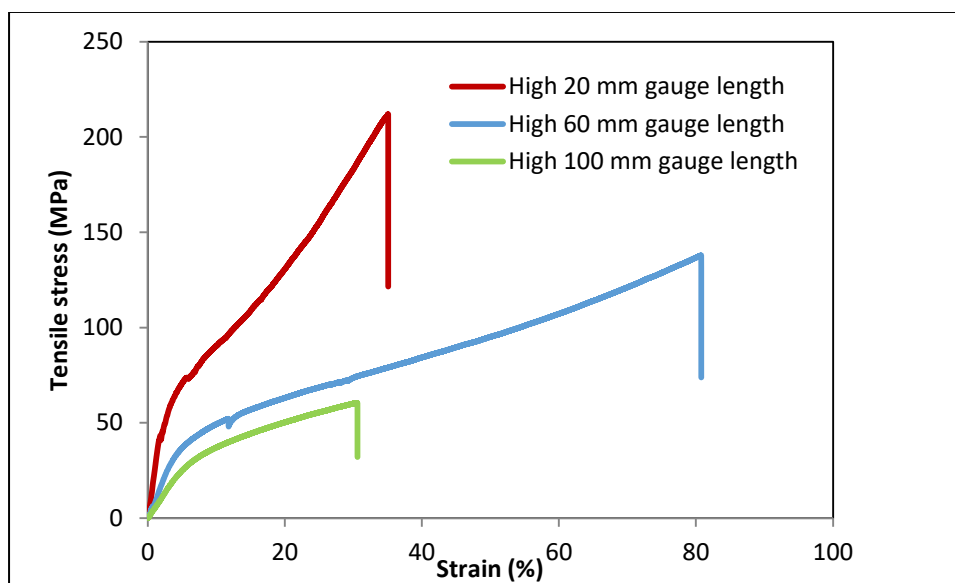


Figure 4.22 Stress/strain curves for as-received coir fibres at a cross-head displacement rate of 1mm/minute and gauge lengths of 20, 60 and 100 mm.

4.3.5 Weibull distribution of as-received coir fibres

Weibull distribution is a continuous probability distribution, the two-parameter Weibull (see Chapter 2 section 2.5.5) was used to characterize the tensile strength results of the as-received coir fibres. The Weibull parameters for as-received coir fibres at the three specified gauge lengths and strain rates are given in Table 4.4 and the Weibull plot shown in Figure 4.23 (Madueke, Kolawole and Tile, 2021). The Weibull modulus for the selected gauge lengths of 20 to 100 mm and cross-head displacement rates of 1 to 10 mm/min in this study is 3 to the nearest whole number unlike that at the displacement rate of 100 mm/minute. The results of the Weibull modulus are in accordance with the Weibull modulus for natural fibres (1 to 6) (Defoirdt, *et al.*, 2010). The Weibull scale parameter is the tensile strength of natural fibre with a probability of survival at 0.63 in the Weibull survival plot (see Figure 4.24). The Weibull scale parameter as shown in the Table 4.4 decreases as the gauge length increases possibly as a result of increased probability of encountering flaws. This trend has also been observed by other authors (Pickering *et al.*, 2007; Guo *et al.*, 2014; Trujillo *et al.*, 2014). However, the tensile properties of sisal fibres were evaluated for varying gauge lengths but at a constant strain rate, no effect on the tensile strength was observed rather a 35% decrease in Weibull modulus was observed with increase in gauge length (Ramesh, Palanikumar and Reddy, 2013). Bamboo fibre has showed very low strength variability with Weibull modulus

of 7.6 when compared with coir fibre. The difference in the level of scatter in the strength of natural fibres depends on the degree of flaws in the fibre. Several factors have been noted to contribute to the level of flaw and flaw distribution in natural fibres, such factors include the level of porosity of the fibre, the diameter of the fibre, processing conditions and fibre treatment (Kulkarni, Satyanarayana and Rohatgi, 1983; Pickering *et al.*, 2007; Zafeiropoulos and Baillie, 2007). Survival probability, P , is used for the Weibull analysis P is the probability of survival of the fibres after a certain stress, (σ) (Kulkarni *et al.*, 1983; Hill and Okoroafor 1995). The sum of the survival probability and failure probability is equal to 1. If the survival probability is equal to 1, then there is no fracture (See section 2.5.1). The survival probability obtained for as received coir fibres as shown in Figure 4.24 was at a stress, σ , of 217.96 MPa.

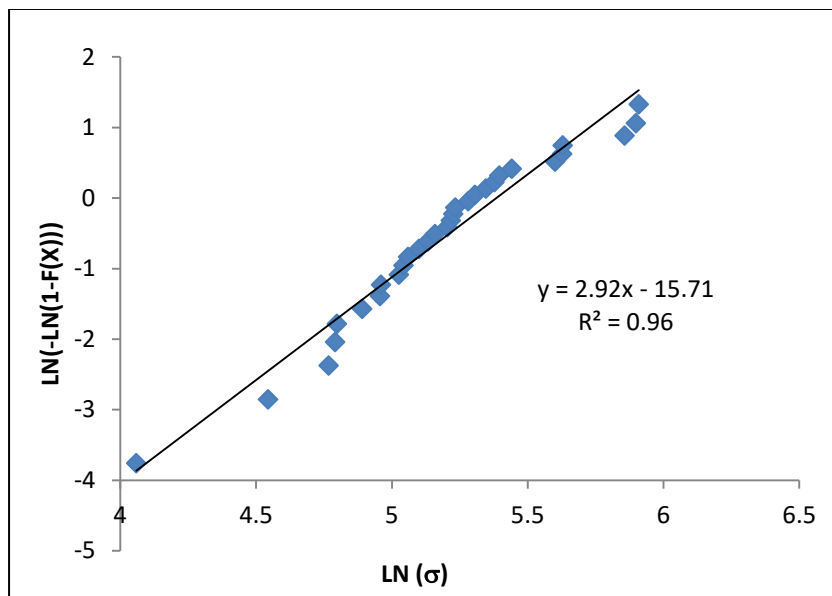


Figure 4.23 Weibull plot of as-received coir fibres at a cross-head displacement rate of 1mm/minute and gauge length of 20 mm.

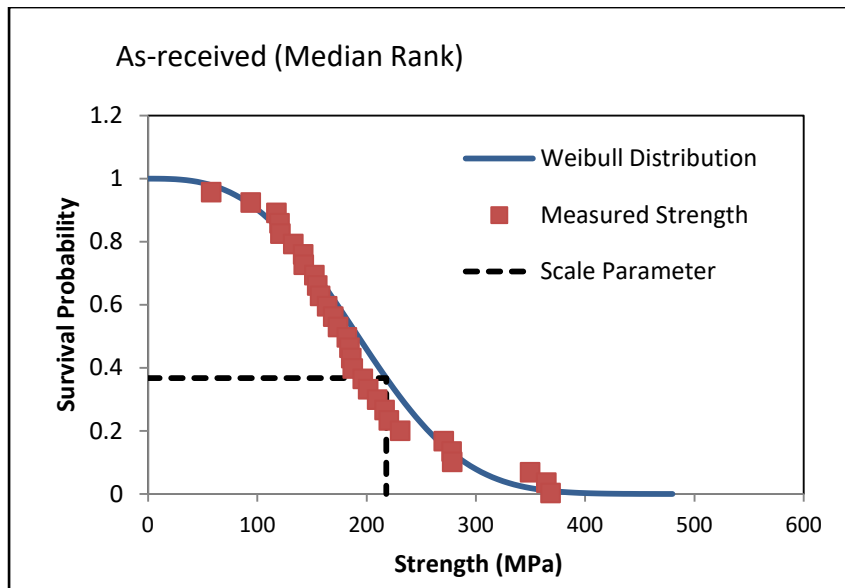


Figure 4.24 Weibull Survival probability of as-received coir fibre at 20 mm gauge length and crosshead speed of 1mm/minute.

The higher the Weibull modulus, the smaller the number of flaws present and the more even the distribution of those flaws.

Table 4.4 Weibull Parameters on the tensile strength of as-received coir fibres at varying gauge lengths and crosshead displacement rates.

Gauge length (mm)	Crosshead displacement rate (mm/minute)	Weibull Modulus, m	Weibull Scale parameter (MPa)	R^2
20	1	2.92	217.96	0.96
60	1	3.26	123.28	0.96
100	1	2.70	107.38	0.95
20	10	3.38	165.74	0.92
20	100	2.30	155.61	0.93

These findings on the diameter of coir fibre being most uniform at about the middle of the fibre, the gauge length of 20 mm and the cross-head displacement rate of 1mm/minute so far have been adopted for subsequent tensile tests in this research.

4.3.6 Microstructural Characterisation of coir fibres

Specimens of coir fibres prepared for optical and scanning electron microscopy views were taken from the head, middle and the tail of coir fibre (see Figure 4.12) for detailed analysis.

The scanning electron micrographs (SEM) of unwashed coir fibre showed in Figure 4.25 shows coir fibre with a flaky surface. Patches of debris can be seen on the surface. The SEM of as-received (as-received=after vacuum-cleaning, washing and drying) shows regularly arranged globular protrusions referred to as tyloses as shown in Figure 4.26. The tyloses in the microstructure of coir fibre have been identified by several authors (Kim, 2014; Rait, 2014). The arrangement of these tyloses may offer an improved fibre/matrix adhesion in composite manufacture. Figure 4.27 shows the longitudinal cross section containing helical spirals. Figure 4.28 (a) shows a continuous central lacuna running all the way through from one end to the other in the middle section of the fibre. As can be seen, the coir fibre is multicellular and the cells are either round or polygonal in shape. However, the arrangement of the cells is quite different at the end section of the coir fibre as shown in Figure 4.28 (b). This suggests that the properties or performance of coir fibre could be a function of the area or section of interest (head, middle or tail) as further observed in the cross-sectional view (this has not been reported elsewhere).

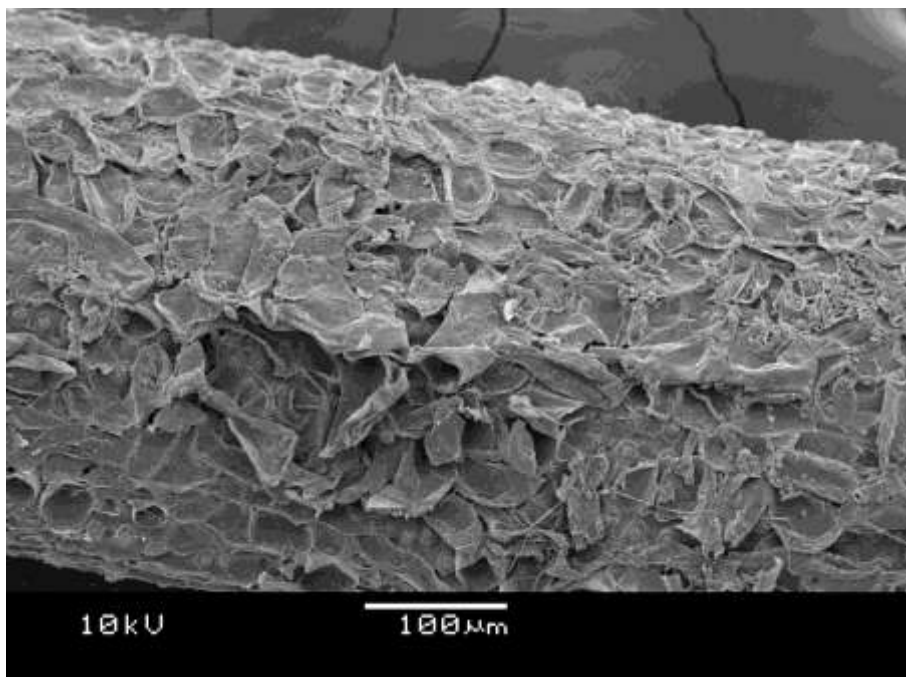


Figure 4.25 Micrograph of unwashed coir fibre showing a furfuraceous (flaky) surface.

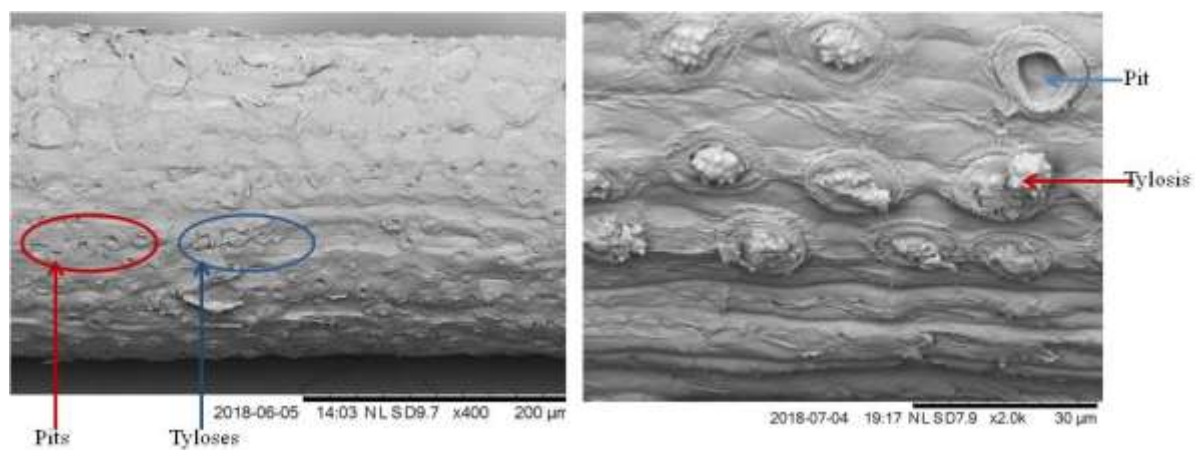


Figure 4.26 Micrographs of as-received coir fibre showing globular protrusions referred to as tyloses.

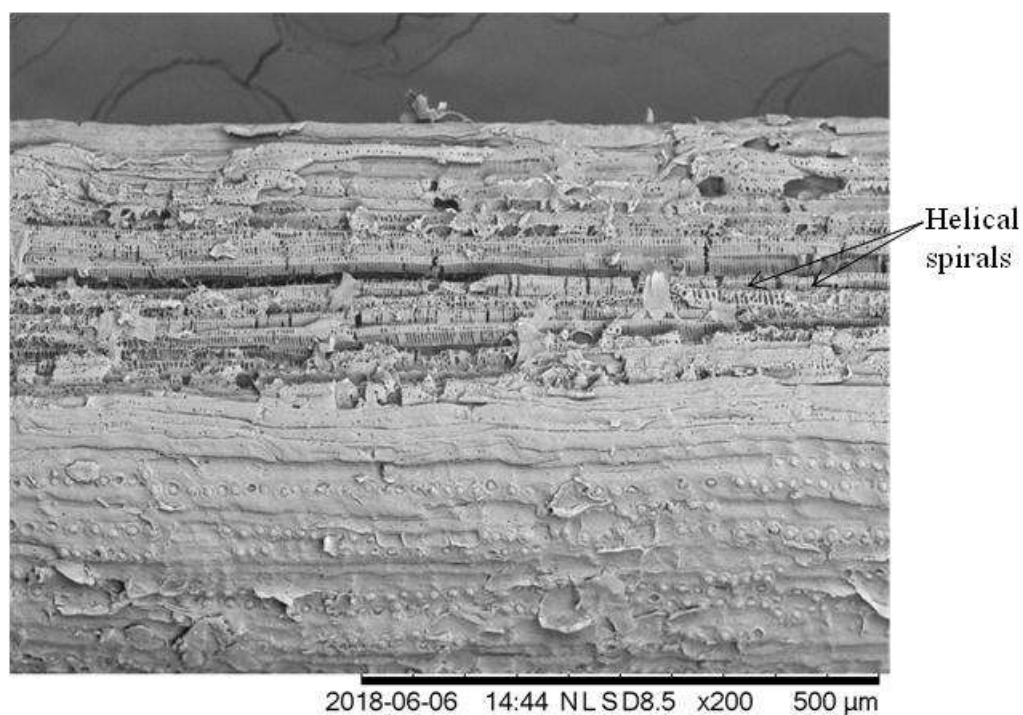


Figure 4.27 Micrograph of as-received coir fibre showing helical spirals arranged in parallel manner.

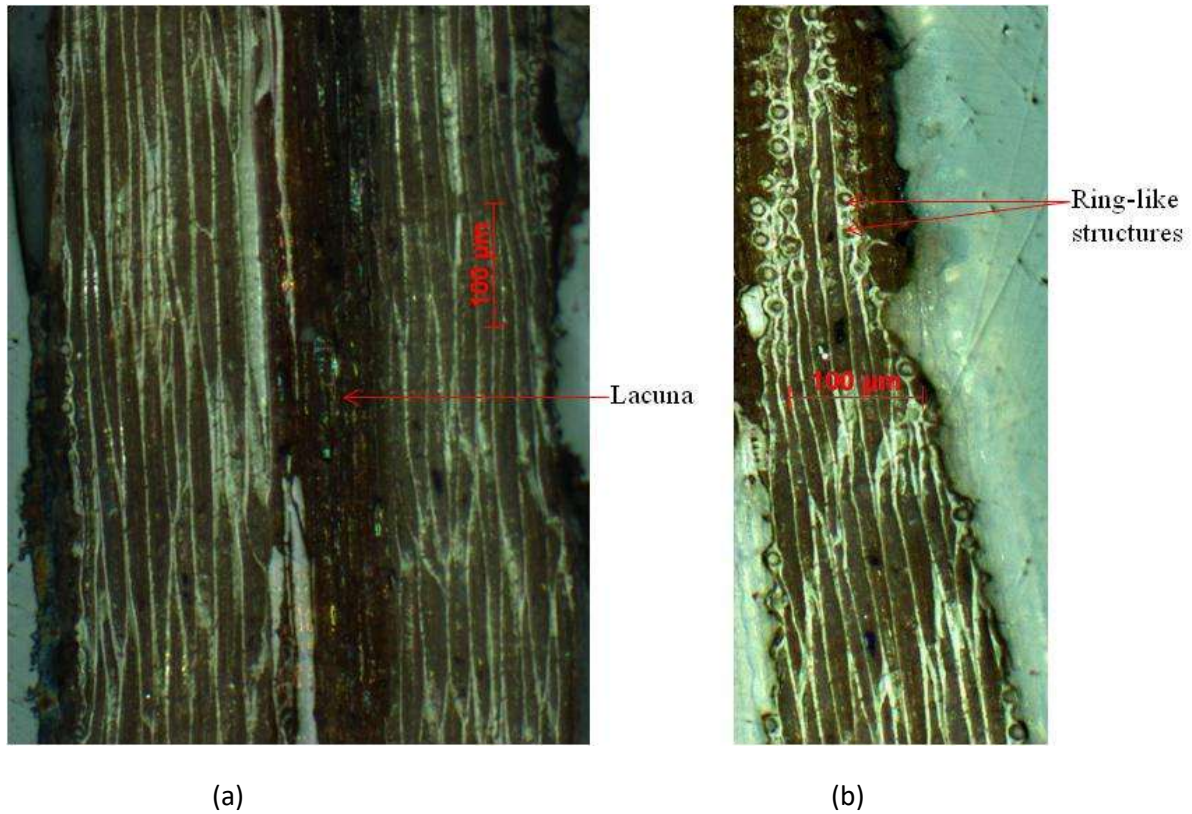


Figure 4.28 Figure 4.28 Optical longitudinal micrographs of as-received coir; (a) middle section of coir showing continuous lacuna from one end through to the other end (b) tail section of coir showing ring-like structures towards the end.

Coir fibres are highly porous as observed in the previous section and in Figures 4.29 and 4.30. The pores possess different sizes and shapes. The porosity of coir has been associated with its low densities such as; 860 kg/m^3 (Muensri *et al.*, 2011) and 1150 kg/m^3 (Ticoalu, Aravinthan and Cardona, 1997).

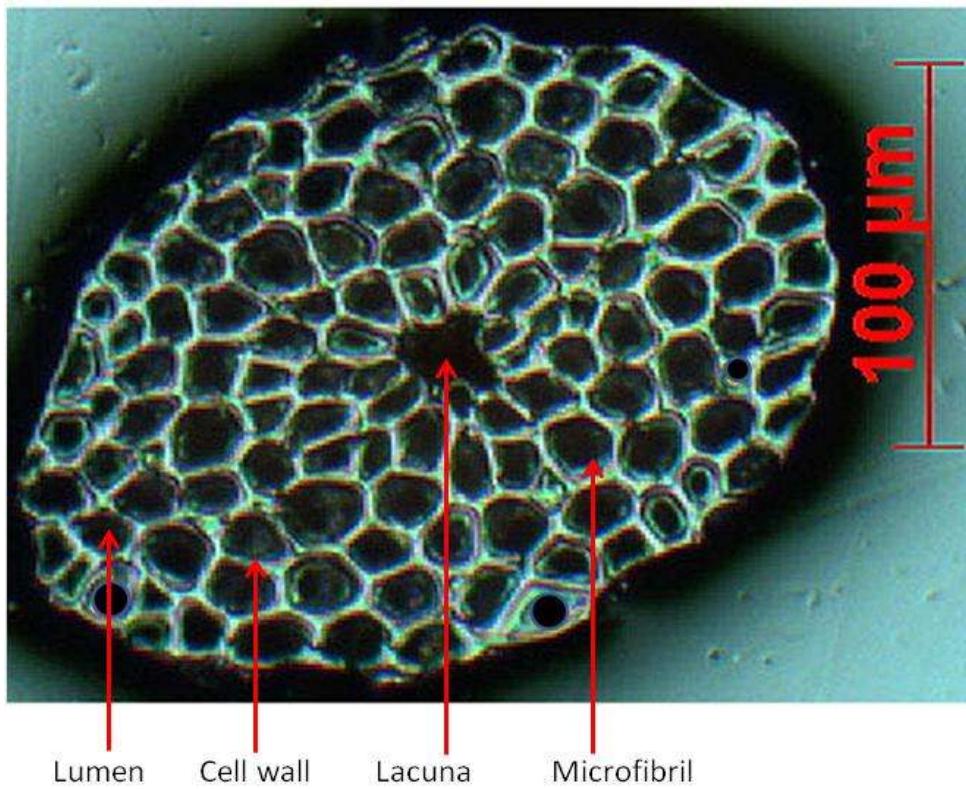


Figure 4.29 Transverse section of an optical micrograph of as-received coir fibre.

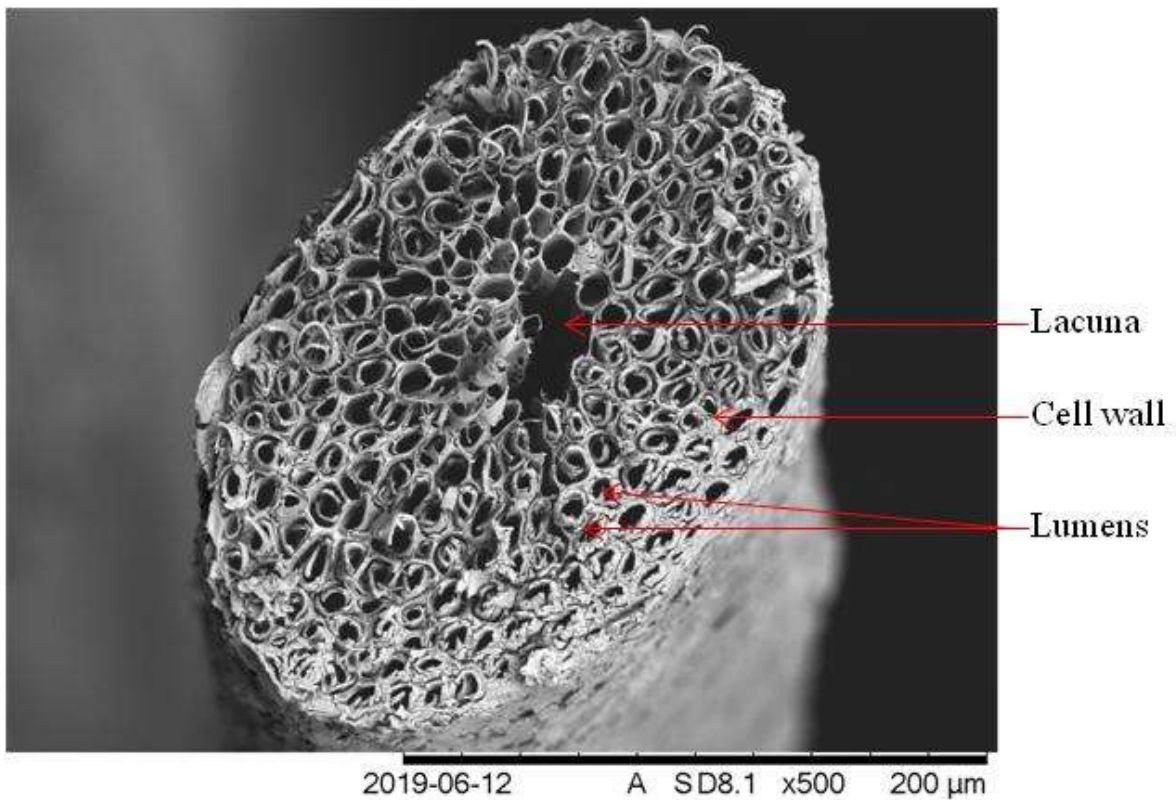


Figure 4.30 A SEM of the cross-section of as-received coir fibre.

Coir fibre contains bundles of cylindrical cells as shown in Figure 4.31. On the plant cell wall are located the microfibrils as shown in Figure 4.32. The amorphous region keeps the cellulose microfibrils in place by bonding (Mathura and Cree, 2016). The cellulose microfibril angle present in the secondary cell wall has been linked to their mechanical properties (Burgert, 2006). As the microfibril angle increases, the stiffness of the cell wall decreases while the strain to fracture increases. The microfibril angle can be measured or determined from its tensile strength as stated in equation 2.22, Section 2.2.3.2. When natural fibres that possess both crystalline and non-crystalline regions such as coir are subjected to tensile force, the fibrils undergo elongation or bend or even twist causing a reduction in the cross section of the fibre and lateral separation of several cell walls (Satyanarayana 1986).

The lacuna as can be observed in the optical and scanning electron microscopy images displayed in Figures 4.33 and 4.34 differ in dimensions. The sizes and dimensions of the lacuna mainly depend on the sectioned area (head, middle or tail) as shown in Figure 4.35. This suggests that the properties of coir could be affected by the sectioned area.

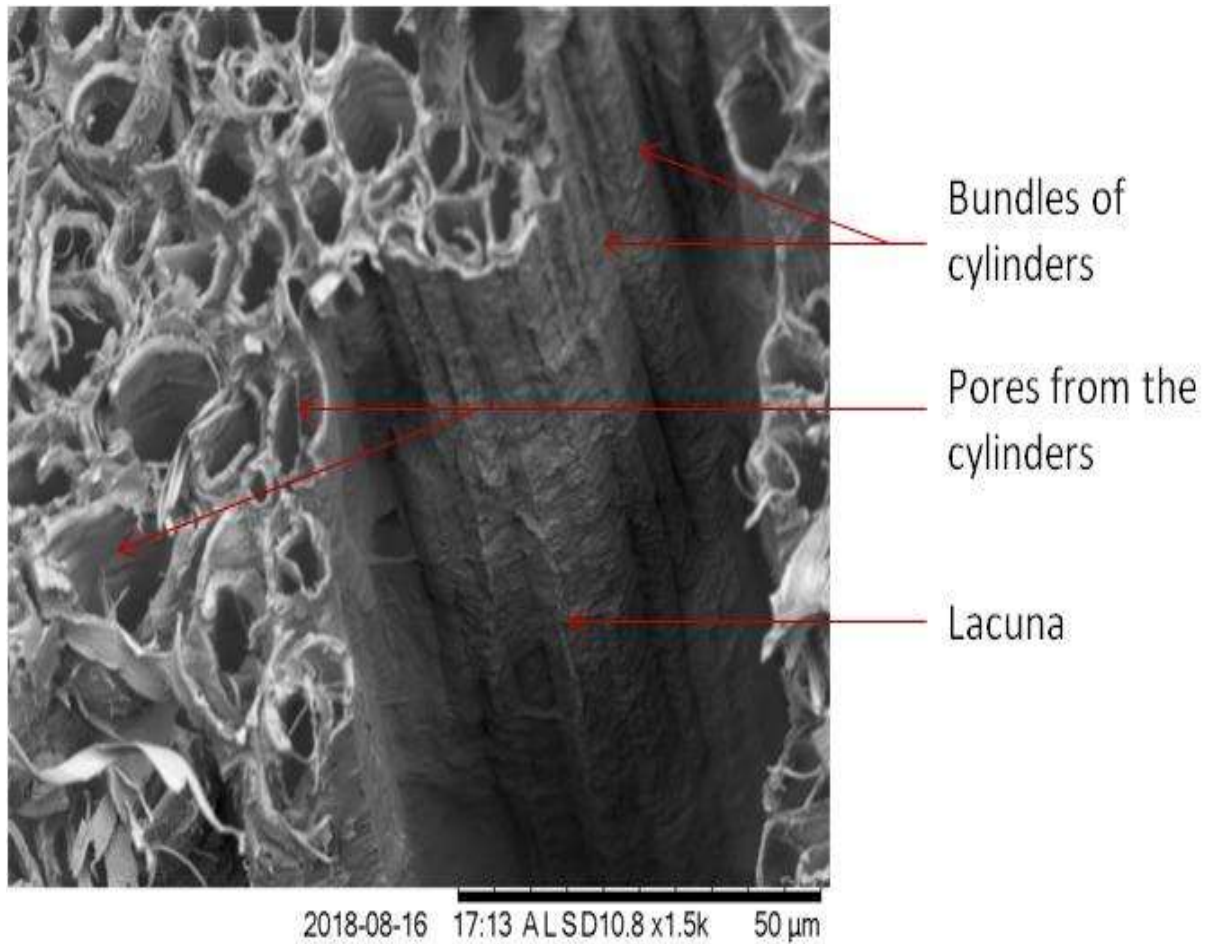


Figure 4.31 SEM of the cross-section of as-received coir fibre showing deep-rooted pores.

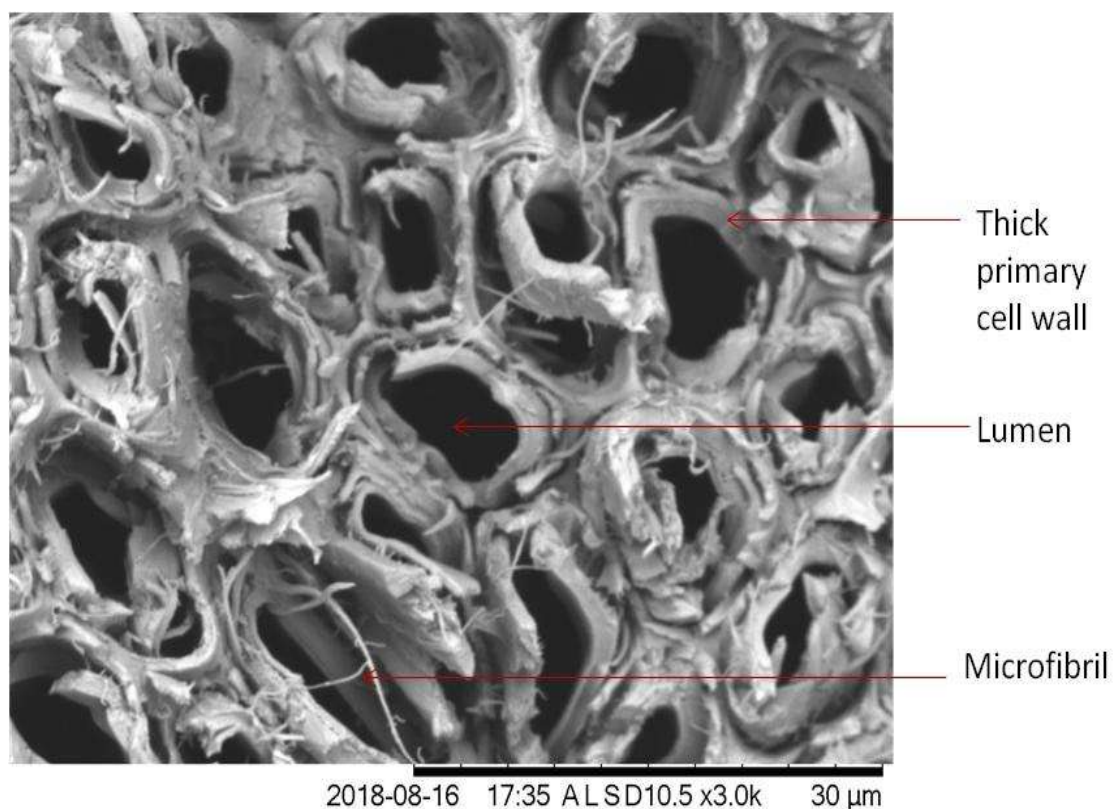


Figure 4.32 SEM of the cross-section of as-received coir fibre showing thick primary cell.

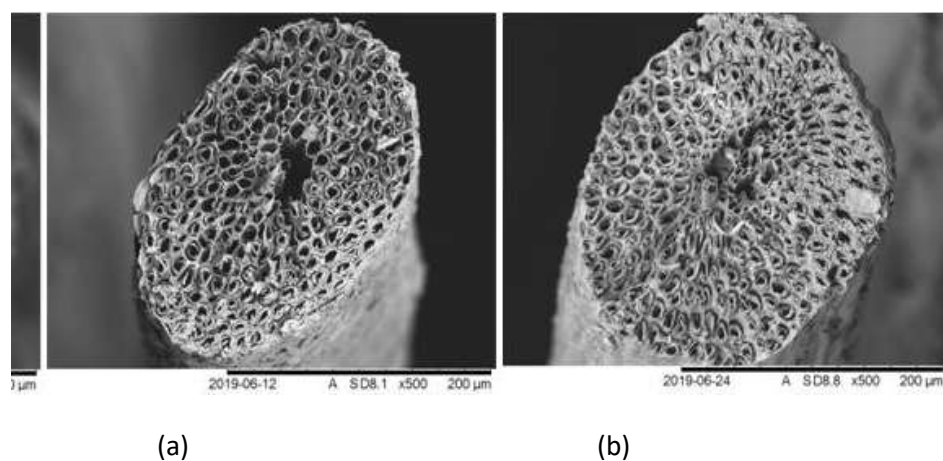


Figure 4.33 Micrographs showing different sizes of lacuna from different sections of as-received coir fibres (a) middle (b) tail.

4.3.6.1 Image analysis of as-received coir fibres

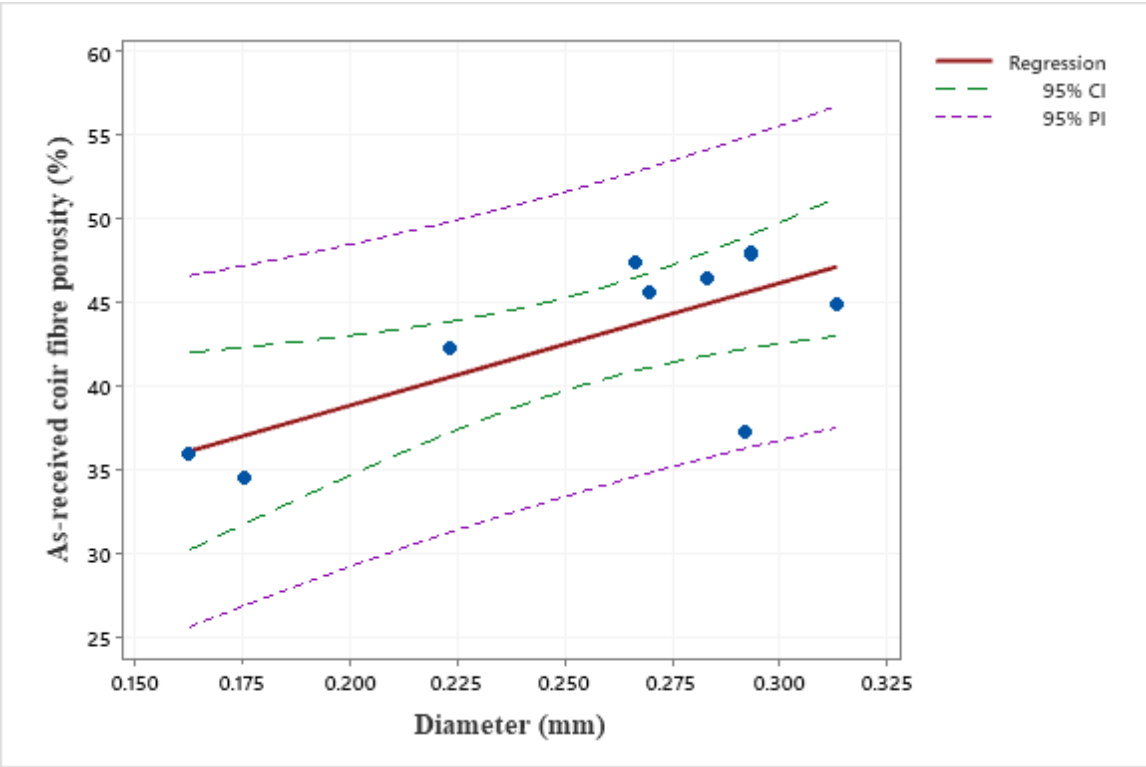
ImageJ was employed to process the microstructural images of coir fibres and to analyse the data obtained.

4.3.6.1.1 Determination of the volume fraction of pores in as-received coir fibre via ImageJ

The optical microscopy images of as-received coir fibres were analysed using ImageJ. Each image was cropped and converted to an 8-bit image. The threshold parameters were adjusted to colour-pick the phase for volume fraction. The slide bars were employed to completely colour-red the phase of interest and then applied and analysed. The analysis was repeated for ten times per sample. The results of the analysed fibres are given in Table 4.5. The diameters of the fibres were determined from the cross-sectional area of the fibre with the assumption that the cross-sectional area of the fibre is that of a circle. The results obtained showed that the porosity of as-received coir fibre is in the range of 34-48%. The average pore count is in the range of 130 to 475. The average cell diameter ranges from 6 to 14 μm . The relationship between the fibre diameter and the porosity of the fibre is shown in Figure 4.34. The porosity of coir fibre increases as the diameter increases. The number of pores as well as the pore size varies with the diameter of the fibre as shown in Figure 4.35. This has been reported by Madueke, Kolawole and Tile, (2021).

Table 4.5 Porosity of coir fibre determined from image analysis.

Sample number	Fiber diameter (μm)	Av. pore size (μm^2)	Porosity (%)	Av. cell diameter (μm)
1	283.20 ± 2.13	135.11 ± 27.90	46.21 ± 2.91	10.94 ± 2.3
2	269.72 ± 2.66	119.39 ± 30.79	45.37 ± 1.38	12.38 ± 2.34
3	313.43 ± 2.26	131.93 ± 9.46	48.05 ± 0.85	6.75 ± 1.71
4	223.26 ± 2.46	106.01 ± 6.81	42.19 ± 1.61	10.16 ± 2.52
5	266.38 ± 1.09	106.60 ± 0.03	47.36 ± 0.17	7.83 ± 1.45
6	291.58 ± 1.61	147.91 ± 5.12	47.32 ± 0.41	10.15 ± 3.88
7	291.82 ± 1.61	150.31 ± 4.71	47.44 ± 0.35	13.72 ± 2.13
8	162.61 ± 0.84	50.82 ± 0.95	32.85 ± 0.51	7.46 ± 0.46
9	175.52 ± 0.36	97.07 ± 18.30	36.82 ± 0.24	6.94 ± 1.54



CI= Confidence Interval; PI=Prediction Interval

Figure 4.34 Relationship between the fibre diameter and the porosity of the fibre

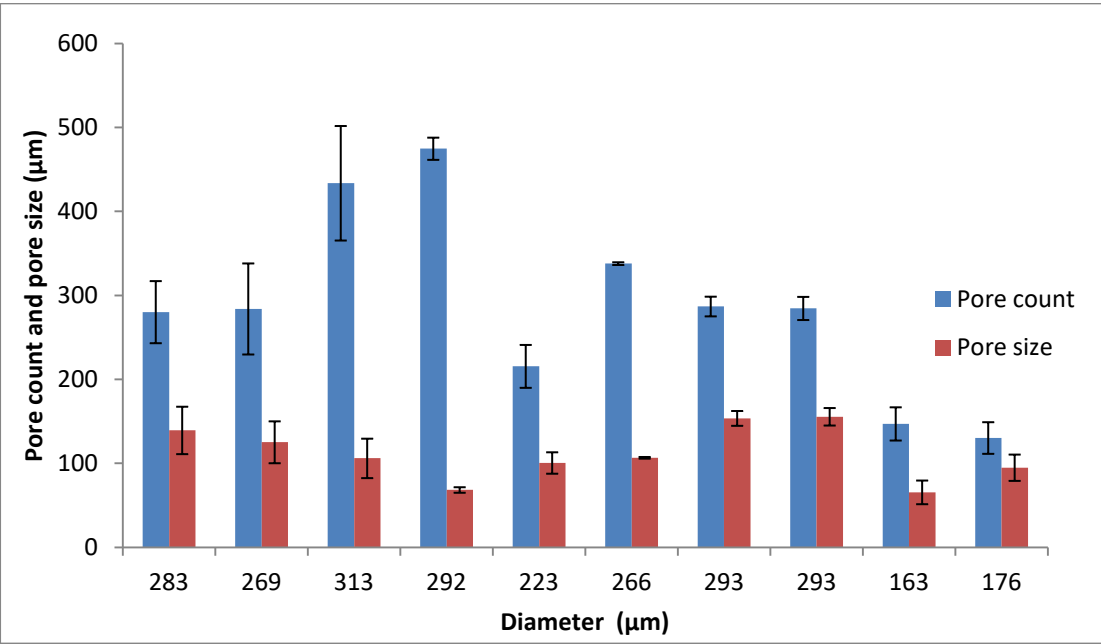


Figure 4.35 Relationship between the fibre diameter and the pore count/size.

Observations

- (i) The microstructure of coir contains bundles of regularly arranged cells.
- (ii) The porosity of coir fibre varies with the diameter of the fibre.
- (iii) The number of pores of coir fibre and the pore size varies with the diameter of the fibre.
- (iv) The size of the lacuna does not depend only on the external diameter of the fibre as stated by Satyanarayana *et al.*, (1986) but also depends on the section of interest (head, middle, tail).

4.4 Treatment of coir fibres using sodium hydroxide (NaOH)

Several conflicting reports on the effects of NaOH on the properties of coir fibre have been highlighted in the literature review (Chapter 2; Section 2.3.3). Many researchers have limited the NaOH treatment temperature of coir fibre to room temperature and temperatures below 30 °C (Bismarck *et al.*, 2001; Nam *et al.*, 2011a; Dixit and Verma, 2012; Karthikeyan and Balamurugan, 2012; Andic-Cakir and Demirci, 2014; Narendar, Dasan and Jayachandran, 2016; Yan *et al.*, 2016). Post-processing of treated fibres were mostly carried out at undisclosed temperatures. The vast majority of the previous researchers adopted the same NaOH treatment concentration of 5% (Varma, Varma and Varma, 1984; Bismarck *et al.*, 2001; Nam *et al.*, 2011a; Hariprasad, Dharmalingam and Raj, 2013; Narendar, Dasan and Jayachandran, 2016; Yan *et al.*, 2016; Manjula *et al.*, 2017). The singular effects of the treatment variables and their statistical significance on the properties of coir fibres were scarcely reported on any of the published works so far. Most of these issues are addressed in this section.

4.4.1 Visual and microstructural observations of NaOH-treated coir fibres

Multifactorial design of experiment on Minitab 19 Software was used in order to establish the variables that exerted predominant effects on the properties of coir fibre. A total of 27 experimental conditions were created through this design. In order to ensure better clarity in the interpretations of the data obtained from tensile tests, the 27 experimental conditions were split into three groups based on the temperature level used for each treatment.

The treatment of coir fibre with NaOH resulted in a change in the colour of the fibres as shown in Figure 4.36. This colour was observed to deepen as the temperature and duration increased. The pH of the effluence was 14 in all cases. This signified strong alkalization of the fibres. This colour change can therefore be attributed to the alkalization of the NaOH-

treated fibres which might have been as a result of the removal of impurities from the surface of the fibres. It can be seen previously, that unwashed coir fibres contain a lot of debris, impurities and residual components which might not be completely washed off as in the as-received. NaOH treatment resulted in the removal of these impurities. NaOH treatment led to a decrease in the size of the lumens and the lacuna as shown in Figure 4.37.



Figure 4.36 Photographs of NaOH-treated fibres showing change in colour at (i) 20% NaOH concentration, 30 °C and for 24 hours duration and (ii) 20% NaOH concentration, 50 °C and for 24 hours duration

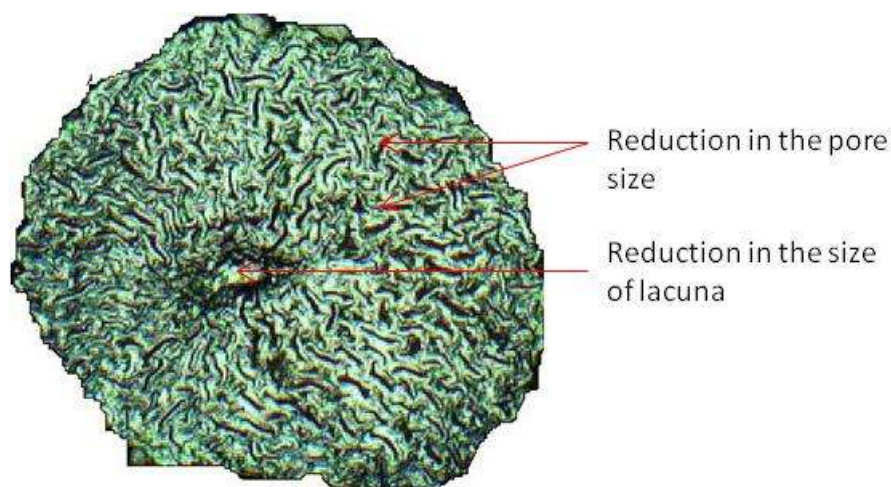
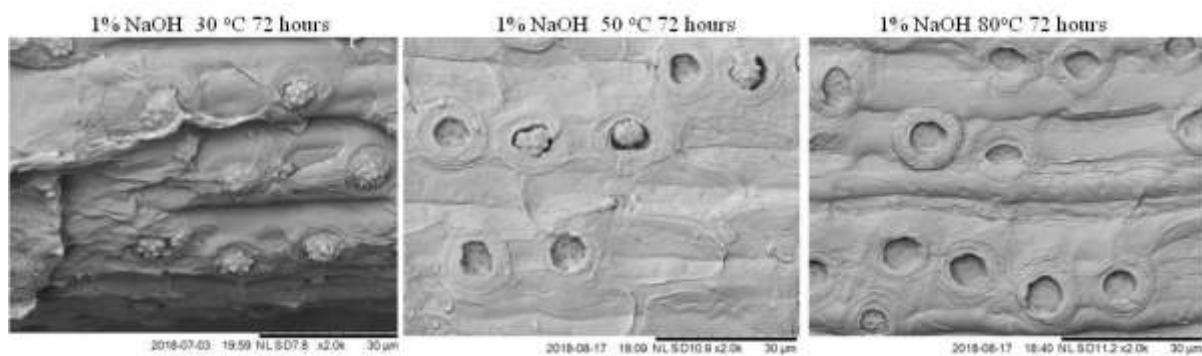


Figure 4.37 An optical micrograph of NaOH-treated coir fibre showing decrease in the size of lumens. This batch was treated at 20 % NaOH concentration, 80 °C and for 24 hours.

The SEM of as-received coir fibres showed coir fibre surface to be full of globular protrusions referred to as tyloses. At increasing temperatures of 30 to 80 °C but at a constant NaOH concentration of 1% and temperature of 72 hours, the globular particles were increasingly removed dissolving into pits as shown in Figure 4.38a giving rise to rougher surfaces. Surface roughness can be determined using laser displacement sensor or statistical parametric mapping (SPM) image processing software.

At a constant temperature and duration of 30 °C and 72 hours respectively but at an increasing NaOH concentration of 1 to 20 %, removal of tyloses also increases with formation of pits as shown in Figure 4.38b. This gives rise to uneven and rough surfaces especially at 5% and 20% NaOH concentrations.

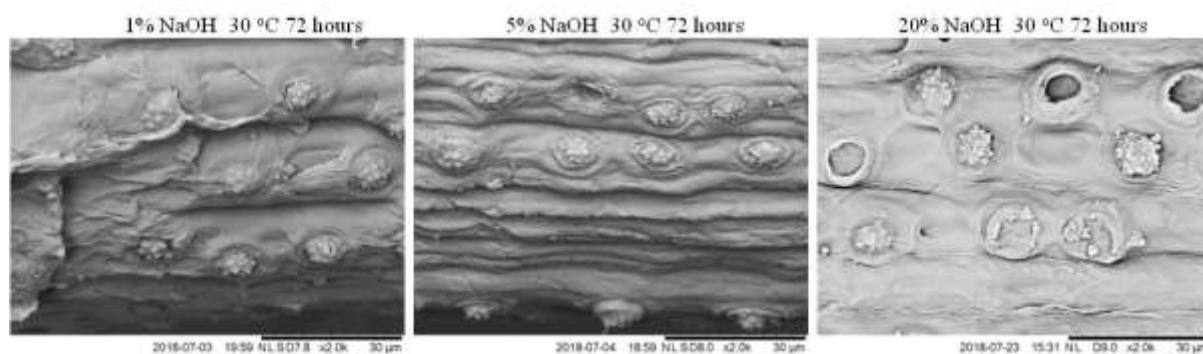
At a constant NaOH concentration of 1% and temperature of 80 °C but at an increasing duration of 24 to 72 hours, all the tyloses present on the fibre surfaces are completely removed as shown in Figure 4.38c rendering the surfaces tylosis-free irrespective of the concentration (1%) of NaOH. This shows that temperature and duration exert a reasonable influence on the surfaces of as-received coir fibers during NaOH treatment. However, it is important to determine the most significant variables in the treatment of coir fibre using NaOH as this will lead to further improvement of its properties.



[a] [(i)

(ii)

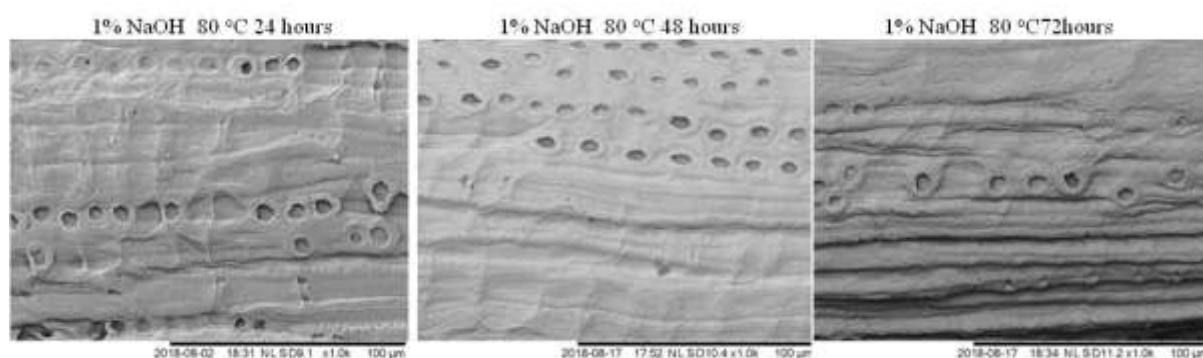
(iii)



[b] (i)

(ii)

(iii)



[c] (i)

(ii)

(iii)

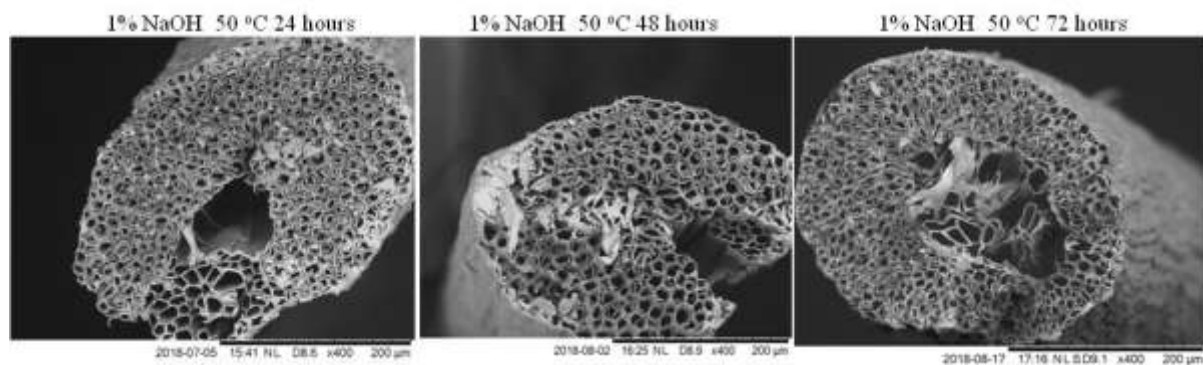
Figure 4.38 (a-c) SEM micrographs of the surfaces of NaOH-treated coir fibres at varying concentrations, temperatures and durations.

The NaOH treatment conditions affected the cross-section of the as-received coir fibres in different measures as shown in Figure 4.39 (a-d). At a constant NaOH concentration of 1% and a constant treatment temperature of 50 °C but increasing duration from 24 to 72 hours, no substantial changes can be observed in the cross-section as shown in Figure 4.39 (a) however at an increased NaOH concentration of 20% and at the same increasing time of 24 to 72 hours, a change in the microstructure can be observed as shown in Figure 4.39 (b). There is a decrease in the size of the pores and the lacuna as the duration increases.

At a constant temperature of 50 °C and a constant duration of 48 hours, but increasing NaOH concentration of 1 to 20%, no significant changes can be observed at 1 and 5% concentrations

but a reduction in the size of the lumens and lacuna can be observed at 20% as shown in Figure 4.39 (c).

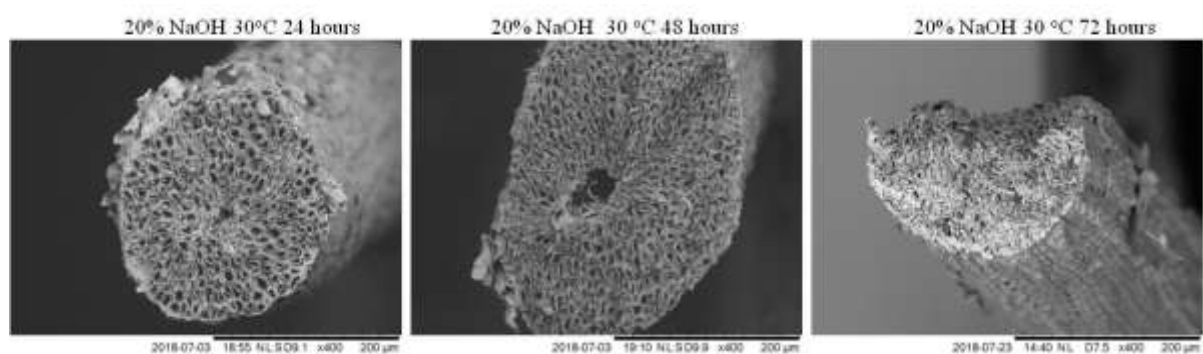
With the NaOH concentration and duration held constant at 20% and 24 hours respectively, but with an increasing temperature from 30 to 80 °C, reduction in the sizes of lumen are visible as shown in Figure 4.39(d). This reduction in the sizes of lumen can be seen to increase as the treatment temperature increases. The increased strength of the NaOH treated fibres has been attributed to the reduction in the size of the lumens (Nam *et al.*, 2011a; Saw, Sarkhel and Choudhury, 2011).



[a] (i)

(ii)

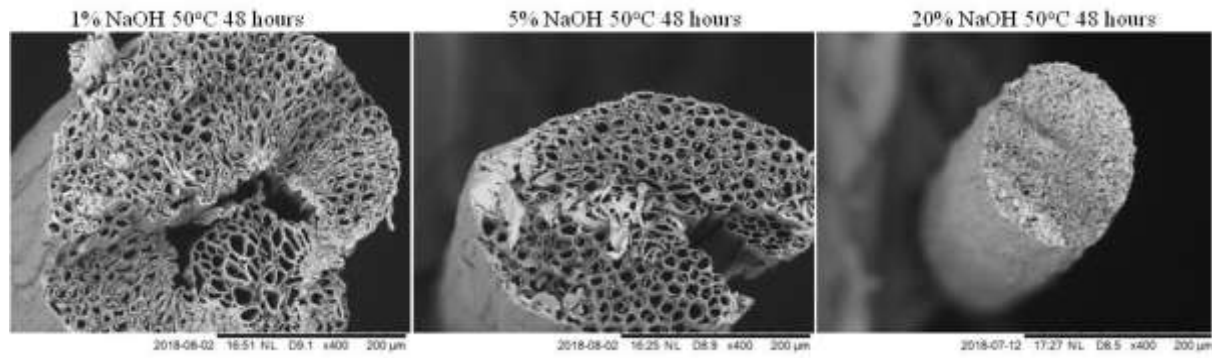
(iii)



[b] (i)

(ii)

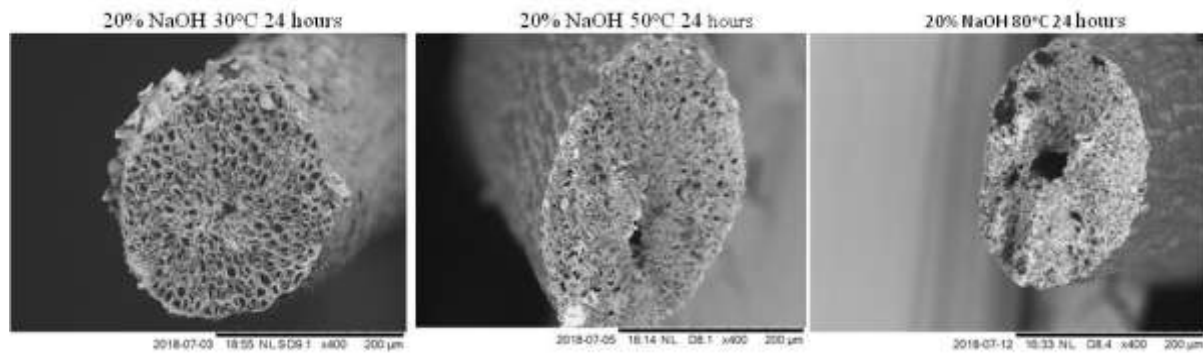
(iii)



[c] (i)

(ii)

(iii)



[d] (i)

(ii)

(iii)

Figure 4.39 (a-d) SEM images of the cross sections of NaOH-treated coir fibres at different NaOH treatment conditions.

4.4.1.1 Image Analysis of NaOH-treated coir fibres

In order to analyse the microstructure of NaOH-treated fibres, ImageJ was employed. The specimens were prepared from the middle section of the fibres. The diameters of the fibres were calculated from the area (Tran *et al.*, 2015). The porosity of NaOH-treated coir fibres were found to be in the range of 10 to 22% as against 34-48% obtained from the untreated coir fibres. About 24 to 26% reduction in the diameter of the treated fibres was observed.

4.4.2 Effects of NaOH treatments on the tensile properties of as-received coir fibres

The results in Table 4.6 (a-c) and Figures 4.40 to 4.42 show that an increase in the tensile strength, Young's modulus and elongation at fracture of as-received coir fibres can be achieved using NaOH treatment. It is worthwhile to note that the strongest and stiffest coir fibres were treated at the same treatment conditions of 20% NaOH concentration and at a

temperature of 50 °C for 24 hours. The maximum elongation of the fibres was achieved at 1% NaOH concentration and at 80 °C for 24 hours.

The tensile strength of the treated fibres was observed to increase with an increase in the concentration of NaOH especially at a temperature of 50 °C at all durations as shown in Figure 4.48. The tensile strength increased by 15% when the NaOH concentration was changed from 1 to 20%. This was about 21% increase in the tensile strength at optimum condition when compared with the untreated fibres. An increase in the strength of coir fibres at concentrations of 5% have been observed in previous reports (A. Bismarck *et al.*, 2001; Nam *et al.*, 2011b; Yan *et al.*, 2016). As the concentration of NaOH increased, a reduction in the pore size, the lacuna and the diameter of the fibres was observed and the cell wall thickened leading to increase in the strength of the treated fibres as previously observed by (Nam *et al.*, 2011b; Saw, Sarkhel and Choudhury, 2011).

No significant difference was observed in the stiffness of the treated fibres as the NaOH concentration was changed from 1 to 5% but at 20% NaOH concentration, the stiffness of the treated fibres increased from 1.99 to 2.73 GPa.

The elongation at break of the treated fibres was found to decrease as the NaOH concentration increased from 1 to 20%. A significant decrease in the elongation of the treated fibres by 34% was observed when the NaOH concentration was swapped from 1 to 20%. However an increase in the elongation of coir fibres by 4 and 10% at a concentration of 5% and treatment time of 72 hours have been observed by a few researchers (Brintz, 2014; Rait, 2014).

A variation exists in the tensile strength of the treated coir fibres as the treatment temperature increased from 30 to 80 °C. At the treatment temperature of 50 °C and at all durations, the strength increases with increase in the concentration. At a treatment temperature of 80 °C and at a 48 hour duration, the tensile strength increases as the concentration increases. However at a treatment temperature of 30 °C, a scatter in the strength was observed at all concentrations and durations. This explains the variable reports obtained from literature at a treatment temperature of 30 °C and durations of 24 to 72 hours. The elongation at break increased as the treatment temperature increased. The elongation at break was found to increase by 14% as the treatment temperature was changed from 30 to 80 °C. An insignificant increase in elongation has been recorded at treatment durations of 5 and 72 hours (Nam *et al.*, 2011b;

Brintz, 2014; Rait, 2014). The increase in the elongation at break as a result of increase in duration has been observed by other researchers (Wenbin *et al.*, 2005).

For most materials, changes in temperature have resulted in changes in volume. A decrease or increase in a material's temperature has been associated with the contraction or expansion of the material.

The tensile strength of the treated fibres decreased as the duration of treatment increased. At the duration of 72 hours, the strength of the treated fibres was found to decrease by 10%. This amounted to about 6% reduction in strength when compared with untreated fibres. However, no significant changes in the stiffness and elongation were recorded as the duration of treatment was varied from 24 to 72 hours. At prolonged treatment time such as 72 hours, during which the coir fibres are still soaked in the NaOH solution, the fibrils are completely exposed to the surface, cell rupture will most likely take place. This contributes to a decrease in the tensile strength of the fibres as the duration of treatment increased.

At the optimum NaOH treatment conditions of as-received coir fibres, the increases in the tensile strength, modulus and elongation at fracture compared to the untreated conditions were 21, 41 and 34% respectively.

Tables 4.6 (a-c) Tensile test results of as-received coir fibres treated with NaOH.

(a) At 30 °C

SC	Factors: Experimental condition			After NaOH treatment			
	Con (%)	Te (°C)	Du (Hr)	Diameter (mm)	Tensile strength (MPa)	Young's modulus (GPa)	Elongation at failure (%)
A	1	30	24	0.21 ± 0.04	190.19 ± 63.39	2.06 ± 0.85	38.63 ± 9.27
B	1	30	48	0.24 ± 0.05	176.48 ± 78.13	2.01 ± 0.91	36.58 ± 11.59
C	1	30	72	0.24 ± 0.04	207.41 ± 60.50	2.23 ± 0.52	39.56 ± 10.81
D	5	30	24	0.23 ± 0.05	227.75 ± 73.27	1.64 ± 0.61	40.48 ± 5.94
E	5	30	48	0.22 ± 0.03	171.12 ± 54.21	1.61 ± 0.58	35.01 ± 7.55
F	5	30	72	0.23 ± 0.03	204.58 ± 66.12	2.40 ± 0.63	34.10 ± 5.74
G	20	30	24	0.21 ± 0.06	197.27 ± 54.79	2.47 ± 0.80	26.89 ± 3.80
H	20	30	48	0.24 ± 0.05	176.85 ± 53.78	2.60 ± 0.98	29.62 ± 6.91
I	20	30	72	0.28 ± 0.03	196.70 ± 40.82	2.35 ± 0.53	29.26 ± 5.13

Con=Concentration; Te= Temperature; SC=Sample code, Du= Duration

(b) At 50 °C

SC	Factors: Experimental condition			After NaOH treatment			
	Con (%)	Te (°C)	Du (Hr)	Diameter (mm)	Tensile Strength (MPa)	Young's modulus (GPa)	Elongation at failure (%)
J	1	50	24	0.25 ± 0.04	171.32 ± 62.35	2.29 ± 0.67	39.56 ± 7.16
K	1	50	48	0.26 ± 0.04	148.25 ± 50.63	1.91 ± 0.74	44.85 ± 16.45
L	1	50	72	0.25 ± 0.04	158.73 ± 48.29	1.72 ± 0.47	47.74 ± 14.98
M	5	50	24	0.24 ± 0.04	184.01 ± 68.97	2.38 ± 0.81	34.87 ± 8.79
N	5	50	48	0.22 ± 0.03	162.14 ± 72.65	1.85 ± 0.84	43.85 ± 9.58
O	5	50	72	0.23 ± 0.03	172.44 ± 46.23	2.31 ± 0.60	42.54 ± 13.36
P	20	50	24	0.21 ± 0.03	246.27 ± 71.86	3.30 ± 0.99	25.18 ± 5.27
Q	20	50	48	0.21 ± 0.03	209.71 ± 51.97	2.53 ± 0.72	32.88 ± 6.27
R	20	50	72	0.22 ± 0.03	205.53 ± 52.93	3.09 ± 0.81	26.89 ± 5.15

(c) At 80 °C

SC	Factors: Experimental condition			After NaOH treatment			
	Con	Te	Du	Diameter	Tensile strength	Young's modulus	Elongation at fracture
	(%)	(°C)	(Hr)	(mm)	(MPa)	(GPa)	(%)
S	1	80	24	0.23 ± 0.04	188.63 ± 60.63	2.19 ± 0.74	48.52 ± 10.24
T	1	80	48	0.23 ± 0.03	168.36 ± 52.12	1.63 ± 0.48	50.74 ± 11.55
U	1	80	72	0.23 ± 0.05	185.37 ± 55.18	1.85 ± 0.72	46.65 ± 12.52
V	5	80	24	0.21 ± 0.03	212.21 ± 57.95	2.21 ± 0.57	43.80 ± 9.59
W	5	80	48	0.20 ± 0.03	203.35 ± 50.74	2.15 ± 0.81	45.71 ± 8.58
X	5	80	72	0.26 ± 0.04	109.51 ± 49.82	1.24 ± 0.52	38.59 ± 10.87
Y	20	80	24	0.21 ± 0.03	208.78 ± 42.24	2.47 ± 0.60	29.30 ± 4.66
Z	20	80	48	0.19 ± 0.02	240.26 ± 46.35	3.13 ± 0.65	27.33 ± 5.97
ZZ	20	80	72	0.22 ± 0.03	196.77 ± 47.19	2.60 ± 0.78	30.67 ± 6.04

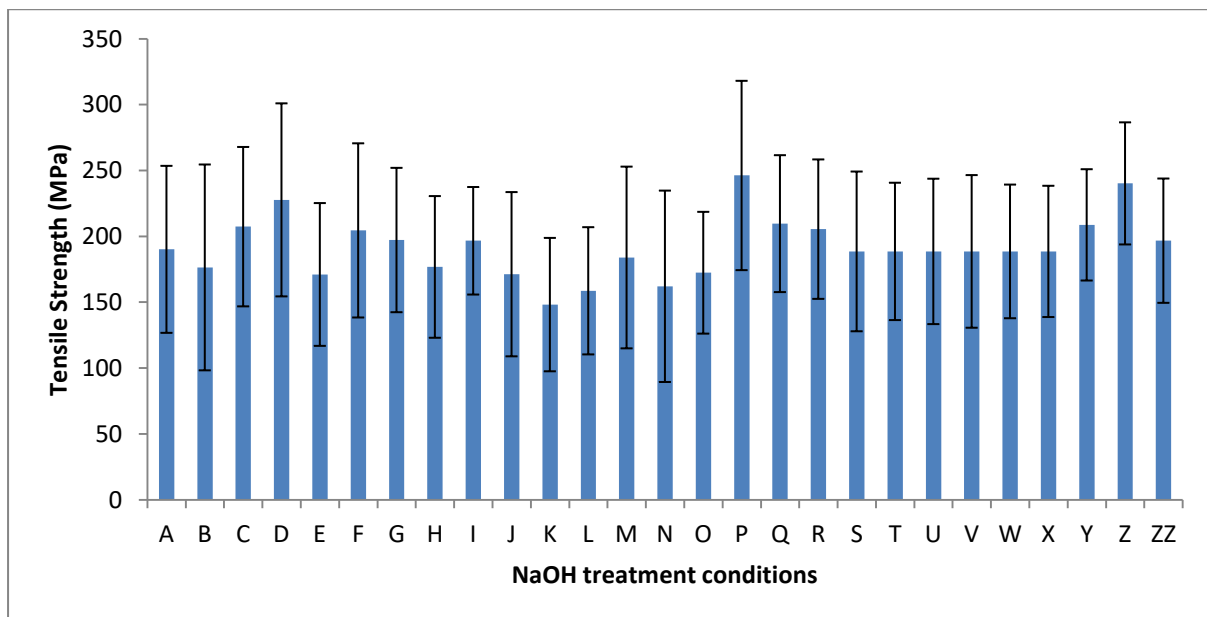


Figure 4.40 showing the effects of NaOH treatment conditions on the tensile properties of coir fibre. The NaOH treatment conditions are as stated in Tables 4.6(a-c).

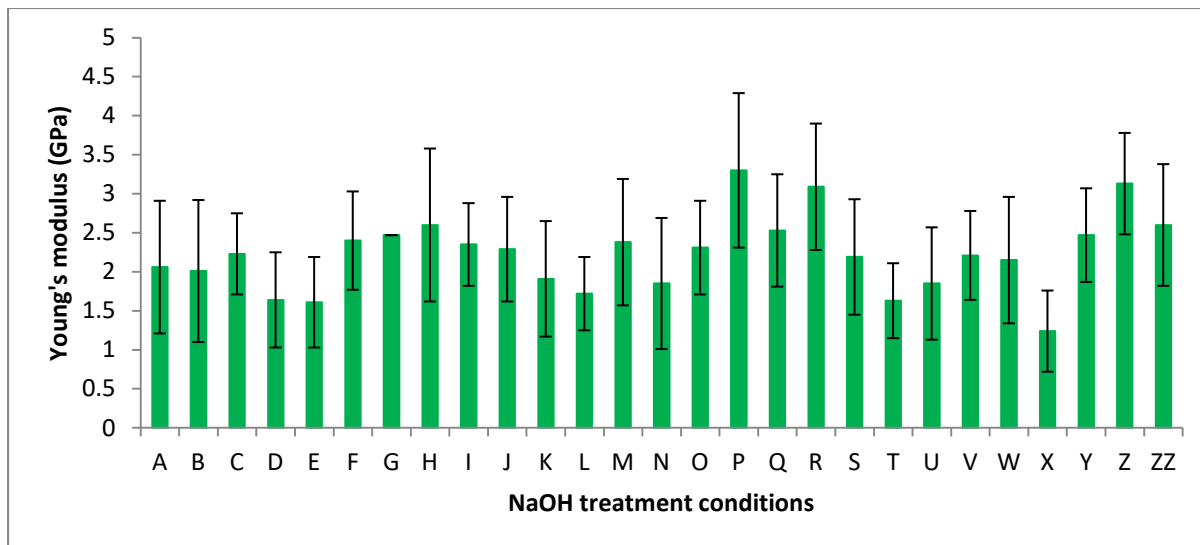


Figure 4.41 showing the effects of NaOH treatment conditions on the Young's modulus properties of coir fibre. The NaOH treatment conditions are as stated in Tables 4.6 (a-c).

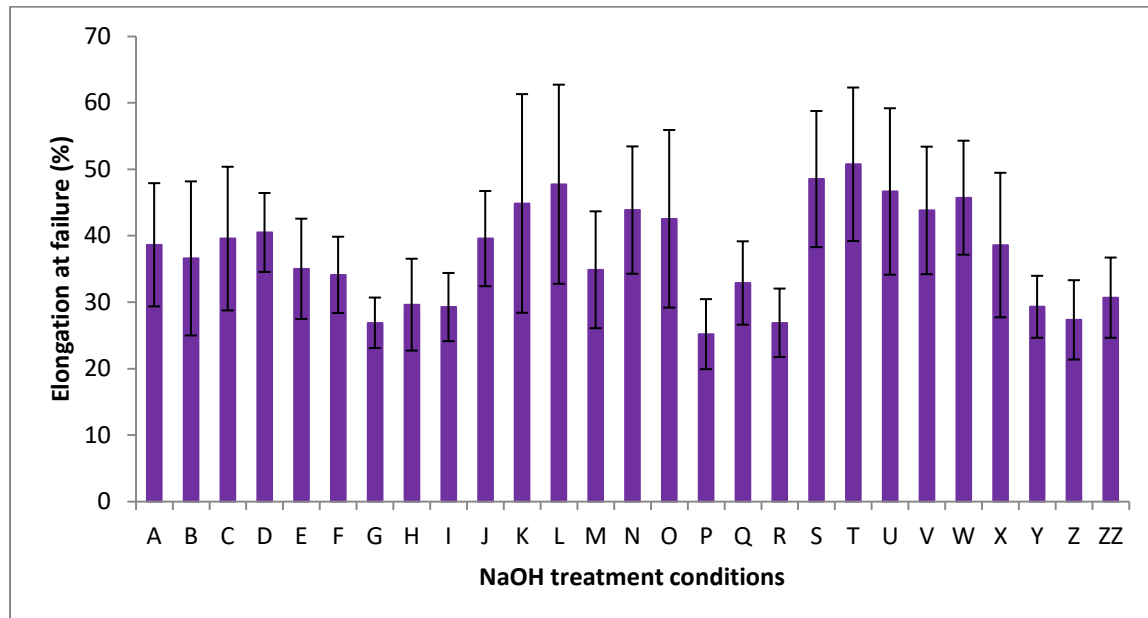


Figure 4.42 showing the effects of NaOH treatment conditions on the elongation at failure of coir fibre. The NaOH treatment conditions are as stated in Tables 4.6 (a-c).

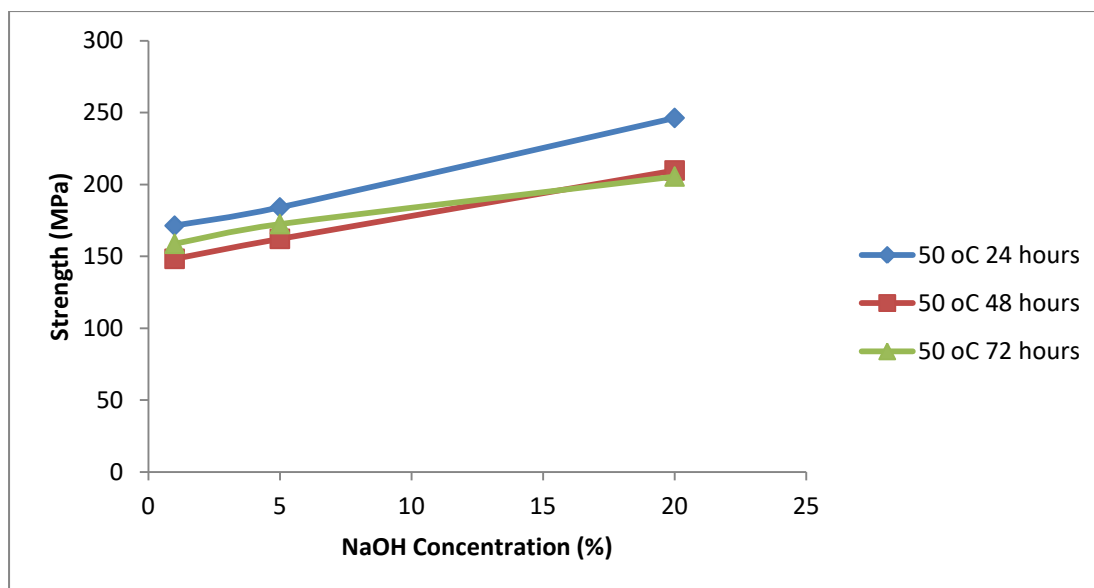


Figure 4.43 Effects of NaOH concentrations on the tensile strength of as-received coir fibres at 50% NaOH concentration.

4.4.2.1 Factorial analysis results for Strength, Stiffness and Elongation for NaOH treated coir fibres

The tensile tests results were fitted through regression via analysis of variance (ANOVA) to determine the variables which have statistically significant effects on the properties of coir fibres and to what extent these variables influence the properties. A Pareto chart is a quality tool used to identify both significant and non-significant categories and focus further improvement where possible on the identifications made ('Machinery Failure Analysis and Trouble-shooting', Geitner and Bloch, Butterworth-Heinemann, 4th ed., 2012). Temperature and duration are not statistically significant for strength and stiffness but only concentration as shown in the Pareto Charts; Figures 4.44 and 4.45 denoted by the reference lines in the Pareto Charts. Concentration and temperature are statistically significant for elongation as shown in Figure 4.46, however, duration is not. Concentration was found to exert the highest influence on all the tensile properties of NaOH treated coir fibres as shown from the longest bars in Figures 4.44-4.46.

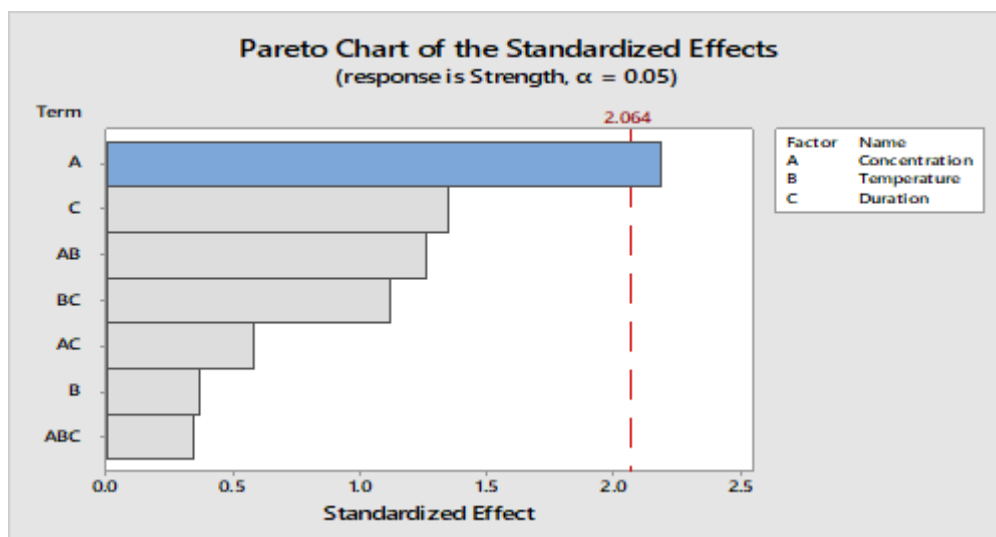


Figure 4.44 NaOH concentration exerted the highest influence on the strength properties of coir fibre.

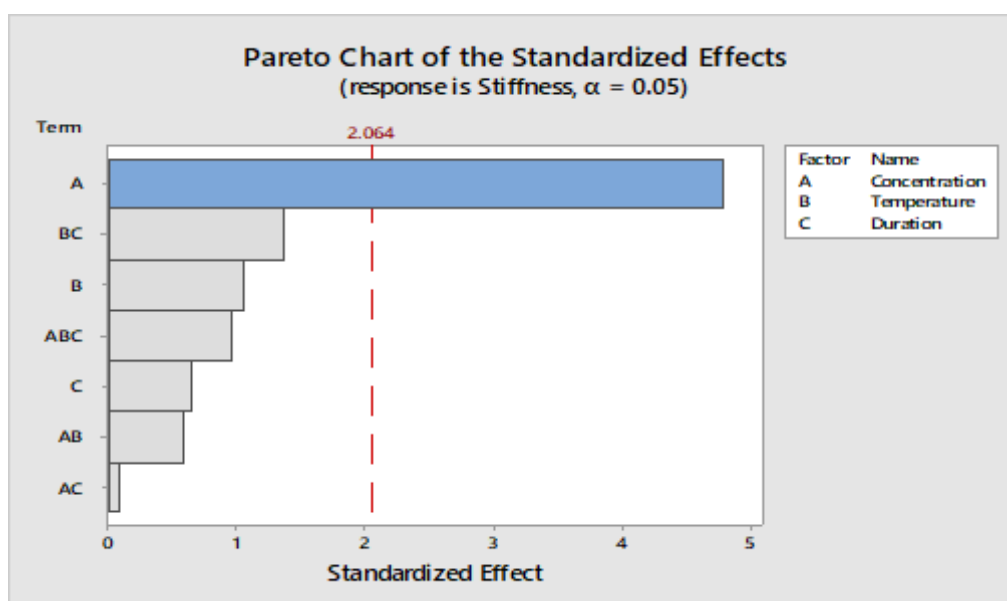


Figure 4.45 NaOH concentration exerted the highest influence on the Young's modulus properties of coir fibres.

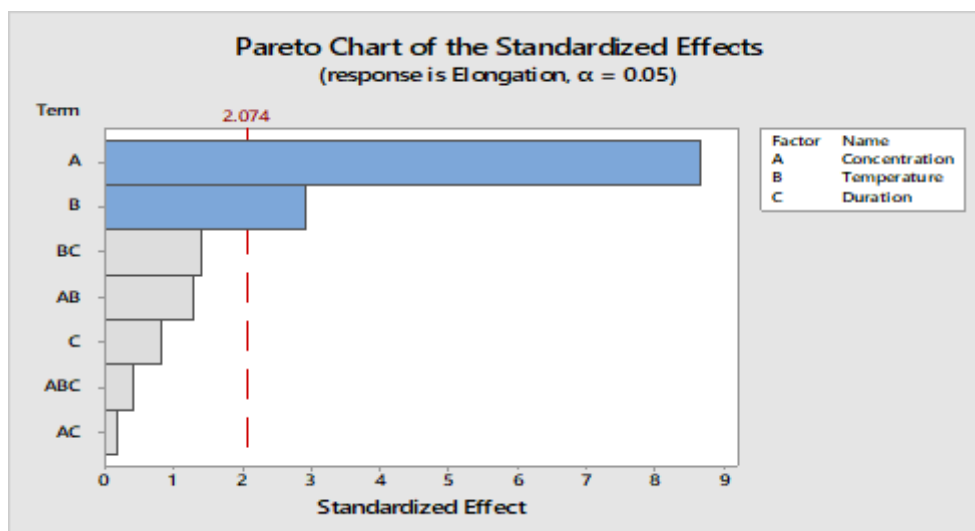


Figure 4.46 NaOH concentration exerted the highest influence on the elongation at break of coir fibres.

4.4.2.2 Weibull modulus of the NaOH-treated coir fibres

Table 4.7 (a-c) shows the Weibull distribution for the tensile strength obtained at temperature levels 30 - 80 °C. The Weibull shape parameter for the NaOH treated fibres fall within the range of 2.38 to 5.50. The lowest Weibull shape parameter 2.38 was at treatment conditions; 5% NaOH, at 50 °C for 48 hours. The Weibull modulus was affected by temperature and NaOH concentration. The Weibull distribution of the NaOH-treated fibres at NaOH concentration of 20%, temperature of 80 °C and at duration of 24 hours is shown in Figure 4.47. This displays the highest Weibull modulus of 5.50 out of the 27 treated samples. Weibull modulus varies as the NaOH treatment condition varies; this is represented in Figure 4.48.

Tables 4.7 (a-c) Weibull analysis for NaOH-treated coir fibres.

(a) Weibull analysis for NaOH-treated coir fibres at temperature level: 30 °C.

Factors: Experimental condition			Weibull Parameters		R ²
Concentration (%)	Temperature (°C)	Duration (Hours)	Shape parameter	Scale parameter (MPa)	
1	30	24	3.15	211.83	0.96
1	30	48	2.58	198.63	0.96
1	30	72	4.15	228.09	0.93
5	30	24	3.52	253.26	0.93
5	30	48	3.56	189.91	0.98
5	30	72	3.37	228.16	0.96
20	30	24	3.52	207.07	0.94
20	30	48	3.74	195.98	0.93
20	30	72	5.36	213.18	0.98

(b) Weibull analysis of NaOH-treated coir fibres at temperature level: 50 °C.

Factors: Experimental condition			Weibull Parameters		R ²
Concentration (%)	Temperature (°C)	Duration (Hour)	Shape parameter	Scale parameter (MPa)	
1	50	24	3.19	191.21	0.95
1	50	48	2.91	167.56	0.94
1	50	72	3.81	176.56	0.84
5	50	24	3.09	205.70	0.96
5	50	48	2.38	183.25	0.98
5	50	72	4.30	189.43	0.93
20	50	24	3.77	254.27	0.93
20	50	48	4.76	228.96	0.93
20	50	72	4.30	225.80	0.97

(c) Weibull analysis for NaOH-treated coir fibres at temperature level: 80 °C.

Factors: Experimental condition			Weibull Parameters		R^2
Concentration (%)	Temperature (°C)	Duration (Hour)	Shape parameter	Scale parameter (MPa)	
1	80	24	3.72	208.75	0.95
1	80	48	3.82	186.20	0.92
1	80	72	3.76	205.16	0.97
5	80	24	4.23	233.13	0.97
5	80	48	4.66	222.24	0.94
5	80	72	2.40	123.68	0.97
20	80	24	5.50	225.02	0.98
20	80	48	5.05	260.80	0.92
20	80	72	4.59	215.36	0.97

Shape parameter: 20%, 80 °C, 24 hours > 20%, 80 °C, 48 hours > 5%, 80 °C, 48 hours

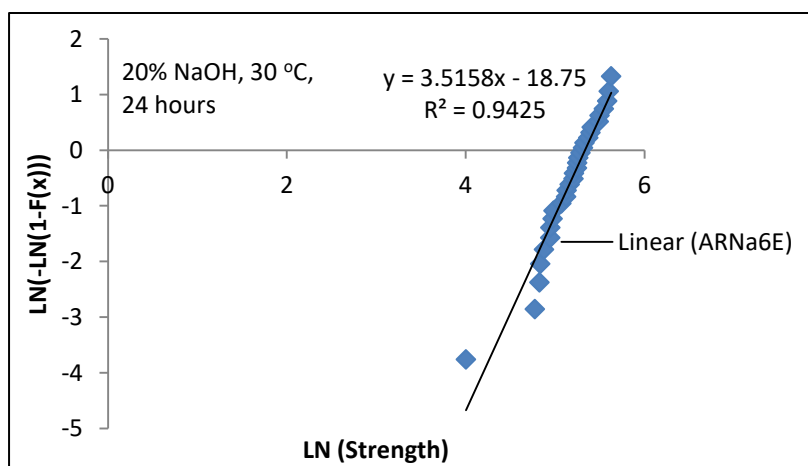


Figure 4.47 Weibull distributions of NaOH-treated coir fibres at 20% NaOH concentration, 30 °C and for duration of 24 hours.

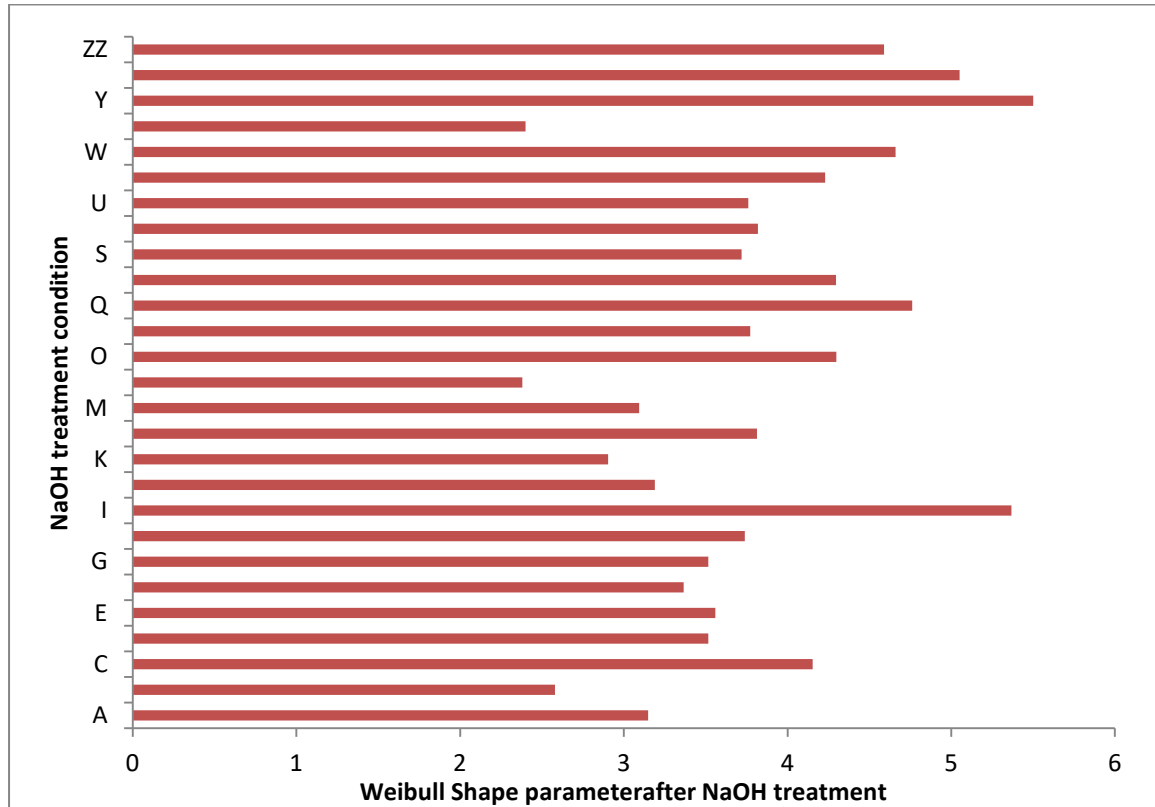
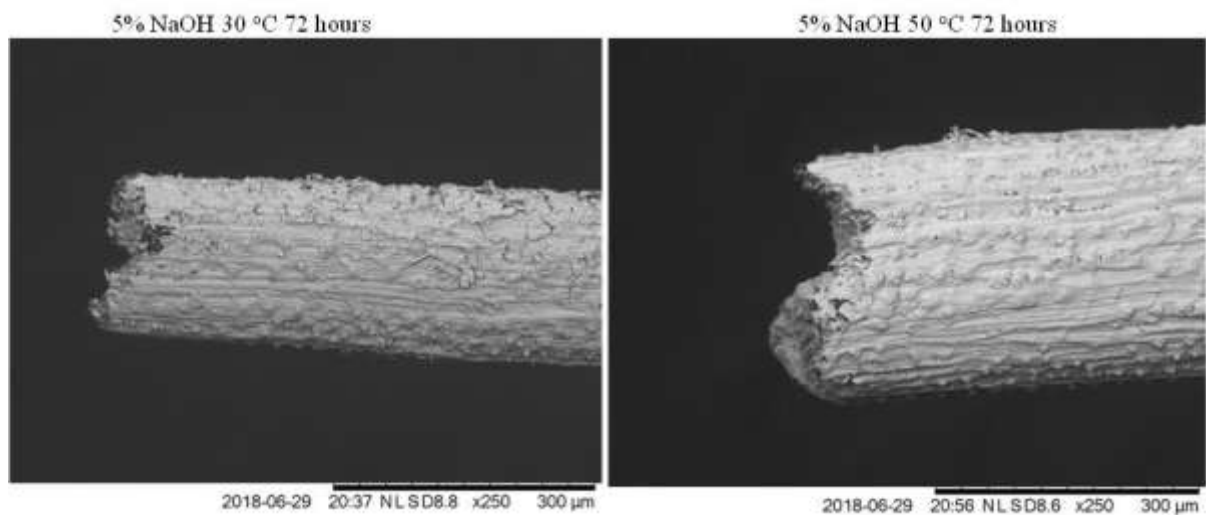


Figure 4.48 Weibull distributions of NaOH-treated coir fibres at 20% NaOH concentration, 30 °C and for duration of 24 hours.

4.4.3 Failure analysis of NaOH-treated coir fibres

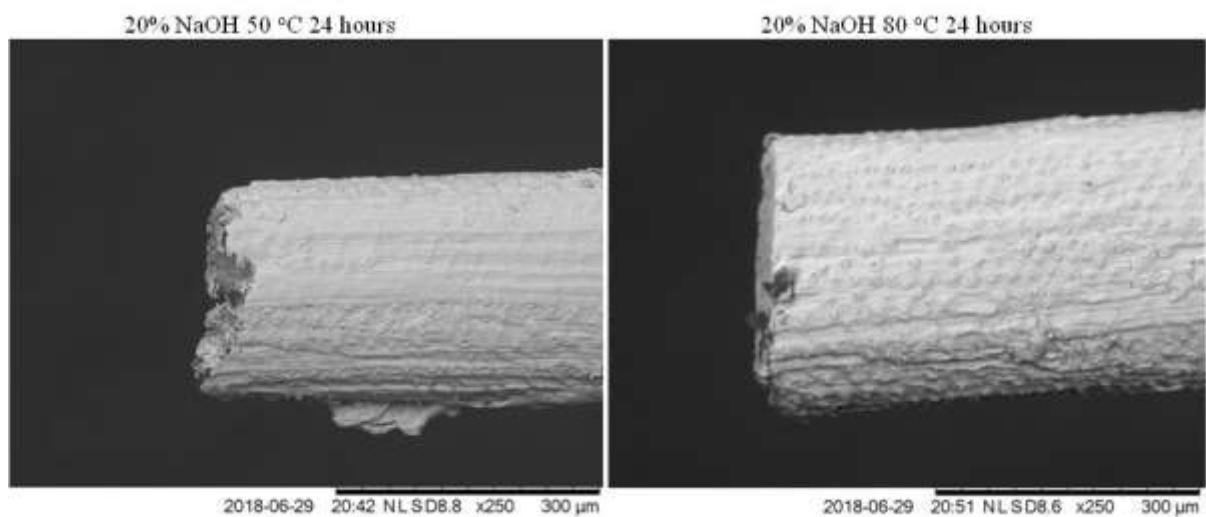
Different failure surfaces were observed with different treatment conditions as shown in Figure 4.49. The failure of the fibers appears to be uneven and irregular. Defects were observed on the failed surfaces. Elongation of the helical structures after brittle failure of the fibres was observed. Pull-out of the cellulose fibril is evident on the failed surfaces.



(i)

(ii)

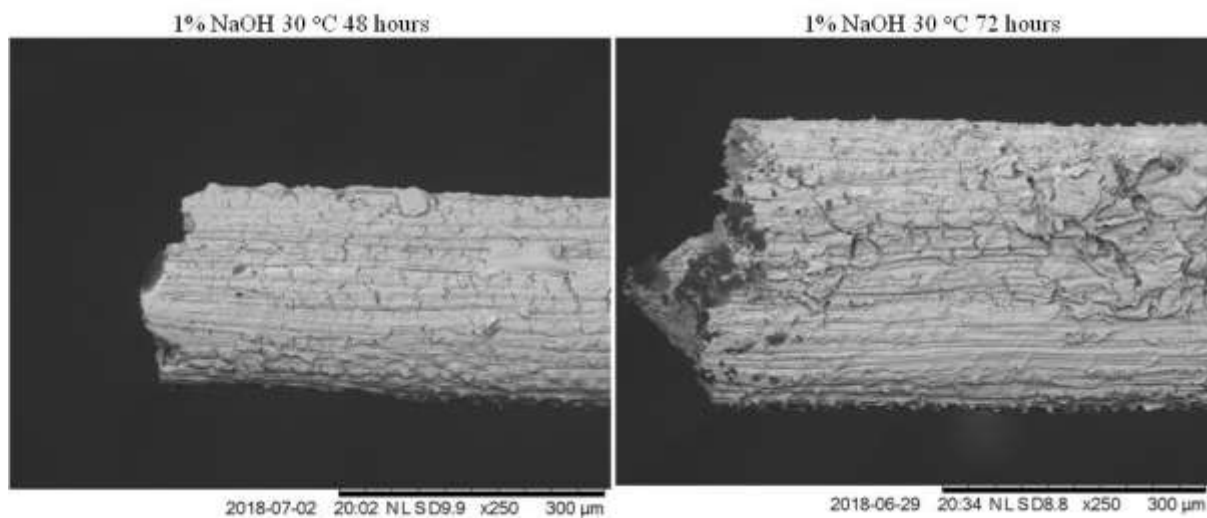
(a) Fracture surface of NaOH treated coir fibres at different temperatures of (i) 30 and (ii) 50 °C but at the same duration of 72 hours and NaOH concentration of 5%.



(i)

(ii)

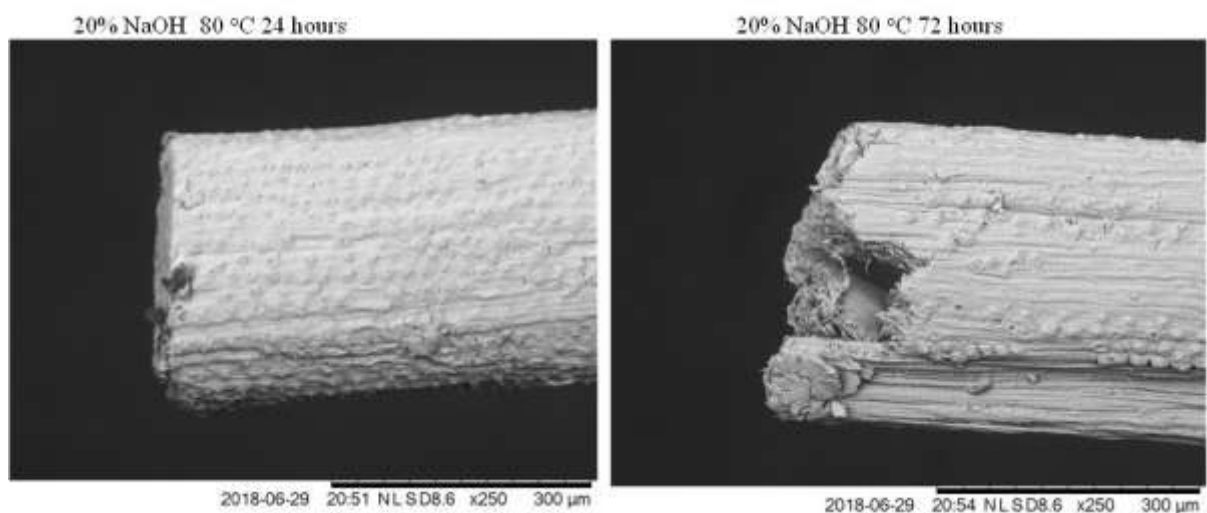
(b) Fracture surfaces of NaOH treated coir fibres at temperatures of (i) 50 and (ii) 80 °C but at the same duration of 24 hours and NaOH concentration of 20 % .



(i)

(ii)

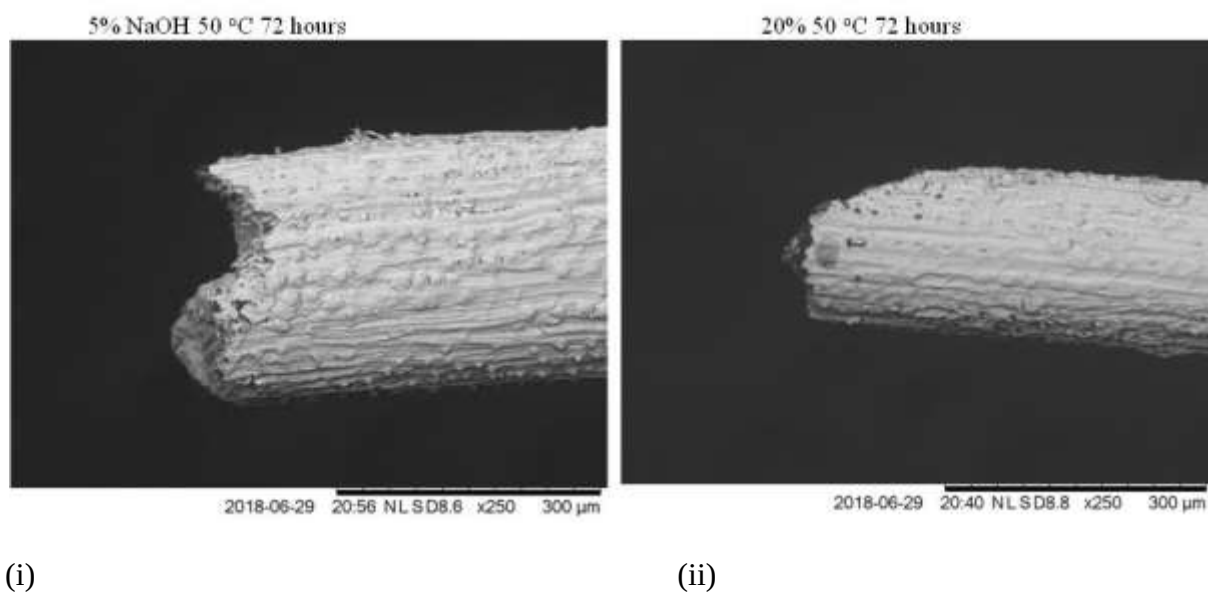
(c) Fracture surfaces of NaOH treated coir fibres at a NaOH concentration of 1%, temperature of 30 °C but at different durations of (i) 48 and (ii) 72 hours.



(i)

(ii)

(d) Fracture surfaces of NaOH treated coir fibres at a higher concentration and temperature of 20% and 80 °C respectively but at different durations of (i) 24 and (ii) 72 hours.



(e) Fracture surfaces of NaOH-treated coir fibres at different concentrations of (i) 5 and (ii) 20 % NaOH concentration but at the same temperature and duration of 50 °C and 72 hours.

Figure 4.49 (a-e) Failed surfaces of NaOH treated coir fibres at different NaOH treatment conditions.

4. 4. 4 Weight losses of coir fibres after NaOH treatment

A loss in weight of as-received coir fibres was observed after NaOH treatment at all treatment temperature levels as shown in Figure 4.50. The weight loss increases as the treatment temperature increases. Therefore the least weight loss was at treatment temperature of 30 °C. A minimum weight loss of 6.99 % occurred at treatment conditions of 1% NaOH concentration at 30 °C for 24 hours.

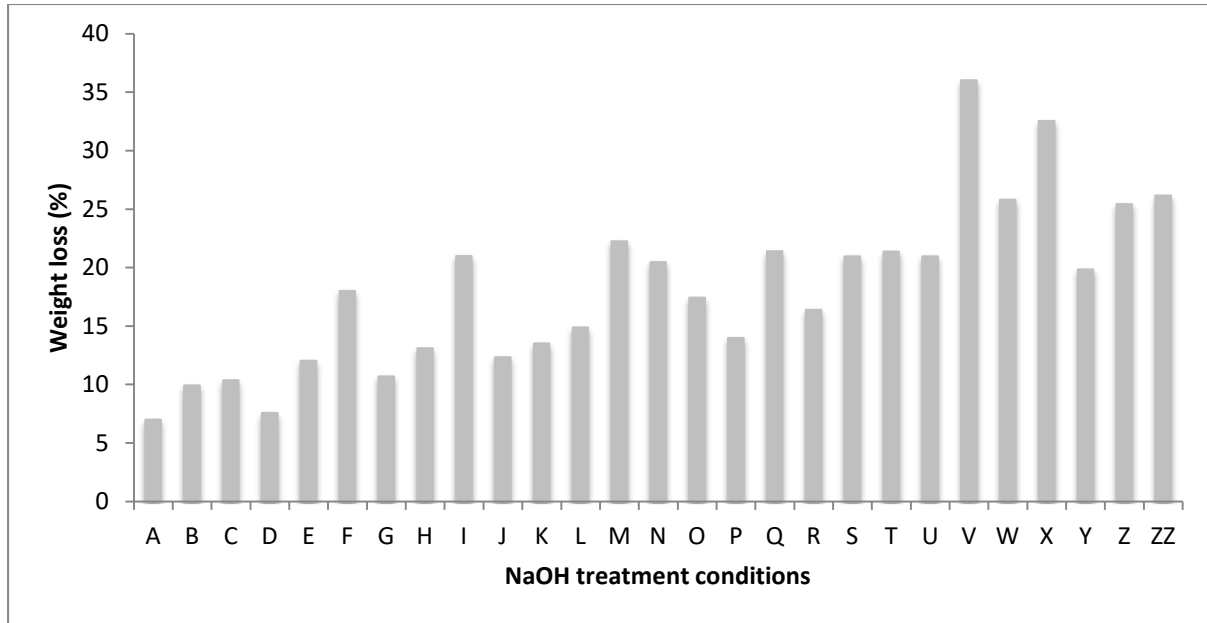


Figure4.50 Bar charts showing the weight losses of as-received coir fibres after NaOH treatment.

4.5 Hot-compaction of coir fibre

Compaction of single coir fibres using hot press so far has not been reported in the literature. However, compaction has been applied to control the porosity of soil, rendering the soil more compact with enhanced strength. Strength has been related to the porosity using the following equation according to Barralet, *et al.*, (2001);

$$\sigma_{P_o} = \sigma_t \exp(-KP) \quad [4.3]$$

where the strength at a given porosity is σ_{P_o} , σ_t is the maximum theoretical strength of the material, K is a constant, P is porosity.

The compaction was carried out on the unidirectionally aligned as-received and water treated coir fibres. The number of experiments carried out and the treatment conditions for the as-received and water treated coir fibres were the same.

In order to determine the optimum compaction conditions that would yield the best mechanical properties, the experiment was designed with temperature, pressure and duration as variables using ANOVA on Minitab 19 Software. The influence of compaction pressure, temperature and duration on the mechanical properties microstructure and porosity of as-received coir fibres under 12 different experimental conditions was characterised. The number of samples per treatment condition was 30.

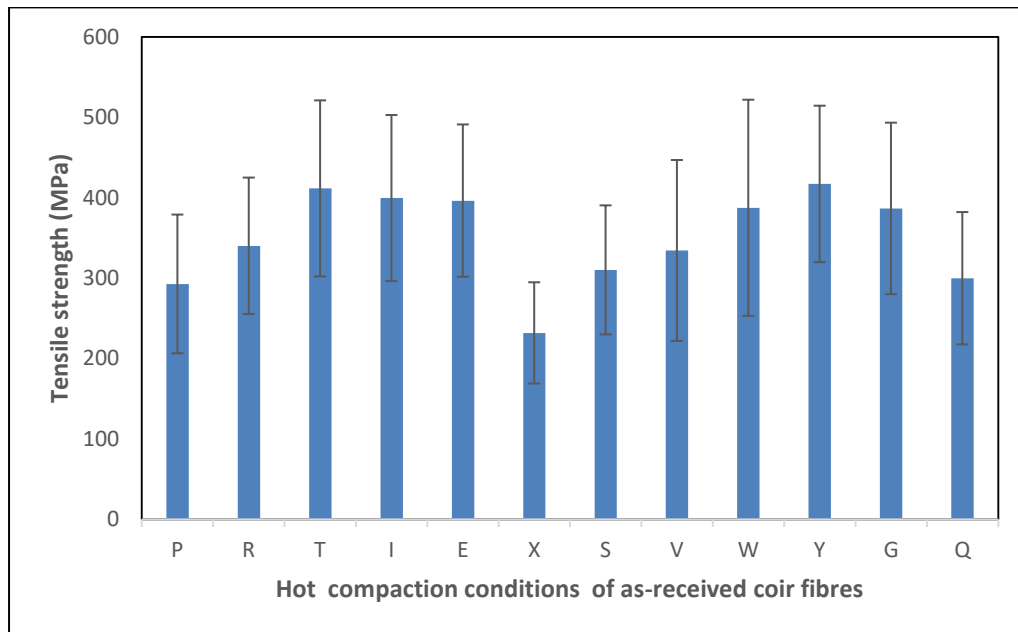
4.5.1 Tensile properties of hot-compacted coir fibres

The average tensile properties of hot compacted coir fibres per condition are given in Table 4.8. Figure 4.51 (a and b) shows the effects of hot compaction on the tensile properties of coir fibres. The tensile strengths of the hot compacted samples were found to be exceptionally high. The highest tensile strength of 417 MPa was obtained at a compaction pressure of 6 MPa, a temperature of 140 °C and at duration of 5 minutes. These values obtained have not been reported for tensile strength of coir fibre in the literature. Further analysis of the results was carried out using ANOVA via F-tests and T-tests to determine their statistical significance. The P-values for strength (0.006) and stiffness (0.000) were found to be less than 0.05 showing that tensile properties are influenced by hot-compaction experimental conditions.

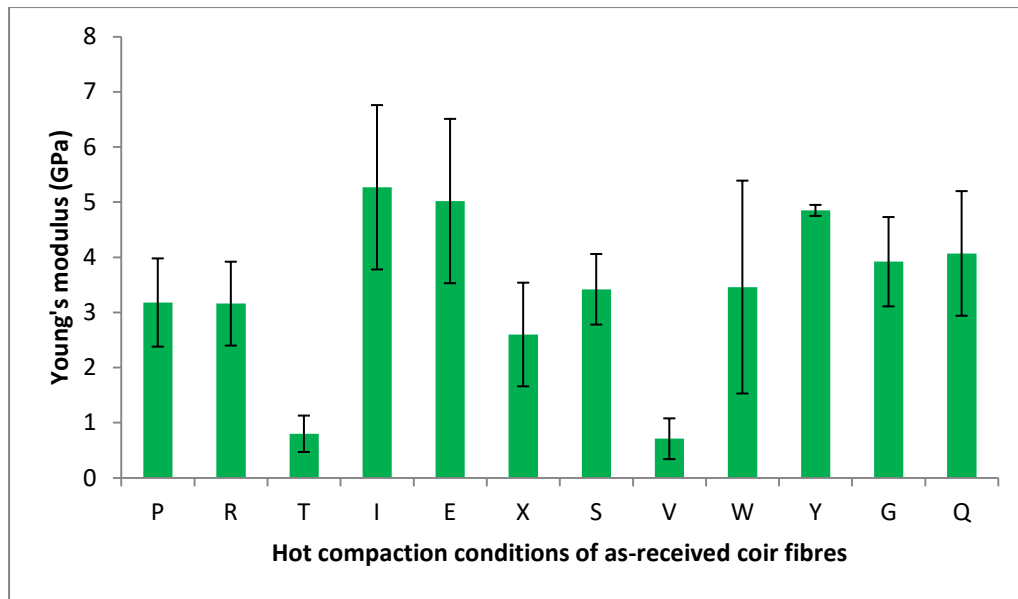
Table 4.8 Tensile test result hot-compacted coir fibres.

SC	Factors: Experimental conditions			diameter (mm)	Tensile properties after hot compaction	
	Pressure	Temp	Duration		Strength	Young's modulus
	(MPa)	(°C)	(Minutes)		(MPa)	(GPa)
P	4	70	5	0.19 ± 0.02	292.64 ± 86.22	3.18 ± 0.80
R	4	70	15	0.18 ± 0.02	340.00 ± 84.85	3.16 ± 0.76
T	4	70	30	0.16 ± 0.02	411.43 ± 109.46	0.80 ± 0.33
I	4	140	5	0.17 ± 0.02	399.42 ± 103.25	5.27 ± 1.49
E	4	140	15	0.16 ± 0.02	396.22 ± 94.73	5.02 ± 1.49
X	4	140	30	0.20 ± 0.02	231.73 ± 62.94	2.60 ± 0.94
S	6	70	5	0.20 ± 0.02	310.04 ± 80.27	3.42 ± 0.64
V	6	70	15	0.17 ± 0.03	334.19 ± 112.53	0.71 ± 0.37
W	6	70	30	0.17 ± 0.03	387.35 ± 134.38	3.46 ± 1.93
Y	6	140	5	0.17 ± 0.02	416.97 ± 97.30	4.85 ± 0.10
G	6	140	15	0.19 ± 0.02	386.54 ± 106.63	3.92 ± 0.81
Q	6	140	30	0.17 ± 0.02	299.70 ± 82.27	4.07 ± 1.13

SC=Sample code; Temp= Temperature



(a)

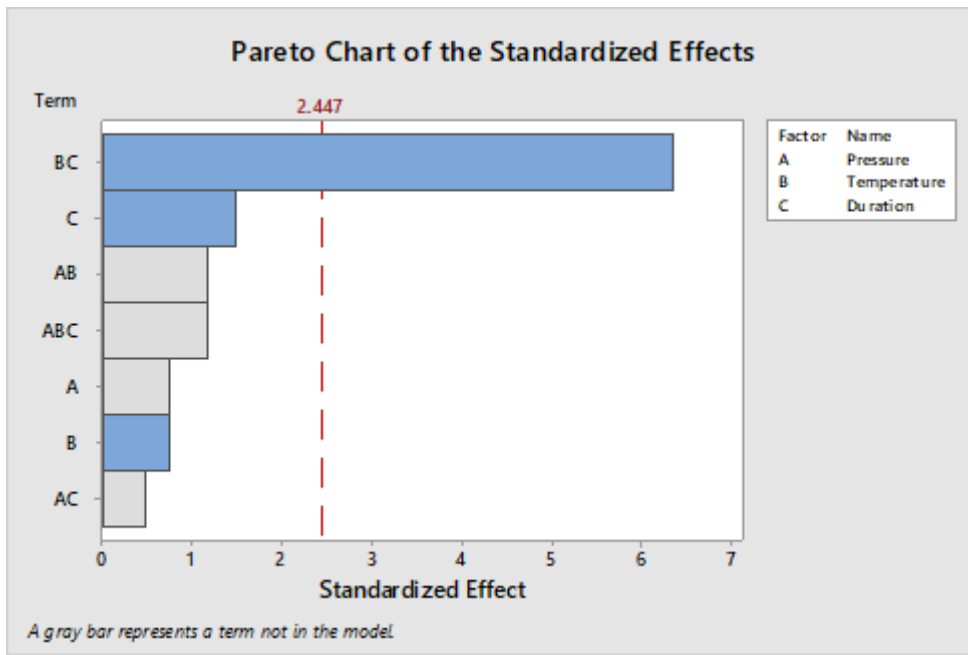


(b)

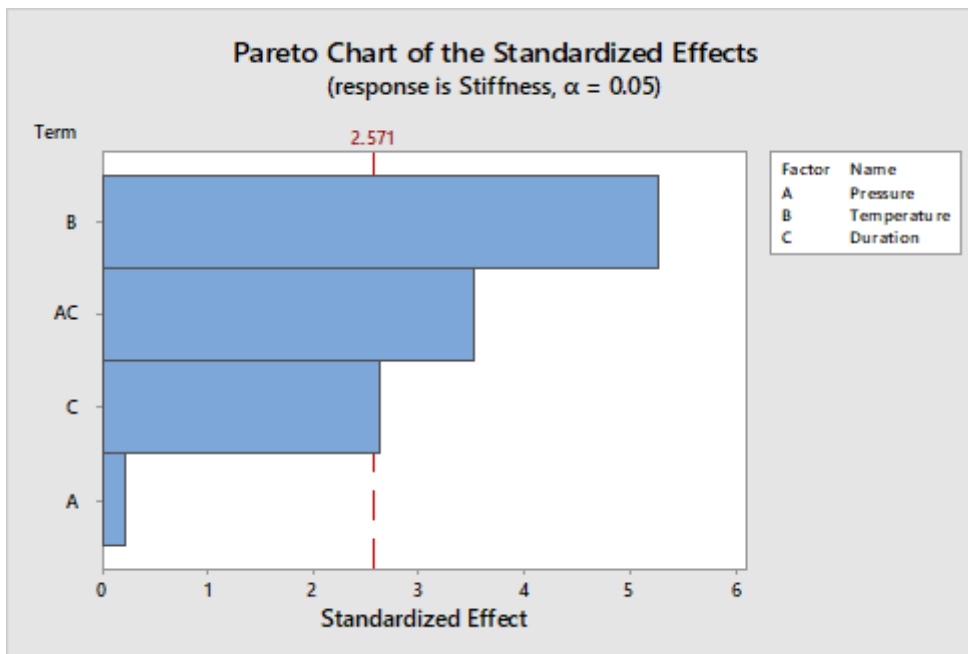
Figure 4.51 Effect of hot compaction on the tensile properties of coir fibres (a) strength and (b) Young's modulus.

4.5.1.1 Factorial regression models for the tensile properties of hot compacted coir fibres

From the ANOVA and the Pareto Chart displayed in Figure 4.52(a) the interaction of temperature and duration is statistically significant for the strength at 85% confidence level. As a result only the temperature and duration appeared in the regression model equation for strength. The interaction of temperature and duration exerted the greatest impact on the strength properties of the as-received compressed coir fibres. However for stiffness, temperature, duration and the interaction of pressure and duration are statistically significant at 95% confidence level as shown in Figure 4.52(b) but the temperature was responsible for the greatest influence on the stiffness of the as received compacted coir fibres .



(a)



(b)

Figure 4.52 Pareto Charts for the hot compacted coir fibres (a) for strength at $\alpha=0.15$ and (b) stiffness at $\alpha=0.05$.

4.5.1.2 Weibull Distribution of hot compacted coir fibres

The Weibull results of the hot compacted coir fibres are shown in Table 4.9. The strength has been related to the porosity as shown in Equation [4.3];

$$\sigma_{p_o} = \sigma_t \exp(-KP) \quad [4.3]$$

For Equation [4.3], the critical flaws of size c are not taken into consideration. These critical flaws are related to strength, σ_p , modulus E , and fracture energy R as given by (Barralet *et al.*, 2001) in Equation [4.4]:

$$\sigma_p = \left(\frac{ER}{\pi c}\right)^{0.5} \quad [4.4]$$

That is

$$\sigma_p = (ER/\pi c)^{0.5}$$

Therefore the ultimate strength at zero porosity σ_{p_o} is a function of the flaw size c inherent in the fibre by substituting Equation [4.4] into [4.3]

$$\left(\frac{E_0 R}{(\pi c)}\right)^{0.5} = \sigma_{p_o} \exp(-KP) \quad [4.5]$$

E_0 is the modulus at zero porosity.

The increase in Weibull modulus as shown in Table 4.9 shows that compaction pressure brings about reduction in the flaw size of the as received compacted coir fibres. Hence Increase in tensile strength and Weibull modulus indicates decrease in both porosity and flaw size.

Table 4.9 Weibull Distribution of the hot-compacted coir fibres.

Hot press experimental condition (AR)			Weibull Parameters		R2
Pressure (MPa)	Temperature (°C)	Duration (minutes)	Modulus	Scale (MPa)	
4	70	5	3.71	324.96	0.94
4	70	15	4.24	374.48	0.97
4	70	30	4.27	451.89	0.98
4	140	5	4.91	431.7	0.87
4	140	15	4.59	437.39	0.87
4	140	30	3.13	263.06	0.88
6	70	5	4.16	341.63	0.97
6	70	15	3.3	372.53	0.97
6	70	30	2.83	437.7	0.98
6	140	5	4.38	469.19	0.94
6	140	15	4.33	424.04	0.95
6	140	30	3.91	331.57	0.97

4.5.1.3 Microstructure of the as-received hot-compacted coir fibres

The microstructure of hot compacted coir fibre is shown in Figure 4.53. The reduction in the pore size and the lacuna is clear especially when compared with the untreated fibres. A significant difference was observed in the porosities of the untreated and hot compacted coir fibres. However, no significant difference was observed in the porosities of NaOH-treated and compacted coir fibres.

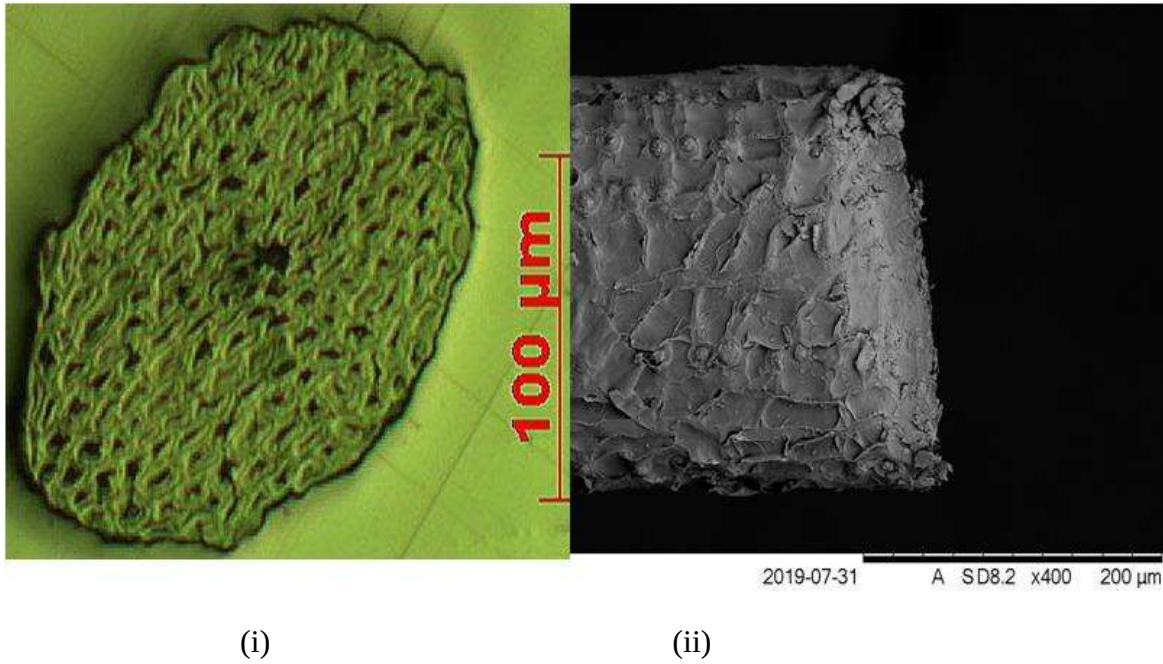
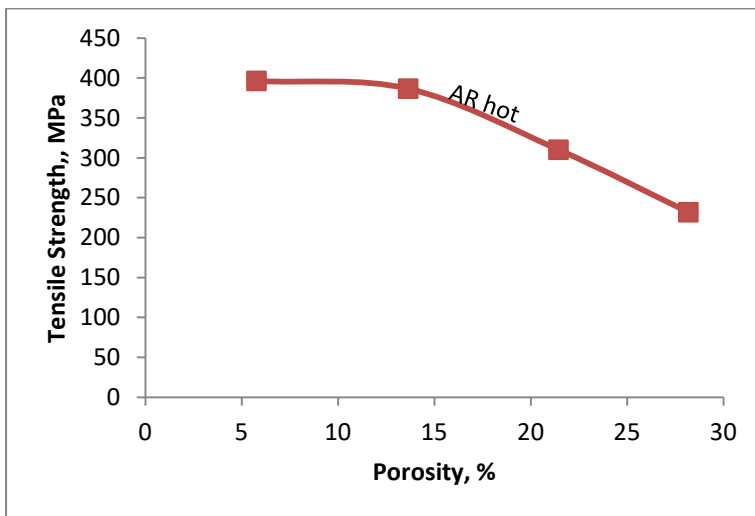


Figure 4.53 Microstructures of as-received hot-compacted coir fibre.

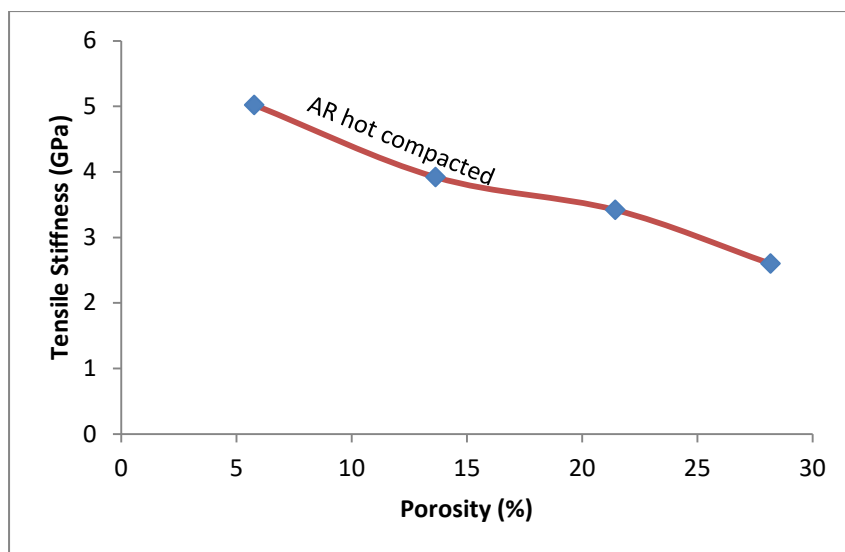
The relationship between the tensile properties and the porosity of hot compacted coir fibres are shown in Figure 4.54 (a and b), where the tensile strength and stiffness of the fibre increase as the porosity decreases. The strength has been related to the porosity as previously shown in Equation [4.3];

$$\sigma_{P_o} = \sigma_t \exp(-KP) \quad [4.3]$$

where the strength at a given porosity is σ_{P_o} , σ_t is the maximum theoretical strength of the material, K is a constant, P is porosity.



(a)



(b)

Figure 4.54 Relationship between (a) the tensile strength, (b) the stiffness and the porosity of hot compacted as-received coir fibre.

4.5.1.4 Failure analysis of AR hot-compacted coir fibres

The as-received hot compacted coir fibres displayed a brittle failure as shown in Figure 4.55. The failure curves were rather smooth. The failure traces of the high and mid ranges were similar although failure occurred at different elongation points. No significant difference was observed between the high and mid failure stresses. However the maximum observed failure stress was 60% higher than the minimum range but this minimum range represents only an insignificant fraction of 0.1 of the total samples tested. Therefore the minimum range could have been anomalies or as a result of significant defects present in those fibres.

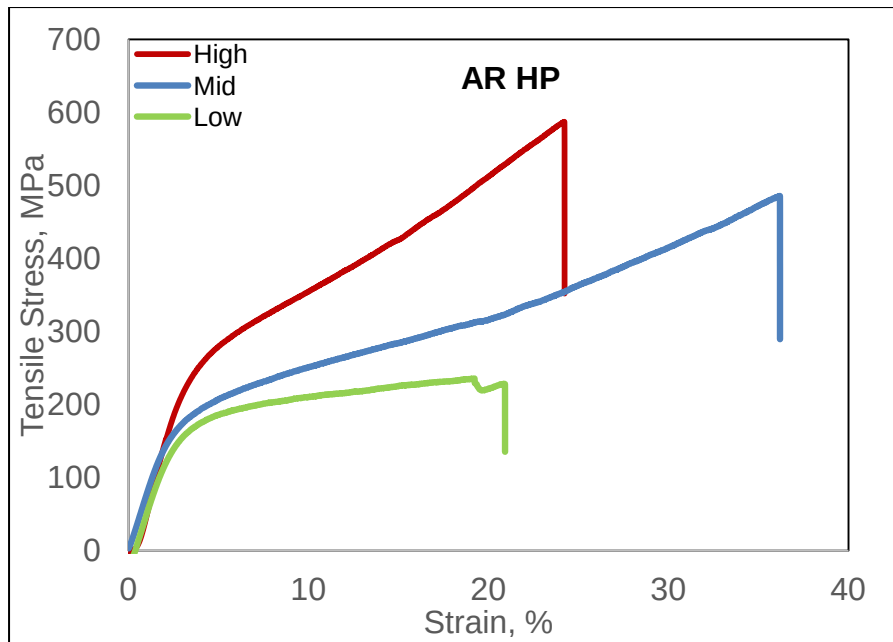


Figure 4.55 Failure traces of as-received hot compacted coir fibres.

4.5.2 Hot-compaction of water-treated coir fibres

30 as-received coir fibres of average diameter 0.231mm were immersed in water at 80 °C for 24 hours. The unidirectionally aligned water-treated coir fibres were compacted using the same conditions as in as-received fibres above.

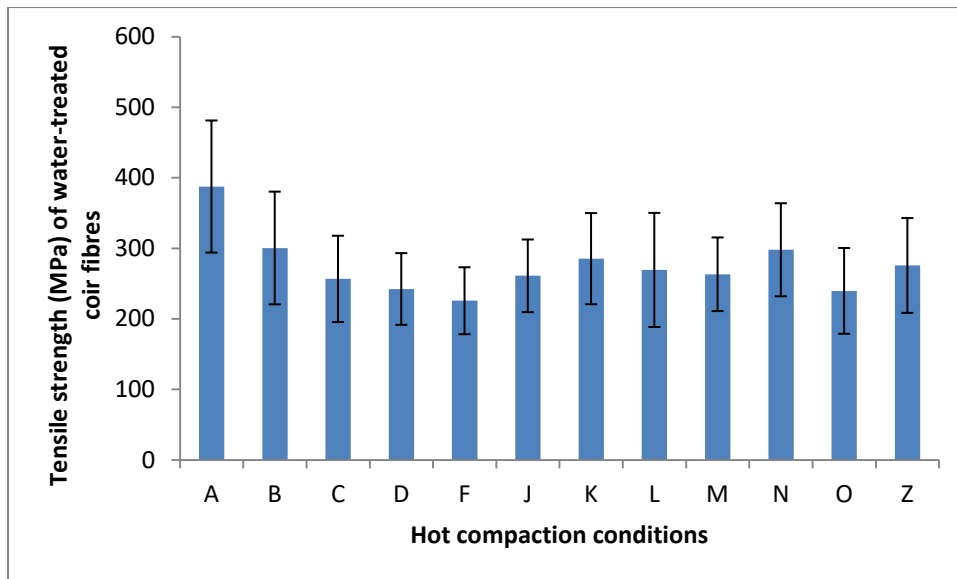
4.5.2.1 Tensile properties of water-treated hot-compacted coir fibres

The tensile properties of water-treated hot compacted coir fibres are shown in Table 4.10. The highest tensile strength and modulus were obtained at treatment conditions A as shown in Figure 4.56 (a) and (b). The average increase in diameter of the as-received coir fibres after water-treatment was found to be 2.75% after immersion in water at 80 °C for 24 hours.

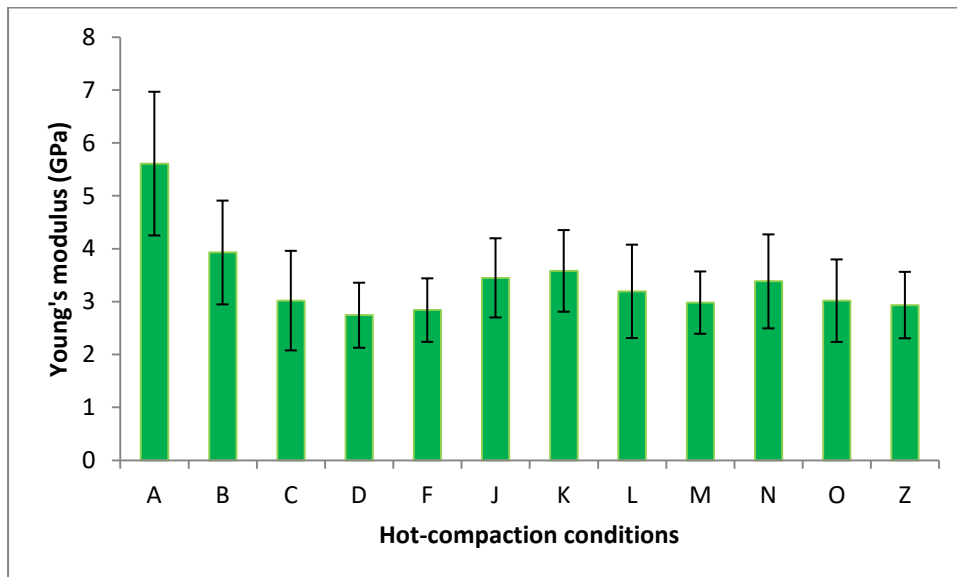
Table 4.10 Tensile properties of water-treated hot compacted coir fibre.

SC	Experimental Conditions: Water-treated			Diameter		Tensile properties after hot compaction	
	Pr	Te	Du	Initial	Final	Tensile strength	Young's modulus
	(MPa)	(°C)	(Ms)	(mm)	(mm)	(MPa)	(GPa)
K	4	70	5	0.25 ± 0.04	0.18 ± 0.03	285.32 ± 64.61	3.58 ± 0.77
Z	4	70	15	0.25 ± 0.04	0.18 ± 0.02	275.65 ± 67.21	2.93 ± 0.63
L	4	70	30	0.25 ± 0.04	0.18 ± 0.02	269.30 ± 80.90	3.19 ± 0.88
F	4	140	5	0.25 ± 0.03	0.19 ± 0.03	225.67 ± 47.46	2.84 ± 0.60
D	4	140	15	0.26 ± 0.05	0.19 ± 0.02	242.33± 50.82	2.74 ± 0.61
J	4	140	30	0.26 ± 0.05	0.18 ± 0.03	260.99 ± 51.51	3.45 ± 0.75
N	6	70	5	0.25 ± 0.04	0.17 ± 0.03	297.2 ± 65.96	3.38 ± 0.89
O	6	70	15	0.24 ± 0.04	0.18 ± 0.03	239.61 ± 60.82	3.02 ± 0.78
M	6	70	30	0.25 ± 0.03	0.18 ± 0.02	263.21 ± 52.20	2.98 ± 0.59
C	6	140	5	0.24 ± 0.04	0.18 ± 0.02	256.63 ± 61.15	3.02 ± 0.94
B	6	140	15	0.27 ± 0.04	0.16 ± 0.02	300.44 ± 79.90	3.93 ± 0.98
A	6	140	30	0.26 ± 0.04	0.16 ± 0.02	387.59 ± 93.63	5.61± 1.36

Pr= Pressure, Ms=minutes, Te= Temperature



(a)



(b)

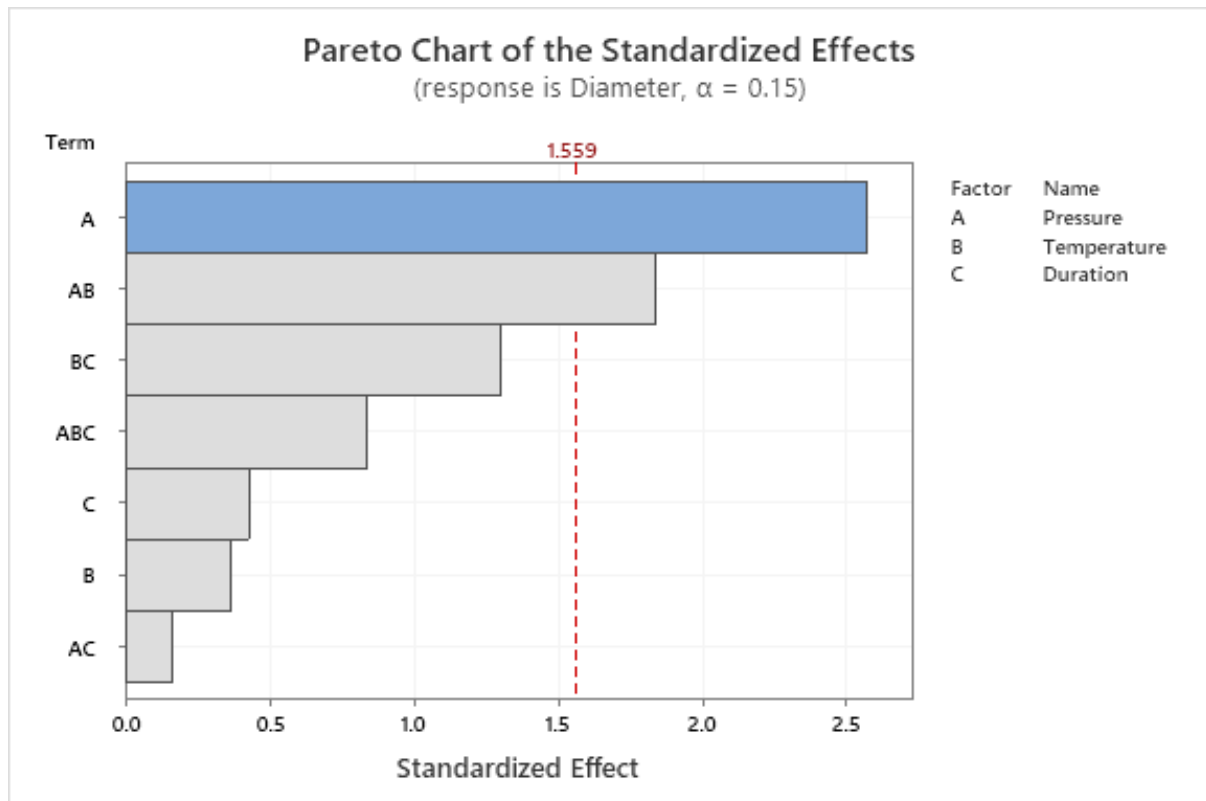
Figure 4.56 Tensile properties of water-treated hot compacted coir fibres for; (a) Strength (b) Stiffness.

4.5.2.1.1 Factorial regression models for the water-treated hot compacted coir fibres for diameter, strength and stiffness

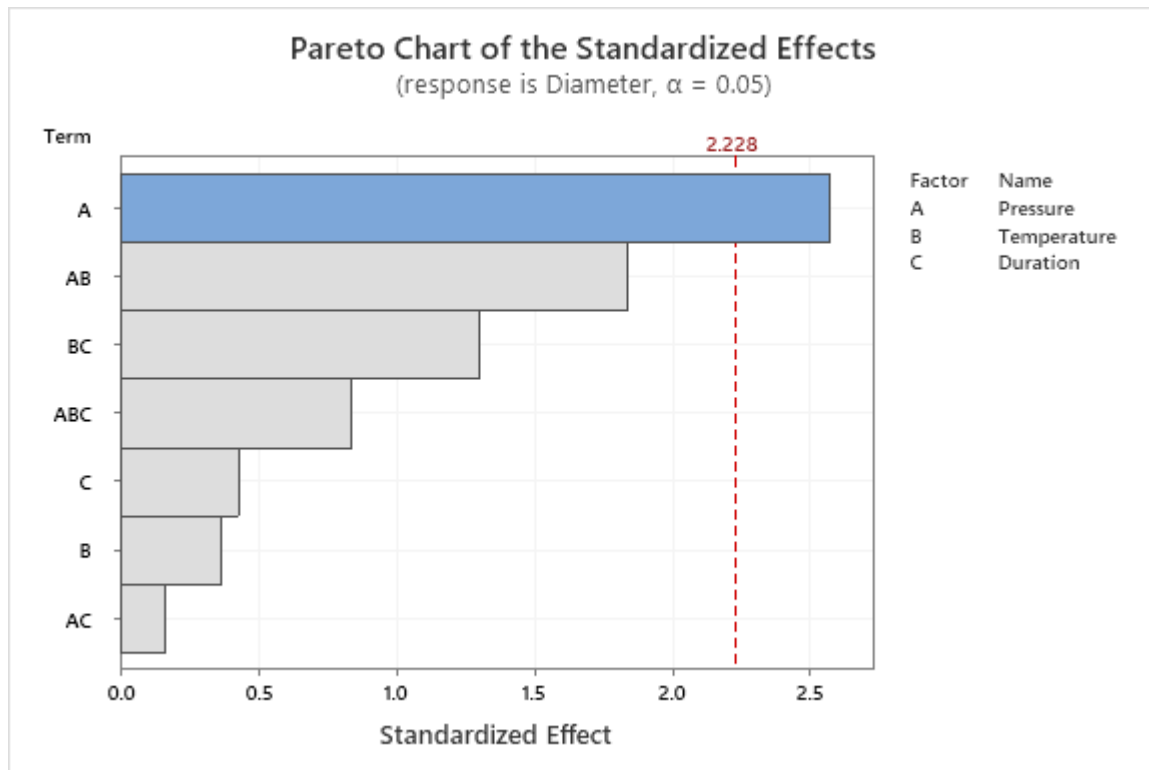
Factorial regression using ANOVA on Minitab 19 Software was carried out in order to determine the impact of the variables on the identified treatment parameter.

4.5.2.1.1.1 Diameter versus Pressure, Temperature and Duration at 85 % and 95 % confidence levels for the water-treated hot-compacted coir fibre

From Figure 4.57 (a); Pressure, temperature and the interaction of pressure and temperature made statistically significant impact on the diameter of the water treated hot pressed coir but not the duration. Pressure exerted the maximum influence on the diameter at 85% confidence level. However at 95% confidence level, only pressure had a significant influence on the diameter as shown in Figure 4.57 (b).



(a)



(b)

Figure 4.57 Pareto Chart for Diameter versus Pressure Temperature and Duration at (a) 85% and (b) 95% Confidence levels.

4.5.2.1.1.2 Strength versus Pressure, Temperature and Duration at 75% Confidence level

The significant parameters affecting the strength of the water treated hot-compacted coir fibres are pressure, the interactions of pressure and temperature and the interactions of temperature and duration at 75% confidence level as shown in Figure 4.58. The interaction of pressure and temperature exerted the highest impact.

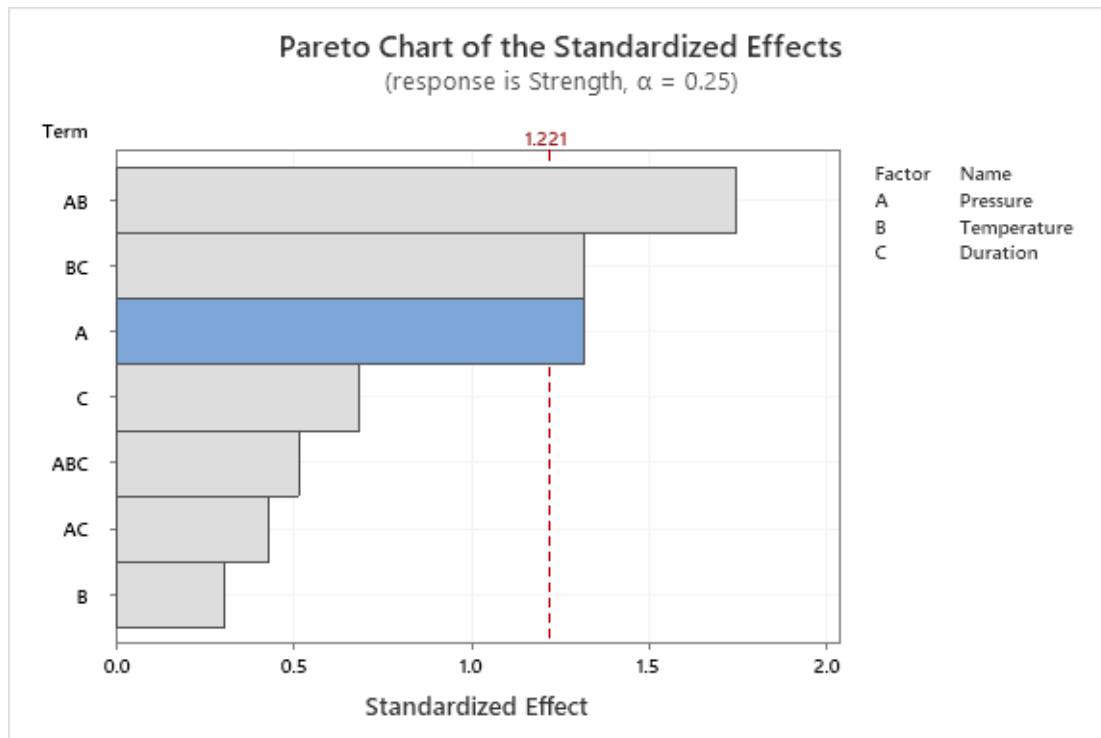


Figure 4.58 Pareto Chart for the strength of water treated hot compacted coir fibre at 75% confidence level.

4.5.2.1.1.3 Stiffness versus Pressure, Temperature and Duration at 70% confidence level

The interactions of pressure and temperature and duration as well as the interactions of pressure and temperature exerted significance influence on the stiffness of water treated hot compacted coir fibres as shown in Figure 4.59.

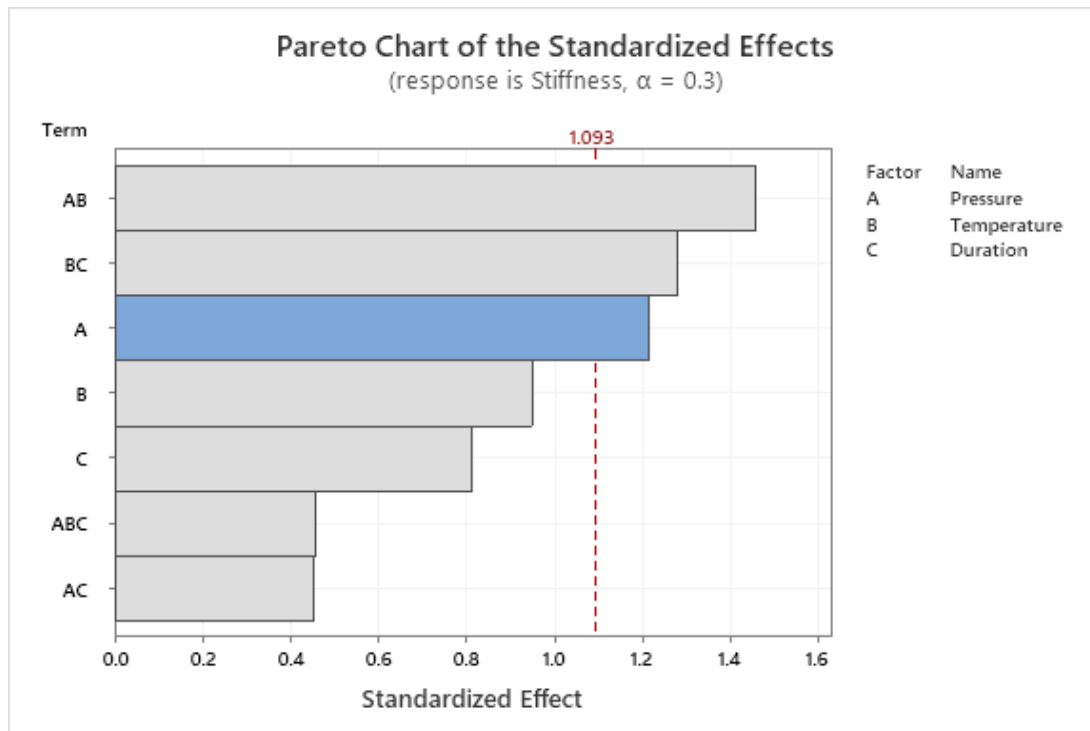


Figure 4.59 Pareto Chart for the stiffness of water-treated hot compacted coir fibre at 70% confidence level.

4.5.2.2 Weibull analysis of water-treated hot-compacted coir fibres

The water-treated compacted coir fibres show a very high Weibull modulus as shown in Table 4.11. The Weibull scale parameter increases with increase in temperature, pressure and duration. The highest shape parameter obtained was at treatment condition M.

Table 4.11 Weibull parameters of water-treated hot-compacted coir fibres.

SC	Hot press experimental condition (Water-treated)			Weibull Parameters		R2
	Pressure	Temperature	Duration	Shape	Scale	
	(Ton)	(°C)	(minutes)		(MPa)	
A	6	140	30	4.85	422.59	0.94
B	6	140	15	4.38	329.39	0.96
C	6	140	5	5.1	279.03	0.89
D	4	140	15	5.14	263.47	0.98
F	4	140	5	5.3	244.81	0.98
J	4	140	30	5.65	282.09	0.97
K	4	70	5	5.15	310.18	0.91
L	4	70	30	3.23	302.87	0.92
M	6	70	30	5.98	283.71	0.87
N	6	70	5	5.34	323.23	0.89
O	6	70	15	4.57	262.06	0.97
Z	4	70	15	4.47	302.36	0.94

4.5.2.3 Microstructure water-treated hot-compacted coir fibres

The microstructure of the water treated hot compacted coir fibres shows a reduction in porosity as shown in Figure 4.60 when compared with untreated. The difference in porosity between the as-received and the water-treated hot compacted coir fibres is yet to be determined.

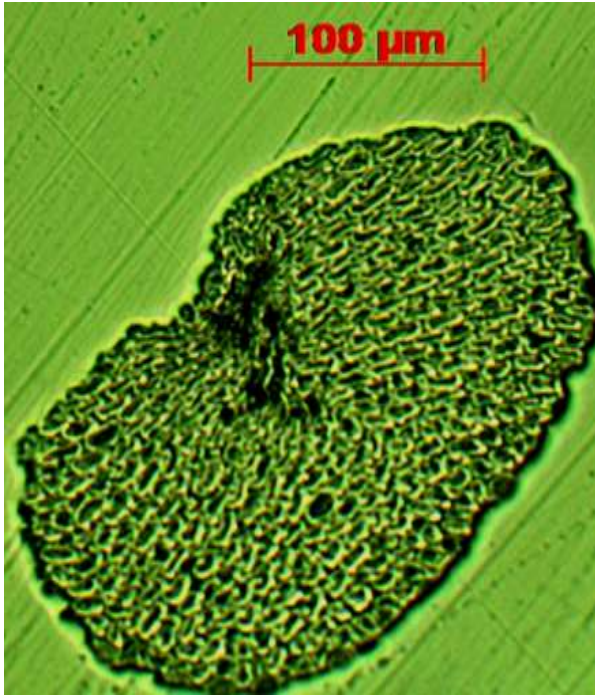


Figure 4.60 Microstructure of water-treated hot-compacted coir fibre at a compaction pressure of 6 MPa, temperature of 140 °C and duration of 15 minutes.

4.5.2.4 Tensile test graphs of water-treated hot-compacted coir fibres

The failure traces of water-treated compacted coir fibre showed smooth surfaces without load drops, an initial elastic region was followed by an extended uniform plastic region before fracture occurred as shown in Figure 4.61. However the initial linear region increases with increasing failure stress. The maximum failure stress was only 14% higher than the mid failure stress but 33% higher than the minimum failure stress. However the sample with the minimum failure stress displayed significantly higher elongation than the samples with the maximum and mid failure stresses. This suggests that the difference in the failure stress is as a result of the differences in the diameters of the fibres.

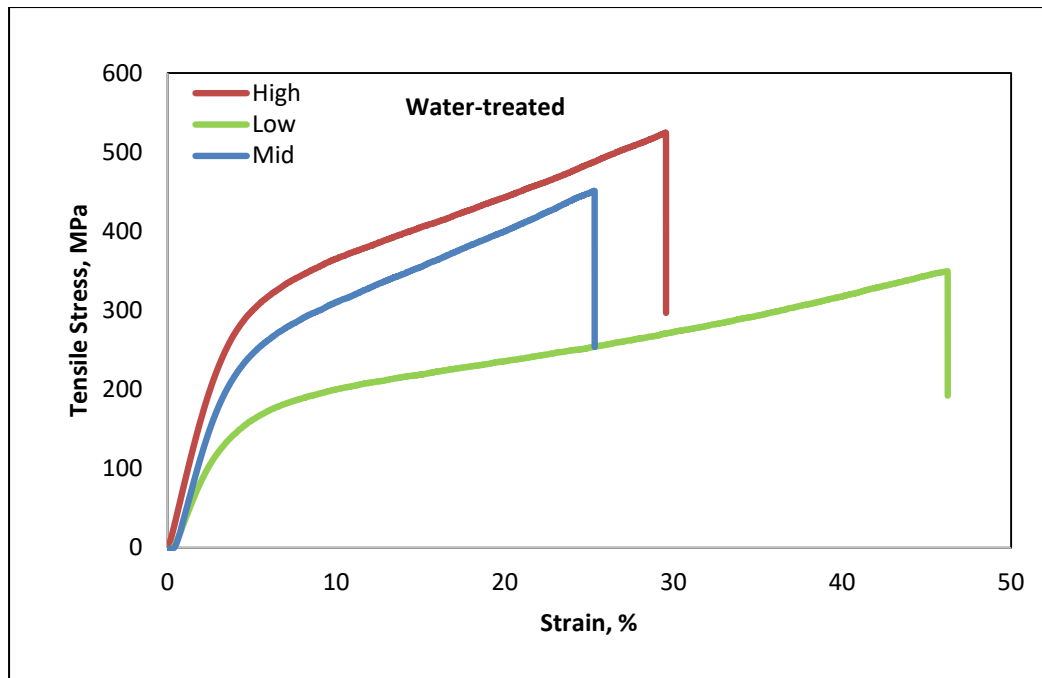


Figure 4.61 Failure traces of water-treated hot compacted coir fibres.

Summary

The optimum compaction pressure and temperature for the as-received and water treated hot-compacted coir fibres are the same; 6 MPa and 140 °C respectively, however the dwell time varies. The dwell time for the as received is lower than that of the water treated, this perhaps is as a result of the effects of water on the processing conditions and needs to be investigated. Dwell time has been observed to have a minor influence on the tensile properties of compacted materials (Swolfs *et al.*, 2014).

There is no significant difference in the strength and stiffness of the as received and water treated hot compacted coir fibres. The two display a deviation from linearity at about the same point followed by a long period of deformation during which there was a slow but uniform increase in stress and then a sharp failure without any load drops as shown in Figure 4.62. The difference in the diameters of the compacted AR and that of water-treated at optimum conditions was found to be 0.01. With the water-treated, additional factors control the transfer of properties. The compacted coir fibres have been found to possess exceptionally high mechanical properties. Investigations showed that this is as a result of reduction in the pore size and hence the porosity and as a result of reduction in the fibre diameter.

The peak in the Young's modulus as a result of increased temperature has been reported in literature on hot compaction of synthetic fibres. The hot compaction brings about thermal stability of the oriented structure hence higher temperature led to increase in tensile strength, and stiffness. At lower temperature, the pores are not sufficiently compacted but as the temperature increases the pore sizes are reduced.

The findings as obtained here are the first of their kind as regards compaction of coir fibres under the given conditions.

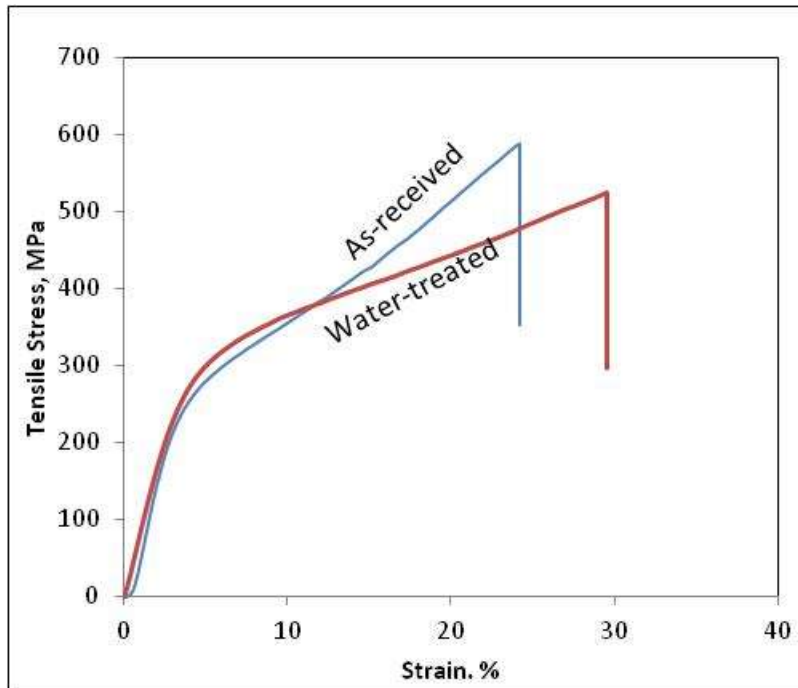


Figure 4.62 Stress-strain curves for AR and water treated samples at optimum compaction conditions.

4.6 Heat treatments of coir fibres

The as-received coir fibres were subjected to heat treatments in the vacuum oven and tube furnace at the same selected temperature range of 40 to 200 °C and duration of 6 hours per treatment temperature for the vacuum oven and the tube furnace (in air). This was carried out with the aim of improving the mechanical properties of coir fibre in preparation for composites manufacture. The tensile tests and other mechanical properties checks were run on the treated samples. The results of the two heat treatment methods were analysed and compared.

4.6.1 Tensile test results of the heat treated coir fibres

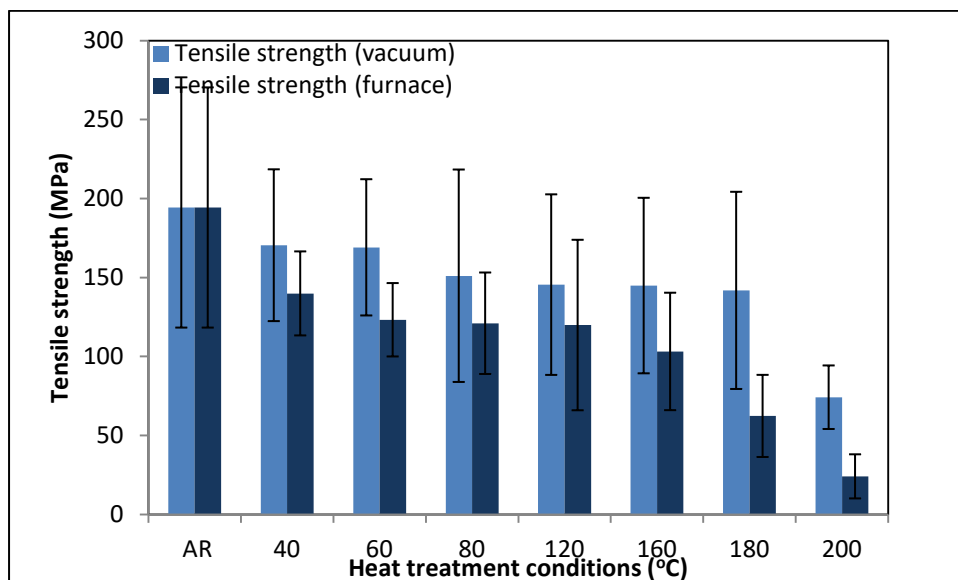
The results of the tensile tests of the heat treated coir fibres are given in Tables 4.12 and 4.13. A reduction of 12 to 27% in strength was observed for the vacuum oven heat-treated coir fibres and a reduction of 28 to 88% observed in the tube furnace (with air) treated fibres. The results compared favourably with literature reports. Figure 4.63 (a) shows the highest strength for the vacuum and furnace treated coir fibres at the same treatment temperature of 40 °C. The strength was found to decrease as the treatment temperature increases. Figure 4.63 (b) shows the maximum stiffness for vacuum-treated coir fibres at a treatment temperature of 180 °C. Elongation at break appears to be maximum at heat treatment temperature of 40 °C as shown in Figure 4.63 (c).

Table 4.12 Tensile tests results of the vacuum oven heat treated coir fibres from 40 to 200 °C.

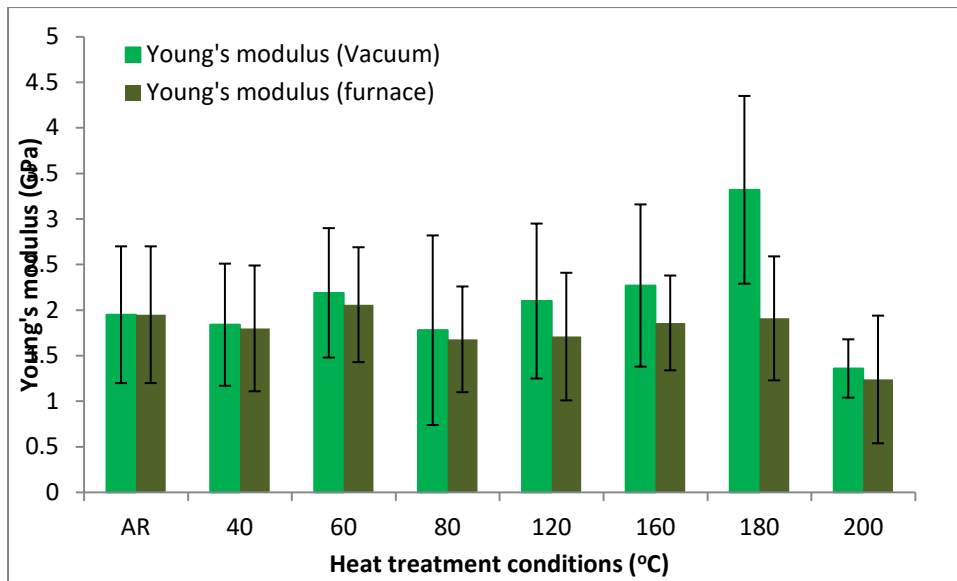
Heat treatment temperature Vacuum (°C)	Tensile strength (MPa)	Young's modulus (GPa)	Elongation at break (%)
AR	194.26 ± 76.03	1.95 ± 0.75	33.3 ± 10.9
40	170.38 ± 48.05	1.84 ± 0.67	32.26 ± 13.13
60	169.04 ± 43.12	2.19 ± 0.71	29.07 ± 6.09
80	151.05 ± 67.24	1.78 ± 1.04	24.24 ± 9.68
120	145.43 ± 57.16	2.1 ± 0.85	27.96 ± 8.14
160	144.84 ± 55.5	2.27 ± 0.89	23.13 ± 8.2
180	141.78 ± 62.41	3.32 ± 1.03	11.26 ± 3.95
200	74.15 ± 20.12	1.36 ± 0.32	16.87 ± 6.47

Table 4.13 Tensile test results of the tube furnace heat treated coir fibres from 40 to 200 °C.

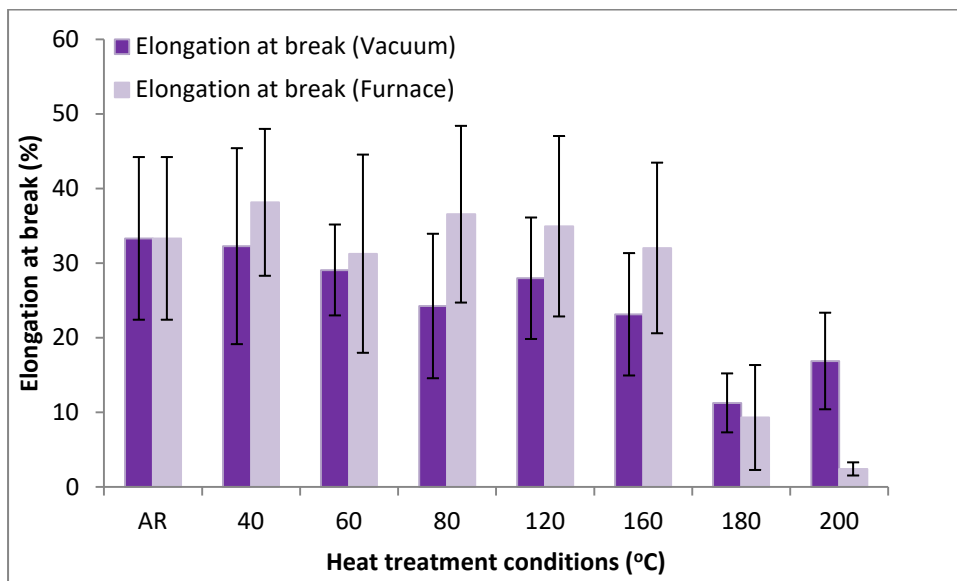
Heat treatment temperature	Tensile strength	Young's modulus	Elongation at break
Furnace (°C)	(MPa)	(GPa)	(%)
AR	194.26 ± 76.03	1.95 ± 0.75	33.3 ± 10.9
40	139.87 ± 26.59	1.80 ± 0.69	38.13 ± 9.84
60	123.19 ± 23.23	2.06 ± 0.63	31.25 ± 13.28
80	120.98 ± 32.12	1.68 ± 0.58	36.54 ± 11.84
120	119.85 ± 53.95	1.71 ± 0.70	34.93 ± 12.09
160	103.13 ± 37.17	1.86 ± 0.52	32.02 ± 11.43
180	62.30 ± 26.00	1.91 ± 0.68	9.30 ± 7.03
200	24.05 ± 13.96	1.24 ± 0.70	2.41 ± 0.88



(a)



(b)



(c)

Figure 4.63 Effect of heat treatment temperature on the (a) tensile strength (b) Young's modulus and (c) elongation at break of coir fibres.

4.6.2 Weibull Analysis of heat-treated coir fibres

The results of the Weibull analysis indicate the highest dispersion of flaws in furnace-treated coir fibres at 60 °C with Weibull modulus of 6.14 as given in Table 4.14.

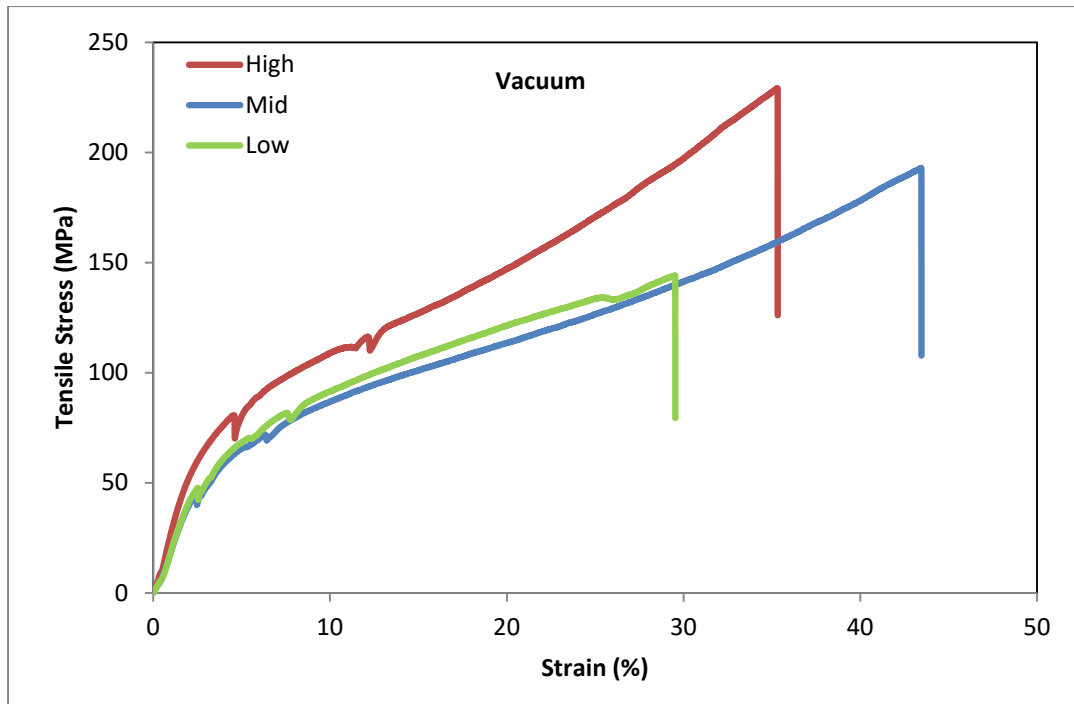
Table 4.14 Summary of the Weibull findings on the heat-treated coir fibres.

Fibre condition	Vacuum		Furnace	
	Weibull Modulus, m	Weibull Scale Parameter, α_0	Weibull Modulus, m	Weibull Scale Parameter, α_0
		(MPa)		(MPa)
AR	2.92	217.96	2.92	217.96
HT 40 °C	3.81	188.61	6.10	150.45
HT 60 °C	3.98	187.25	6.14	132.80
HT 80 °C	2.66	169.79	4.18	133.10
HT 120 °C	2.75	163.60	2.49	135.20
HT 160 °C	2.67	144.52	2.63	119.34
HT 180 °C	2.50	160.45	2.89	69.74
HT 200 °C	4.09	81.59	1.91	27.11

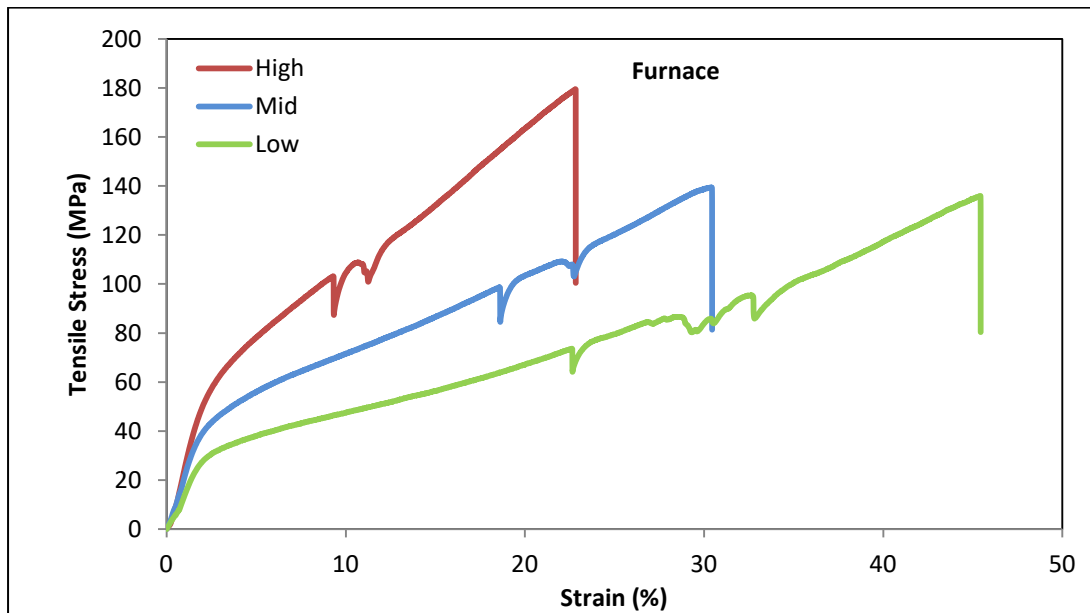
4.6.3 Failure analysis of the heat-treated coir fibres

The stress-strain curves of the vacuum oven heat-treated coir fibres displayed initial elastic deformation as shown in Figure 4.64 (a), this was followed by plastic deformation during which load drops were observed about the mid regions. A brittle failure occurred in all the samples. The highest stress observed was 229.2 MPa. This was 37% higher than the minimum failure stress. The minimum failure stress was characterized by several load drops. The load drops must have contributed to its reduced failure stress.

The failure traces of the furnace oven heat treated coir fibres as shown in Figure 4.64 (b) was characterised with load drops about 30% away from the initial linear portion. The maximum failure stress recorded was 186.01 MPa. This was about 25 and 24 % .higher than the mid and minimum failure stresses. The sample with the minimum failure stress displayed higher elongation at break than the samples with the maximum and mid failure stresses hence the reduced failure stress could be attributed to the differences in their diameters.



(a)



(b)

Figure 4.64 Stress-Strain curves of (a) vacuum oven and (b) furnace oven heat treated coir fibres at optimum strength heat treatment temperature (40 °C).

4.6.4 Scanning electron microscopy analysis of coir fibres before and after heat treatment

Figure 4.65 compares the SEM micrographs of (a) untreated and (b) heat-treated coir fibres with the changes in the microstructure of the heat treated fibres shown Figure 4.66. Changes in the fracture surfaces and failure pattern of the heat-treated samples are displayed in Figure 4.67. Failures were observed to be uneven. Colour change of the fibres to dark brown and cracking of the cell were observed. These can influence the mechanical behaviour of the heat treated fibres. These phenomena are especially evident from SEM of fibres treated from 120 °C but became more obvious at 180 °C.

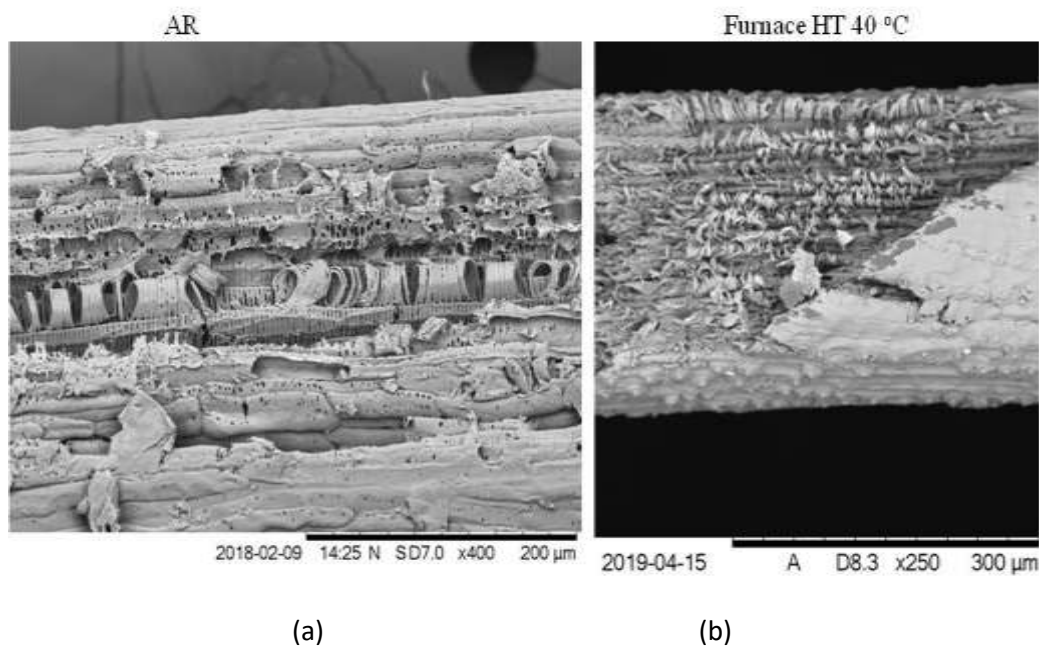


Figure 4. 65 shows the SEM micrographs of (a) untreated (b) heat-treated coir fibres.

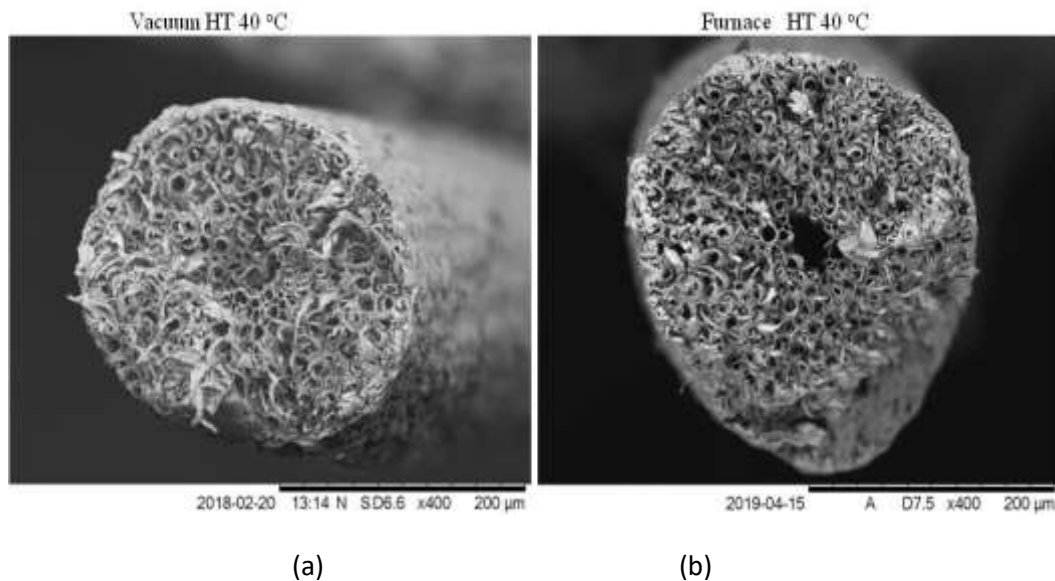
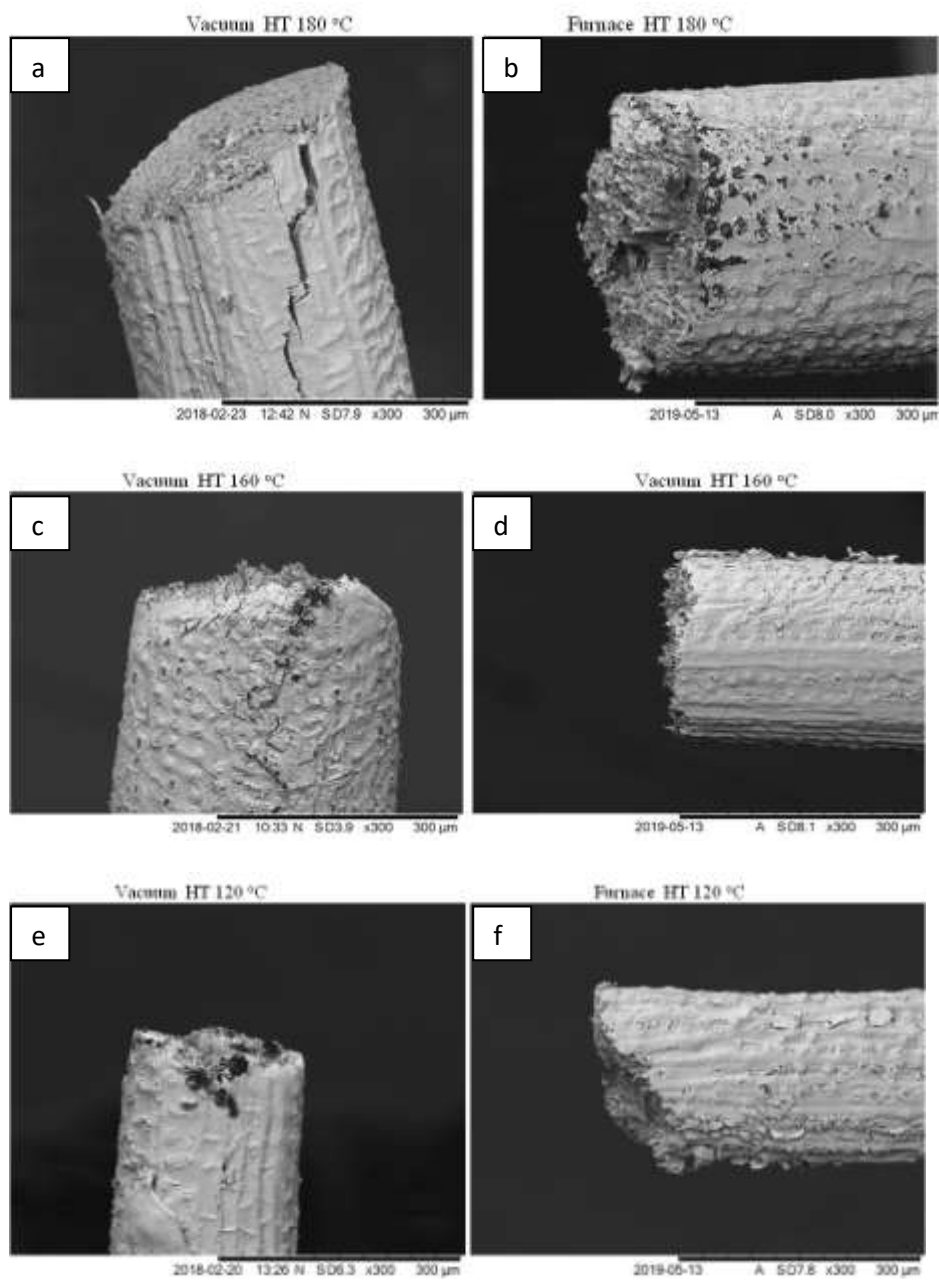


Figure 4.66 SEM micrographs of heat-treated coir fibres at 40 oC in the (a) vacuum and (b) tube furnace.



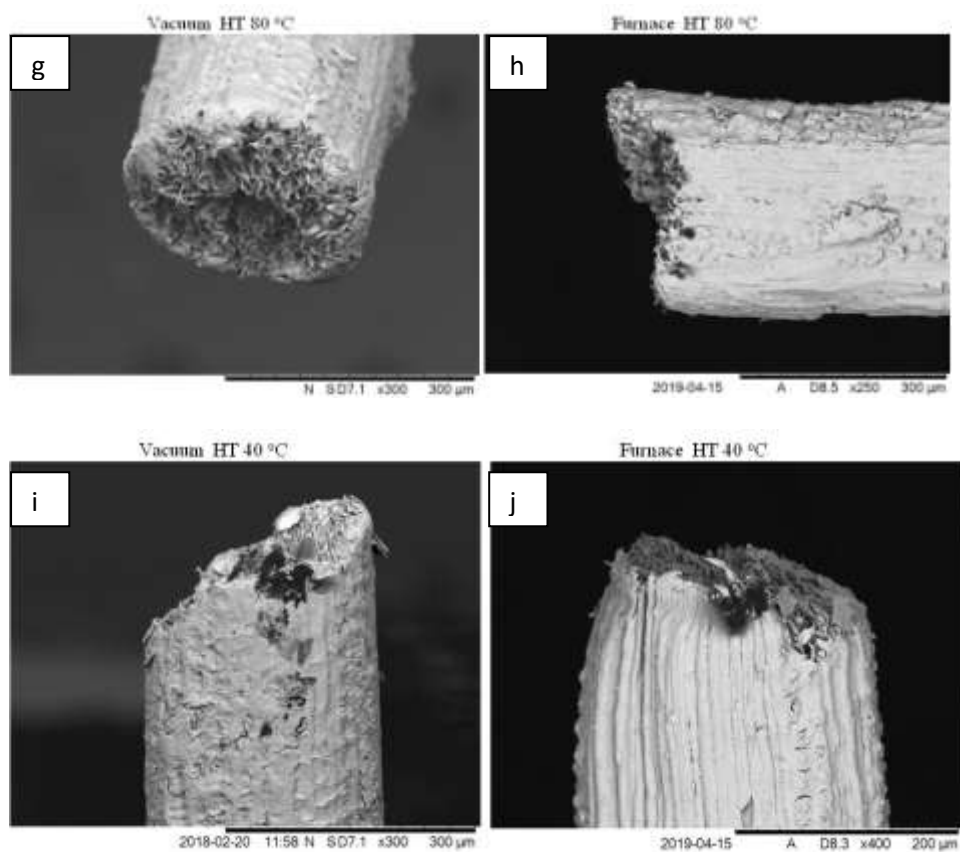


Figure 4.67 (a-j) SEM of fractured heat-treated coir fibre samples.

4.7 Treatment of coir fibres using silane

The aim of the treatment of coir fibre using silane is to improve the interfacial bond strength between the fibre and the polymer matrix in subsequent composite manufacture. As received coir fibres were treated with different concentrations of (3-aminopropyl) trimethoxy silane and (3-aminopropyl) triethoxy silane for varying durations as shown in Table 4.15. The properties of the silane treated fibre were tested and analysed.

Table 4.15 Concentrations of amino and epoxy silane and the treatment durations.

Run order	(3-Aminopropyl) trimethoxy silane and (3-glycidyloxypropyl) trimethoxy silane	
	Concentration (%)	Duration (minutes)
1	0.5	15
2	0.5	30
3	0.5	60
4	1	15
5	1	30
6	1	60
7	1.5	15
8	1.5	30
9	1.5	60

4.7.1 Tensile properties of the silane treated coir fibres

From Figure 4.68, increase in the concentration of epoxy silane led to increase in the tensile strength of the fibres however, no significant difference was observed in the tensile strength of the fibres as the concentration of the amino silane increased. The tensile properties of the silane treated coir fibres are shown in Table 4.16. As was found in the heat treatment of coir fibres, silane treatment led to a reduction in the tensile strength of coir. Drops in the strength by 24% and 12% were recorded for (3-Aminopropyl) trimethoxy silane and (3-glycidyloxypropyl) trimethoxy silane treatments respectively at a treatment concentration of 1.5%. From Figure 4.69 (a) and (b), (3-glycidyloxypropyl) trimethoxy silane treated coir fibres yielded higher strength and stiffness than (3-Aminopropyl) trimethoxy silane. A reduction in strength with an increase in Young's modulus was recorded after epoxy silane concentrations of 0.25 and 2.00 % at room temperature for 60 minutes preceded by pre-treatment with 10% NaOH at 30 °C for 4 hours (Varma,Varma and Varma, 1984). Abdullah and Ahmad, (2012) observed a 25% increase in strength with NaOH pre-treated coir fibers. The tensile strength of hemp fibres decreased by 33% and 23% after silane treatment and when the silane treatment was preceded by NaOH treatment respectively (Kabir, Wang, Lau,

Cardona, *et al.*, 2012). Therefore, improvement in the tensile strength of single fibres can be achieved when the silane treatment is preceded by NaOH treatment. However an increase in the tensile strength of silane-treated coir fibre composites have been recorded as a result of improved adhesion of fibre on the polymer matrix due to a reduction in the cellulose hydroxyl group at the fibre/matrix interface (Munirah Abdullah and Ahmad, 2012) furthermore, the stored energy of composites manufactured with silane treated coir fibres have been reported to be higher than those with raw coir fibre (Arrakhiz *et al.*, 2012).

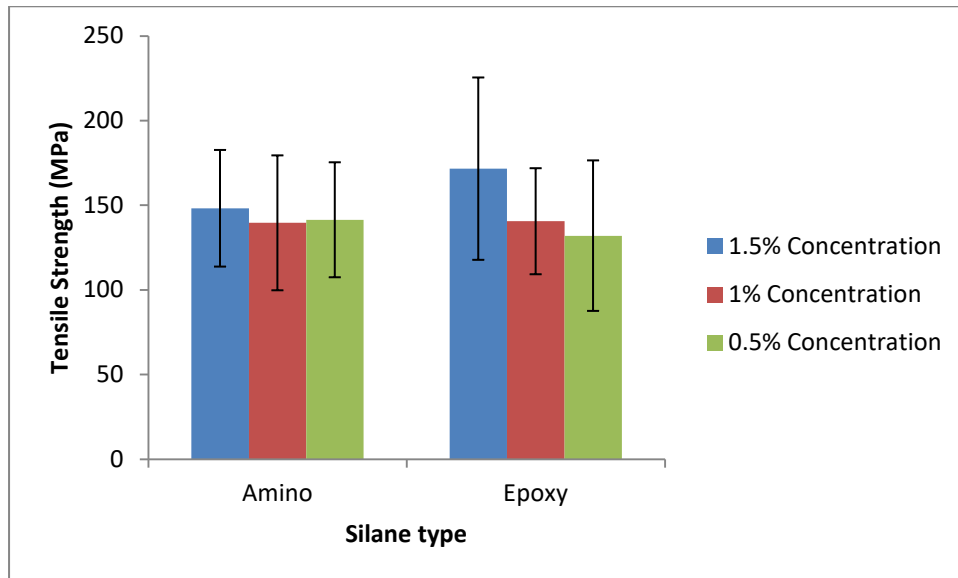
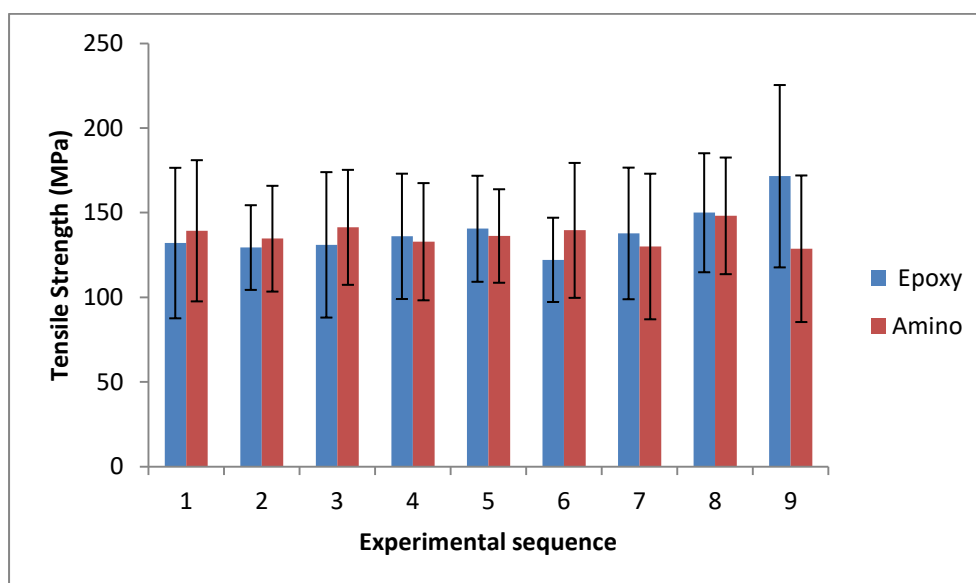


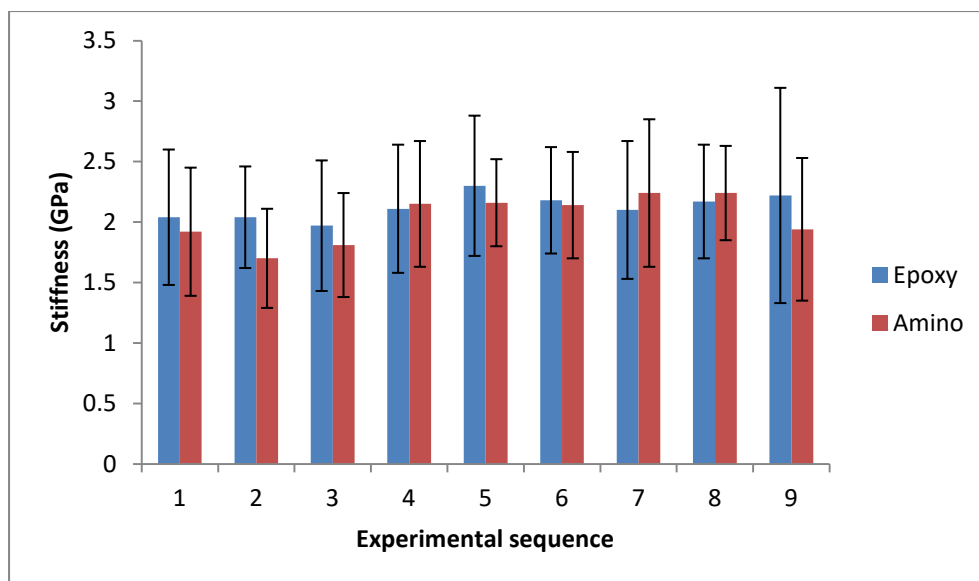
Figure 4.68 Effects of concentrations of silane on the tensile strength of coir fibres.

Table 4.16 Tensile properties of coir fibres treated using (3-Aminopropyl) trimethoxy and ((3-glycidyloxypropyl) triethoxy silane.

Concentration (%)	Duration (minute)	(3-Aminopropyl) trimethoxy silane		((3-glycidyloxypropyl) trimethoxy silane	
		Tensile strength	Young's modulus	Tensile strength	Young's modulus
		(MPa)	(GPa)	(MPa)	(GPa)
0.5	30	134.66 ± 31.24	1.70 ± 0.41	129.43 ± 25.02	2.04 ± 0.42
0.5	60	141.37 ± 33.6	1.81 ± 0.43	131.03 ± 42.96	1.97 ± 0.54
0.5	15	139.34 ± 41.70	1.92 ± 0.53	132.08 ± 44.46	2.04 ± 0.56
1	60	139.57 ± 39.85	2.14 ± 0.44	122.16 ± 24.89	2.18 ± 0.44
1	15	132.89 ± 34.62	2.15 ± 0.52	136.05 ± 37.02	2.11 ± 0.53
1	30	136.25 ± 27.62	2.16 ± 0.36	140.52 ± 31.32	2.30 ± 0.58
1.5	15	130.05 ± 43.00	2.24 ± 0.61	137.77 ± 38.88	2.10 ± 0.57
1.5	30	148.16 ± 34.45	2.24 ± 0.39	149.91 ± 35.15	2.17 ± 0.47
1.5	60	128.73 ± 43.29	1.94 ± 0.59	171.57 ± 53.86	2.22 ± 0.89



(a)



(b)

Figure 4.69 Effects of Amino and Epoxy silane on (a) tensile strength (b) stiffness of coir fibres.

4.7.2 Weibull characterization of silane treated coir fibres

The silane treated coir fibres showed a low degree of scatter in the range of 3.45 to 6.09 as shown in Table 4.17. The shape parameter indicates the degree of dispersion of flaws. The average Weibull modulus for (3-Aminopropyl) trimethoxy silane is 4.33 while the average Weibull modulus for (3-glycidyloxypropyl) trimethoxy silane is 4.56. The highest average Weibull modulus for both silanes is at 1% concentration.

Table 4.17 Weibull distributions of silane treated coir fibres.

Treatment conditions			(3-Aminopropyl) trimethoxy silane		(3-glycidyloxypropyl) trimethoxy silane	
Conc (%)	Duration (Minutes)	Temperature (°C)	Weibull modulus	Scale parameter (MPa)	Weibull modulus	Scale parameter (MPa)
0.5	30	30	4.48	147.55	6.09	139.37
0.5	60	30	4.86	154.29	3.45	145.79
0.5	15	30	3.68	154.5	3.47	147.05
1	60	30	3.87	154.34	5.72	131.96
1	15	30	4.56	145.48	4.33	149.26
1	30	30	5.58	147.33	4.92	153.18
1.5	15	30	3.5	144.5	4.22	151.51
1.5	30	30	5.15	161.07	5.07	163.01
1.5	60	30	3.31	143.89	3.8	213.33

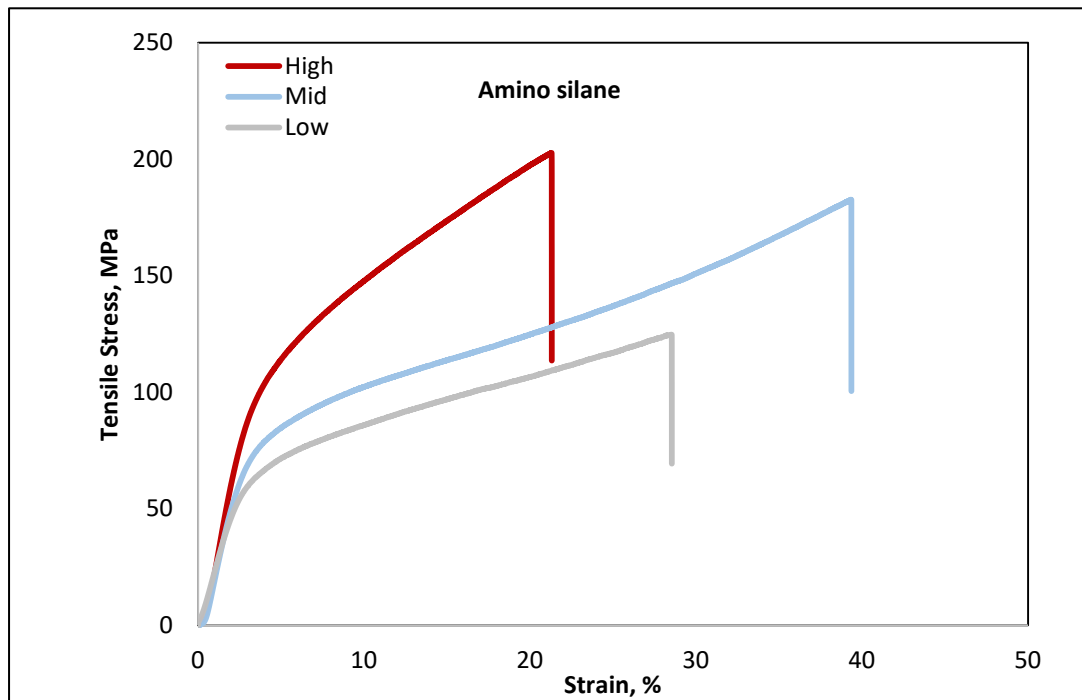
Conc=concentration

4.7.3 Failure traces of amino and epoxy silane-treated coir fibres

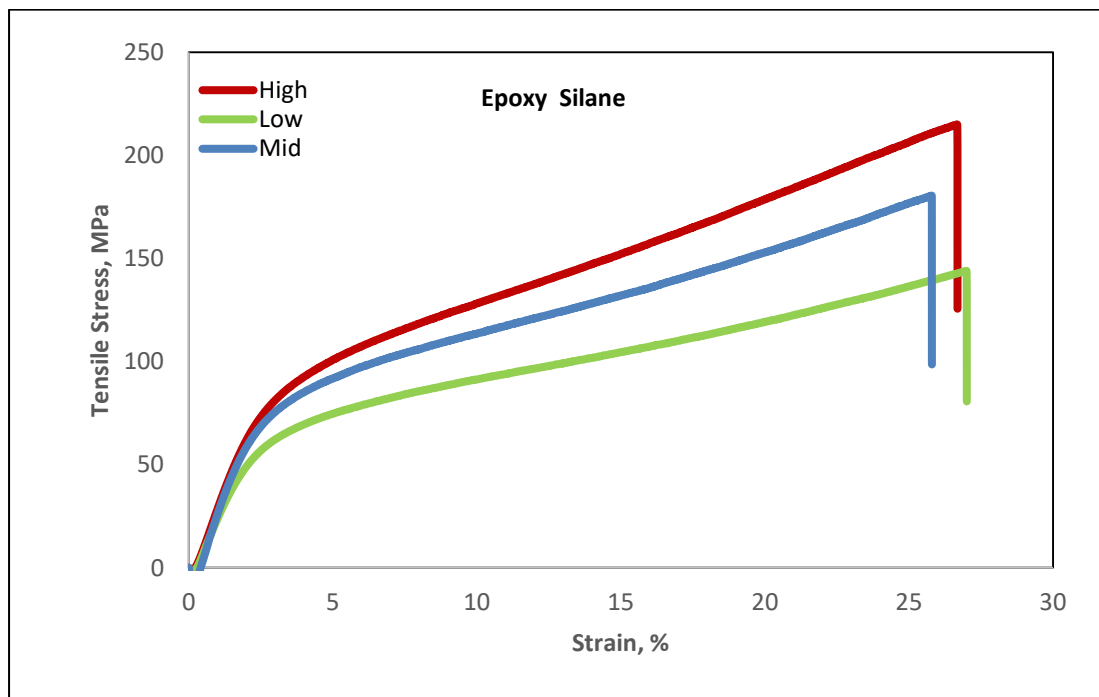
Failure curves of the (3-Aminopropyl) trimethoxy silane and ((3-glycidyloxypropyl) trimethoxy silane treated coir fibres were characterised with smooth traces without load drops as shown in Figure 4.70 (a) and (b). The shapes of the tensile curves are in line with natural fibres. An initial linear region was followed by a non-linear region before fracture occurred. Silane-treated fibres displayed a very high ductility. No significant difference existed between the maximum and mid failure stresses however the maximum failure stress was 38 % higher than the minimum failure stress for the amino silane treated coir fibres.

The failure traces in epoxy silane treated fibres are more consistent than in the amino silane treated fibres. A drop of 16% was observed between the maximum and mid failure stresses for the epoxy-treated coir fibres. This can be attributed to the presence of debris or slight

differences in the diameter of the fibres.



(a)



(b)

Figure 4.70 Failure traces of (a) amino and (b) epoxy treated coir fibres.

4.7.4 Scanning Electron Microscopy of silane treated coir fibres

The surface of the as-received coir fibre changed after treatment with silane. The silane-treated coir fibres show rougher surfaces and striations than the untreated as shown in Figure 4.71. This might enhance better interlocking between the fibre and the matrix in composite manufacture.

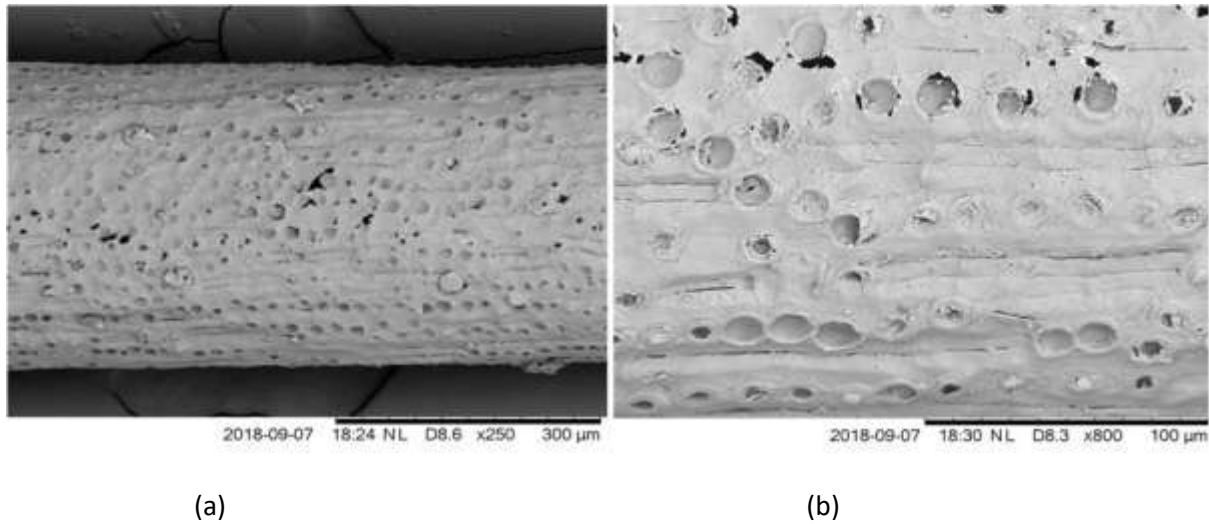
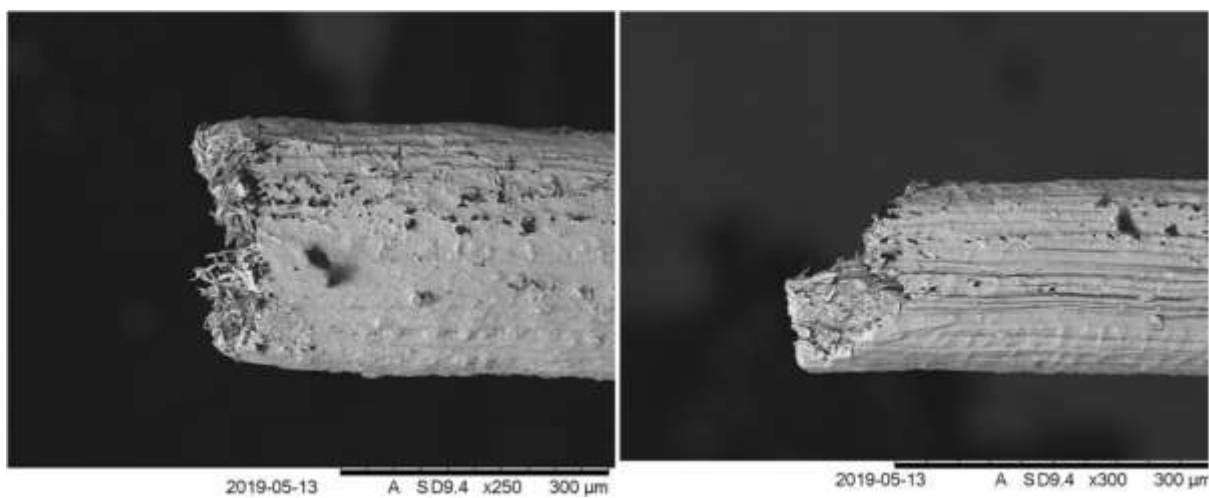


Figure 4.71 Micrographs of amino silane-treated coir fibre at (a) X250 and (b) X800 at 1.5% concentration of amino silane.

The scanning electron micrographs of failed surfaces of silane treated coir fibres shown in Figures 4.72 (a) and (b) show brittle fracture, uneven fracture surfaces and fibre pull out.



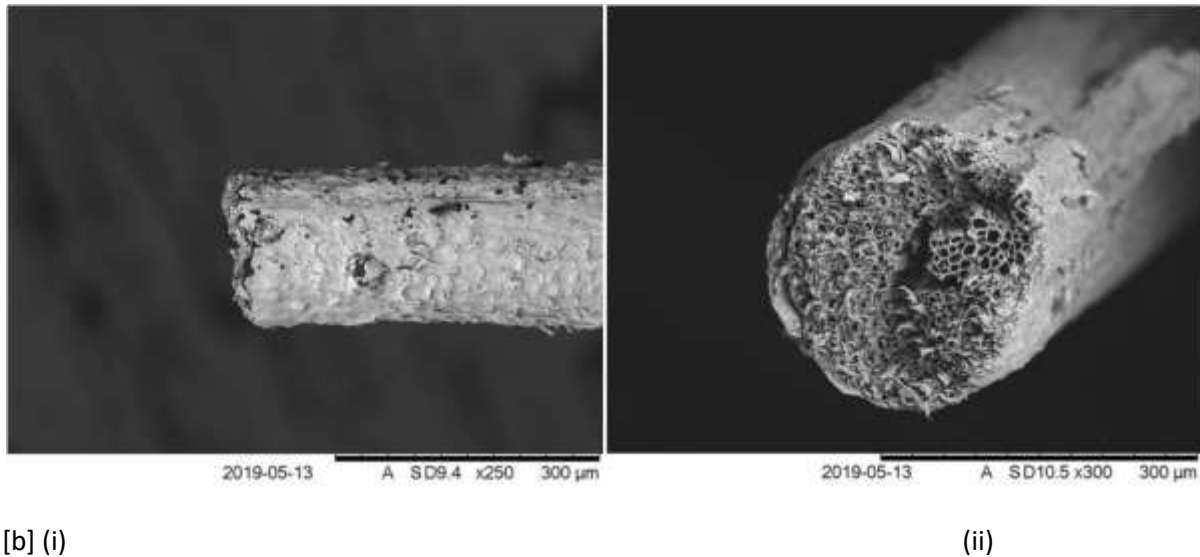


Figure 4.72 SEM of fractured (a) amino and (b) epoxy silane treated coir fibres.

4.8 Water absorption of coir fibres

Moisture ingress reduces the crystallinity of natural fibres, gives rise to swelling and promotes microcracks in the composites (Sreekala *et al.*, 2002; Khalil *et al.*, 2012; Muñoz and García-Manrique, 2015; Londhe, Mache and Kulkarni, 2016), (see Chapter 2, Section 2.2.2.2 for details). Therefore it is important to determine to what extent and at what temperature moisture can significantly affects the properties of coir fibre.

Figure 4.73 shows the percentage weight gain as a result of water absorbed by the coir fibre samples as a function of the square root of time (Madueke, Kolawole and Tile, 2021). The water absorption curves are similar to those obtained on bowstring hemp, okra and bethel nut fibres (Saikia, 2010), sisal and jute (Giridhar, Kishore and Rao, 1986) and Napier grass (Haameem *et al.*, 2016). The two stage absorption characteristic of natural fibre is displayed in the graph. The first stage was a rapid linear uptake resulting to more than 8% weight gain in the fibre. This rapid linear uptake is proportional to the square root of time. The initial rapid linear response has been attributed to the presence of hydroxyl group and the capillary action with respect to Fick's law of diffusion (Saikia, 2010). The percentage weight loss after the first 5 minutes was found to be 5.6, 6.4, 7.4, and 7.6 % at 40, 50, 60 and 80 °C respectively. The weight gain increases non-uniformly with increase in the temperature. The second stage of weight gain was much slower than the first stage until full saturation was achieved. The second stage has been attributed to Non-Fickian diffusion (see Section 2.4).

The diffusion coefficient at temperatures of 40, 50, 60 and 80 °C are shown in Table 4.18. The diffusion coefficient was found to increase with increase in temperature.

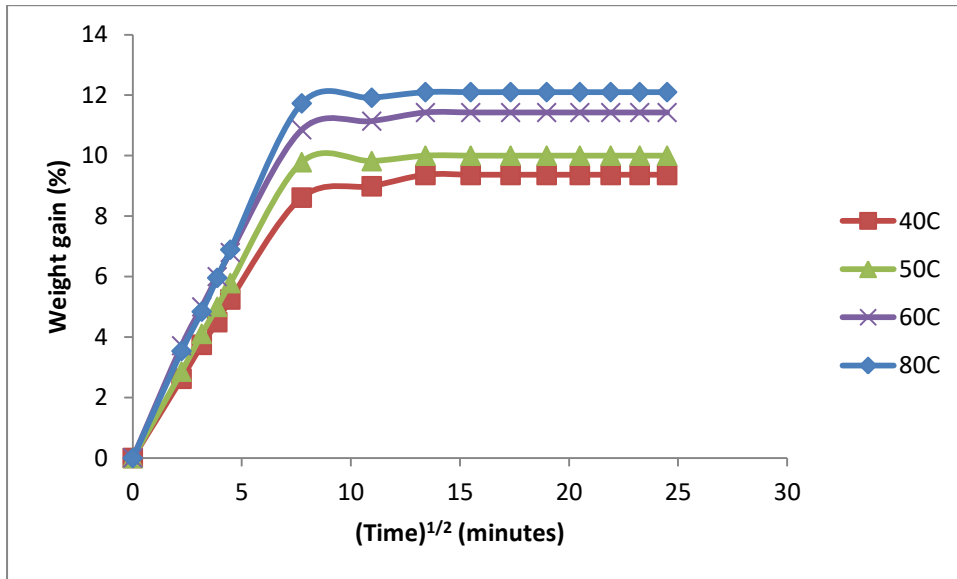


Figure 4.73 Moisture absorption of as received coir fibres as a function of the square root of time at different temperatures.

Water absorption is calculated using the following equation:

$$W_a = \left[\frac{M_w - M_d}{M_d} \times 100 \right] \% \quad [4.6]$$

M_d = constant mass (g) of dry coir fibre sample

M_w = mass (g) of wet coir fibre sample after time

From the slope of the linear portion of weight gain curve versus the square root of time, the diffusion coefficient D of the coir fibre samples for the specified temperatures could be determined using:

$$D = \pi \left(\frac{k d}{4 M_\beta} \right)^2 \quad (\text{Muñoz and García-Manrique, 2015}) \quad [4.7]$$

d = thickness;

k = slope of weight gain versus SQRT (time);

M = maximum weight gained at constant

Table 4.18 Weight gain after water absorption and the diffusion coefficient of as received coir fibre at different temperatures.

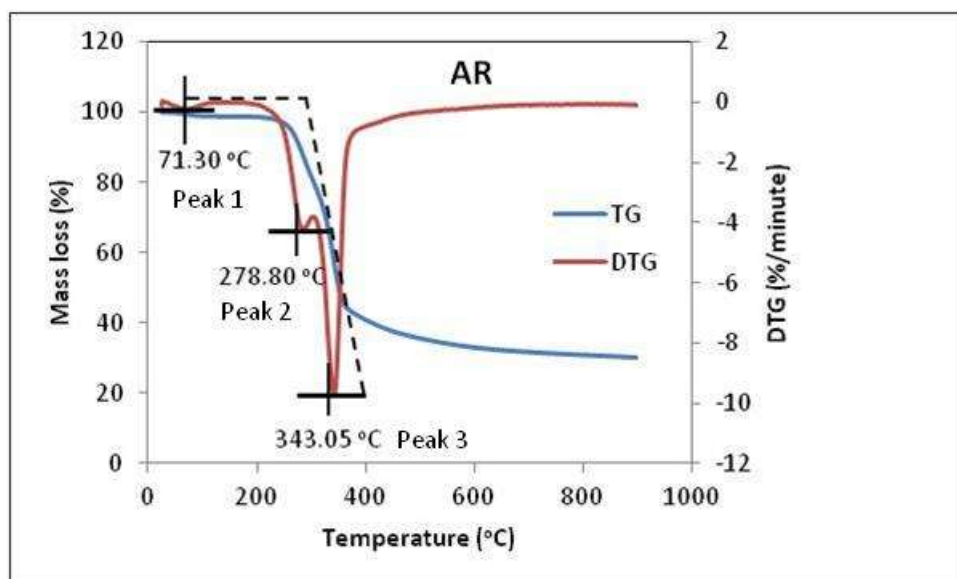
Temperature (°C)	Temperature (K)	Initial Diameter (cm)	Weight gained (absorbed H ₂ O) (%)	Slope	Diffusion coefficient (10 ⁻² cm ² .s ⁻¹)
40	313.15	0.024	9.36	1.25	1.548
50	323.15	0.024	10	1.31	1.941
60	333.15	0.032	11.43	1.33	4.647
80	353.15	0.030	12.10	1.4	5.072

4.9 Thermal properties of coir fibre

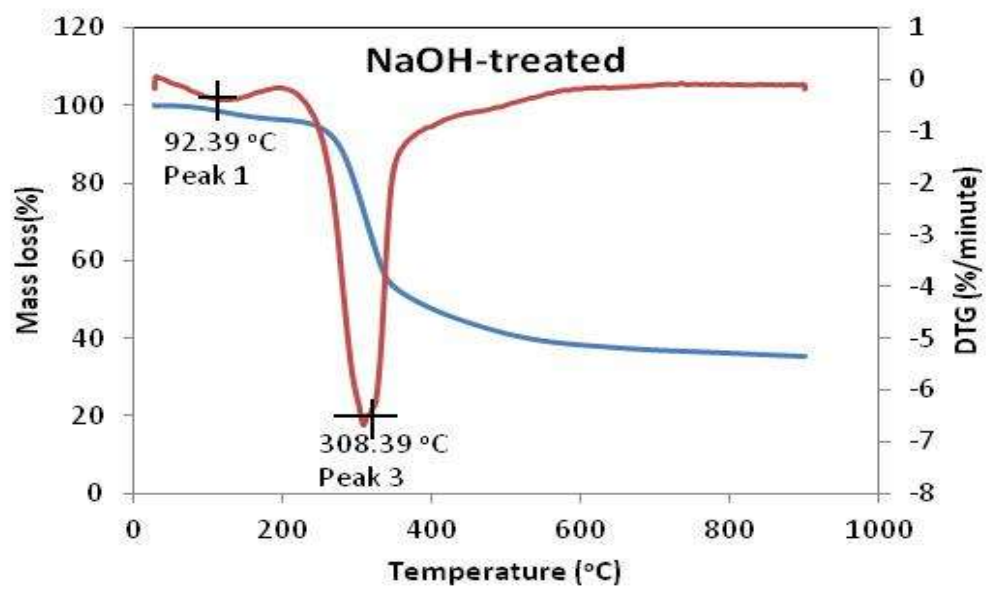
Thermal properties of coir fibres are important in order to determine their level of application as reinforcement in elevated temperature environment, they are a measure of the thermal stability of coir fibre. Thermogravimetric analysis was carried out on as received and treated coir fibres under a nitrogen atmosphere. The coir specimens were heated from room temperature to 900 °C at a heating rate of 10 °C/minute. The results of the thermogravimetric analysis for the treated and untreated coir fibres are shown in Figure 4.74 (a-f; TGA and DTG traces) and a summary of the results is given in Table 4.19. The decomposition profiles of the untreated and treated coir fibres occurred in three stages of weight loss: (i) 0 to 200 °C; due to evaporation of water (ii) 200 to 360 °C attributed to the degradation of hemicellulose and (iii) above 360, which has been linked to the thermal degradation of cellulose (Tomczak, Sydenstricker and Satyanarayana, 2007; Claramunt, Ardanuy and García-hortal, 2010; Santos *et al.*, 2010). The stages are characterized by three peaks (onset temperatures) except for NaOH-treated fibres. NaOH- treated fibres were characterized by two peaks. For as received coir fibres, the first peak was between 71.30 °C. The second peak occurred at about 278.8 °C (c) the third peak was at about 343.05 °C.

Table 4.19 TGA of treated and untreated coir fibres.

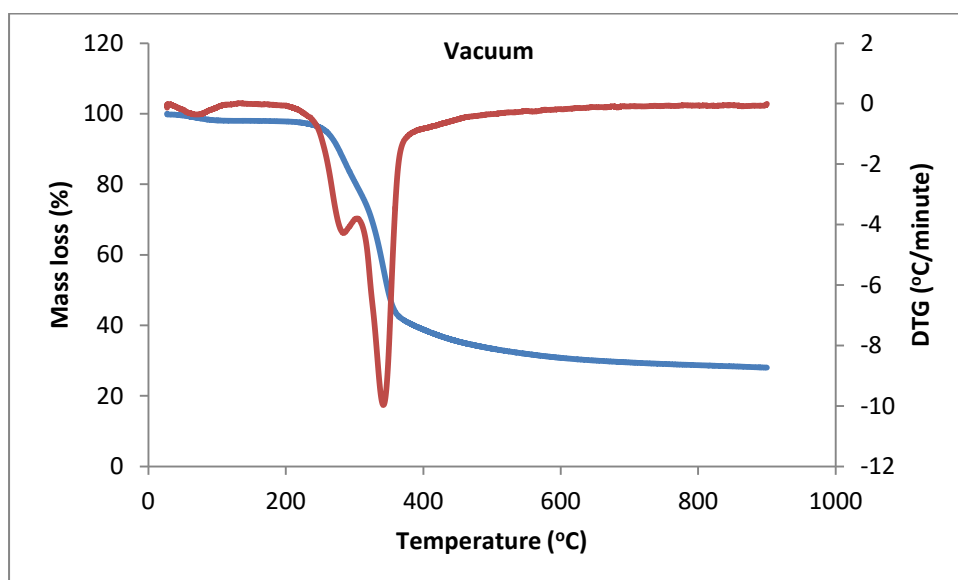
Fibre Condition	Temperature at Peak			Actual Mass Loss		
	Peak1 (°C)	Peak 2 (°C)	Peak 3 (°C)	At Peak 1 (g)	At Peak 2 (g)	At Peak 3 (g)
As-received	71.30	278.8	343.05	0.65	9.47	42.47
Vacuum at 40 °C	72.12	280.32	346.32	0.81	9.95	44.22
Furnace at 40 °C	64.19	289.89	359.09	0.85	12.6	46.67
Amino silane treated	79.08	287.88	364.58	0.70	10.36	50.22
Epoxy silane treated	79.03	294.63	362.73	0.96	13.61	49.20
NaOH-treated	92.39	NA	308.39	0.86	NA	27.28
AR hot compacted	79.09	288.99	357.69	1.13	13.43	46.72



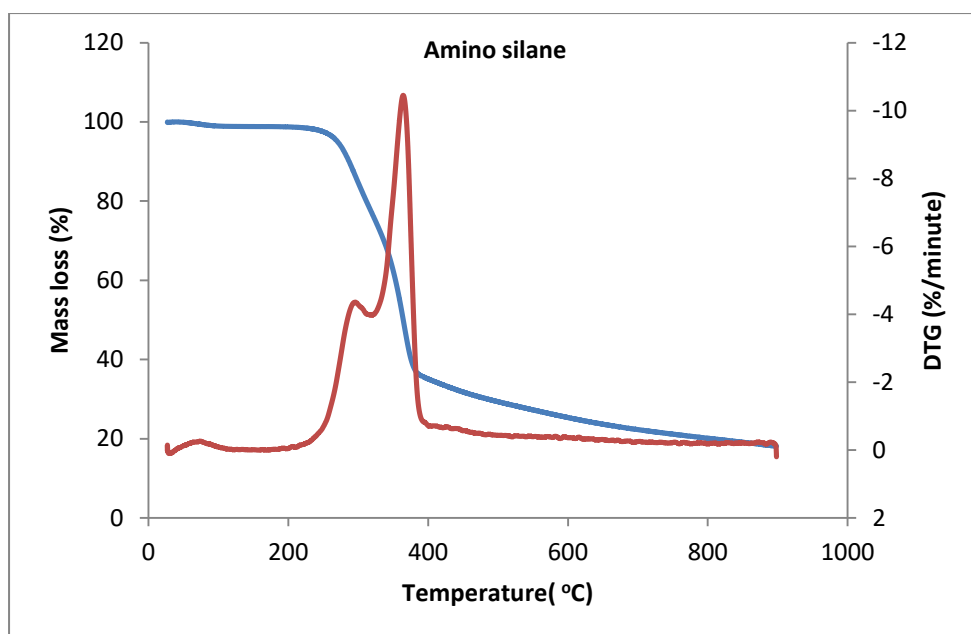
(a)



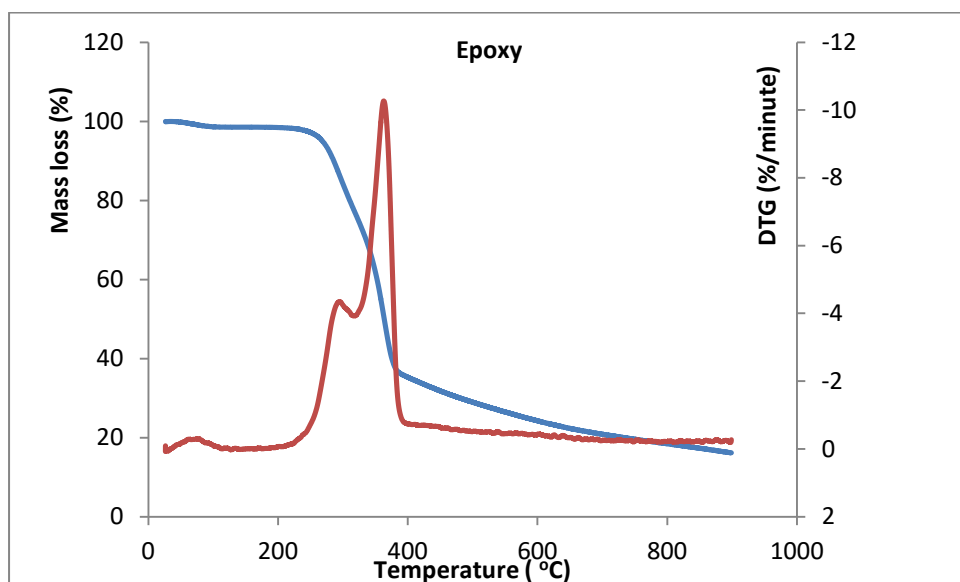
(b)



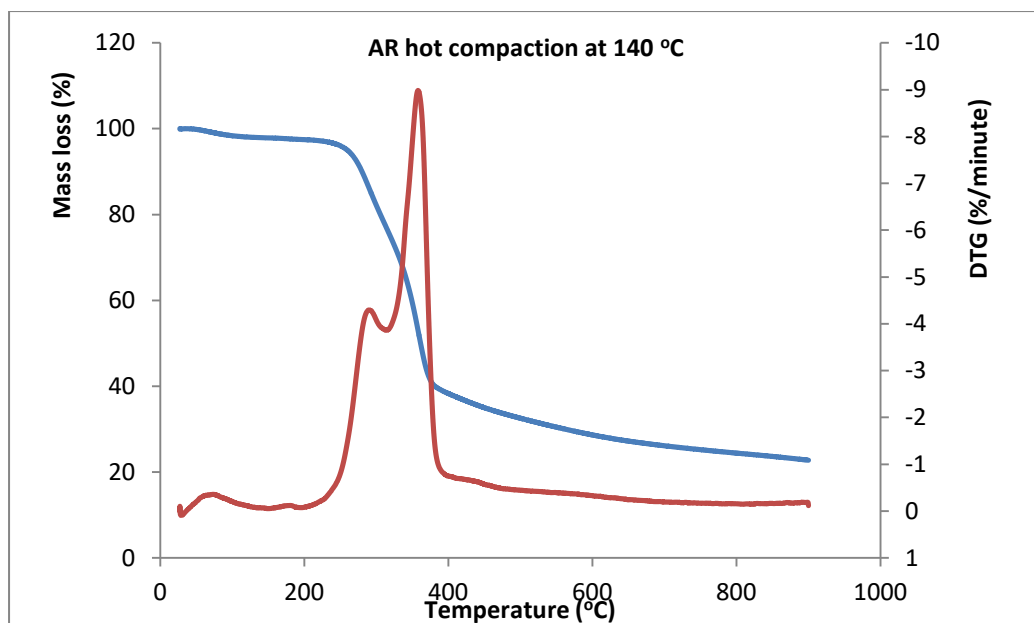
(c)



(d)



(e)



(f)

Figure 4.74 TGA and DTG of untreated and treated coir fibres (a) As received (b) NaOH- treated (c) vacuum oven dried (d) amino-treated (e) epoxy-treated and (f) AR hot compaction at 140 °C.

Summary of the properties of the untreated and treated single coir fibres

The properties of the treated and untreated single coir fibres are as summarized in Figures 4.75- 4.78. Table 4.20 shows Fisher pairwise comparisons of the fibres.

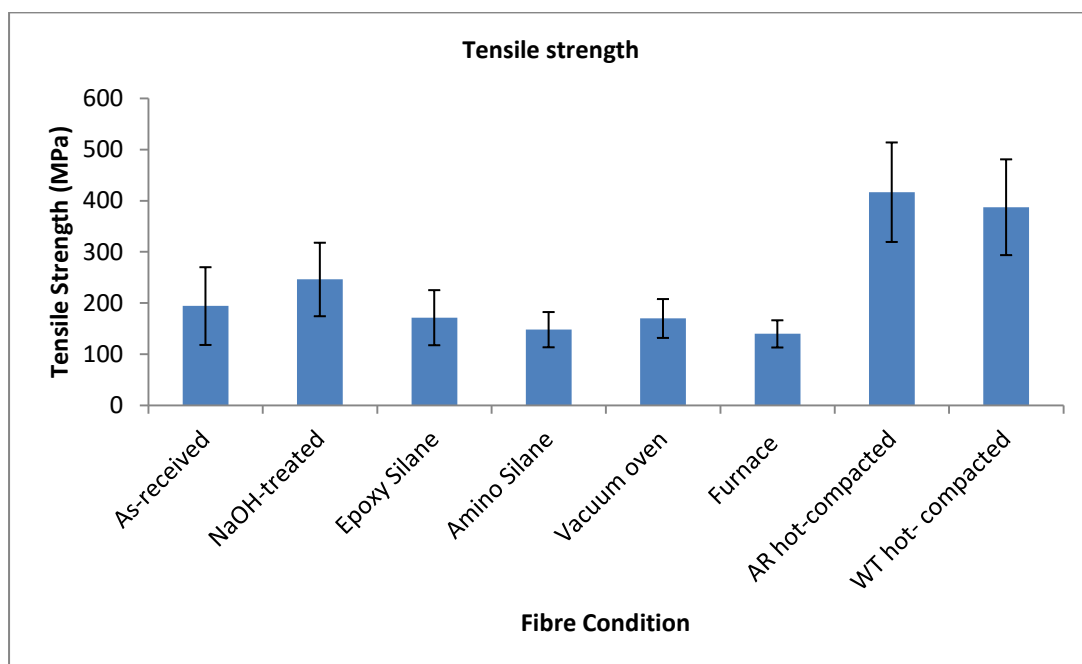


Figure 4.75 Tensile strengths of AR coir fibre in comparisons with the treated coir fibres.

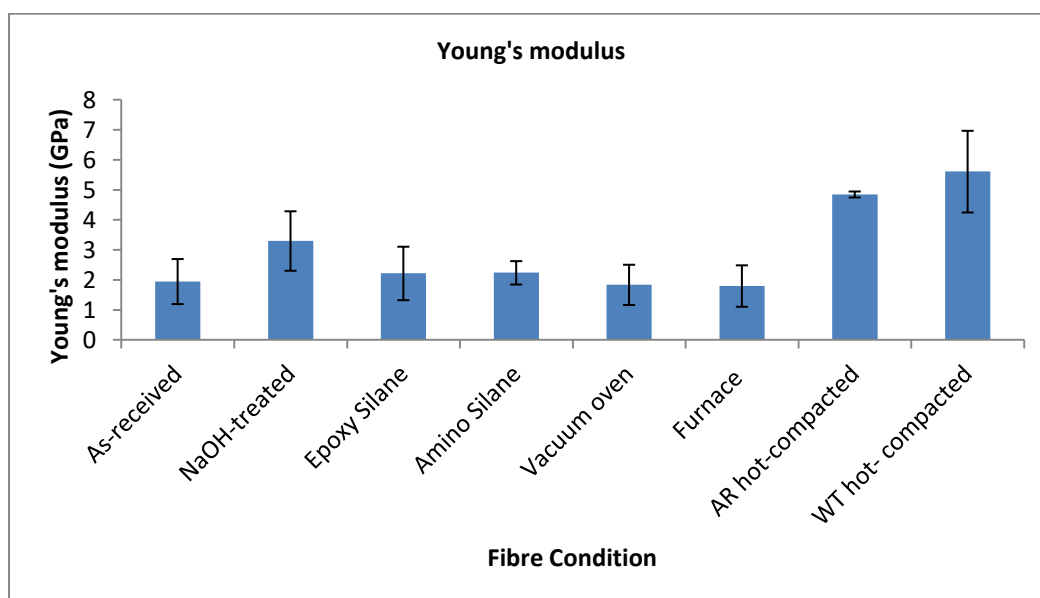


Figure 4.76 Young's modulus of AR coir fibre in comparisons with the treated fibres.

Table 4.20 Fisher Pairwise Comparisons of the tensile properties of treated and untreated coir fibres at 95% Confidence level (all treatments).

Fibre condition	N	Tensile strength		Young's modulus	
		Mean	Grouping	Mean	Grouping
AR hot compacted	30	426.5	A	4.9	G
Water-treated hot compacted	30	387.6	B	5.6	F
NaOH-treated	30	246.3	C	3.3	H
AR	30	194.3	D	2.0	I
Epoxy silane treated	30	171.6	D E	2.2	I
Vacuum oven treated	30	170.4	D E	1.8	I
Amino silane treated	30	148.2	E	2.2	I
Furnace oven treated	30	139.9	E	1.8	I

N=number of samples

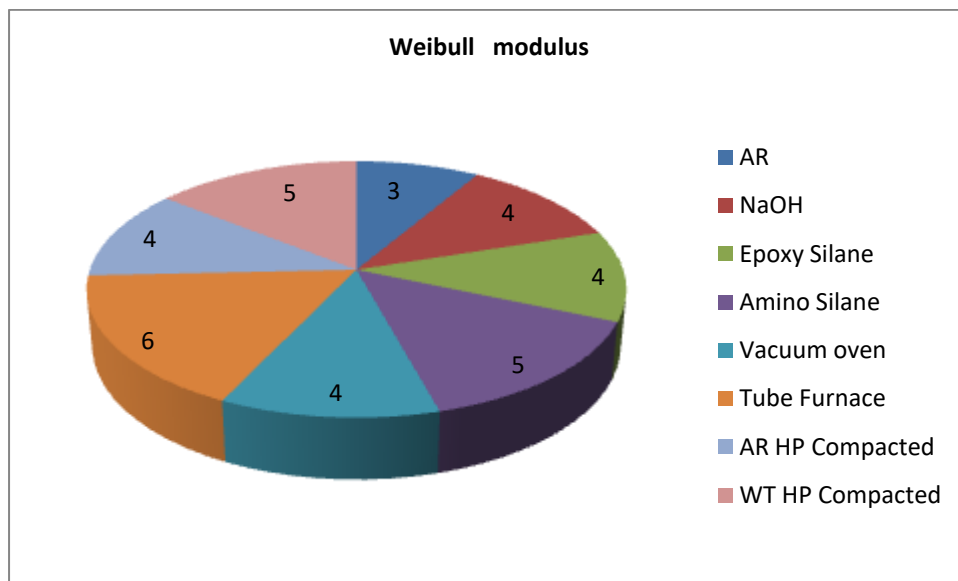


Figure 4.77 Values of the Weibull modulus obtained from AR coir fibre in comparisons with the treated fibres.

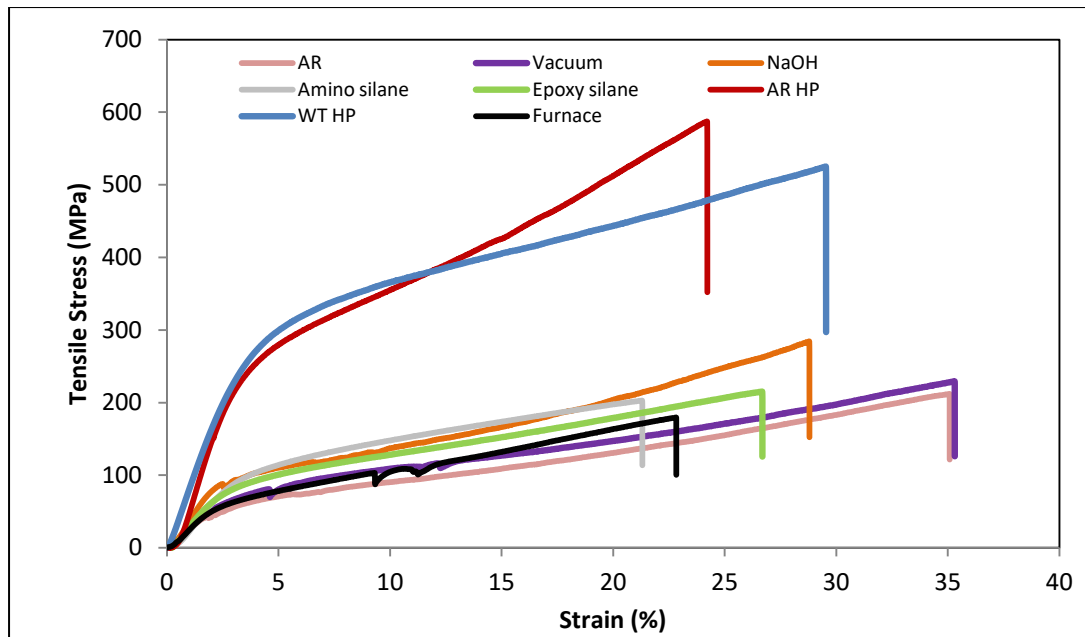


Figure 4.78 Failure curves of AR coir fibre in comparisons with the treated fibres.

4.10 Synthetic fibre reinforcements and matrices

The synthetic fibres considered for use as reinforcement in composite manufacture for this research are E-glass fibre and T700 carbon fibre in the forms of spread E.glass and spread T700 carbon fibres. The matrices considered for the composite manufacture are the synthetic and bioepoxy resins.

4.10.1 Spread E-glass fibre

The creels of E-glass fibre bundle of 2400 TEX Owens-Corning Advantex R25H were supplied by Halyard Precision Composites UK. The electrically-graded glass fibres contained approximately 2400 individual filaments in each bundle. The E-glass fibres were used in the fibre spreading experiments (see Section 3.7.5) and in the manufacture of composites. The properties of the E-glass have been stated in the manufacturer's data sheet reported in the experimental section. The spreading techniques were discussed at length in Section 3.8.

4.10.1.1 Tensile and Weibull properties of the spread E-glass fibre

Sections were taken directly from the mandrel containing the spread E-glass fibre and end-tabbed with the dimensions as given as in Chapter 3. The tensile tests were carried out on the spread E-glass and spread T700 carbon fibres using Instron 5566 at a head displacement rate of 1mm/min and a gauge length of 100 mm. The load cell was 100 N.

The average tensile strength and Young's modulus obtained were 800.47 MPa and 56.86 GPa respectively. The tensile strengths and the Young's modulus of E-glass fibre bundle at varying gauge lengths have been reported (Pardini and Manhani, 2002). It has been reported that spreading the fibre brings about a non-significant decrease in the tensile properties. This has been attributed to fibre damage during mechanical spreading and friction as a result of fibre to fibre contact.

Figure 4.79 presents three representative stress-strain curves from the spread E-glass fibre. Linear loading behaviour was observed in the samples that displayed maximum (1039.80 MPa) and mid-range (833.34) failure stresses. The lowest observed failure stress was 620.72 MPa. The maximum and lowest failure stresses obtained by R. Murray (2016) were 793.89 MPa and 563.02 MPa respectively. From Figure 4.79, the sample that exhibited the lowest failure stress did not load linearly up to failure; this non-linear behaviour could be attributed to the misalignment of the filament or filament slippage during tensile testing. The Weibull distribution of the spread E-glass is shown in Figure 4.80.

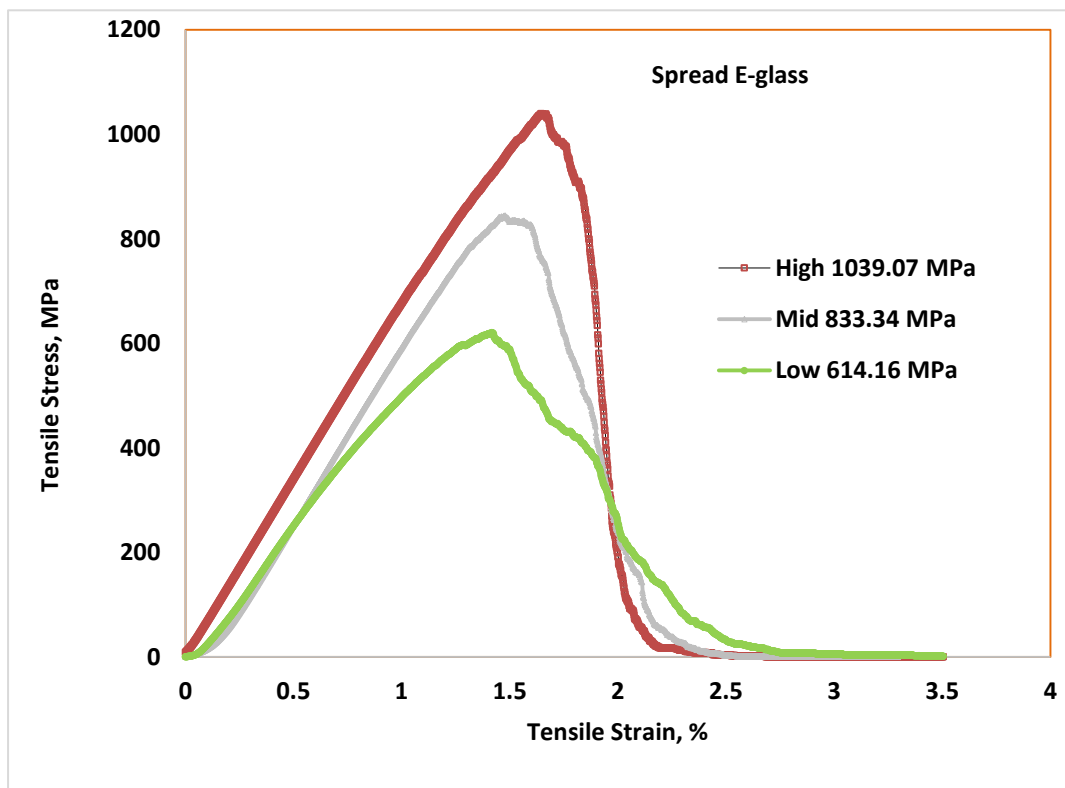


Figure 4.79 Stress-strain curves of spread E-glass fibre.

Weibull modulus is subject to the degree of flaw and the flaw distribution in a sample. If the flaws are evenly distributed, the strength becomes less dependent on the length. The Weibull modulus obtained was 9.78 as shown in Figure 4.80. Weibull modulus values of 9.4 and 5.89 .have been reported by Hill and Okoroafor, (1995); Pardini and Manhani, (2002) respectively. The tensile and Weibull properties of the spread E-glass fibres obtained are shown in Table 4.21.

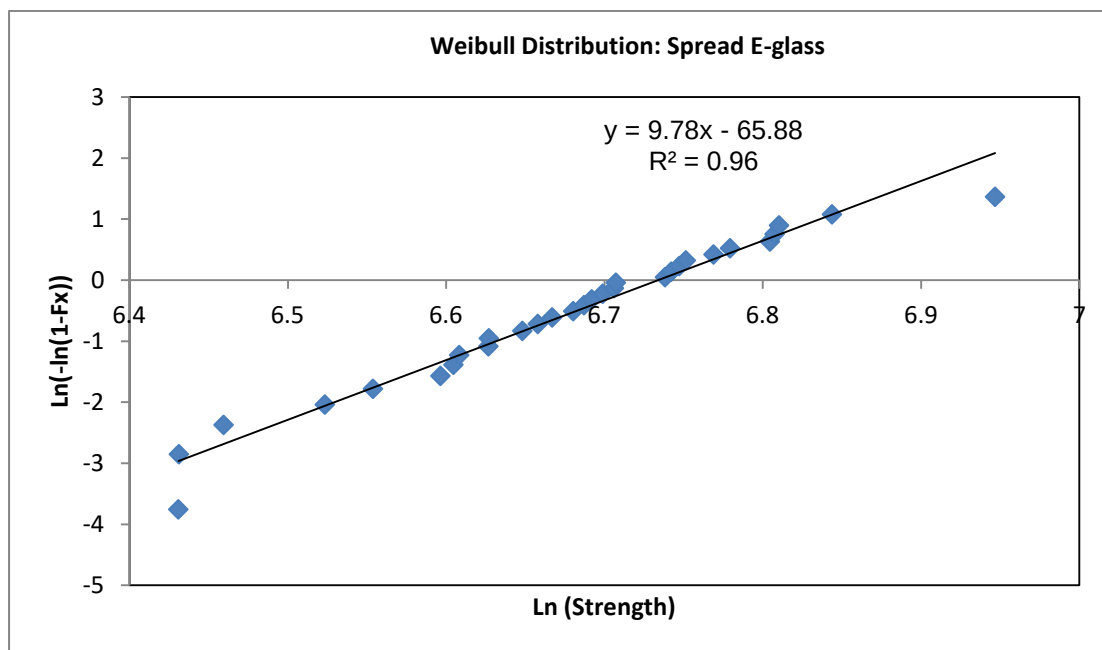


Figure 4.80 Weibull distribution of spread E-glass fibres.

Table 4.21 Tensile and Weibull properties of the spread E-glass fibre.

Researcher	N	Tensile properties of spread E-glass			Weibull Properties		
		Av. Strength	Av. Stiffness	Av. Elongation	Shape parameter	Scale parameter	R ²
		(MPa)	(GPa)	(%)		(MPa)	
Present work	30	800.47 ± 95.18	56.86 ± 6.78	1.59 ± 0.23	9.78	842.35	0.96

N=number of samples tested

4.10.2 T700 Spread carbon fibre reinforcement

The T700 carbon fibre bundle supplied by Toray carbon fibre Europe was spread using a custom-designed fibre spreading rig discussed in Section 3.5. The spread fibres were used in the production of composites with and without coir fibres. The properties of the fibre were supplied on the manufacturer's data sheet.

4.10.2.1 Tensile and Weibull properties of T700 Spread carbon fibre

Tensile tests were carried out on the spread carbon as was done for spread E-glass using the same test conditions. Figure 4.81 shows the stress-strain curves obtained from the tensile tests. The maximum, mid and minimum failure stresses obtained from the test were 2718.14, 2580.05 and 2400.09 MPa respectively. The average failure stress was 2528.95 MPa . From literature, an average failure stress of 2466.68 MPa for T700 spread carbon at 100mm gauge length was obtained, 2540 MPa was obtained by Pardini,; Manhani, (2002) on carbon fibre bundle. The Weibull distribution of the spread T700 carbon fibre is shown in Figure 4.82.

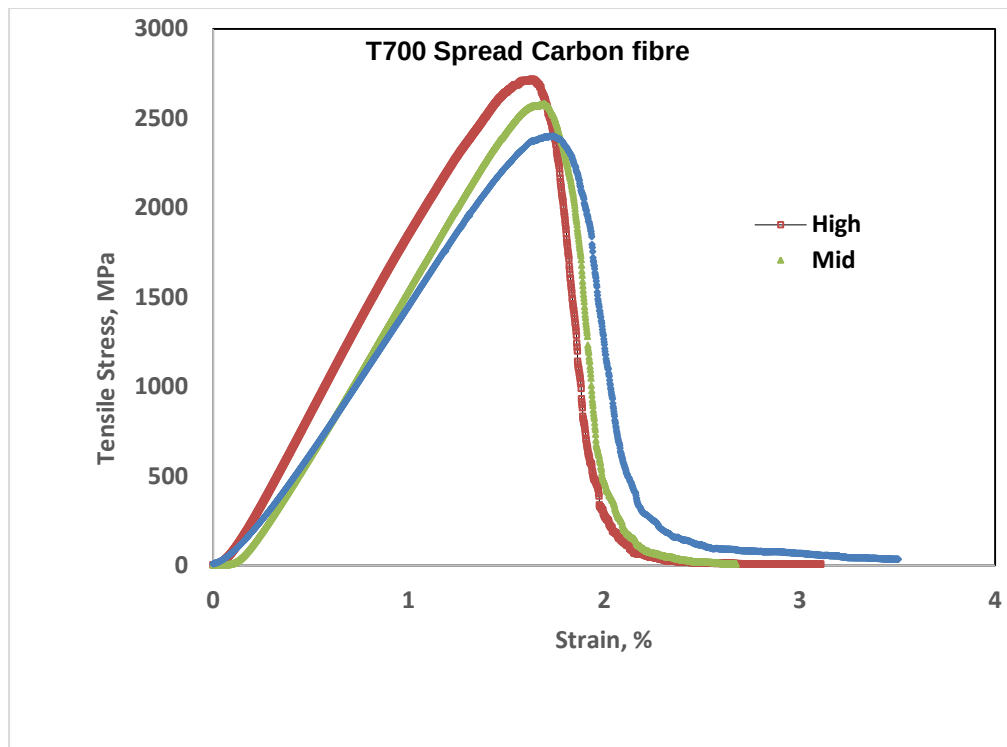


Figure 4.81 Stress-Strain curves of T700 Spread Carbon fibre.

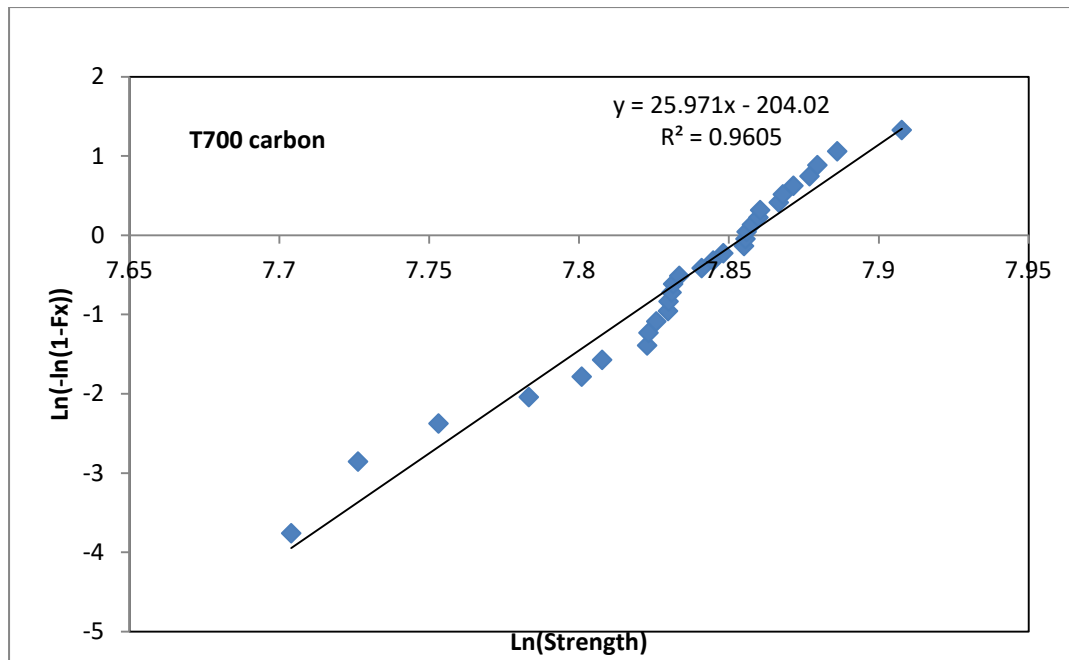


Figure 4.82 Weibull distribution of T700 Spread Carbon fibre.

4.10.3 Matrices for composites manufacture

The matrices considered for use in the composites manufacture were synthetic and bio-epoxy. These were chosen as a result of their different properties.

4.10.3.1 Synthetic epoxy/amine thermosetting resin

Synthetic epoxy/amine thermosetting resin by name; Araldite LY 3505 (epoxy resin) and hardener XB 3403 (amine hardener) are commercially available laminating resin system used for manufacturing composites. The tensile properties of the cast epoxy/amine resins were determined. The stress-strain curves obtained from the tensile tests are as shown in Figure 4.83. The average tensile strength and modulus obtained from the tensile tests were 43.87 ± 0.86 MPa and 2.67 ± 0.93 GPa respectively. These values are lower than those obtained by (Kim, 2014). This can be attributed to the ageing and storage conditions.

The epoxy amine failure shows an initial elastic linear region. The epoxy/amine failure displays a brittle failure at high, mid and low tensile stresses. The maximum, mid and minimum failure stresses observed were 44.73 MPa, 43.04 MPa and 41.89 MPa respectively.

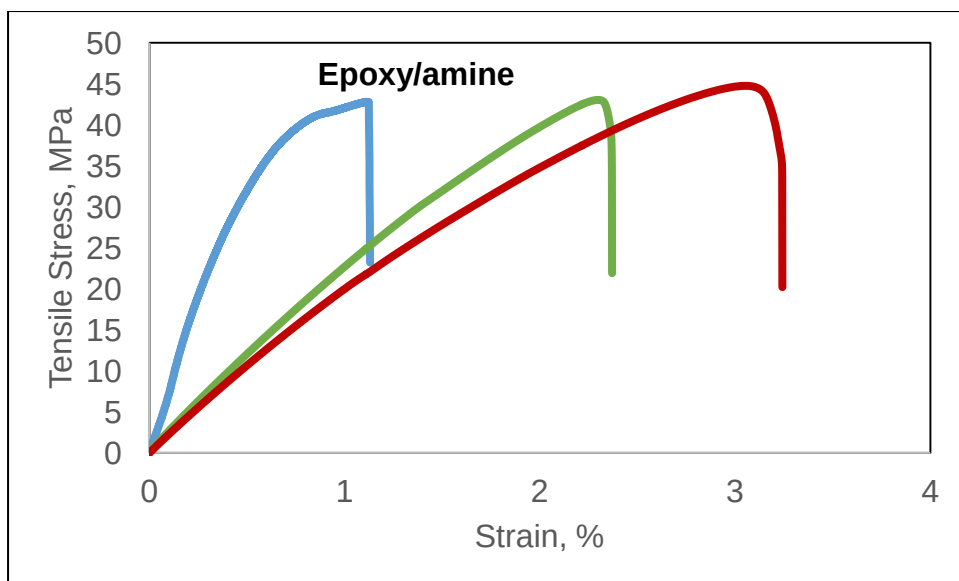
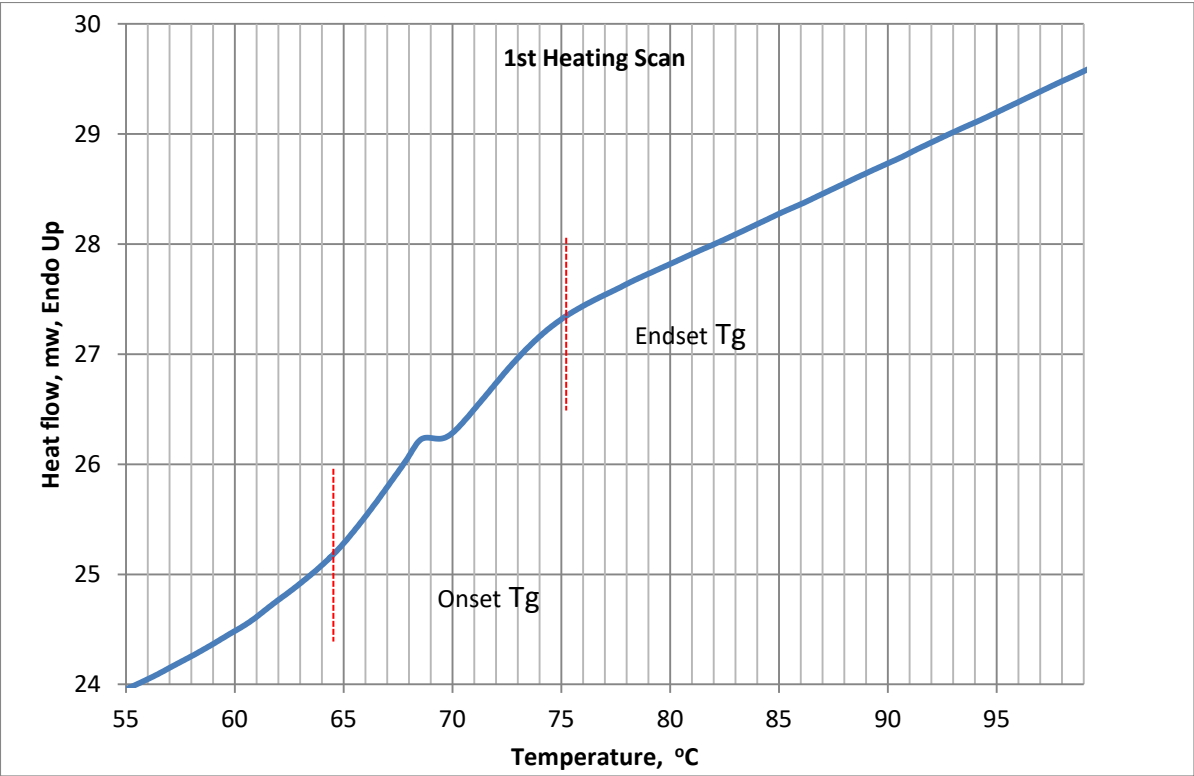


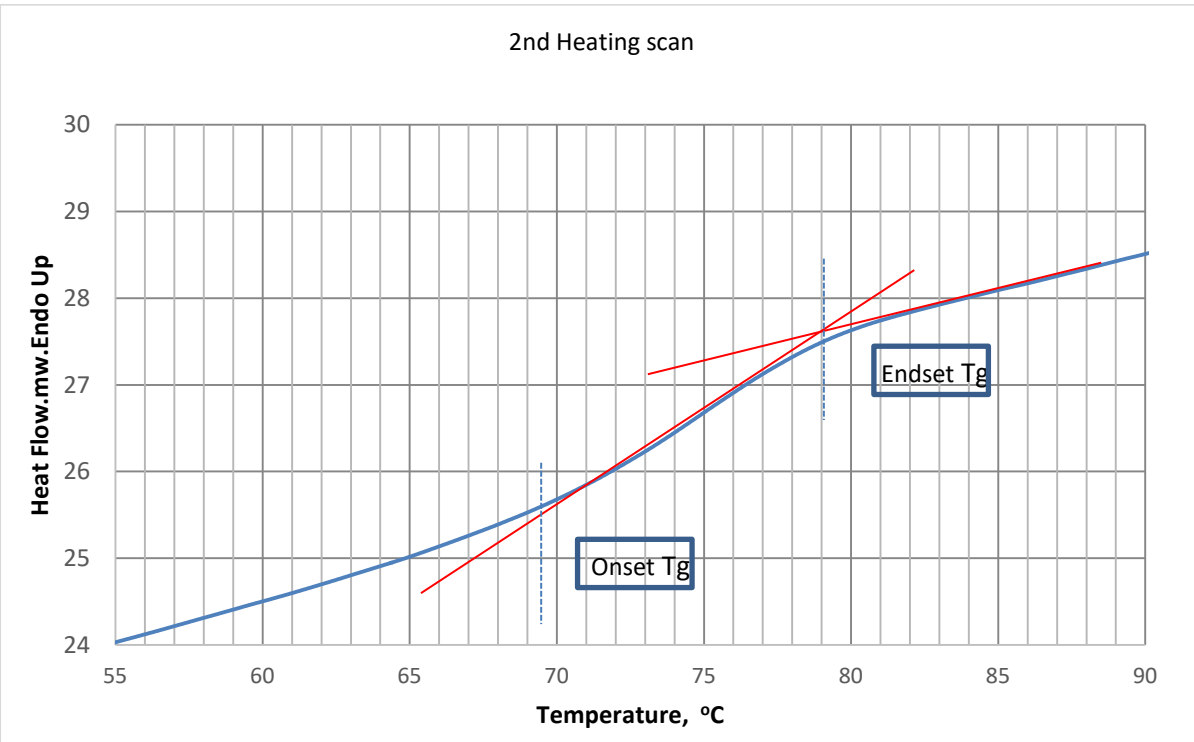
Figure 4.83 Stress-Strain curve of Araldite LY 3505 (epoxy resin) and Hardener XB 3403 (amine hardener).

4.10.3.1.1 Differential Scanning Calorimetry (DSC) traces of cast synthetic epoxy/amine thermosetting resin (Araldite LY3505 and XB 3403 hardener)

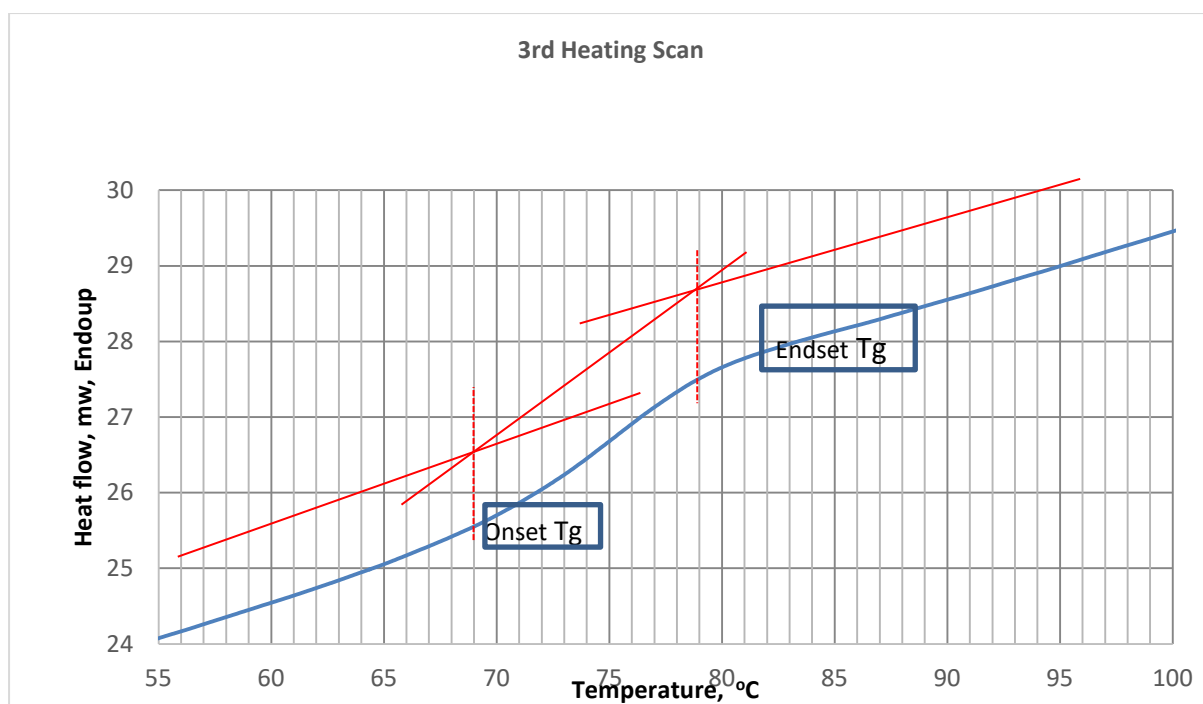
The glass transition temperature (T_g) is the temperature at which a solid material when heated becomes viscous. The glass transition temperature is usually lower than the melting temperature of the material (for semi-crystalline materials). DSC tests were carried out on the cast sample of LY3505-epoxy resin and XB 3403 hardener to determine the glass transition temperature of the cast epoxy/hardener. The T_g temperature was obtained using the Perkin Elmer thermal analysis technique. Through the tangent method the glass transition temperature value of the sample was obtained as shown in Figures 4.84. The T_g of the cast epoxy/hardener obtained via the first, second and third heating scans is shown in Table 4.22. The average T_g value of the epoxy/hardener obtained was 73.05 °C.



(a)



(b)



(c)

Figure 4. 84 (a) first heating scan (b) second heating scan and (c) third heating scan of epoxy/hardener.

Table 4.22 Glass transition temperatures for the LY3505 epoxy resin and XB3403 amine hardener.

Heating scan	Onset Tg (°C)	Endset Tg (°C)	Midset Tg (°C)
1 st	64.9	75.1	70.00
2 nd	69.5	79.1	74.30
3 rd	69.9	79.8	74.85
Average			73.05

4.11 Manufacture of composites

The composites were manufactured using hand layup, vacuum pump and autoclave methods as previously described in Section 3.7.

z composites were manufactured using vacuum pump (VP), autoclave (AC) and hand lay up (HLU) in single, double and triple layers.

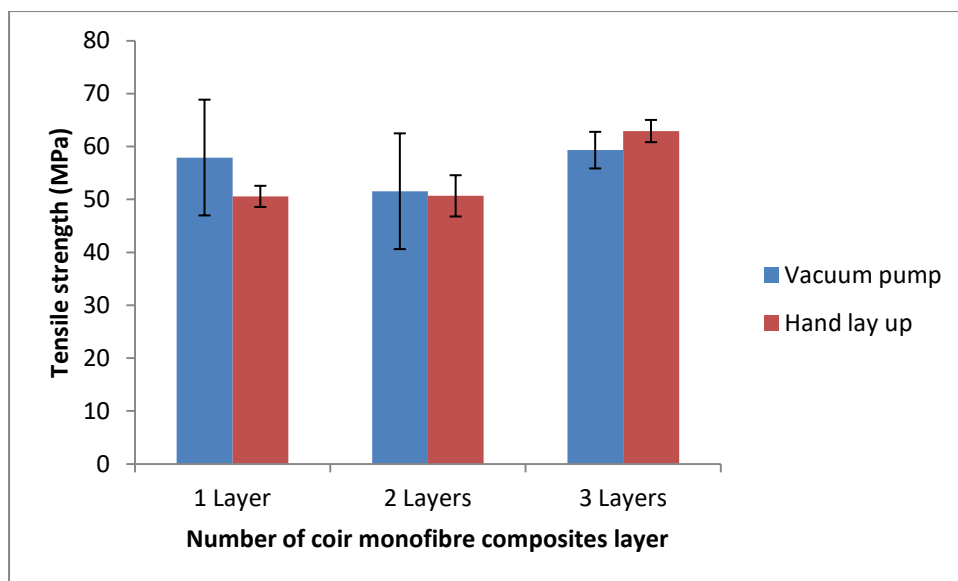
4.11.1.1 Tensile test results of the manufactured coir composites

The average tensile strength obtained using hand lay up for single, double and triple layers of coir composites were 50.57 MPa, 50.67 MPa and 62.92 MPa respectively. These values are higher than the values obtained from the literature. The average tensile strengths obtained from the literature for coir composites are 30.44 MPa (Rait, 2014) and 28.3 MPa (Kim, 2014). The difference in properties could be as a result of fibre origin, specie or age. Table 4.23 shows the tensile properties of the manufactured coir composites. Figure 4.85 displays the comparison of the tensile properties of coir composites with their manufacturing methods.

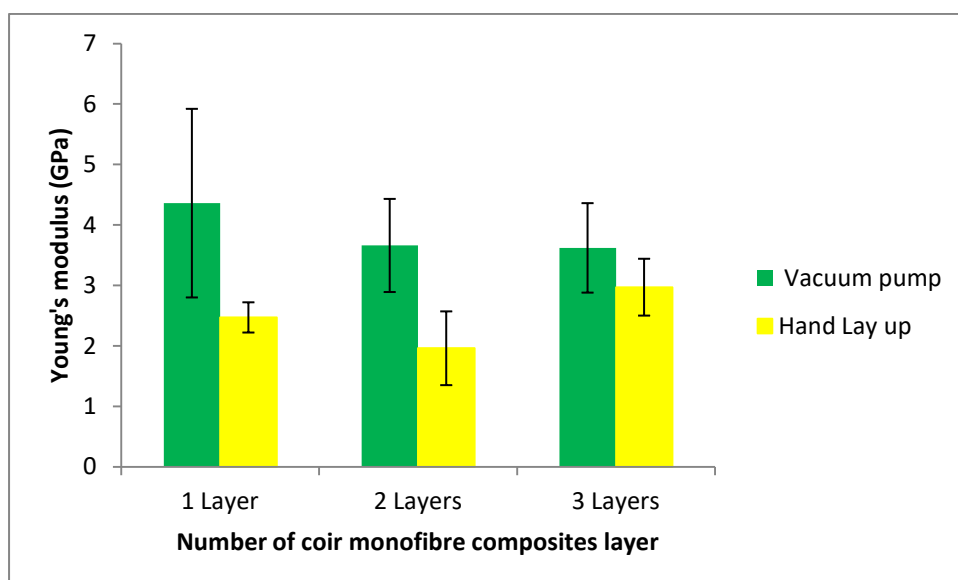
Table 4.23 Tensile properties of the manufactured coir composites.

Manufacture method	No of layers	Thickness (mm)	Width (mm)	Tensile strength (MPa)	Young's modulus (GPa)
Vacuum pump (VP) (untreated)	1	0.61 ±0.19	12.82 ± 1.83	57.91±10.94	4.36 ± 1.56
	2	1.25 ± 0.26	13.80 ± 1.55	51.54 ±10.94	3.66 ± 0.77
	3	1.58 ± 0.26	15.00 ± 0.00	59.31 ± 3.46	3.62 ±0.74
Hand Lay Up (HLU) (untreated)	1	1.04 ± 0.22	16.86 ±2.51	50.57 ± 2.00	2.47 ± 0.25
	2	1.49 ± 0.27	20.47 ± 0.77	50.67 ± 3.90	1.96 ± 0.61
	3	1.73 ± 0.12	20.40 ± 0.50	62.92 ± 2.10	2.97 ± 0.47
VP (S-treated)	2	1.6 ± 0.19	17.06 ± 0.45	47.77 ± 4.77	1.11 ± 0.39
Autoclave (Untreated)	1	0.38 ±0.08	15.91±1.10	34.14 ± 7.30	1.12 ± 0.64

S-Treated=Silane treated; No=number; treated/untreated=coir fibre treated/untreated



(a)



(b)

Figure 4.85 Comparisons of the tensile properties of coir composites with their manufacturing methods (a) strength (b) Young's modulus.

An analysis of variance (ANOVA) was carried out on the tensile properties of the coir composites on Minitab 19 Software to establish their statistical significance. A P-value of 0.000 was obtained showing that there is significant differences in the tensile properties of the coir composites manufactured using different processing techniques. Further analysis using Fisher pairwise comparison by difference of means as shown in Table 4.24 shows that the tensile strengths of HLU 3L (3 layer by Hand layup) and VP 3L are similar because they share the same letter (A) and therefore they are not statistically different from each other. The

same reasoning goes for VP 3L and VP 2L as well as VP 2L, HLU 2L, and HLU 1L. However, the tensile strength of HLU 3L is completely different from those of VP 1L, VP 2L, HLU 2L, HLU 1L and VP 2L because they contain different letters as shown in Table 4.24.

Table 4.24 Fisher Pairwise comparisons of coir composites.

Strength			Stiffness		
Factor	Mean	Grouping	Factor	Mean	Grouping
HLU 3L	64.13	A	VP 2L	3.78	E
VP 3L	58.54	A B	VP 3L	3.61	E
VP 2L	52.99	B C	VP 1L	3.34	E F
HLU 2L	51.58	C	HLU 3L	2.91	E F G
HLU 1L	51.44	C	HLU 1L	2.42	F G
VP 1L	43.57	D	HLU 2L	1.93	G

4.11.1.2 Correlation between the thickness and the tensile properties of coir composites

A difference of 51% in thickness was recorded between single and double layer in the VP method. A reduction in the thickness of one layer by 41%, two layers by 16% and three layers by 9% were observed when the composite manufacture was switched from HLU to VP.

The correlation between the thickness of the composites and the composites properties is shown in Table 4.25. For the vacuum pump manufacturing technique; no correlation was observed between the strength and the thickness in single layer coir composites, this is probably because the thickness of the single layer is more like the diameter of single fibre. However strength is found to increase with decreasing thickness in double and triple layer for vacuum pump manufacturing. The effect of specimen thickness on the stiffness for both single and double layer is non-significant however as the specimen increases for triple layer; the stiffness also increases for the vacuum pump technique. As in the vacuum pump, no correlation exists between the strength and the specimen thickness in the single layer for

HLU. Furthermore, no significant relationship exists between the strength and thickness in triple layers.

Table 4.25 Relationship between the thickness and the tensile properties of the monolayer coir composites manufactured using HLU and VP techniques.

Single layer					
MT	Thickness (mm)	Tensile strength (MPa)	Correlation between strength and Thickness	Young's modulus (GPa)	Correlation between YM and thickness
HLU	1.04 ± 0.22	50.57 ± 2.00	0.05	2.47 ± 0.25	-0.79
VP	0.61 ± 0.1	57.91 ± 10.94	0.02	4.36 ± 1.56	-0.10
Double layer					
HLU	1.25 ± 0.26	51.54 ± 10.94	-0.62	3.66 ± 0.77	0.09
VP	1.49 ± 0.27	50.67 ± 3.90	0.43	1.96 ± 0.61	0.73
Triple Layer					
HLU	1.58 ± 0.26	59.31 ± 3.46	-0.72	3.62 ± 0.74	-0.80
VP	1.73 ± 0.12	62.92 ± 2.10	-0.00	2.97 ± 0.47	0.32

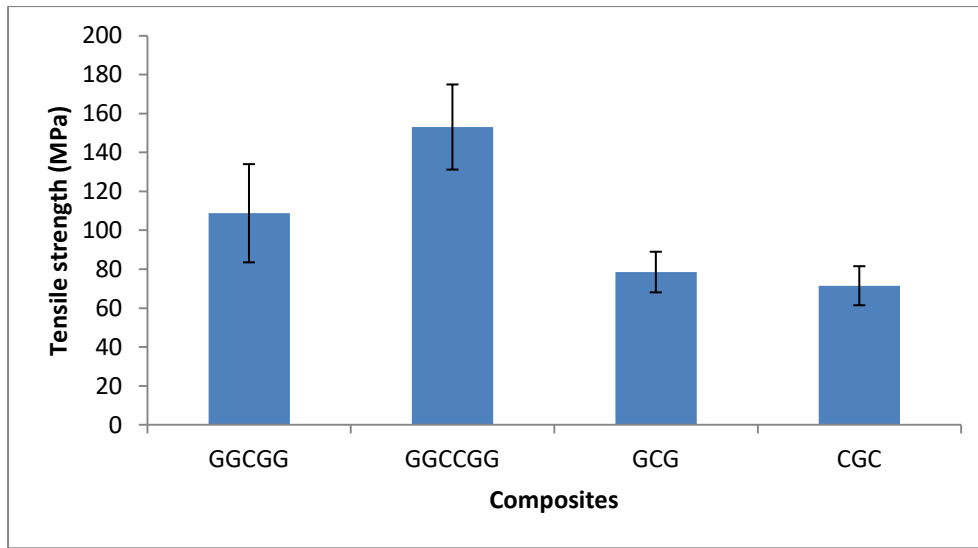
MT=manufacturing technique

4.11.2 Tensile properties and statistical analysis of coir sandwich composites

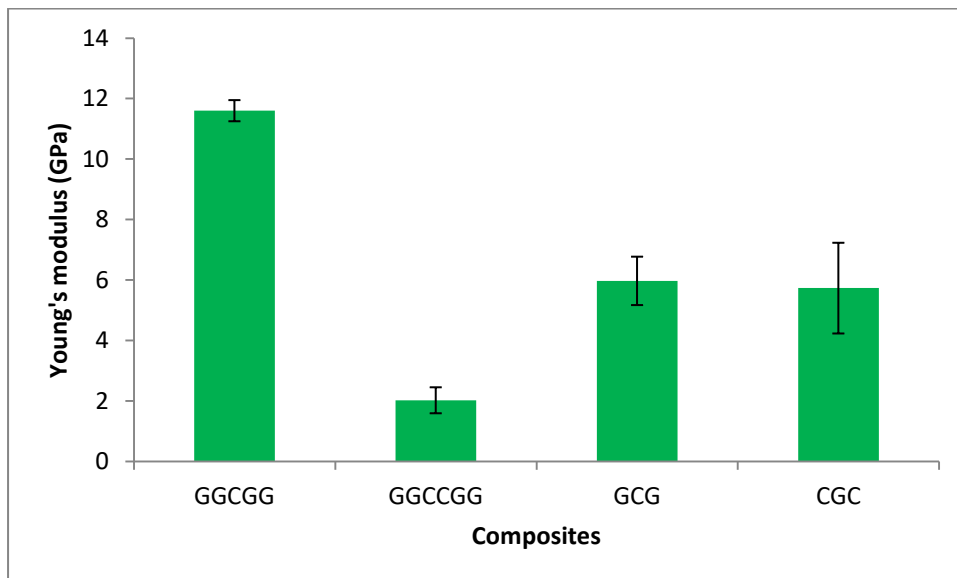
The tensile properties of the manufactured sandwich composites are as shown in Table 4.26. An average tensile strength and Young's modulus of 153.07 MPa and 5.97 GPa respectively were obtained from 2Glass 2Coir 2Glass. The single stack GCG and CGC had average tensile strengths of 78.55 and 71.52 MPa respectively. Figure 4.86 shows the highest tensile strength at GGCCGG. However the tensile strength obtained elsewhere for GCG and CGC were found to be 47 MPa and 52 MPa respectively (Jayabal, Natarajan and Sathiyamurthy, 2011). The tensile properties obtained are higher than the ones in the literature.

Table 4.26 Properties of coir sandwich composites.

Manufac turing. method	Sandwich Pattern	Thickness (mm)	Width (mm)	Tensile strength (MPa)	Young's modulus (GPa)
Hand Lay Up	2Glass 1Coir 2 Glass	1.11 ± 0.07	17.33 ± 1.23	108.77 ± 25.24	11.60 ± 0.35
	2Glass 2Coir 2 Glass	1.84 ± 0.19	18.32 ± 0.49	153.07 ± 21.88	2.02 ± 0.43
	Glass Coir Glass	1.09 ± 0.16	20.58 ± 0.96	78.55 ± 10.44	5.97 ± 0.80
	Coir Glass Coir	1.34 ± 0.08	14.83 ± 0.41	71.52 ± 10.04	5.73 ± 1.50



(a)



(b)

Figure 4.86 Comparisons of the tensile properties of coir sandwich composites (a) strength and (b) Young's modulus.

The P –Values obtained from ANOVA for the tensile strength and Young's modulus are less than 0.05. However further analysis using Fisher pairwise grouping as shown in Table 4.27 shows that 2G2C2G is totally different from the rest. However 2GC2G, GCG, CGC are not different from one another.

Table 4.27 Fisher Pairwise comparison for difference of means for coir sandwich composites.

Tensile strength			Young's modulus		
Factor	Mean	Grouping	Factor	Mean	Grouping
2G2C2G	153.4	A	2GC2G	11.60	A
2GC2G	108.8	B	CGC	6.8500	B
GCG	81.87	B	GCG	5.827	B
CGC	78.91	B	2G2C2G	2.223	C

4.11.2.1 Coir sandwich composites: Correlation between the specimen thickness and the tensile properties

The increase in thickness of the coir sandwich composites is as follows; 2G2C2G > CGC > 2GC2G > GCG thus 2G2C2G is thicker than CGC, 2GC2G and GCG by 27%, 39.67%, and 40.76% respectively. Strength decreases thus; 2G2C2G > 2GC2G > GCG > CGC and stiffness 2GC2G > GCG > CGC > 2G2C2G. The correlation between the thickness of the composites and the composites properties is as shown in Table 4.28. For all the sandwich composites except 2GC2G, both the strength and the stiffness increase as the thickness decreases. 2GC2G rather shows a dip in the tensile strength as the thickness dips though it is insignificant. This appears to be an anomaly, therefore further investigations may be required to re- analyse this group of composites. An increase of approximately 47 MPa was observed in 2G2C2G when compared with 2GC2G; this can be attributed to the two layers of Carbon fibre present in 2G2C2G. However, 2GC2G gained about 9.5 GPa higher than 2G2C2G in Young's modulus, this appears to be an anomaly.

Table 4.28 Correlation between the specimen thickness and the tensile properties for coir fibre sandwich composites.

Composite Type	Thickness (mm)	Tensile strength (MPa)	Correlation between Strength and thickness	Young's modulus (GPa)	Correlation between Young's modulus and thickness
GCG	1.09 ± 0.16	78.55 ± 10.44	-0.97	5.97 ± 0.80	-0.59
CGC	1.34 ± 0.08	71.52 ± 10.04	-0.54	5.73 ± 1.50	-0.52
2GC2G	1.11 ± 0.07	108.77 ± 25.24	0.32	11.60 ± 0.35	0.28
2G2C2G	1.84 ± 0.19	153.07 ± 21.88	-0.54	2.02 ± 0.43	-0.39

4.11.3 Failure modes of the manufactured composites

The failure modes of the manufactured coir and sandwich composites differ. These differences were attributed to their manufacturing techniques, the number of layers and their stacking sequence.

4.11.3.1 Failure mode of composites manufactured using Vacuum pump

The failure modes of the coir composites manufactured using vacuum pump were similar in all the layers as shown in Figure 4.87. The initial linear regions are about 70-80% of the failure trace in both 2 and 3 layer composites but about 60% in single layer. The maximum failure stress for 2 and 3 layers was 60 MPa. However the maximum failure stress for the single layer was 48 MPa. The minimum failure stress of 52 MPa was recorded for the triple layer. This is about 14 and 25 % .higher than the minimum failure stress for the double and single layer respectively.

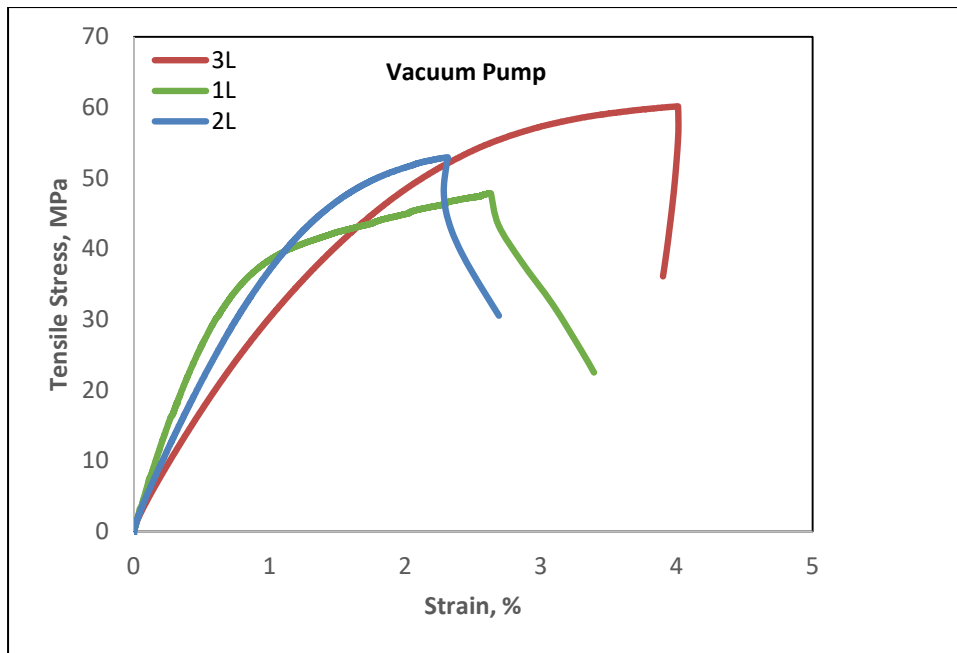


Figure 4.87 Comparison of stress-strain curves of single, double and triple layer coir composites manufactured using vacuum pump.

4.11.3.2 Failure mode of composites manufactured using Hand layup (HLU)

The initial elastic region is at about 70% of the failure trace in all the layers for composites manufactured using hand lay up as shown in Figure 4.88. In the single layer, the difference between the maximum failure stress and the minimum failure stress was 1.12 (2%). For the second layer, a difference of 7% was observed between the maximum failure stress and the minimum failure stress. A difference of 8% existed between the maximum failure stress and the minimum failure stress in the third layer. As the number of layers increased, the disparity in the failure stresses also increased.

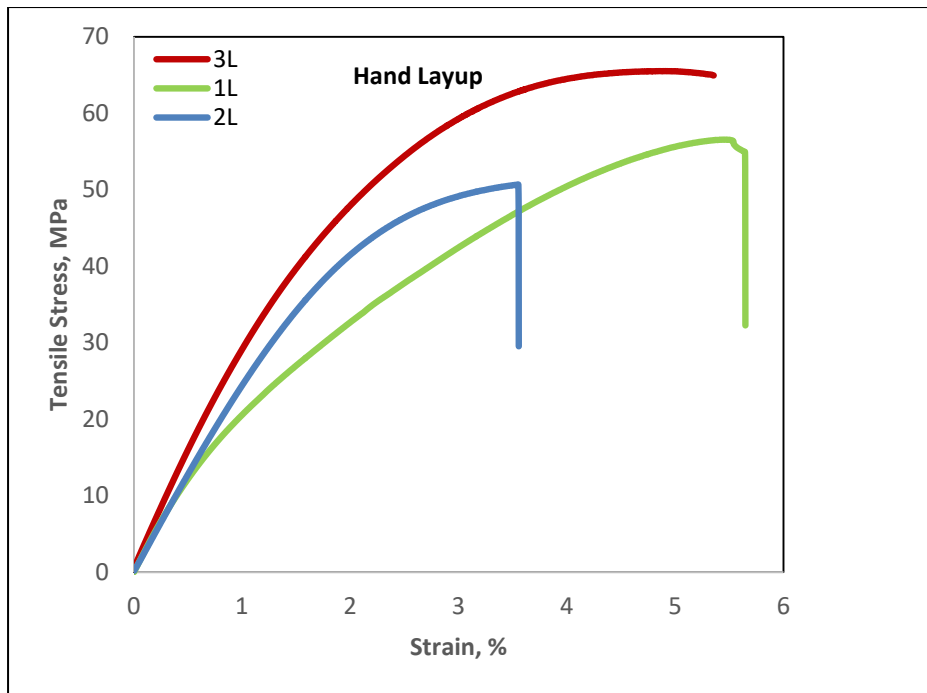


Figure 4.88 Comparison of stress-strain curves of single, double and triple layer coir composites manufactured using hand layup technique.

The failure traces for a single layer from the different manufacturing techniques are somewhat similar however failure occurred at different strain points as shown in Figure 4.89. Coir composites manufactured via hand layup exhibited the highest failure stress with 15 and 22% higher than those manufactured via the vacuum pump and autoclave respectively.

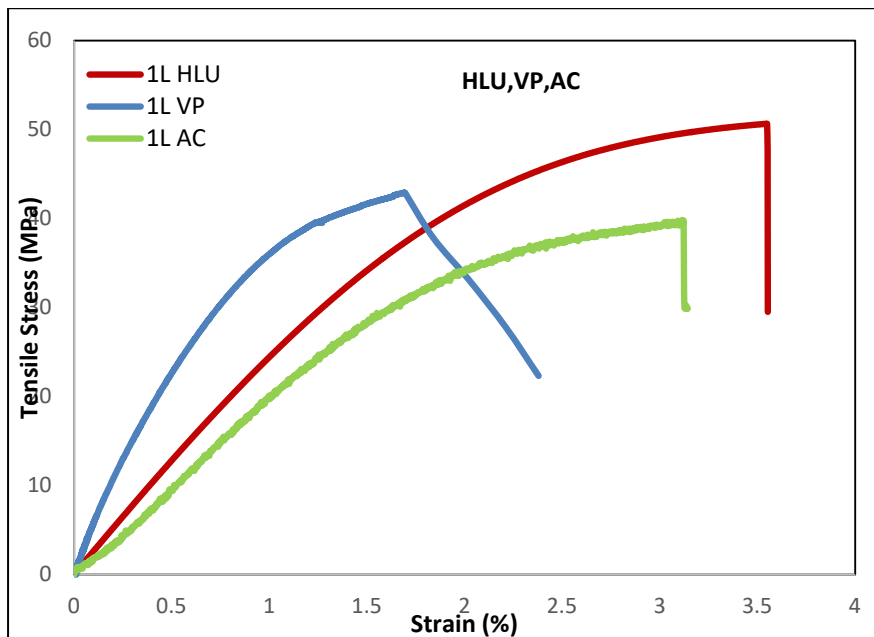


Figure 4.89 Comparison of stress-strain curves of coir composites manufactured using hand layup, vacuum pump and autoclave techniques.

4.11.3.3 Failure mode of amino silane treated coir composites manufactured using hand layup

The failure traces for amino-silane treated coir shows a long initial linear region of about 60% of the curve as shown in Figure 4.90. A brittle fracture occurred after the plastic deformation. No significant difference was observed between the mid and minimum failure stresses. The maximum failure stress was .higher than the minimum failure stress by 17%.

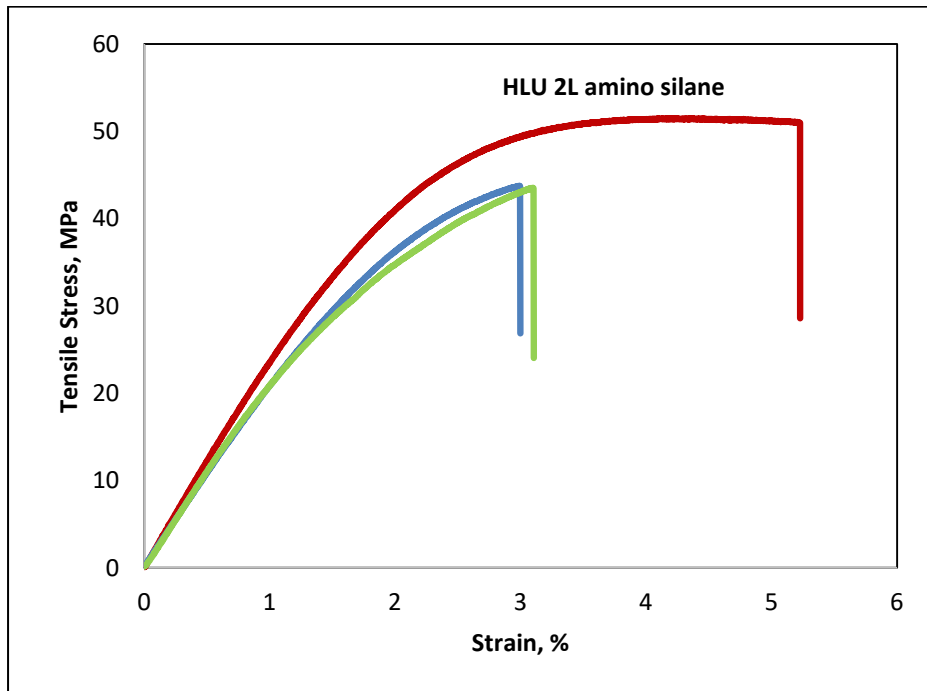


Figure 4.90 stress-strain curves of amino silane treated coir composites.

4.11.3. 4 Failure mode of single layer Spread E-glass composites manufactured using hand layup

Figure 4.91 presents three representative stress-strain curves from the spread E-glass composites. The failure of the unidirectional spread E-glass composites was mainly characterised by longitudinal splitting. Linear loading behaviour was observed in all the failure traces. The maximum and minimum observed failure stresses were 740.25 MPa and 555.88 MPa respectively.

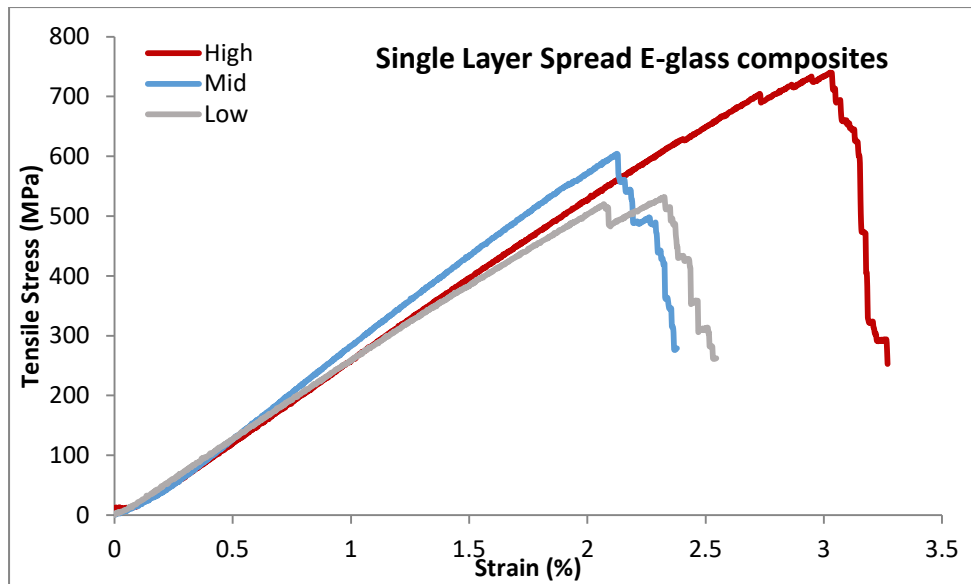


Figure 4.91 Stress-Strain curves of single layer spread E-glass composites.

4.11.3. 5 Failure modes of GCG in comparison with CGC composites manufactured using hand layup

GCG and CGC display similar failure pattern except that GCG traces show linear response up to the fracture point as shown in Figure 4.92. The failure for both is a typical sudden fracture characteristic of non-metals. The maximum failure stress of GCG is only 6% higher than the maximum failure stress of CGC. This suggests that one can be used in place of the other depending on the ease and availability.

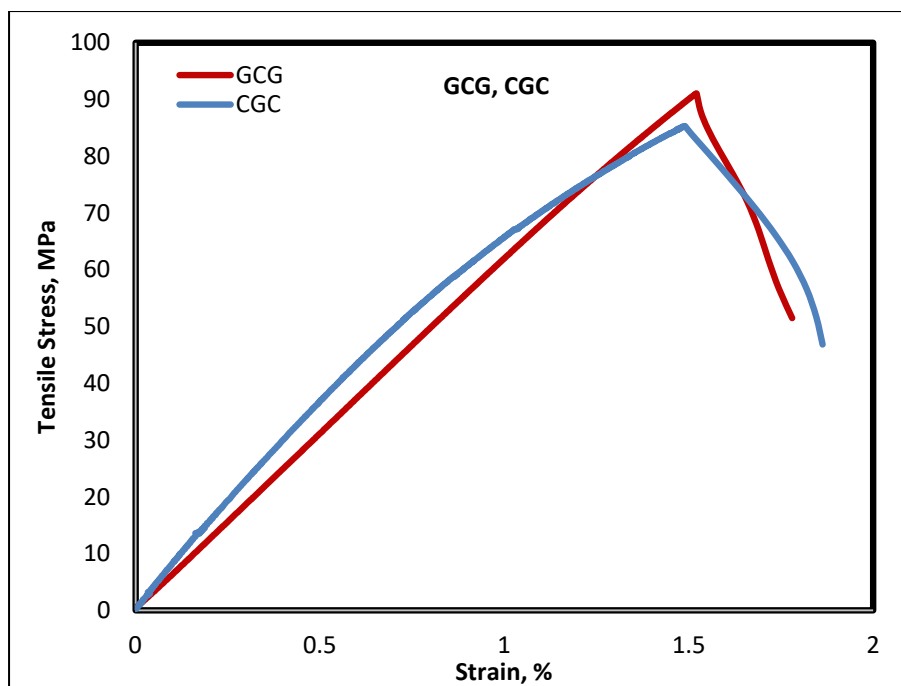


Figure 4.92 Comparison of Stress-Strain curves of GCG and CGC.

4.11.3. 6 Failure mode comparisons of sandwich composites manufactured using hand layup

The maximum and minimum failure stresses for the 2C2G2C was 146.23 MPa and 128.84 MPa respectively. The maximum failure stress for the 2GC2G was 80 MPa, this is about 55% and 38% short of the maximum and minimum failure stresses of 2G2C2G. This can be attributed to the increase in the thickness of the faces rather than the core.

4.11.4 Microstructures of the manufactured coir composites and the fibre volume fraction in the composites

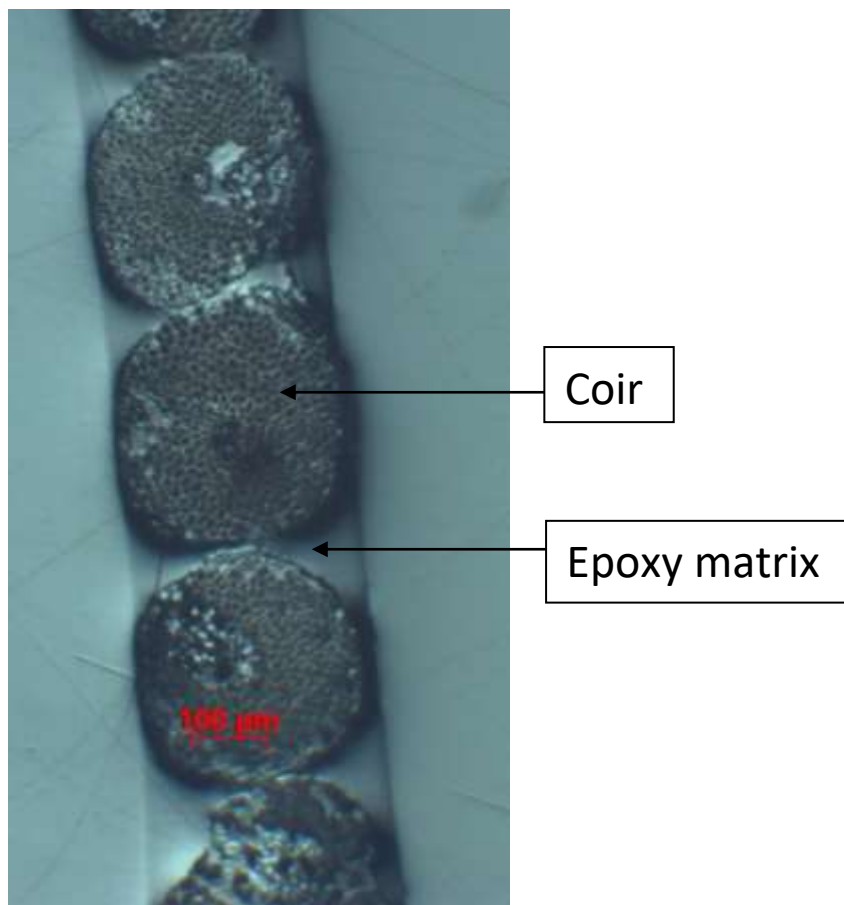
A laboratory microscope Zeiss Axioskop-2 was used to obtain the optical images of polished coir fibre composites used in the determination of the volume fraction of fibres in the composites.

The micrographs of coir composites to be analysed were opened on ImageJ and the procedure carried out as previously described. The analysis was repeated for five times per sample. Similar procedures were carried out for double and triple layers. The results obtained from the analysis are shown in Tables 4.29-4.31.

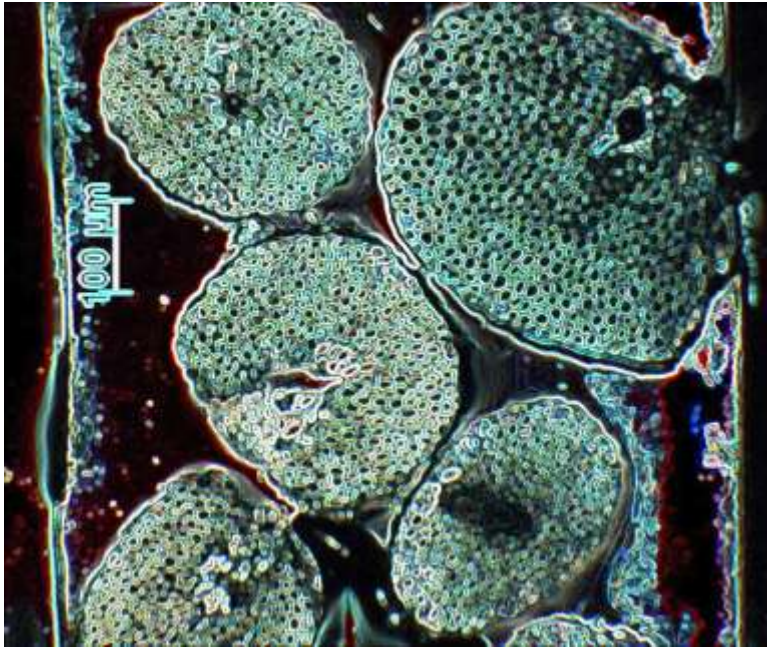
The average volume fraction of the fibres obtained for single, double and triple layers were 82%, 65% and 49%. The high volume fraction of the single layer is as a result of the close

contacts between the fibres, obvious in its microstructure as shown in Figure 4.93a. Less resin was required to impregnate the single layer than the double and triple layer fibres. The reduction in the volume fraction of the double and triple layer could be as a result of gaps existing between the fibres (fibre to fibre) and between the layers (layer to layer) within the composite as shown in Figure 4.93 (b) and (c) respectively. A significant difference exists between the volume fractions of single, double and triple layers.

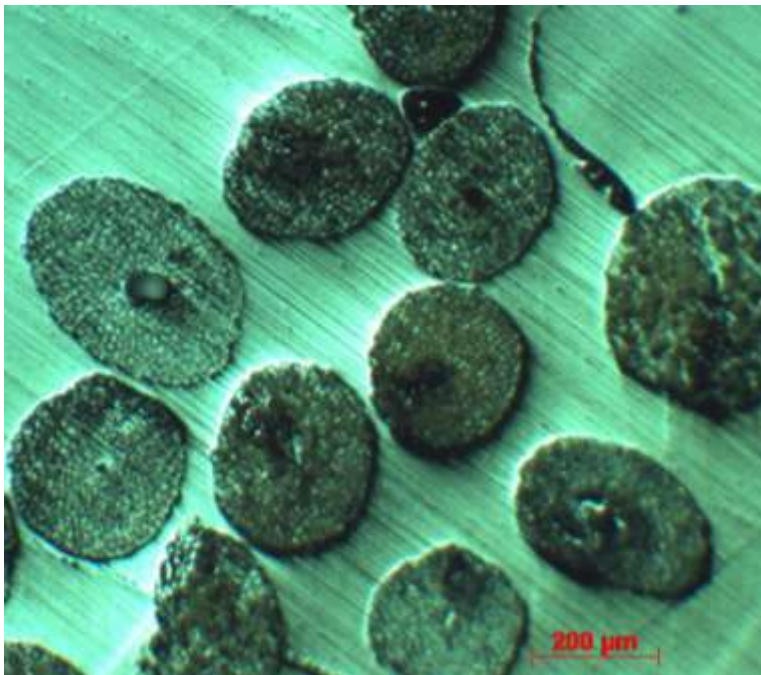
From Section 4.11.3, the tensile strength increased significantly as the number of coir layers increased from single to triple layer. Strength has been reported to increase with the increase in the fibre volume fraction up to a certain threshold point. 34%, 40% and 50% fibre volume fractions improved the tensile strength of coconut/epoxy, sisal/epoxy and coir/ polybutylene succinate composites respectively (Silva *et al.*, 2013; Bisanda and Ansell, 1991; Joseph *et al.*, 2002). Hence the highest tensile strength of coir composites was obtained in the triple layer irrespective of its lower fibre volume fraction when compared with single and double layer.



(a)



(b)



(c)

Figure 4.93 (a) Single (b) double (c) triple layer coir fibre composites.

4.11.5 Microstructure of spread E-glass and coir sandwich composites

The microstructures of spread E-glass and coir sandwich composites were investigated to determine their contributions to the mechanical properties of the composites. A monolayer post-spread E-glass fibre reinforced epoxy composite was successfully produced as shown in

Figure 4.94 from a high quality post-spread architecture. The retention of the quality architecture during manual impregnation in the manufacture of sandwich composites could not be guaranteed. This must have affected the tensile properties of coir-glass sandwich composites though strategies were adopted to produce coir-glass sandwich composites with minimal void content.

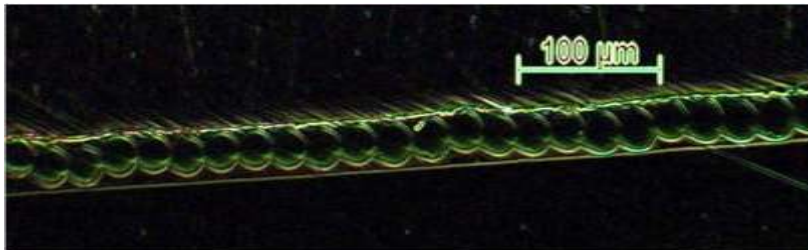
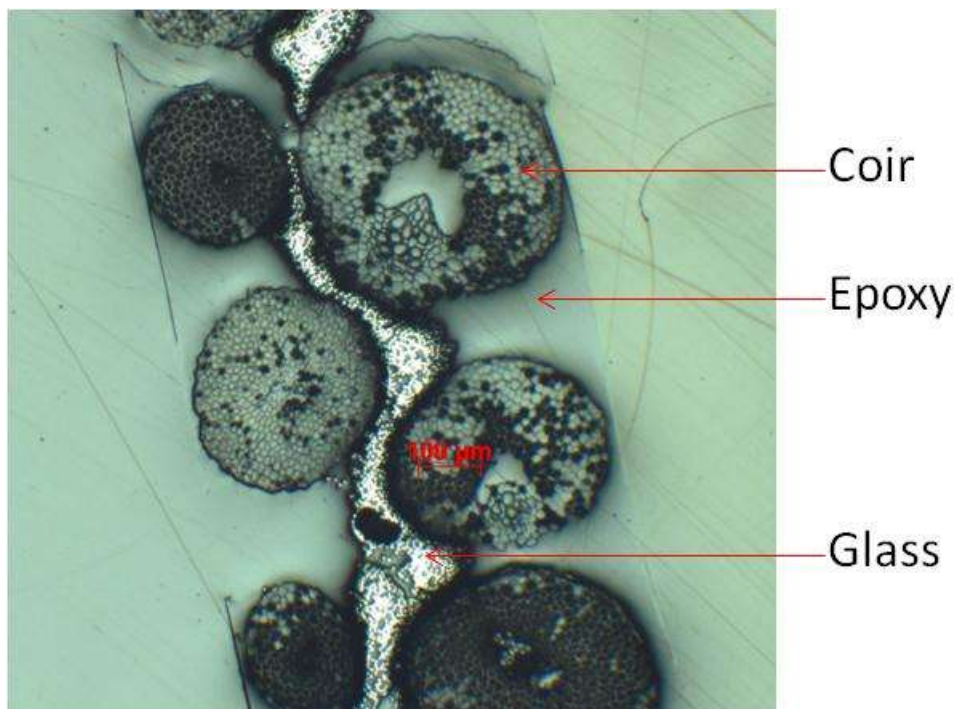


Figure 4. 94 Micrograph showing a monolayer of post-spread 2400 Tex E-glass fibre reinforced epoxy resin composite; magnification x10. Work undertaken in collaboration with T. Ma (PhD student, University of Birmingham).

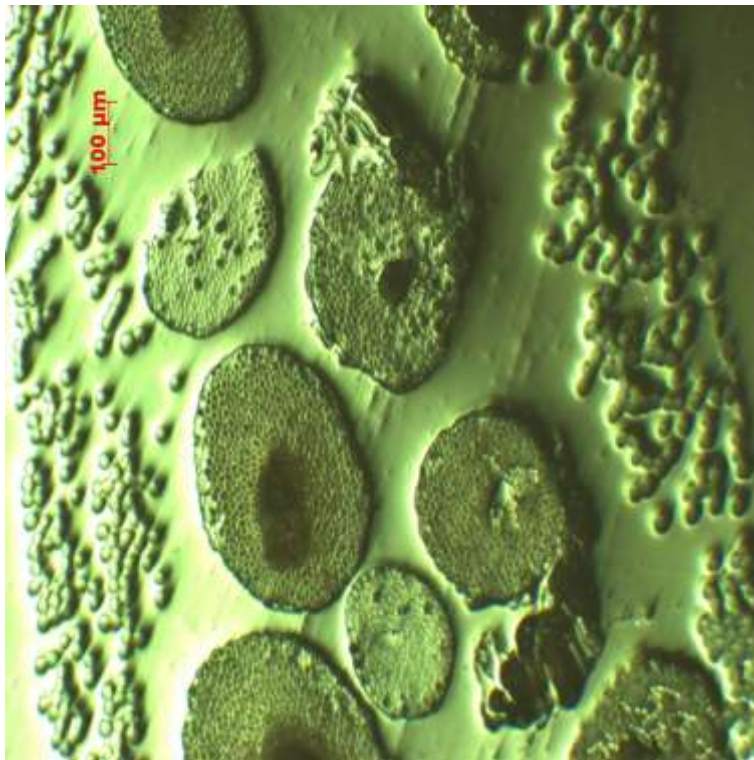
Figure 4.95 (a-c) shows micrographs of coir-spread glass-coir (CGC) composite; the E-glass-coir fibre interactions are not 100% effective as a result of the differences in the shapes and sizes of the coir fibre and E-glass. This non-uniformity could hamper the effective tensile properties of the composites.



(a) CGC



(b) 2C2G2C



(c) 2G2C2G

Figure 4.95 (a-c) shows micrographs of coir-spread-glass-coir (CGC) composite.

The breakage of the glass in Figure 4.95 could have been as a result of pressure exerted on the composite in trying to wet out excess resin during manufacture.

Summary

- (i) Coir fibre composites and coir-glass fibre hybrid composites have been produced using unidirectional coir fibre preforms.
- (ii) The volume fraction of fibre in the single layer of the coir composites was 21 and 40% higher than those of double and triple layer respectively. This was attributed to the close-packing of the fibre in the single layer.
- (iii) The highest tensile strength was achieved in triple layer coir composites.
- (iv) Glass-coir sandwich hybrid composites showed higher tensile strength by 59% than coir composites.

5 Conclusions

The following conclusions have been drawn from the research findings and analysis presented and discussed in the previous chapters:

The tensile properties of single coir fibres have been significantly improved. The percentage improvement in the tensile strength and Young's modulus were between 27 to 115% and between 41 to 188%. This was achieved via treatment with NaOH and hot-compaction. Tensile properties were influenced by the sample gauge length. The Weibull survival probability of as-received single coir fibre was obtained at a stress of 217.6 MPa. By zoning, coir fibre diameters were found to be most uniform towards the mid-section. The porosity of coir fibre is in the range of 34-48%. The number of pores as well as the pore size varies with the diameter.

NaOH brought about removal of tyloses and reduction in pore size with concentration exerting the highest influence on the coir fibre tensile strength and stiffness.

Hot compaction brought about reduction in the pore size with the interaction of temperature and duration exerting the highest influence on the tensile properties of AR compacted coir fibres.

Drying of coir fibres under vacuum and in the tube furnace resulted in decrease in the tensile strength properties of coir fibres by a minimum of 28 and 12% respectively however the Young's modulus was found to increase by up to 41% especially above 120 °C when the fibres were heat-treated in a vacuum.

The best method for improved mechanical properties of single coir fibre is via fibre treatment using NaOH and by hot compaction.

Unidirectional coir fibre composites have been produced in layers with significant increase in strength by 107% when compared to the results obtained in literature. A hybrid coir sandwich composite have been manufactured. A new technique was used via hand layup to achieve an increased tensile strength by 143% when compared to non-hybridized coir composites with the highest tensile strength achieved in sandwich composite; GGCCGG.

6 Recommendations for further work

- (i) An enhanced method of selecting fibres of fairly uniform diameter needs to be developed in order to obtain uniform and improved mechanical properties of the formed composites.
- (ii) Fibre treatment conditions need to be optimised to accommodate NaOH concentrations of more than 20% for possibly better mechanical properties.
- (iii) Further work is needed to be undertaken to use the treated coir fibres as reinforcements in the manufacture of composites and determine the degree of the interfacial bond strength.
- (iv) The manual method of aligning coir fibres to manufacture unidirectional coir fibre preforms needs to be improved upon. This is necessary to achieve gap-free unidirectional arrays with improved tensile strength.
- (v) Different compositions and stacking sequence of hybrid composites is necessary in order to establish the hybrid effect on the mechanical properties on the composites.
- (vi) Infilling of the pores for improved mechanical properties under pressure using suitable and compatible fluid needs to be carried out for enhanced tensile strength properties of single fibres and composites.

7 References

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