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Natural and Artificial Fiber-Reinforced Composites as Renewable Sources

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NATURAL AND ARTIFICIAL FIBER-REINFORCED COMPOSITES AS RENEWABLE SOURCES

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Notice

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Meet the editor

Ezgi Günay is presently an Associate Professor in the Mechanical Engineering Department at Gazi University in Turkey. Ezgi Günay graduated from the Engineering Sciences Department (Applied Mechanics Department) of the Engineering Faculty at the Middle East Technical University in 1985 and received her Master of Science degree in 1989. She received her PhD degree at Gazi University from the Mechanical Engineering Department in 1996. She has worked as a research assistant between 1993 and 1999 and studied academically as an assistant professor between 1999 and 2010 at the same department. She has been given courses in the following subjects: technical drawing, computer programming languages, engineering mathematics, statics, dynamics, strength of materials, numerical analysis, composite materials, finite element analysis, plate and shell theories, and elasticity. She has authored about 40 papers published in international conference proceedings and journals. She has a chapter published in *Progress in Sustainable Energy Technologies Vol. II* (2014). She has written two programming books (co-author) for the engineering students on Fortran language (*Workbook for FORTRAN 77* and *Programming Book for FORTRAN 77*) (2016). She has a chapter in the published book *Perusal of the Finite Element Method* (2016). She has received a certificate of appreciation for her outstanding contributions in reviewing journal papers from Elsevier publisher in 2016. She is presently working on the following subjects: finite element linear and nonlinear analyses of fiber composite-stiffened shell and plate structures, buckling analyses of fiber composite-stiffened plates, micromechanical investigation of fiber composites and natural composites, and experimental and numerical studies on wood materials.

Contents

Preface

Nano- and micro-sized natural fibers of vegetable origin are fully biodegradable in nature. However, the nano- and micro-sized synthetic fibers are fully man-made. Fiber-reinforced composites composed of stiffened fiber and matrix are well-known engineering materials. Fiber-reinforced materials have been used in industrial production. Natural fibers can be obtained from many sources in nature such as wool, sisal, ramie, kenaf, jute, corn tassel, hemp, grass, flax, cotton, coir, bamboo and abaca, banana, and sugarcane bagasse. Artificial fibers have been produced from more stiff materials such as glass, single-walled carbon nanotubes, double-walled carbon nanotubes, carbon, aramid, boron, and polyethylene (PE). The cyclic reusability of materials is an important qualification in protecting the environment from waste pollution. Three important factors can be mentioned in terms of material properties in the recycling process. The first factor is "the rate of cyclic usage," the second one is "less material loss in each recycle," and the last one is "the role of waste products in the self-renewal of ecosystem." In engineering area, the usage of waste materials has taken into account in production of composite materials. The use of waste materials as particulate-type composite production is also possible in the industry. Fiber-reinforced materials can be grouped into two categories: "the natural fiber-reinforced materials" and "the artificially produced fiber reinforced materials."

The book consists of seven chapters. In the first four chapters of this book, the properties of green and natural fiber materials as a reinforcing material have been mentioned. In the fifth chapter, epoxy material fibers for double-walled carbon nanotubes have been examined and tested for artificial reinforcement, and crack problem solution has also been performed. In the sixth chapter, the properties of the natural fibers and their artificial use in "man-made" fiber composite materials have been explained. Additionally, recycling of reinforced cement and textile materials as a waste material has been examined. In the seventh chapter, the advantages of sustainable bio-composites have been emphasized and their usage in the industrial area has been mentioned.

The first chapter that belongs to the editor has been mainly subjected about the actual detailed literature survey on "Natural Fiber Plastic Composites." This review covers a general overview of the preparation phase (chemical procedures), test techniques (experiments), and results and conclusion summaries (the gainings) of current studies on hemp fiber-reinforced plastic composites.

In the second chapter, "Interfacial Modification of Hemp Fiber Reinforced Composites ", the micromechanical studies on the characteristics of fiber and matrix interface and stress distribution have been summarized by Dr. Yekta Karaduman and his colleagues. The content described the industrially used hemp fiber-based composites and their interfacial modifications

in the consideration of shear stresses. In this century, determination of the most successful bonding between fibers and matrix by experimental and numerical studies is one of the most important research areas in mechanics and materials sciences. The aim of this chapter was to describe the methods to obtain a full transfer of applied external loads from weak matrix material to fibers as internal stress distributions. In this scope, firstly, the biological structure of the hemp fiber was explained. Mechanisms of fiber/matrix bonding in composite materials were outlined by theoretical and experimental scientific researches based on the literature. Samples of available techniques for the determination of interfacial strength were presented. Finally, the interface modification methods in the hemp fiber-reinforced composites were introduced.

In the third chapter titled "Development of Hemp Fibers: The Key Components of Hemp Plastic Composites," the results of recent scientific researches about this research area were explained by researchers Dr. Chernova T., Mikshina P., and their colleagues. The chapter's concept was based on the scientific researches for the material properties of processed green composite materials by applying the crop processing methodology. The growing strategies of the fibers are based on their extremely complex natural states within the planta. In this chapter, fiber and matrix combination in the green materials such as flax and hemp was expressed by examining their growing stages in nature and with their located environmental conditions. Considering the material and geometrical properties of the cells, thick cell walls, and their layers, the final dimensions of secondary fibers were compared with the primary fibers. Basically, in this research, studies were explained in detail by declaring the basic two concepts. The first one was the intrusive elongation, and the second was the deposition of thick cell wall layers. The microscopic views and pictures were given for detailed information about the mentioned results.

The fourth chapter titled "Effect of Fiber Waviness on Tensile Properties of Sliver-Based Natural Fiber Composites" by Prof. Dr. Eng. Koichi Goda and his team presented the results of their original research by explaining the two different specimen preparation methods. The mentioned two methods were different from each other as the sliver-based natural fiber composites with and without fiber waviness. The tensile test results showed that tensile strengths of direct method (DM) specimens accompanied by the fiber waviness were lower than those of sheet laminate method (SLM) specimens. The properties of fiber waviness on the specimens were quantified by two types of spatial autocorrelation analyses: the first one was Local Moran's I and the second was Local Geary's C. In the research, three-dimensional finite element analysis (3D FEA) was used to obtain the stress distribution numerically. At the end of the study, two failure criteria "Tsai-Hill" and "maximum stress" were used to discuss the tensile failure modes of the fiber composite material.

The fifth chapter titled "Mechanical and Fracture Surface Analysis of Higher Viscous Epoxy/Multi-Walled Carbon Nanotube Nano-Composites Subjected to Flexural Loading" waspresented by Prof. Dr. Aidy Ali and PhD degree student and researcher A.H. Muhammad Ismail and M.S. Risby. In their study, the results of the developing fracture surface in the fiber composite material were explained by experimental studies. The effects of MWCNT weight percentage (wt%) to flexural and fracture toughness properties were investigated for Mode I fracture behavior. In the chapter content, two important points were explained in an order: firstly, the significant enhancement as achieving the optimum state of dispersion and, secondly, the interfacial adhesion between epoxy and MWCNT. It was declared that the well-dispersed MWCNTs could easily alleviate the stress concentration of the matrix and eliminate

the adverse effect of voids. In this study, the flexural strength and fracture toughness of the material at the break were improved with CNT additives.

In the sixth chapter, the industrial usage areas about "Waste and Recycled Textiles as Reinforcements of Building Materials" were studied by researchers Patricia Peña Pichardo, Prof. Dr. Gonzalo Martínez-Barrera, and their colleagues. In this chapter, the outcomes of the literature surveys were summarized by comparing the results of the recent scientific studies about this subject. In this chapter, the recently used basic topics on natural reinforcement applications were described, and these subjects were grouped under three subtitles such as (a) environmental impact of textile waste as a result of massive consumption of clothing, (b) recycling and reuse of textile waste, and (c) waste and recycled textile materials used as building materials. By learning the techniques for the use of natural textile fibers as reinforcement in cement, improvements on the reinforcement-matrix interface in fiber composite materials, upgrading of natural polymers, obtaining polymer concrete by combining polymers and minerals, and improvement in the environmental properties were achieved. In this chapter, these topics were presented and described in detail.

In the seventh chapter titled "Natural Fibers for Sustainable Bio-Composites" by Dr. Tri-Dung Ngo, the advantages of natural fiber bio-composites used in the industrial applications were demonstrated by literature survey. This study was based mainly on six topics as follows: (i) concept development, (ii) material design, (iii) material fabrication, (iv) product manufacturing, (v) marketing, and (vi) regulations. In this chapter, the potential utilization of natural fiber for the development of green polymer composite materials was emphasized with the objective to elucidate the possibility of using these bio-based materials for various important industrial applications. The other basic subtitles of the work were as follows: thermoplastics, biopolymers, natural plant fiber polymer composites, eco-friendly bio-composites from crop-derived plastics, and plant-derived fibers. The results of the collocated values were summarized in charts and tables.

Finally, we conclude that this book consists of mainly summarized three subject headings within the two specific book subsections : The first group contains the main subjects related to the natural and artificial fibers obtained by literature review; second, experimental and numerical studies are made in order to perform the necessary arrangements in the production stages and to establish a decision mechanism on the specification of the technical properties of the fiber-reinforced composites. The third group of studies focused on the use of sustainable bio-composites and recycled textile wastes as reinforcements in construction.

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Section 1

Micromechanical Investigations on Natural Fiber Composites and Epoxy/MWCNT Nanocomposites

Chapter 1

Introductory Chapter: Natural Fiber Plastic Composites - A Brief Review

Ezgi Günay

Additional information is available at the end of the chapter

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1. Introduction

The natural composites are classified mainly in three biomaterial categories: green composites, hybrid biocomposites, and textile biocomposites. In recent times, biological materials have become essential materials for the construction and automotive industry. Natural fibers and particles have been already used in various types of materials such as plastics, concrete, and textile products as strengthening part of the fiber/matrix combination. These composite materials have very good mechanical thermal and acoustical properties; therefore, they have been used in various engineering applications. The wood-plastic composites (WPCs) have been used in many application areas as automotives, constructions, marine, electronic and aerospace areas instead of fiber glass composites and steel materials. As a wood derivative, hemp fibers have been used in generating thermoplastic matrix composites. These composites can find its application area in the following five sectors: the first area is to modify some parts of the internal and external automobile structures and electric cars. The second area is to obtain strong cementation in building construction. Another area is the production of durable clothes for army suppliers. The fourth one is to produce small electric hand tools and the last one is to build supercapacitors in carbon nanosheets which are as strong as graphene. This review covers a general overview of the preparation phase (chemical procedures), test techniques (experiments) and results, and conclusions summaries (the gaining) of current studies on hemp fiber plastic composites. The data obtained by literature search of the 63 publications have been shown in **Table A1** [1–63].

2. Scientific researches about natural hemp composites

The quality of the produced compounds depends on the elastic constants of the natural fibers used in the composition and the shape and size of the fibers as well as the properties of the matrix material. Factors influencing the strength of the composite material can be listed in more detail as follows: Morphic structure, chemical composition, density, thickness of wood plastic

composites (WPCs) as well as the type and amount of bonding agent and fiber percentage used in composite material. According to the literature, the usage rate of the herb composites (WPCs) in the industrial areas is stated as follows: in the field of aviation 1%, in the area of consumer products 8%, in various fields 8%, in the field of electronics 10%, in the maritime field 12%, in the construction industry 26%, in the automotive sector 31%, and miscellaneous 4% [46].

2.1. General properties of natural fibers in plastic composites

Interface conditions have been influenced at the nanoscale level depending on the thermal sensitivity and the water content of the green materials in the process of preparing composite material from natural fiber embedded in a polymeric matrix. The natural fiber and the polymeric matrix interface features and the cell wall structure of the natural fibers influence: (i) the mechanical properties, (ii) the durability, and (iii) the recyclability of the

Test type	Experimental methods	Test type	Experimental methods
1	Accelerated weathering testing	20	Microscopy (optical light microscope, SEM, confocal laser scanning microscopy (CLSM))
2	Acoustic emission monitoring	21	Moisture absorption method
3	Biodegradability test	22	Nanoindentation test
4	Chemical techniques	23	Nondestructive longitudinal and flexural free/forced vibration test
5	Compression test	24	Sample thickness measurements
6	Compression molding	25	Shear testing
7	Density measurements	26	Single-fiber pullout test
8	Diffusion measurement	27	Static/dynamic/vibration-damping testing
9	Differential scanning calorimetry (DSC)	28	Surface energy and dynamic contact angle measurement
10	Digital images recording	29	Taguchi's technique
11	Fatigue testing	30	Temperature field measurement
12	Flexural test	31	Tensile testing
13	Fourier transform infrared spectroscopy (FTIR)	32	Thermal techniques (annealing)
14	Fracture toughness	33	Torsion test
15	Growth test	34	Vicat test
16	Impact test	35	Water absorption and volume change test
17	Liquid chromatography (HPLC)	36	Weibull statistics
18	Mass spectrometry	37	X-ray microtomography
19	Microbond test		

Table 1. The list of performed experimental studies on natural fiber plastic composites.

industrially produced green composites. The literature survey results both on mechanical and chemical properties of the hemp fiber plastic composites and their usage in industrial areas have been listed in **Table A1**. **Table A1** gives brief information on: (i) the aim of this research, (ii) the experimental methods used in compound production, and (iii) conclusions according to the obtained results. In **Table A1**; 63 research articles have been listed according to the fiber/matrix material characteristics. Of these 63 studies, 59 were related to fiber composites, while only 4 of them were related to particle composites. In the literature survey, the investigations were carried out in two groups: (a) original research articles and (b) review articles. The 52 articles of the 63 articles were original research articles. Researchers performed a series of experimental studies to obtain the information about the following main subjects related with the natural fibers and plastic matrices in biocomposites: (a) mechanical elastic constants, (b) strength, (c) failure stages, (d) the effect of moisture content, (e) biodegradability, (f) fiber matrix interface stresses, (g) cell wall properties, (h) hardness, and (i) the effect of chemical processing. The statistical information according to the performed experiments has been presented in **Table 1** and **Figure 1**. The basic tests have been performed by tensile loading (23%) to obtain the Young's modulus and tensile strength of the composite material in addition to the microscopic visualizations (optical light microscope, SEM) (17%) to observe deformation patterns of the loaded specimens in micro and nanoscales.

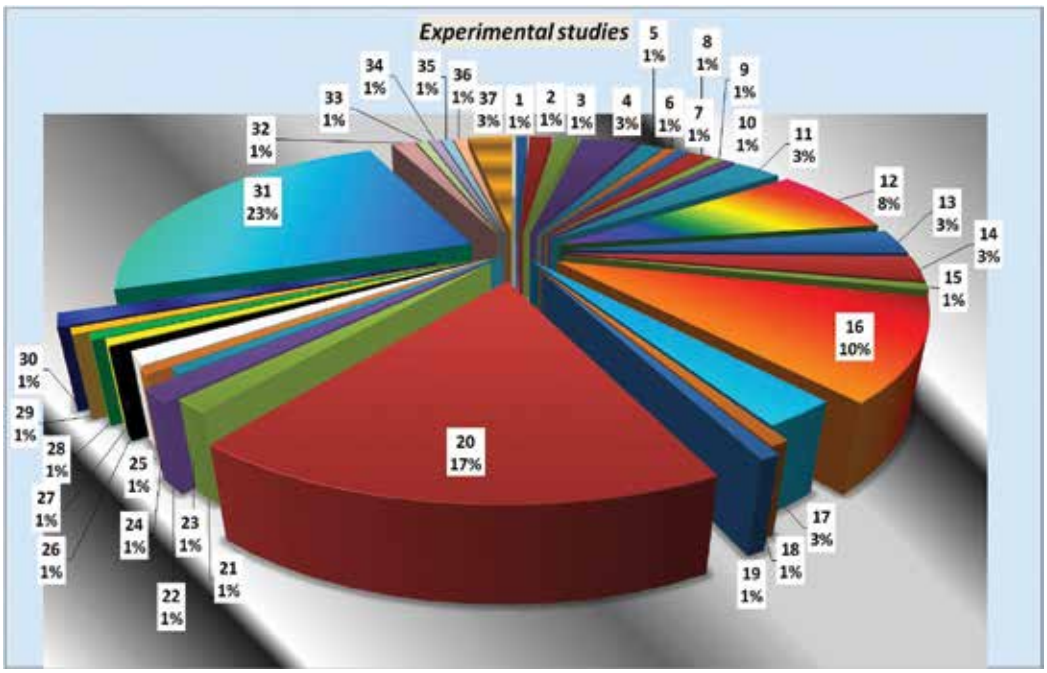

Figure 1. Curve represents the hemp fiber plastic composites and percentage distributions of the performed experimental studies in literature survey.

3. Conclusions

The results obtained by the literature search were summarized below and very important keypoints about fiber and matrix compositions, the physical features of the hemp fibers and hemp fiber plastic composites were emphasized. The main results were as follows.

(1) It was found that tensile strength, Young's modulus, and impact strength of the hemp short fiber reinforced composites were increased in proportion to the increase in fiber content. (2) Flexural strength of the hemp fiber reinforced polylactide and unsaturated polyester composites were found to be decreased with increased fiber content. Additionally, flexural modulus was found to be increased in proportion to the increased fiber content. (3) The impact energy required to damage hemp composites was higher than in conventional laminates. (4) The deformation characterization of hemp/epoxy composites has been developed in three stages. (5) It was shown that natural fibers when compared to flexible fibers showed scattered and lower mechanical properties. (6) Minor variations in terms of the mechanical properties of the woody hemp core (WHC) cell walls were investigated at the nanoscale level. (7) The fiber/polymer interface was modified by using two functionalized chemical procedures simultaneously, and in this way, better adhesion capacity was obtained at the interface (between hemp fibers and thermoplastic matrix).

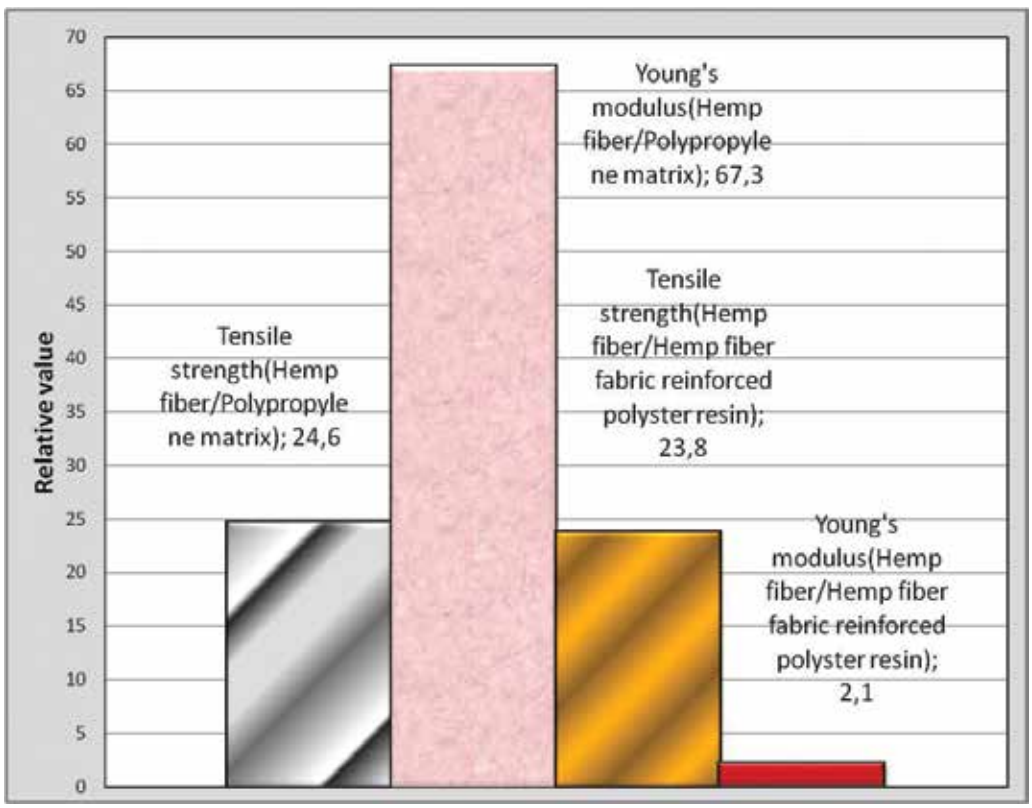

Figure 2. The comparison for the hemp fiber, polypropylene and polyester resin materials according to the tensile strength and Young's modulus.

(8) Variations obtained using nanodrawing tests showed slight variation in the cell wall properties, while the polymer composition was more variable. (9) Hemp fiber composites showed a greater resistance to crack formation and growth than glass fiber composites, although they had lower fatigue strength. (10) Testing natural fiber composites under low impact loading provides important information on the failure mechanisms of hemp (*Cannabis sativa* L.) fiber epoxy composites.

Characteristics of the deformed material such as matrix cracking, delamination, fiber breakage, and fiber pullout phenomenon were examined microscopically [15, 16, 25, 44, 58]. In literature, there were rare experimental studies on the characteristic determination of hemp cell wall structures. In a study on this subject, minor variations at the nanoscale level related to the mechanical properties of the cell wall have been identified [19]. The comparison between the hemp fiber-polypropylene matrix and hemp fiber-fabric reinforced polyester resin in terms of material properties were summarized in Refs. [30, 46, 47, 60, 62]. The graphical results on mentioned values were given in **Figure 2**.

A. Appendix

Author, Reference number	Research subject	Testing methods	Results
Moyeenuddin et al. [1]	Mechanical properties of hemp fiber reinforced composites	Tensile testing, impact testing, and fracture toughness testing	Tensile strength, Young's modulus, and impact strength
Summerscales et al. [2]	A review to obtain high quality fiber	Growth, harvesting, and fiber separation techniques	A review
Summerscales et al. [3]	Properties of natural fiber reinforced composites	Microscopy, mechanical, chemical, and thermal techniques	A review
Pickering et al. [4]	Plane-strain fracture toughness (KIc)	Heat treatment "annealing"	KQ of random short hemp fiber reinforced (PLA).
Sawpan et al. [5]	Flexural strength and flexural modulus	Flexural test	Flexural strength of the composites increased with fiber content
Summerscales et al. [6]	The rules-of-mixture (effects of porosity)	Weibull statistics	A review
Newman et al. [7]	Wood fiber reinforced in polypropylene	Tensile testing	Micromechanical models predicted the tensile modulus
Rachini et al. [8]	Characteristics for hemp fibers and thermoplastic matrix	Chemical process, tensile and impact testing, and SEM	Organosilane coupling agents affecting the tensile strength
Michel et al. [9]	Failure modes of (PHB) and PHB-hemp fiber reinforced composites	Tensile testing and accelerated weathering testing	Changes in the mechanical properties
Vasconcellos et al. [10]	Typical tests of a woven hemp fiber reinforced epoxy composite	Tensile and fatigue testing, optical microscopic, X-ray micro-tomography,	Three stages of damage mechanisms

Author, Reference number	Research subject	Testing methods	Results
		temperature field measurement, and acoustic emission monitoring (AE)	
John et al. [11]	The classification of composites	–	A review
Ude et al. [12]	A summary about *Bombyx mori* woven silk fiber and its composite	–	A review
Misnon et al. [13]	An overview of describing natural textile materials	–	A review
Shah [14]	Ashby-type materials selection charts	Tensile testing	A review
Caprino et al. [15]	Natural composites in applications	Impact loading, optical microscope, penetration test, and indentation test	An higher impact energy is necessary to obtain damage inside hemp composites
Kim et al. [16]	The effect of pH on the tensile properties	Tensile testing, microbond test, SEM.	The fracture toughness of the composites
Shah et al. [17]	The mechanical properties of silk	Charpy impact testing and short beam shear testing	Silk fiber composites (SFRPs) offer advantages
Marrot et al. [18]	The mechanical properties of hemp fibers (Fedora 17)	Tensile testing, nanoindentation test, and X-ray diffraction test	Plant fibers showed low mechanical properties
Beaugrand et al. [19]	The micromechanical properties (WHC) cell walls	Nanoindentation test, density measurements, flexural tests, and biochemical analysis	The mechanical properties of the cell walls
Salentijn et al. [20]	Fiber hemp (*Cannabis sativa* L.) breeding programs	–	A review
Liu et al. [21]	The mechanical properties of hemp fibers	Tensile testing, chemical analysis, microscopic tests, and digital images recording	Less variable and high strength fibers
Almusawi et al. [22]	The particle sizes, the moisture content, and the heating temperatures	Three point flexural testing and tensile testing	The importance of particle size
Christian et al. [23]	Diffusion properties and mechanical properties	Tensile testing and diffusion measurement	Larger change in properties of the hemp/cellulose
Yan et al. [24]	The effect of fibers (MAPP)	Tensile testing, flexure testing, and impact testing	(MAPP) improved the fiber-PP adhesion
Lu et al. [25]	The improvement of the fiber-matrix interface by 5 wt% NaOH treatment	SEM and Fourier transform infrared spectroscopy	Mechanical and thermo-mechanical properties
Shah et al. [26]	Investigation of the mechanical testing cases	Tensile testing and fatigue testing	Flax is a potential structural replacement to E-glass
Kabir et al. [27]	The tensile properties of single hemp fibers	Optical microscopy (OM) measuring and tensile testing	The tensile strength of chemically-treated fibers

Author, Reference number	Research subject	Testing methods	Results
Vasconcellos et al. [28]	The resistance for low velocity impact of hemp/epoxy	Tensile testing, impact testing, fatigue testing, acoustic, emission (AE) monitoring, and microscopic observations	A decrease of the residual tensile strength
Landro et al. [29]	The characteristics and performance of a thermoset bioepoxy resin	Static/dynamic/vibration-damping testing	Laminated composites reinforced with hemp fibers
Sukmawan et al. [30]	Steam exploded bamboo (SEB) fibers	Tensile testing	The tensile strength of alkali treated bamboo fiber
Vukcevic et al. [31]	The optimal production of hemp fibers (ACh)	SEM, mass spectrometry, and temperature programmed chemical reaction	High efficiency in pesticides removal on hemp fibers
Kord [32]	Mechanical properties of (PP)/hemp fiber	Tensile and impact testing, and X-ray diffraction (XRD) testing	The PP/hemp fiber composites
Kavianiboroujeni et al. [33]	The effects of different design parameters	Three-point bending flexural testing, and SEM	Hemp content parameters
Alhuthali et al. [34]	A new infiltration method	Three point bending testing, impact testing, and fracture testing	The development of new composite materials
Saikia [35]	The mechanical properties of the composite	SEM, the gravimetric moisture absorption method, and three point bending test	The thermodynamic parameters of the absorption process
Dalmay et al. [36]	The effects of adding natural fibers	SEM, Vicat test, and three point bending testing.	Flax, lowered the crack propagation
Wretfors et al. [37]	The improvement of fiber-matrix interactions	Tensile testing, SEM, (HPLC), (CLSM)	Getting a better fiber distribution
Erchiqui et al. [38]	The mechanical and structural properties	–	The variations of the process parameters
Etaati et al. [39]	The distribution and effects of the interfacial shear strength (IFSS)	Tensile testing	The hemp fibers had lower tensile properties
Fotouh et al. [40]	The effect of strain rate	Tensile testing, (SEM)	The mechanical behavior is dominated by the matrix
Han et al. [41]	Surface treatment effects	(FTIR), moisture analysis, (DSC), and Tensile testing	Improvement of the thermal stability of hemp fiber
Sawpan et al. [42]	Mechanical properties of chemically treated random short fiber	Impact testing and fracture testing	Mechanical properties of (PLA) increased
Mantia et al. [43]	Polymer composites filled with natural-organic fillers.	–	A review
Sawpan et al. [44]	The interfacial shear strength (IFSS)	Optical light microscope, Fourier transform infrared spectroscopy (FT-IR), scanning electron microscope (SEM).	(IFSS) of PLA/hemp and UPE/hemp fiber samples
John et al. [45]	Cellulosic fiber reinforced polymeric composites	–	A review
Ashori et al. [46]	Wood-plastic composites (WPC).	–	A review

Author, Reference number	Research subject	Testing methods	Results
Koronis et al. [47]	Green composites	–	A review
Schirp et al. [48]	Wood and hemp fibers	Tensile testing, three-point bending test, Charpy impact testing, water absorption and volume change test, and dynamic mechanical analysis test.	Flexural strength values.
Wretfors et al. [49]	Reinforcing wheat gluten (WG) plastics with hemp fibers	Compression molding, tensile testing, SEM, and sample thickness measurements	Hemp fibers in composite material
Ismail et al. [50]	The influence of drilling parameters and fiber aspect ratios	Taguchi's technique	The delamination factor and surface roughness of drilled holes
Jalili et al. [51]	The acoustic parameters of three different polyester composites	Non-destructive longitudinal and flexural free/forced vibration tests	The results obtained from longitudinal free vibration method
Kakroodi et al. [52]	Hemp fibers and particles, in hybrid composites	Tensile, flexural, three-point bending, torsion and impact tests, and SEM	The mechanical properties of the composites
Ochi, et al. [53]	The hemp fiber reinforced biodegradable plastics	Tensile testing, SEM, and biodegradability test	The tensile strength of the composites
Shahzad et al. [54]	The effects of alkalization surface treatment on hemp fiber properties	Tensile testing, impact testing, fatigue testing, and SEM	The tensile properties, interfacial shear strength
Shahzad et al. [55]	The fatigue properties of nonwoven hemp fiber	Tensile testing and fatigue testing	The hemp fiber composites with less fatigue sensitivity.
Terzopoulou et al. [56]	The study about fully biodegradable ("green") composite materials	Tensile testing, impact testing, Fourier transform, infrared spectroscopy, X-ray diffraction, differential scanning calorimeter, and scanning electron microscopy	Tensile and impact strength
Toupe et al. [57]	Phase compatibilization of four mechanical properties	Tensile testing, flexural testing, and impact testing	Fiber concentration parameter
Kabamba et al. [58]	The effect of hemp fibers.	Shear testing and elongation test.	The rheological properties
Muneer [59]	Hemp fiber reinforced wheat gluten (WG) composites	Biodegradability test using the ASTM D5988-03 standard	A review
Scutaru [60]	The mechanical properties hemp fiber composites	Tensile testing	The measured Young's modulus distribution
Gassan [61]	The elastic properties	Analytical solution, SEM.	The experimental data and calculations

Author, Reference number	Research subject	Testing methods	Results
Shahzad [62]	Physical and mechanical properties of hemp fibers	Tensile testing, thermal characterization, single fiber pull-out test, surface energy and dynamic contact angle measurement, and SEM	The tensile strength, Young's modulus, and surface energy
Niyigena [63]	The impact properties of hemp concrete	Compression test, impact test	Mechanical behaviors of hemp concrete material

Table A1. Literature survey: the main characteristic properties of the hemp fiber reinforced epoxy composites.

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Chapter 2

Interfacial Modification of Hemp Fiber–Reinforced Composites

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Additional information is available at the end of the chapter

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Abstract

Natural fiber–reinforced biocomposites are increasingly used in various industries such as automotive, construction, biomedical, and recreation, thanks to their distinctive advantages over traditional glass fiber–reinforced plastics. Natural fiber composites are sustainable, environmentally friendly, low cost, low density, and easy to process as well as have high mechanical properties. The quality of fiber-matrix interface is of critical importance since it determines the load distribution capability of the material. The interface between natural fibers and polymer resins has always been problematic because of the low compatibility between cellulose-based hydrophilic natural fibers and hydrophobic polymer resins, which leads to poor fiber-matrix adhesion and therefore inefficient load distribution between fibers and matrix. To date, several interfacial modification methods have been implemented to address this issue and improve the properties of the resulting composites. This chapter focuses on the interfacial modification of hemp fiber–based composites. First, hemp fiber structure and the nature of fiber-matrix interface were explained. Mechanisms of fiber/matrix adhesion as well as qualitative and quantitative methods for the determination of interface strength were outlined. Finally, the interface modification methods for hemp fiber–reinforced biocomposites were presented in the light of scientific literature.

Keywords: hemp fiber, natural fiber–reinforced biocomposites, fiber-matrix interface, interfacial modification, engineering applications

1. Introduction

Over the last few decades, the search for more eco-friendly and sustainable materials led to the use of natural fibers for composite reinforcement. Natural fibers are renewable, low cost, and readily available. They have low density when compared to commodity fibers such as glass,

carbon, and aramid. Composites reinforced with natural fibers have high specific mechanical properties comparable to those of glass fiber–reinforced plastics. Therefore, natural fiber–reinforced biocomposites provide a gateway toward more eco-friendly and sustainable economies [1, 2]. Some common applications of biocomposites include automotive, packaging, civil engineering, sports, and recreation [3, 4].

The overall performance of biocomposites depends on several factors such as fiber and resin type, fiber/matrix volume fraction, fiber architecture, and fiber-matrix interface quality. The interface between natural fibers and polymer resin has a large impact on the mechanical and degradation properties of biocomposites. In a composite structure, fibers are the main load-bearing component, whereas the matrix phase keeps the fibers together and protects them from external damage. A good bonding between fibers and matrix is necessary for the successful transfer of applied loads from weak matrix material to stiff and strong fibers. The fiber/matrix interface in these composites has always been problematic because of the incompatibility between the surface energies of hydrophilic natural fibers and hydrophobic polymer resins. Research efforts generally focused on enhancing the fiber-matrix bonding by using various chemical and physical modification techniques such as silane treatment, acetylation, use of coupling agents, and alkali treatment [5].

This chapter deals with the interfacial modification of hemp fiber–based composites. In this scope, first, the structure of hemp fiber was explained. Mechanisms of fiber/matrix bonding in composite materials were outlined. A range of available techniques for the determination of interfacial strength was presented. Finally, the interface modification methods used for hemp fiber–reinforced composites were introduced in the light of scientific work in this field.

2. Hemp fiber structure

Hemp is a member of bast fibers (**Figure 1**) [6] which are extracted from the phloem layer of plant stem. Like all bast fibers, hemp fiber has a multicellular structure (**Figure 2**) [7, 8]. A single hemp fiber is made up of lumen, primer wall, and seconder wall [9, 10]. Seconder wall can be further divided into three sub-layers such as S1, S2, and S3. S2 layer is particularly rich in cellulose microfibrils that are responsible for the fiber strength and stiffness [11, 12]. Physical properties of hemp fiber along with some other common natural fibers are given in **Table 1** [9, 13, 14]. **Table 2** shows the chemical composition of hemp and other natural fibers [15, 16].

The most important component of hemp fiber is cellulose. Cellulose is a linear polymer of glucose ($C_6H_{12}O_6$). Glucose molecules add on successively through β-1,4 linkages to form long cellulose chains (**Figure 3**) [17]. These cellulose chains form hydrogen bonds with one another through their pendant —OH or —CH_2OH groups to form microfibrils. Typical diameter and length of these microfibrils are 2–20 and 100–40,000 nm, respectively [18]. There are different regions in cellulose with respect to the arrangement of these microfibrils, i.e., crystalline regions where the microfibrils are well oriented and run parallel to each other creating ordered structures and amorphous regions, which have a more disordered and openly packed structure. Crystalline regions are so closely packed that they are hard to penetrate for the majority

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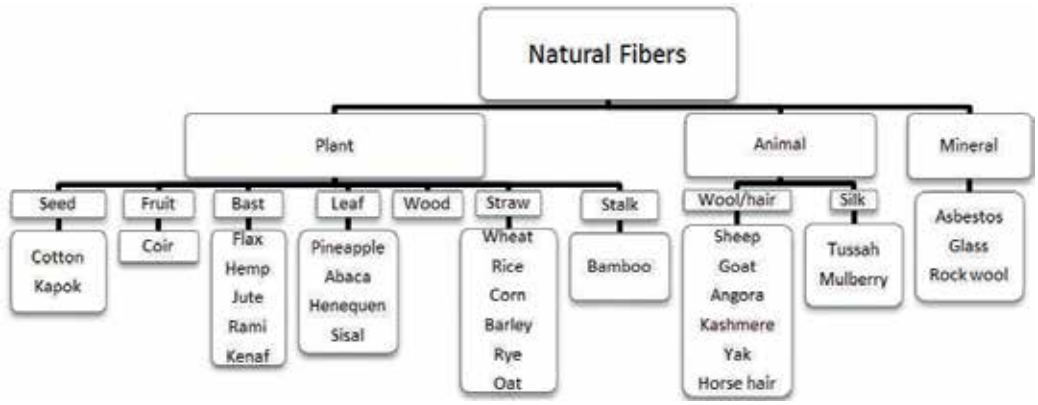

Figure 1. Classification of natural fibers [6].

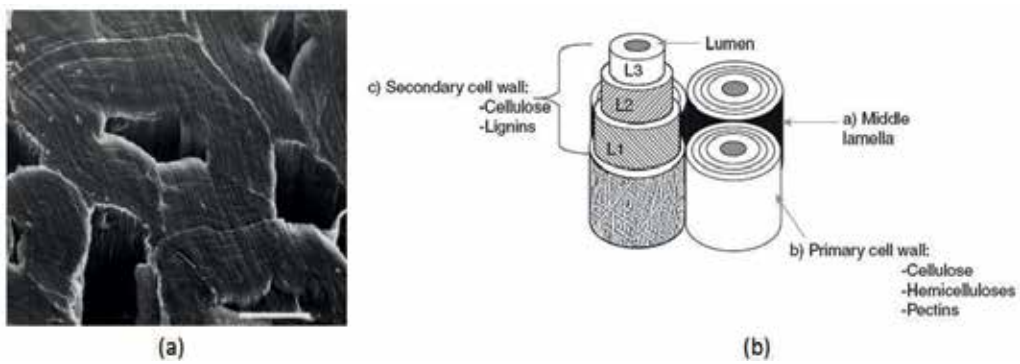

Figure 2. (a) Bundled hemp bast fibers [7]. (b) Schematic representation of hemp fiber structure [8].

Fiber	Length, *l* (mm)	Diameter, *d* (μm)	Aspect ratio (*l*/*d*)	Microfibril angle (°)	Density (kg/m^3)	Moisture uptake (%)
Cotton	20–64	11.5–17	2752	20–30	1550	8.5
Flax	27–36	17.8–21.6	1258	5	1400–1500	12
Hemp	8.3–14	17–23	549	6.2	1400–1500	12
Jute	1.9–3.2	15.9–20.7	157	8.1	1300–1500	12
Kenaf	2–61	17.7–21.9	119	–	1220–1400	17
Ramie	60–250	28.1–35	4639	–	1550	8.5
Sisal	1.8–3.1	18.3–23.7	115	10–22	1300–1500	11
Coir	0.9–1.2	16.2–19.5	64	39–49	1150–1250	13

Table 1. Physical properties of natural fibers (single fiber) [9, 13, 14].

Fiber	Cellulose (% wt.)	Hemicellulose (% wt.)	Lignin (% wt.)	Pectin (% wt.)	Moisture (% wt.)	Wax/oil (% wt.)
Cotton	85–90	5.7	–	0–1	7.85–8.5	0.6
Jute	61–71.5	13.6–20.4	12–13	0.2	12.5–13.7	0.5
Flax	71	18.6–20.6	2.2	2.3	8–12	1.7
Hemp	70–74	17.9–22.4	3.7–5.7	0.9	6.2–12	0.8
Rami	68.6–76.2	13.1–16.7	0.6–0.7	1.9	7.5–17	0.3
Sisal	66–78	10–14	10–14	10	10–22	2
Pineapple	70–82	–	5–12.7	–	11.8	–
Coir	32–43	0.15–0.25	40–45	3–4	8	–

Table 2. Chemical composition of plant fibers [15, 16].

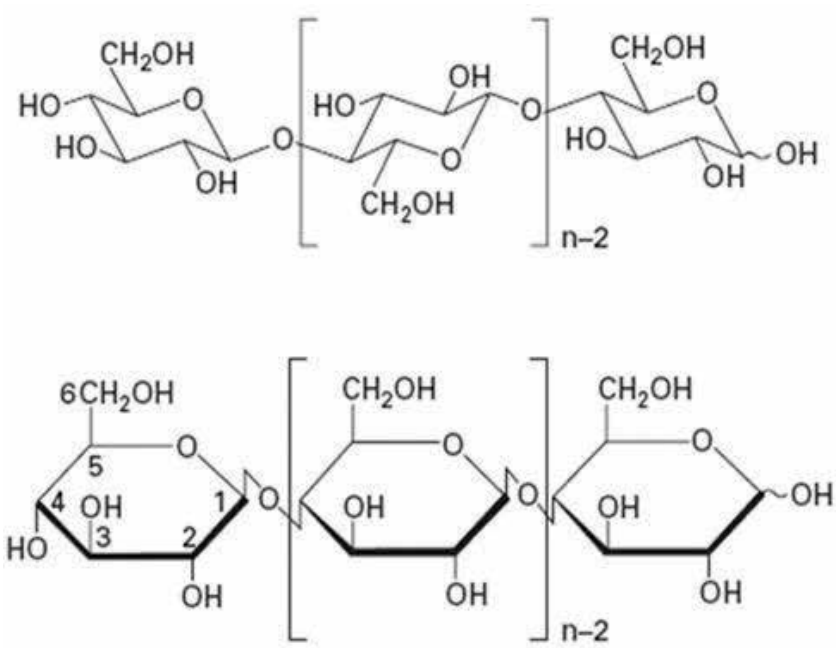

Figure 3. Chemical structure of cellulose [17].

of chemical agents. Amorphous regions, on the other hand, are more accessible and easier to manipulate. Therefore, almost all fiber modifications aim at modifying the structure of amorphous regions as well as the surfaces of crystalline regions. Hemicelluloses refer to a group of polysaccharides which bind individual cellulose microfibrils in cell walls [18, 19]. They generally have amorphous structures and are easier to remove or manipulate by using chemical agents. Lignin is also an amorphous material with a branched structure. It typically consists of oxyphenyl propan units [9, 18, 20, 21]. Pectin is a common name representing a range of complex pectic polysaccharides [22]. Like hemicelluloses, pectin serves as a binding agent in

plant cell wall. There are various other aromatic compounds in plant cell wall such as condensed tannins [23] and low-molecular-weight phenolic acids, e.g., ferulic and *p*-coumaric acids. Other ingredients of lignocellulosic plant fibers include low (often negligible) amounts of fats, waxes, and lipids [24].

3. Fiber-matrix interface in composite materials

3.1. Mechanisms of fiber-matrix adhesion

3.1.1. The concept of wetting and intermolecular interactions

Wetting is the ability of a liquid to make and sustain contact with a solid surface, when the two phases are brought together. Fiber wetting occurs when intermolecular interactions between the fiber and resin molecules are greater than the cohesive forces between the resin molecules. There are various intermolecular interactions that may take part in the adhesion process such as ion-ion, ion-dipole, dipole-dipole, van der Waals interactions, and hydrogen bonding [25]. Fiber wetting is the first necessary step before any further interaction or bonding between fibers and matrix can occur. A successful wetting means that the resin spreads over the greatest possible surface area of the fibers (contact angle, $\theta = 0°$) (**Figure 4**) [26].

Wetting behavior of fibers can be fully described by determining four wetting parameters such as thermodynamic work of adhesion (W_a), interfacial energy (γ_{SL}), spreading coefficient (S), and wetting tension (ΔF_i) [27]. Thermodynamic work of adhesion W_a is the energy required to separate a unit area of interface and can be described with Dupré equation [26]:

$$W_a = \gamma_{SV} + \gamma_{LV} - \gamma_{SL} \tag{1}$$

Where γ stands for surface energy; subscripts S, L, and V indicate solid, liquid, and vapor phase, respectively. According to Dupré equation, wetting is favored if the surface energies of the two constituents are large, and their interfacial surface energy is small.

The interfacial energy is defined as the work necessary to increase the interfacial surface area by unit area [27]. Hence, a small value of interfacial energy indicates a good wetting.

The spreading coefficient is the ability of a liquid to spontaneously spread over a solid. A positive value of the spreading coefficient indicates instantaneous spreading.

$$S = \gamma_{SV} - (\gamma_{SL} + \gamma_{LV}) \tag{2}$$

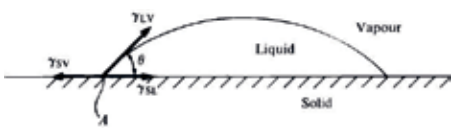

Figure 4. Contact angle, θ and surface energies, γ for a liquid drop on a solid surface [26].

Finally, the wetting tension can be defined as the work needed against wetting a porous network by eliminating a unit area of the solid-liquid interface while exposing a unit area of the solid vacuum interface. A positive value of wetting tension is an indication of good wetting [27].

$$\Delta F_i = \gamma_{SV} - \gamma_{SL} \tag{3}$$

In general, wetting is strongly favored when the surface energy of the fiber greatly exceeds that of the resin. For instance, glass fibers (γ_{SV} = 560 mJ m^{-2}) are easily wetted by polyester resin (γ_{LV} = 35 mJ m^{-2}), whereas polyethylene fibers (γ_{SV} = 31 mJ m^{-2}) are not [26].

3.1.2. Chemical bonds

Chemical bonds include ionic, covalent, and metallic bonds. Covalent bonds are the most common form of chemical bonding in the case of natural fiber composites and are much stronger compared with intermolecular interactions. These bonds generally form between the hydroxyl groups of natural fibers and the functional groups of the polymer resin. Coupling agents with these functional groups can also be used to improve fiber/matrix bonding.

3.1.3. Coulombic interactions

Coulombic interactions can occur when the surfaces of fibers and resin have charges of opposite signs. Coulombic forces are weaker when compared to other forms of interactions and are not stable due to the fact that they can change with humidity, pH, and temperature.

3.1.4. Mechanical interlocking

Another important mechanism for interface adhesion for natural fiber composites is mechanical interlocking. In this case, the resin in liquid form penetrates into the surface cavities of fibers during composite production and is locked inside these cavities upon curing/hardening. It is noteworthy that this mechanism works only for fibers with a rough surface. As the surface roughness of the natural fiber increases, so does the effectiveness of mechanical interlocking. Thus, some fiber modification techniques such as alkali treatment are employed to increase the surface roughness of natural fibers and promote the mechanical locking between fibers and resin.

3.2. Interfacial bond strength measurement

Single-fiber or bulk laminate methods can be employed to determine the interfacial bonding strength of composite materials. The most common single-fiber tests are pull-out, microbond, and full fragmentation tests. These test methods use a single fiber embedded in a matrix. The interfacial shear strength (IFSS) is correlated with the shear debonding stress of the single fiber. One major limitation of single-fiber tests is that the shear behavior of fibers in real composite material may be different from that of the single fiber because of the effect of neighboring fibers. Yet, single-fiber tests yield a reasonable quantification of interfacial bond strength under tensile loading. Bulk laminate experiments, on the other hand, include transverse tensile and bending tests, short beam shear test and Iosipescu shear test. Complementary methods used

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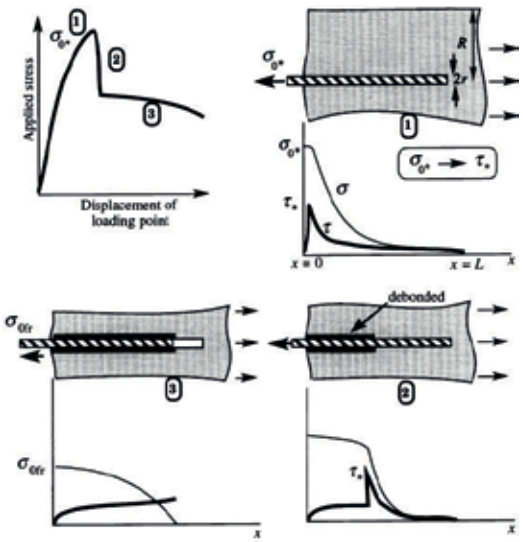

Figure 5. Schematic stress distributions and load-displacement plot during the single-fiber pull-out test [26].

for the assessment of interfacial adhesion include X-ray photoelectron spectroscopy (XPS), ToF-SIMS, SEM, and surface energy analysis. Only single-fiber test methods which are the most common are explained in this section, since the full discussion of all the available techniques goes beyond the scope of this chapter.

3.2.1. Single-fiber pull-out test

Single-fiber pull-out is among the most common testing methods for interface characterization of natural fiber composites [28, 29]. In this method, a single fiber that is half embedded in a matrix is pulled out of the matrix under a tensile load. The apparent interfacial shear strength (IFSS, τ_{app}) is determined using the peak force (F_{max}):

$$\tau_{app} = F_{max}/\pi dL \tag{4}$$

in which d is the fiber diameter and L is the embedded fiber length. The apparent IFSS provides an approximate value for the interface bonding.

More sophisticated treatment of single-fiber pull-out test requires calculating the debonding shear stress by using shear lag theory [30, 31]. **Figure 5** shows axial distributions of the normal stress in the fiber and shear stress at the interface [26]. There are three stages for the complete pulling out of the fiber, i.e., elastic loading up to debonding, propagation of the debonding front, and pull-out by frictional sliding. Shear lag theory assumes that there is no shear strain in the fiber and no transfer of normal stress across the fiber ends [26].

The peak in the load-displacement curve (σ_{0^*}) is indicative of the debonding event. The debonding shear stress τ_* can be determined using σ_{0^*} with the following relation [31]:

$$\tau_* = \frac{n\sigma_{0*}\coth\left(\frac{nL}{r}\right)}{2} \tag{5}$$

Where r is the fiber radius; n is a dimensionless constant; n is given by

$$n = \left[\frac{2E_m}{E_f(1+v_m)\ln(1/f)}\right]^{1/2} \tag{6}$$

in which E_m and E_f are the matrix and fiber modulus, respectively; f is the fiber volume fraction; v_m is the matrix Poisson's ratio.

3.2.2. Microbond test

Microbond test is generally used for fibers with small diameter and is similar to the fiber pull-out test. In this method, a small droplet of resin is applied to the fiber and the specimen is positioned between two knife edge–shaped plates (**Figure 6**) [32]. A tensile force is then applied to the free end of the fiber to pull the resin droplet against the knife edges so that the load is transferred to the fiber-matrix interface. The same equation as in the pull-out method is used to calculate the IFSS. This technique requires very small droplets and is suitable for very soft matrix materials [33].

3.2.3. Full-fragmentation test

In the full-fragmentation test, a single fiber is fully embedded in a matrix, and the matrix is elongated in tension parallel to the fiber axis (**Figure 7**) [34]. Eventually, the fiber breaks into a number of fragments with varying lengths. The shortest fiber length obtained in this way is referred to as the critical fiber length, L_c. Shear strength, τ, is determined by using the aspect ratios of the fiber fragments considering Weibull modulus of the fiber [26]. Fiber fragmentation test gives the best approximation to real composite material compared with other single-fiber tests. Another advantage of this method is that it also takes the heterogeneity of natural fibers into consideration [33].

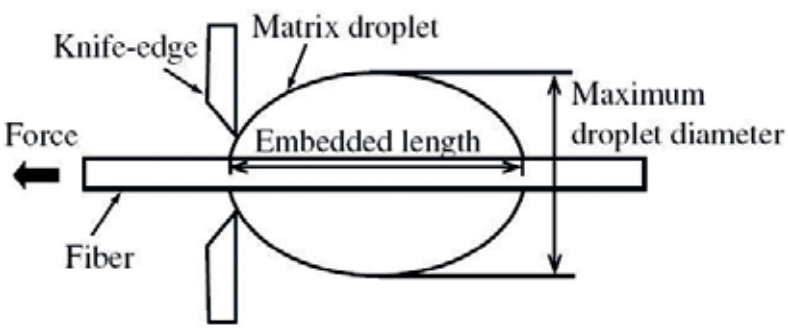

Figure 6. Schematic representation of the microbond test [32].

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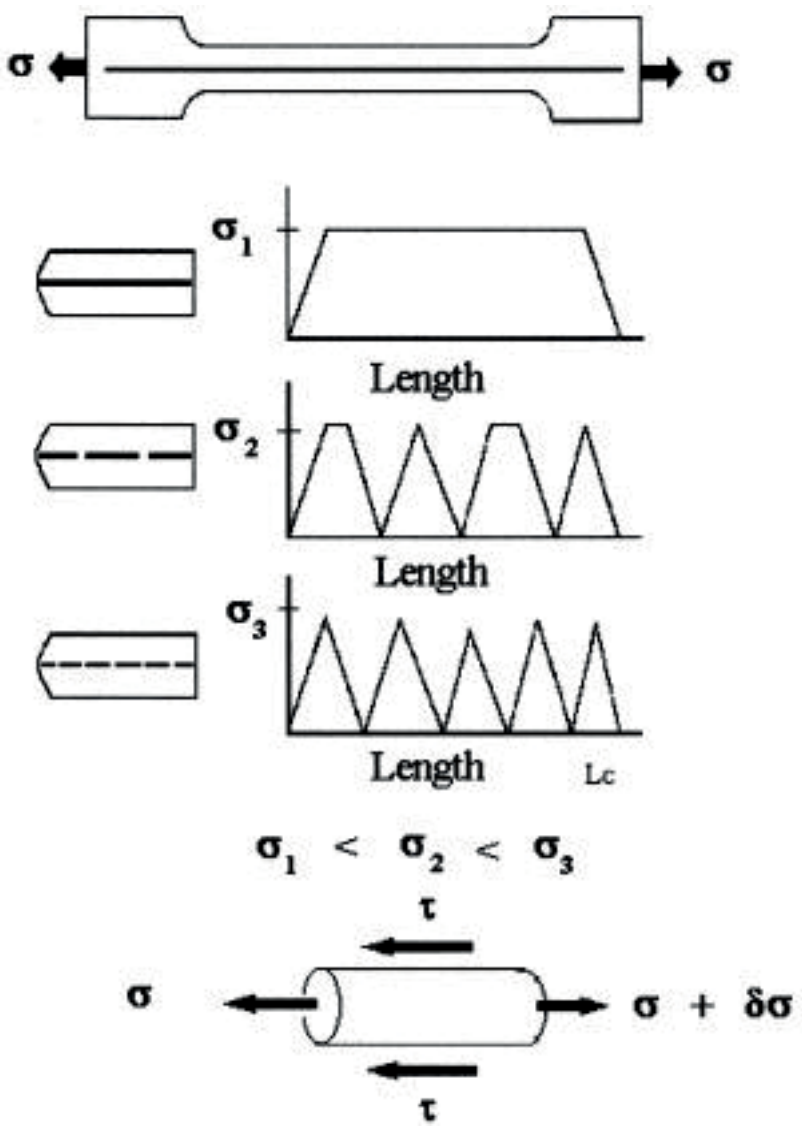

Figure 7. Schematic representation of the full fragmentation test [34].

In a real composite structure, the interface is usually subject to various loads, and its failure may comprise different modes such as debonding, cracking, and sliding, most of which involve plastic deformation of the matrix. Single-fiber experiments generally consider shear debonding and sliding as the failure mechanisms with no regard to normal stress across the interface because of the difficulty in applying such stresses to a cylindrical interface. The basic assumption in all these tests is that the tensile load applied to the fiber end is transferred as a shear load across the fiber/matrix interface. Thus, higher tensile loads indicate better fiber/matrix adhesion at the interface. It is important to bear in mind that the single-fiber tests yield

only approximate values, and the real-life structure can be subjected to different distribution of loads and may show very complex interface failure mechanisms.

4. Interfacial modification methods for hemp fiber composites

4.1. Physical modification methods

4.1.1. Corona treatment

This method is based on "corona discharge", i.e., an electrical discharge appearing around the surface of charged conductors, caused by ionization of the surrounding fluid. This ionization creates radicals on the surface of the fibers and promotes fiber-matrix bonding. Ragoubi et al. [35] investigated the mechanical properties of composites obtained from different combinations of untreated and corona-treated hemp fibers and PP matrix. The corona treatment resulted in a significant increase in tensile strength with 30% enhancement in Young's modulus. Authors reported that the etching effect generated by corona treatment which is evidenced by microscopy increased the mechanical locking between fibers and the matrix and therefore resulted in enhanced mechanical properties.

4.1.2. Plasma treatment

In plasma treatment, plasma of different gases is used to modify the surface of natural fibers. It is similar to corona treatment in that an ionized region including excited species such as ions and radicals is formed around the fiber surface. This technique requires a vacuum chamber and gas feed to maintain the desired pressure and composition of the gas mixture. Jimenez et al. [36] studied the impact of atmospheric air pressure plasma (AAPP) treatment on the mechanical properties and interfacial behavior of hemp fiber–reinforced cellulose-acetate-butyrate (CAB) biocomposites. During testing, the untreated and AAPP-treated fibers exhibited a linear region and then exhibited a sharp drop in strength after reaching ultimate failure stress. After the treatment, the tensile strength, Young's modulus, and elongation at break of hemp fibers reduced dramatically. However, the heat generated during the plasma treatment causes dehydration of the fibers and the etching effect of plasma results in rougher fiber surfaces. It was shown that the interfacial shear strength (IFSS) increases after 1-min AAPP treatment due to enhanced surface roughness and accompanying increment in fiber/matrix mechanical locking. The introduction of functional groups and cleaning of contaminant substances were other factors that enhanced fiber/matrix bonding efficiency.

4.1.3. Alkali treatment

Alkali treatment is among the most popular fiber modification methods for its simplicity, low cost, and effectiveness. This technique involves using a dilute solution of sodium hydroxide (NaOH) or other bases to modify the fiber surface. Alkali treatment creates a rougher fiber surface by removing hemicelluloses polymers and other polymeric materials from the fiber surface thus improving mechanical locking between fibers and resin. Alkali treatment also separates fiber bundles by removing pectin and hemicellulose polymers that bind the individual

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fibers together resulting in an increased effective fiber surface area for resin adherence. Mwaikambo and Ansell [37] treated hemp fibers with different concentrations of NaOH. After the treatment, the surfaces of hemp fibers were cleaner and rougher. X-ray diffraction and thermal analysis indicated that the crystallinity index of the fibers slight increased at low NaOH concentration but reduced at higher concentrations of NaOH. Alkalization increased the surface roughness and resulted in better mechanical locking between fibers and the matrix, thus improving the composite mechanical properties (**Figure 8**) [37]. In another study [38], the same research group treated hemp fibers with NaOH and reported that alkalization caused a rapid degradation of the cellulose between 0.8 and 8% NaOH concentrations. At higher concentrations, the degradation was reported to be marginal. Similarly, there was a marginal decrease in the crystallinity index at NaOH concentrations between 0.8 and 30%. SEM study indicated that alkalization resulted in rougher fiber surface, favoring fiber-matrix interlocking and adhesion.

Aziz and Ansell [39] investigated the effect of alkalization and fiber alignment on the performance of hemp fiber composites. Alkali-treated fiber composites possessed higher flexural strength and modulus compared with untreated fiber composites. SEM micrographs indicated that the surfaces of hemp fibers were cleaner after the treatment. Dynamic mechanical analysis showed that the alkalized fiber composites have higher storage modulus values corresponding to higher flexural moduli. Ouajai and Shanks [40] investigated the properties of hemp fibers after mercerization with NaOH solution. The greater activation energy of treated hemp fiber compared with untreated fiber suggested an increase of purity and improvement in structural order. FTIR study indicated that mercerization removed noncellulosic substances from the fiber. However, NaOH treatment caused structural degradation of fibers especially at higher concentrations and longer treatment times as evidenced by X-ray measurements. Park et al. [41] investigated the interfacial properties of PP composites reinforced with as-received and alkaline and silane treated hemp fibers by using acoustic emission (AE) and dynamic contact angle measurement. Surface energy of the fibers was increased after alkaline treatment due to the removal of the weak boundary layers and resulting increase in surface area. The IFSS also increased after alkali treatment. Islam et al. [42] produced short and long hemp fiber reinforced polylactic acid (PLA) composites by film stacking method. Interfacial shear strength (IFSS) measurements proved that the interfacial bonding was favored by alkali treatment of hemp fibers which also led to improved composite mechanical properties. In another study [43],

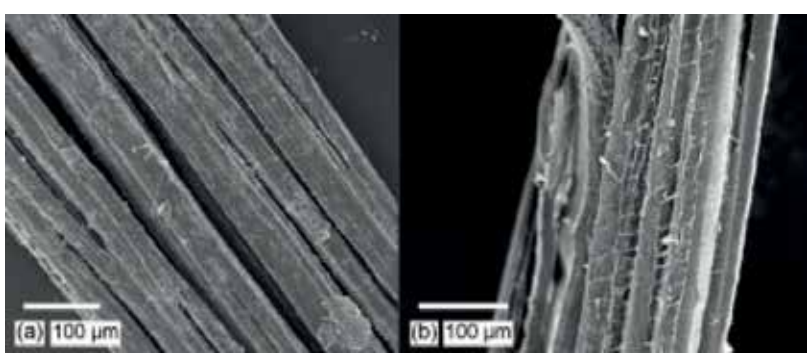

Figure 8. (a) Untreated hemp fiber and (b) alkali treated hemp fiber [37].

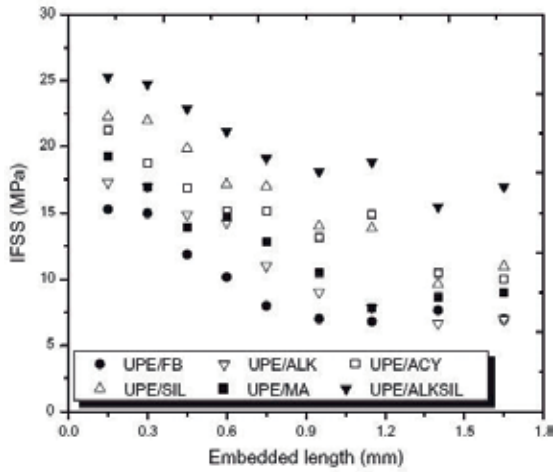

Figure 9. IFSS as a function of embedded length for UPE/hemp fiber (untreated and treated) samples [44].

industrial hemp fibers were treated with a 5-wt% NaOH solution at 120°C for 60 min to remove noncellulosic fiber components. The results showed that alkali treatment removed lignin, separated fiber bundles, exposed cellulose hydroxyl groups, and enhanced thermal stability of the fibers by increasing cellulose crystallinity via better packing of cellulose chains. Alkali treatment also improved the strength and stiffness of resulting hemp/epoxy composites. Sawpan et al. [44] treated hemp fibers with a range of chemicals including NaOH, acetic anhydride, maleic anhydride and silane in an attempt to improve interfacial shear strength (IFSS) of hemp fiber–reinforced polylactide (PLA) and unsaturated polyester (UPE) composites. **Figure 9** shows IFSS as a function of embedded length for UPE/hemp fiber (untreated and treated) samples [44]. IFSS of alkali-treated hemp fiber composites (UPE/ALK) generally increased when compared to untreated samples (UPE/FB), which was attributed to better fiber/matrix bonding, increased matrix transcrystallinity, and increased chemical bonding as evidenced by FTIR results. In a recent work, Dayo et al. [45] prepared bisphenol A-aniline–based benzoxazine (BA-a) composites reinforced with 5% NaOH-treated short hemp fibers. The study showed that the rich —OH groups in treated fibers can promote the ring opening of oxazines at lower curing temperature and improve the adhesion between fiber and matrix. Considerable improvements were recorded in mechanical properties of the composites by using treated fibers.

4.2. Chemical modification methods

4.2.1. Esterification-based treatments

An ester refers to the product of the reaction between a carboxylic acid and an alcohol. In esterification technique, carboxyl groups, —COOH, in carboxylic acids react with hydroxyl groups, —OH, in cellulose macromolecules. After the reaction, —OH groups are eliminated resulting in more hydrophobic fibers making them more compatible with polymer matrix. Esterification techniques include acetylation, benzylation, propionylation and treatment with stearates [33]. Acetylation is the most popular and effective esterification technique for the

treatment of natural fibers. Tserki et al. [46] investigated the effect of acetylation and propionylation on various natural fibers including hemp. It was shown that ester bonds formed on the fiber surface and noncrystalline substances were removed, hence changing the surface topography. The moisture absorption of the fibers reduced after the treatment. **Figure 10** shows ester content versus reaction time plots of various treated fibers [46].

Mwaikambo and Ansell [37] applied acetylation treatment to hemp and other natural fibers. Acetylation was carried out using acetic anhydride with and without an acid catalyst to graft acetyl groups onto the cellulose structure (**Figure 11**) [37]. The surface of treated fibers was

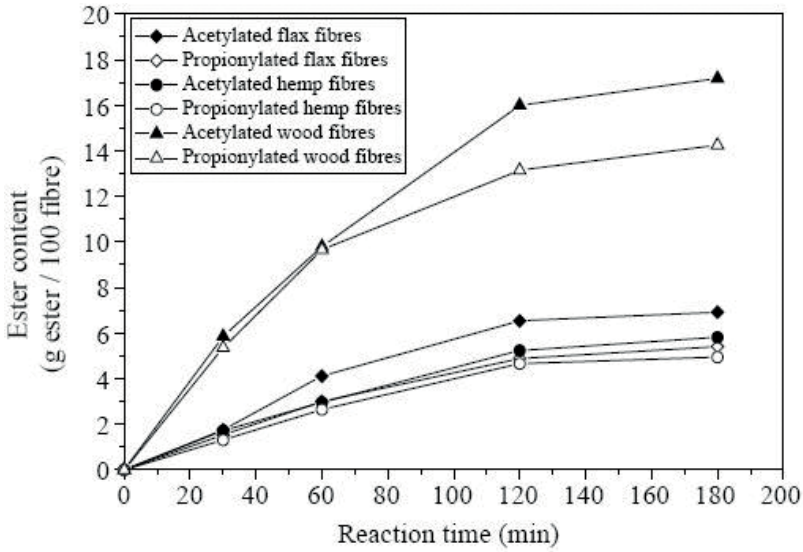

Figure 10. Ester content of chemically treated fibers [46].

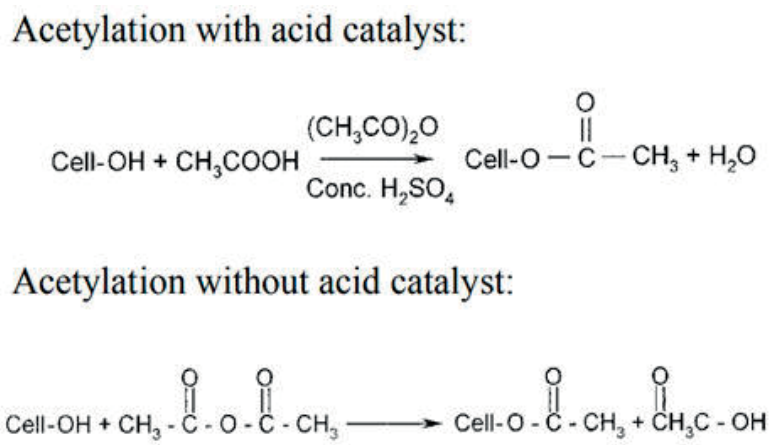

Figure 11. Acetylation process [37].

rougher compared with untreated fibers. Acetylation significantly enhanced the performance of natural fiber composites by promoting better fiber to resin bonding. Gulati and Sain [47] examined the effect of acetylation on acid–base characteristics of hemp fibers by using inverse gas chromatography. The results suggested that acetylation caused hemp fibers to be more basic due to esterification of hydroxyl groups. Composites manufactured with acetylated fibers showed improved flexural properties.

4.2.2. Silane coupling agents

Traditionally, organosilanes are used in glass fiber–reinforced composites in order to enhance their strength and durability, and they are still the largest group of coupling agents used in composite industry today. A silane that contains at least one carbon-silicon bond (Si—C) structure is referred to as an organosilane. The organosilane molecule can be represented by the formula $R—(CH_2)_n—Si(OR')_3$, where n = 0–3; R is a nonhydrolyzable functional organic group that is reactive toward various groups such as amino, epoxy, vinyl, methacrylate, sulfur; OR′ is a hydrolyzable group like an alkoxy group that can react with hydroxyl groups present in inorganic or organic substrates such as natural fibers. Organosilanes can significantly improve fiber/matrix bonding by serving as bridges between them. **Figure 12** shows the general mechanism of alkoxysilane reaction with the fiber surface [5].

Panaitescu [48] investigated the combined effect of 3-aminopropyl triethoxy silane and MAPP treatments on the morphology, thermal, and mechanical properties of hemp fiber–reinforced high-flow PP modified with poly[styrene-b-(ethylene-co-butylene)-b-styrene] (SEBS). Thermal stability of hemp fibers enhanced after silane treatment. Better dispersion of fibers and enhanced static and dynamic mechanical properties of their composites were observed with treated fibers compared with untreated fibers and their composites. In another study [49], the impact of silane treatment on the properties of nonwoven hemp fabric–reinforced unsaturated polyester resin (UPE) composites was investigated. The silane treatment resulted in a better interface adhesion and mechanical and thermal properties of the composites. Rachini et al. [50] reported that silane treatment of hemp fibers increased their hydrophobic character through a condensation reaction between hydrolyzed silane and hydroxyl groups of hemp fibers. Recently, Panaitescu et al. [51] treated hemp fibers with various silane coupling agents to improve the mechanical properties of hemp fiber/PP composites for automotive parts. The silane treatment resulted in bundle splitting and separation of elementary fibers. All the silane treatments were shown to increase the mechanical and thermal properties of the resulting hemp fiber/PP composites.

4.2.3. Graft copolymerization

In graft copolymerization, the cellulosic fiber is treated with an aqueous solution of selected ions and is then exposed to high energy radiation. As a result, the cleavage of cellulose macromolecules takes place, and radical groups are formed. Then, the cellulose material is grafted using a suitable polymer that is compatible with polymer matrix such as acrylonitrile, vinyl monomers, polystyrene, and methyl methacrylate. The most popular and efficient grafting method is the treatment of natural fibers with maleic anhydride grafted polypropylene (MAPP) copolymers.

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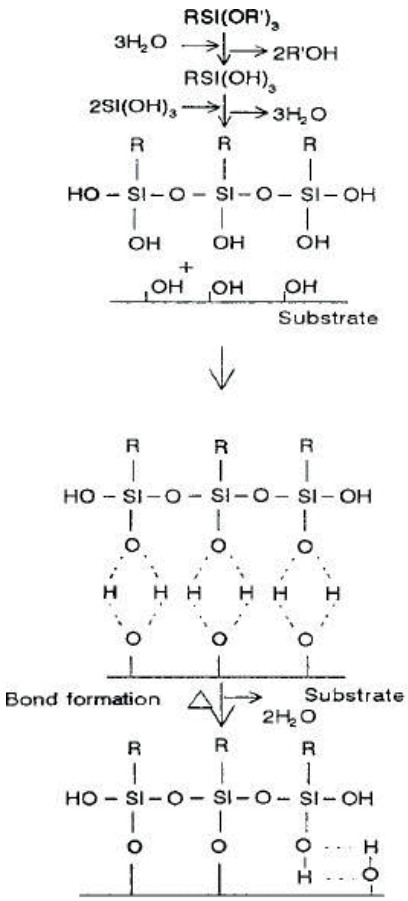

Figure 12. The general mechanism of alkoxysilane reaction with fiber surface [5].

This process results in the formation of covalent bonds across fiber-matrix interface. The reaction takes place in two steps as shown in **Figure 13** [5].

Vignon et al. [52] studied the properties of hemp bast fibers purified by steam treatment compounded with polypropylene (PP), either directly or after surface treatment with polypropylene-maleic anhydride co-polymer. The treatment increased the mechanical properties, tensile modulus, and tensile strength at yield of the resulting PP composites due to a better adhesion between the matrix and the fibers. Mishra et al. [53] investigated the effect of maleic anhydride treatment on the mechanical properties of hemp/novolac resin composites. The treatment resulted in more hydrophobic character in hemp fibers. The impact strength, Young's modulus, flexural modulus, and Shore-D hardness were reported to be higher in maleic anhydride–treated fiber composites compared with as-received hemp fiber composites. Wielage et al. [54] investigated the influence of MAH-PP on storage modulus and loss factor of hemp fiber polypropylene composites. SEM analysis confirmed that the coupling agent causes a significantly better wetting of the natural fibers when compared to untreated composites.

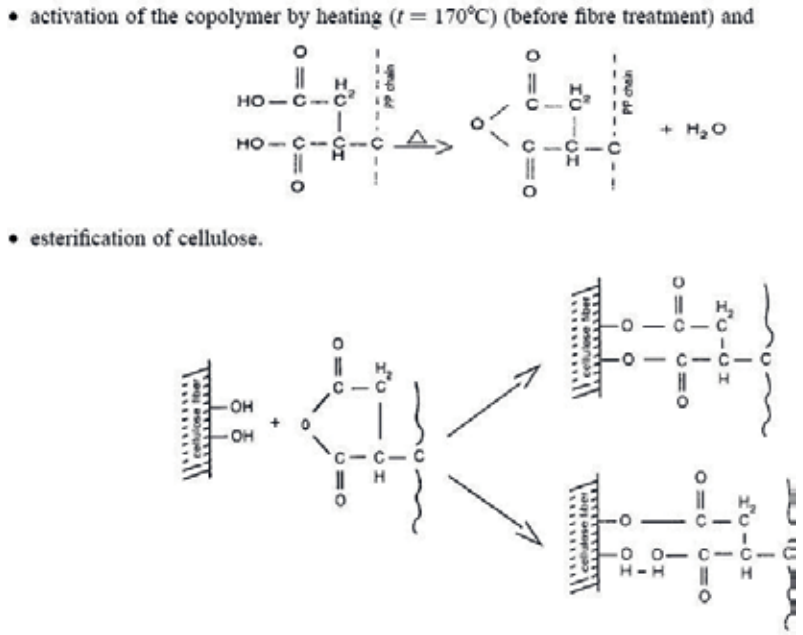

Figure 13. The reaction between natural fibers and maleic anhydride grafted polypropylene (MAPP) copolymers [5].

Some researchers combined MAH-PP treatment with mercerization to obtain a synergistic effect. Bledzki et al. [55] modified hemp fibers by mercerization and MAH-PP coupling agent and used for the production of epoxy and PP composites. The tensile modulus increased after the chemical treatments. Mishra and Naik [56] treated hemp fibers with maleic anhydride and fabricated their polystyrene composites. They reported that maleic anhydride treatment resulted in significant enhancement in Young's modulus, flexural modulus, impact strength, and Shore-D hardness of the composites when compared to the untreated fiber composites. Wang et al. [57] investigated the effect of maleic anhydride-grafted polyethylene (MAPE) on the compressive dynamic behavior and flammability of short hemp fibers/high-density polyethylene (HDPE) composites. The compressive modulus and yield stress increased with MAPE treatment. Etaati et al. [58] investigated the static and dynamic mechanical and viscoelastic properties of short hemp fiber polypropylene composites. The maleic anhydride–grafted polypropylene (MAPP) and maleic anhydride–grafted poly(ethylene octane) (MAPOE) were used as coupling agents for modifying the matrices. The damping ratio analysis suggested that the bonding between fibers and resin was improved by MAPP. This was also evidenced by tensile strength experiments and scanning electron microscope (SEM) observations. Recently, Sullins et al. [59] studied the impact of NaOH and maleic anhydride–grafted polypropylene (MAPP) on the properties of hemp fiber/PP composites. It was reported that the treatments resulted in composites with better mechanical properties. The composites with 5 wt% MAPP displayed the highest mechanical properties.

4.2.4. Treatment with isocyanates

Polymethylene-polyphenyl isocyanates (PMPPIC) can make strong covalent bonds with —OH groups of cellulose through their –N=C=O functional groups. The isocyanate treatment is very effective and can be used to modify both fibers and the polymer matrix. The reaction of isocyanates depends upon the catalysts and temperature. The main disadvantage of this method is the toxicity of the chemicals used. Hemp fiber were modified with 3-isopropenyl-dimethylbenzyl isocyanate (TMI), using dibutyltin dilaurate (DBT) as a catalyst and then used to reinforce unsaturated polyester (UPE) composites [60]. The treatment significantly increased the tensile strength, flexural strength, and water resistance of the resulting composites. SEM study of the fractured surfaces confirmed that the pretreatment significantly improved the interfacial adhesion between hemp fibers and UPE resins. FT-IR spectra and X-ray photoelectron spectroscopy (XPS) analysis indicated that the treated fibers made covalent bonds with the resin. In a recent study, Liu et al. [61] prepared an environmentally friendly composite from hemp fibers and acrylated epoxidized soybean oil (AESO) and N-vinyl-2-pyrrolidone copolymer. The properties of the composites were enhanced through the incorporation of isophorone diisocyanate (IPDI). The FTIR and ^{13}C NMR spectra reveal that IPDI could react with the —OH groups of both hemp fibers and AESO by forming urethane connections thus acting as a bridge between the fibers and matrix. As a result, the tensile and flexural properties, storage modulus, and glass transition temperature of the composites were significantly increased.

5. Conclusions and future trends

Natural fiber–reinforced composites have a growing popularity in various industries such as automotive, construction, recreation, sports, and biomedical due to their high mechanical properties and environmentally friendly nature. However, these composites suffer from problems related to weak interfacial bonding between natural fibers and polymer resins as well as the hydrophilic nature of plant fibers. Over the last few decades, studies mostly concentrated on improving fiber/matrix bonding in natural fiber composites in an attempt to enhance their mechanical properties and at the same time reduce their vulnerability to moisture. Several physical and chemical modification methods have been implemented such as corona, plasma, and alkali treatment, esterification-based treatments, silane coupling agents, graft copolymerization, and isocyanate treatment. Significant improvements have been recorded in the mechanical, thermal, and moisture absorption properties of biocomposites by applying these modification methods. Alkali treatment and graft copolymerization stand out among other treatments for their low cost and efficiency. Interface modification efforts are likely to continue in the future with the implementation of new physical and chemical methods. It is expected that more sophisticated techniques will come into play with an effective use of nanotechnology in the field. It is expected that, with the use of cellulose nanofibrils together with advanced nano-modification methods, stronger, more durable, and cost-effective natural fiber composites will dominate the composite industry in the near future.

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Chapter 3

Development of Hemp Fibers: The Key Components of Hemp Plastic Composites

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Additional information is available at the end of the chapter

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Abstract

Plant fibers in general and hemp fibers in particular have great prospects for their use in various innovative applications such as ecological, biodegradable, and renewable resources with unique properties. Such properties together with the increased strength due to high-cellulose content and specific morphological parameters are widely used to produce plant fiber–based plastic composites. The properties of plant fibers that may influence the properties of composites depend on crop processing, but the basis for them is provided during fiber development *in planta*. It is known that two types of bast fibers are developed in the hemp stem: primary fibers formed from procambium cells and secondary fibers that originate as a result of cambium activity. Both types of fibers may significantly vary in their yield and quality depending on the variety and growth conditions. Differences in the anatomical and morphological characteristics of the two types of hemp fibers, together with peculiarities in the composition and architecture of cell wall, influence the technical parameters of the raw material quality. Based on our study of both primary and secondary fiber development in hemp stem that was focused on the two key stages, intrusive elongation and deposition of thick cell wall layers, we suggest the set of parameters that can influence the quality of the mature fibers and trace their biological origin.

Keywords: plant fibers, gelatinous fibers, hemp, intrusive growth, plant cell wall, rhamnogalacturonan I

1. Introduction

Hemp plastic is a bio-based composite that may vary in composition and applications. The components of plant material that provide valuable properties and usually constitute 25–65% of the composite [1] are bast fibers. These cells are developed within hemp stem and have specific morphological and mechanical parameters. Hemp fibers, same as fibers of flax, ramie, nettle, and some other fiber crops, are distinguished by high-cellulose content [1–3]. The quality of such fibers varies depending on both the processing and the properties developed *in planta* [4–6]. We will consider the biological determinants of hemp fiber quality, comparing primary and secondary hemp fibers. Being developed within the same plant, primary and secondary hemp fibers differ significantly in morphology, cell wall organization, and as a consequence in quality [7–9], giving the possibility to understand how developmental processes influence quality parameters.

2. Origin of primary and secondary fibers in the hemp stem

The usually considered primary and secondary hemp fibers are bast fibers, meaning that they belong to phloem and are located closer to stem periphery than cambium (**Figure 1**). Primary phloem fibers located behind the epidermis and collenchyma are larger than the secondary

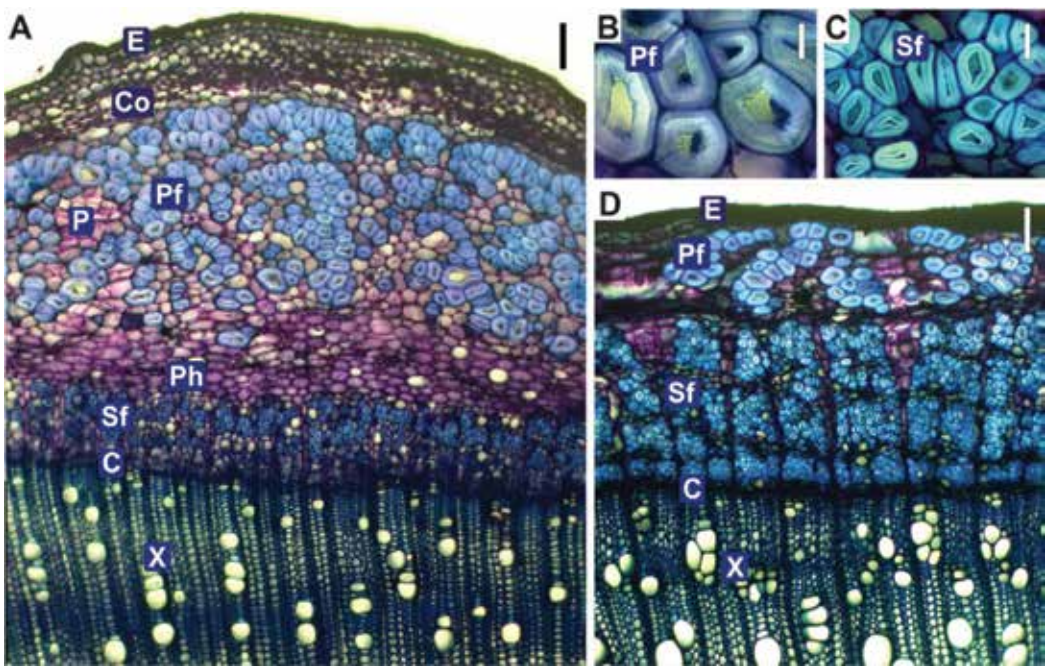

Figure 1. Primary and secondary phloem fibers within hemp stem. Cross-section of hemp stem, stained with toluidine blue. Stem bottom of plant at (A) flower formation and (D) seed maturation stages; (B) primary and (C) secondary phloem fiber bundles. Bar scale = 100 μm (A and D), 20 μm (B and C). C, cambium; Co, collenchyma; E, epidermis; P, parenchyma; Pf, primary phloem fibers; Ph, phloem; Sf, secondary phloem fibers; and X, xylem.

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ones (**Figure 1A**, **B**). Secondary fibers located closer to the cambium and arranged in compact bundles, the number of which grows during plant development (**Figure 1A**, **C**, **D**). Hemp stems, same as in many other dicotyledonous plants, have also fibers located within xylem, but they do not form bundles, are mixed with other cell types (vessels, parenchyma) and have different properties and applications [10]. Xylary fibers are not considered further in this chapter.

By definition, primary fibers originate from primary meristem: procambium, in the region close to apical meristem [8]. Their initiation is coupled to leaf trace formation and occurs within the very top millimeters of the developing stem (**Figure 2A**, **B**). Apical meristem provides the

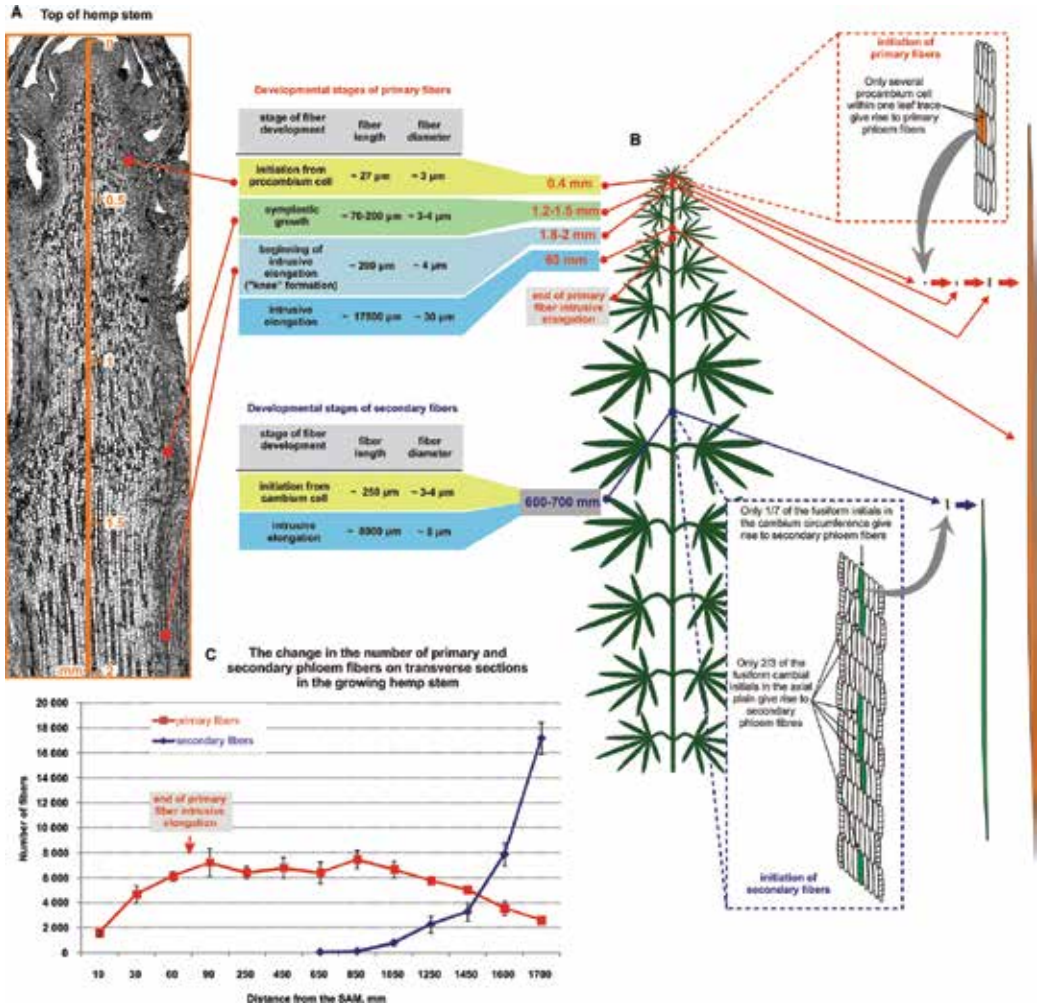

Figure 2. Scheme of initiation and intrusive elongation of primary and secondary phloem fibers in hemp stem. (A) The top of hemp stem (2 mm) [8] with primary fibers at the stages of initiation, symplastic growth, and beginning of intrusive elongation. (B) Primary and secondary phloem fibers at different stages of development, their characteristics, and localization within the stem (on the left); initiation of primary and secondary fibers from procambium and cambium, correspondingly, and morphology of fibers at different developmental stages (on the right); the length of fibers at different stages is scaled to real proportions, while the diameter/length ratio is larger than its actual value. (C) Changes in the number of primary and secondary phloem fibers on transverse section in the developing hemp stem.

increase of stem height [11]. The more downwards the stem, the older the primary fibers. Thus, developmental gradient of primary fiber can be observed along the same stem. However, it is better to characterize it, analyzing fibers at the same height through plant development, same as it was done for flax [12]. This is because primary fibers located at different stem heights originated at different periods of plant development and might be differentially affected by various endogenous and environmental factors. For example, primary fibers from the stem bottom are known to be shorter than the ones from the middle stem part [13]. Fibers traced at the same stem height through plant development have a similar background and can be correctly compared. The total amount of primary fibers in the hemp stem calculated from the total volume of primary fiber bundles and average volume of a fiber cell is around 700–800 thousand [14].

Secondary fibers originate from secondary meristem: cambium that largely provides cells for stem thickening [11]. Secondary fibers start to develop more than half a meter away from stem top (**Figure 2B**), as demonstrated for several monoecious varieties [7, 8, 14–16]. Their developmental gradient can be traced within the stem radius: the closer to the cambium, the younger the secondary fibers. Within hemp stem, secondary fibers may form several distinct concentric layers separated by other cell types. The number of such rings may reach 3–4 (**Figure 1D**). The total amount of secondary fibers in the hemp stem is around 2 million, much higher than the number of primary fibers [14]. From that, only small proportion of cambium cells (around 10%) give rise to secondary phloem fibers [8].

3. Intrusive elongation of primary and secondary fibers

Plant fibers are distinguished by their extreme length [11, 17–19]. It is mainly attained by the special type of cell elongation: intrusive growth [11, 17, 19]. This process has an enormous, though often overlooked, effect on fiber yield and quality. Intrusive elongation is characterized by the higher rate of a cell growth as compared to its neighbors. It is distinct from symplastic (also called coordinated) growth [20], when all involved cells increase their surface with the same rate, as it happens in the growth zones with most of the tissues [11]. Fibers are the classical example of cells performing intrusive elongation [11, 17, 21, 22]. During such growth, a fiber has to split middle lamellae of the cells on the way and intrude between them. Herewith, new contacts are made along the increased fiber surface, so that the stem tissues do not fall apart.

Primary phloem fibers of hemp stem start intrusive elongation rather soon after being initiated from meristematic cells [8] (**Figure 2A**, **B**). Only shortly primary fibers grow symplastically with the surrounding cells and then increase the rate of elongation. By symplastic elongation, primary phloem fibers of hemp attain the length of 200 μm [8] (**Figure 2B**). The start of the primary fiber intrusive growth is marked by the formation of the so-called "knee": the flat tip of the symplastically growing cell is transformed into the tapered one to effectively intrude the surrounding tissues [8, 19, 22]. In hemp stem, such structures are observed at 1.8–2.0 mm from the very top (**Figure 2A**, **B**). By means of intrusive elongation, primary phloem fibers of hemp stem increase their length roughly hundredfold so that their average length gets

around 18,000 μm. The volume of a fiber is increased even more, since intrusive elongation is accompanied by the increase of fiber width, so that the cell diameter gets 30 μm instead of 3–4 μm at the end of symplastic growth [8] (**Figure 2A**). The final fiber dimensions of primary phloem fibers in hemp may differ depending on the variety and growth conditions. The final length of such fiber is reported between 5000 and 100,000 μm, with modal class being around 20,000 μm, and variations in fiber diameters, which could vary between 15 and 40 μm [2, 15, 23–25]. Thus, the ratio of cell length and cell width in a primary fiber of hemp may constitute several thousands, being among the highest in plant cells.

The duration of intrusive elongation can be traced by the increase in the number of fibers on the stem cross-section. As described above, the formation of new primary phloem fibers occurs only at the region close to apical meristem [8], then during intrusive elongation, the number of primary fibers in the cross-section of hemp stem increases till approximately 60–90 mm from the apex (**Figure 2C**); end of the intrusive elongation occurs within this stem region. Further downward the stem, fibers do not elongate and their number on cross-section does not increase with time.

Elongation of fibers goes bidirectionally, as indicated by the presence of "knees" at both cell ends [8, 26]. The intrusive elongation of primary fibers goes for several days and is completely split in time with cell wall thickening, which starts later in the course of fiber development [8, 12, 26]. After the start of cell wall thickening, intrusive elongation cannot be restored any more.

During intrusive elongation, primary fibers initiated at different leaf traces reach each other, this leads to the formation of fiber bundles (**Figure 3**). The structure of the fiber bundles is almost exclusively determined by fiber intrusive growth [8, 12, 22]. The longer the fibers in the course of elongation, the thicker the fiber bundles.

It is due to intrusive growth that fibers in a bundle are so tightly packed to each other and have no intercellular spaces. The large surface of the contacts between neighboring fibers formed during intrusive elongation permits them to stay together during the retting process [4, 23, 27]. These tight contacts may be further reinforced by the special type of the "glue" between cells—pectic compounds that are constituents of middle lamellae and primary cell walls [28], and may have peculiarities in composition in fibers performing intrusive growth [22]. In the course of intrusive growth that leads to the enormous increase of cell surface, pectins are actively deposited and modified. Low-molecular mass phenolic compounds may also be involved in strengthening of interactions between polymers of fiber primary cell wall [29].

Unfavorable environmental factors, like drought, may influence the extent of fiber intrusive elongation. This would lead to the shorter individual fibers and thinner fiber bundles in the region of the stem that contained elongating fibers [30]. For primary phloem fibers, this region is located at the top of the developing stem, usually constitutes 6–9 cm, and corresponds to the maximum length of individual fibers that are elongating in this region. After the end of unfavorable conditions, this stem portion would contain fewer fibers on the cross-section and would remain the weaker part of the whole bundle until the end of plant development.

Secondary fibers do not have the stage of coordinated growth, since the stem portion ceases elongation before they are initiated from the cambium. The final length of secondary fibers is

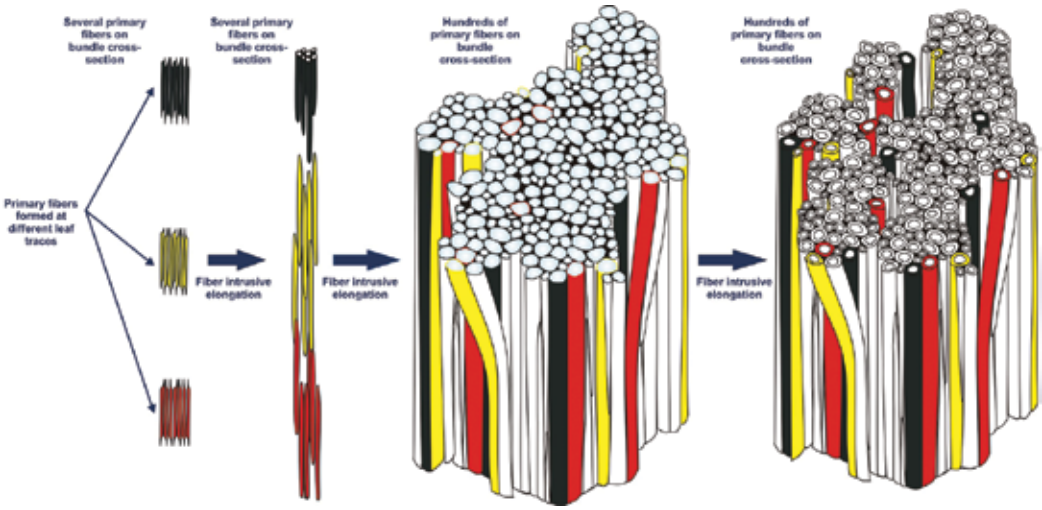

Figure 3. Formation of primary phloem fiber bundle within the hemp stem. Fibers formed within one leaf trace are given in one color; fibers formed in different tree leaf traces are given in different colors (black, yellow and red); fibers formed within other leaf traces are given in white. The scheme illustrates the formation of primary phloem fiber bundle of hemp stem and participation of fibers formed within tree leaf traces in this process.

achieved solely by intrusive elongation (**Figure 2B**). The final dimensions of secondary fibers are much lower than that of primary fibers. Their average length accounts for 2000–8000 μm, while the cell diameter is usually about 6–8 μm [8, 10, 15, 24]. However, these smaller dimensions are still considerably larger than those of cambium initials, the length of which in hemp stem was estimated to be around 250 μm [8]. As mentioned above, secondary phloem fibers start to emerge approximately at the middle part of hemp stem (600–700 mm from the apex) and their initiation continues down to stem bottom. The amount of secondary fibers on the stem cross-section increases toward the base of the stem due to both initiation of additional fibers from cambium and intrusive elongation of already existing fibers (**Figure 2C**). Due to smaller size of secondary fibers, their proportion in total yield of bast fibers in the hemp stem does not exceed 45% [10], despite the higher amount of individual cells [14]. This demonstrates the importance of the intrusive elongation stage for the final yield of bast fibers.

The bundle of the secondary fibers within the certain concentric ring is formed by a very similar process as described above for primary fibers (**Figure 3**). Fiber initiated from a cambium cell elongates and joins other intrusively growing fibers that originated from different cambium initials. The structure of fiber bundles is determined not only by the extent of fiber length increase, but also by the direction of elongation. The analysis of fiber-enriched peels demonstrates that the bundles of primary fibers in flax look like "straight columns," while the bundles in hemp form the ramified net (**Figure 4**). The degree of ramification is especially high for secondary fibers [8, 31]. Such difference is due to the "joint" or "individual" behavior of elongating fibers. In flax, the elongating fibers follow the way made by the "oldest" fiber in the forming bundle. This can be due to the special mechanical properties of middle lamellae that have been already split. For primary fibers of hemp, the situation is rather similar, but

some fibers escape from the bundle, elongating in a different direction and may reach another bundle, leading to formation of a ramified net. Such difference may be related to the large increase of stem circumference in hemp due to secondary growth, which is far less in flax.

The importance of intrusive growth for fiber yield and quality demands the approaches to regulate it. However, the mechanisms of intrusive growth are understood quite poorly. Nothing is known about the mechanisms that trigger and stop it. The peculiarities of fiber physiology at this stage of development are barely characterized. The reason for that is the difficulty to study this process, since it occurs within the depth of tissues and has never been reproduced *in vitro*. Fibers at this stage of development, being quite long cells, have only primary cell wall and can be easily damaged during sample preparation [26, 32]. This makes it quite difficult to obtain intrusively growing fibers for analysis by methods of biochemistry or molecular biology. The important step to identify molecular players involved in various stages of hemp fiber development was performed by the analysis of the transcriptome in hypocotyls of different age [33] and in different parts of young stems [34], both of which may contain intrusively growing fibers. However, the analyzed samples contained complex mixture of tissues and the early stages of fiber development were not fully identified. The indication of the stage-specific participants of fiber intrusive elongation in hemp may come from the analysis of whole transcriptome of intrusively growing fibers of flax. Such fibers were obtained by cryosectioning of stem and further, laser microdissection of fibers specifically at the stage of intrusive elongation [35]. However, elucidation of the mechanisms that perform and regulate the intrusive growth of fibers still has a long way to go.

To summarize the importance of fiber intrusive elongation, it (1) determines the final size of each individual fiber, (2) leads to the formation of fiber bundles and dictates their structure,

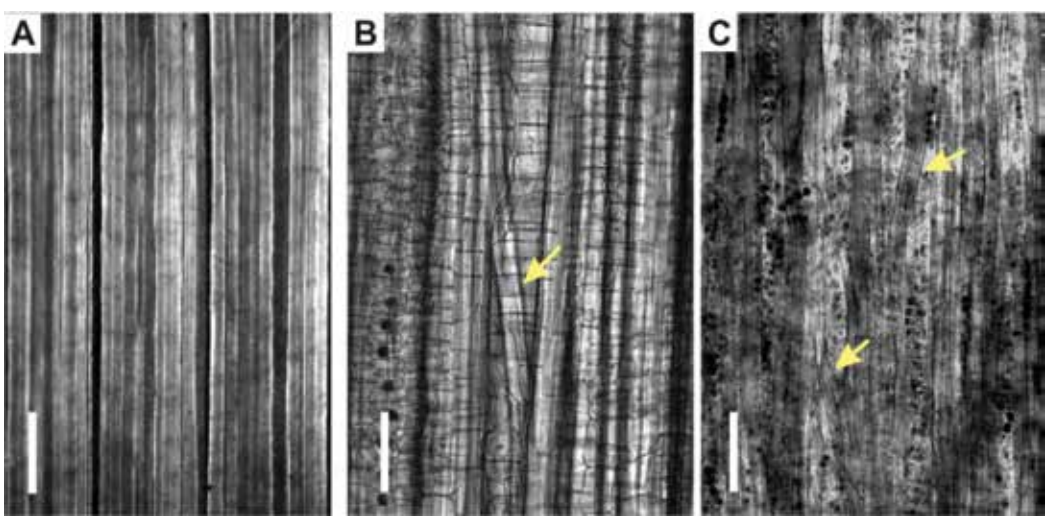

Figure 4. Comparison of phloem fiber bundles in flax and hemp. Structure of (A) primary phloem fiber bundles of flax, (B) primary and (C) secondary phloem fiber bundles of hemp [8] on the strips peeled off from the stems. Fiber bundles of hemp frequently split and merge along the stem forming numerous anastomoses (marked by arrows) unlike fiber bundles of flax. Bar = 100 μm.

(3) provides the tight contacts between the fibers that help to withstand retting process, and (4) provides the large surface for the further deposition of thick cell wall that is the major component of mature fibers.

4. Cell wall thickening

The mechanical properties of mature individual fiber largely come from thickened cell wall. In hemp fibers, cell wall may get 15 μm thick and occupy over 90% of cell cross-section [15]. The thick cell wall is not uniform and contains several layers with distinct properties. After the primary cell wall that is formed during fiber elongation, the secondary cell wall layer (S1) is deposited (**Figure 5**). The rest of the two layers of the thickened cell wall in hemp fibers are often also considered as secondary cell wall layers and named correspondingly, S2 and S3 [15, 31]. However, they differ significantly in composition and structure from S1. As revealed by fluorescent microscopy, LM10 and LM11—antibodies specific for xylan [36]—label only the outer layer of fiber cell wall [37]. Electron microscopy coupled with immunocytochemistry demonstrates that only S1 of hemp phloem fibers, both primary and secondary, is labeled by antixylan antibody [38]. The rest of the thickened cell wall does not contain epitopes for anti-xylan antibodies (**Figure 6**). This is very distinct with xylem cells, which are heavy labeled by these antibodies throughout all secondary cell wall layers [37].

The difference between S1 and the other layers of thickened cell wall is also obvious after labeling with antibody against fiber-specific galactosidase: S1 is not labeled, while the epitope is quite abundant in the inner two layers (**Figure 5B**). The antibody was raised against the enzyme isolated from flax, but it also binds cell wall in hemp phloem fibers, same as G-layers of tension wood fibers in poplar [39]. This tissue- and stage-specific β-1,4-galactosidase is necessary for maturation of cell wall structure in flax fibers, and is involved in partial trimming off the β-1,4-galactan side-chains from the backbone of rhamnogalacturonan I [40]. Similar polymer—rhamnogalacturonan I with β-1,4-galactan side-chains is also present in hemp fibers [38, 41]. Same as in flax [42, 43], fraction of this polymer is so tightly retained by cellulose that can be obtained only after complete cellulose degradation [38]. The antibody RU2 against rhamnogalacturonan I backbone [44] does not recognize any epitopes within S1 layer, but binds to the thick inner cell wall layers of hemp fibers, both primary and secondary, same as to the primary cell wall/middle lamellae region [38] (**Figure 6**). Cytochemical staining for pectin is also positive in the inner layers of hemp fibers [6]. Presence of acidic component indicates that inner layer of hemp fibers is similar to G-layers of tension wood and flax fibers [45, 46]. Notably, hemp fiber cell wall is not labeled by LM5 antibody specific for β-1,4-galactan [37, 38], despite the fact that the presence of corresponding polymer is biochemically proven [38, 41].

Difference between S1 and the rest of the thickened cell wall layers is further evidenced by the character of hemp fiber lignification. As usual for secondary cell wall layers, S1 gets lignified, especially at an advanced stage of hemp fiber development (**Figures 5C** and **6**). Lignification occurs only in the outer cell wall layers of hemp fibers (middle lamellae, primary cell wall and S1): autofluorescence that is characteristic of lignin under UV light (**Figure 5C**), and staining for

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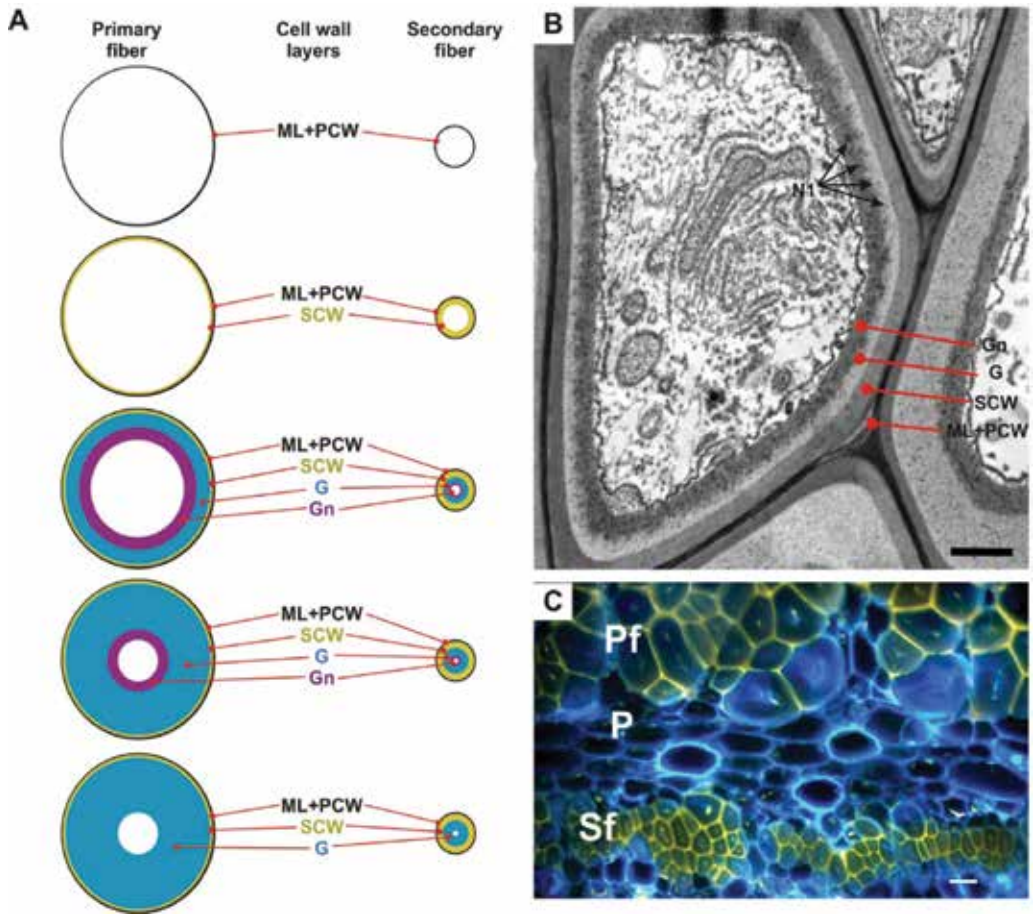

Figure 5. (A) Scheme of the sequential deposition of cell wall layers during thickening of primary and secondary phloem fibers of hemp stem. Primary and secondary fibers differ significantly in diameter as well as in the ratio of cell wall layers. The thickness of the primary cell wall and middle lamellae (ML + PCW) is comparable in primary and secondary fibers. Later deposited layer of secondary cell wall (SCW) is more developed in secondary fibers and exceeds that of primary fibers in width roughly twofold. Then, newly deposited layer of the tertiary (gelatinous) cell wall (Gn) is formed, which is later transformed into mature layer of tertiary (gelatinous) cell wall (G). In mature fibers, the layer Gn can be absent. (B) Cross-section of hemp secondary phloem fibers with different cell wall layers labeled with antibodies N1 raised against flax fiber-specific β-galactosidase [39]; label is absent in SCW, but present in G and especially Gn layers. (C) Primary and secondary phloem fibers on the cross-section of hemp stem stained with Calcofluor White, under UV light (lignified layers of cell walls look yellow due to lignin autofluorescence, nonlignified cell wall layers look blue); only outer layers of fiber cell wall are lignified. Bar = 1 μm (B) and 20 μm (C).

lignin by specific dyes like phloroglucinol are not observed in the thick inner layers, though they are obvious for S1 [6, 15]. The rest of cell wall remains nonlignified even in the fully formed fibers, which is especially obvious for primary fibers. Since the proportion of S1 layer in total cell wall is higher in secondary fibers than in primary ones, the degree of their lignification is higher [6, 15, 38]. This makes secondary fibers more coarse and rigid, and is often considered as the major reason for their lower quality [9].

The main component of the inner layers of cell wall is cellulose. This is evidenced by binding of CBM3—carbohydrate-binding module specific for crystalline cellulose [37] and also by high content of cellulose in hemp fibers [2, 3]. The content of hemicelluloses in hemp fibers is reduced at advanced stages of hemp fiber development [15], which may be due to the increasing proportion of cellulose-enriched inner cell wall layers. The average microfibril angle (MFA) toward the longitudinal fiber axis in hemp fibers is low; in the major cell wall layer (G/S2), it constitutes an average of 2.65° [47], meaning that the orientation of all microfibrils is close to axial. In S1, MFA is over 80° [47].

Same as in flax phloem fibers, cell wall of hemp fibers is a dynamic structure with intensive post-synthetic modifications of the deposited RG-I (**Figure 5**). The newly deposited portions, designated as Gn look rather loosened and contain larger amount of electron-dense material than the mature G-layer. Gn is a transient layer and is transformed into G in the course of fiber development, while the new portions of Gn are deposited (**Figure 5A**) [38]. The transition of Gn- into G-layer is coupled to the action of fiber-specific galactosidase [40] (**Figure 5**).

Together with the cell wall polymers discussed above (xylan, lignin, rhamnogalacturonan I with β-1,4-galactan side chains), immunodot analysis of isolated cell wall constituents reveals other polysaccharides, namely glucomannans, polygalacturonic acid, arabinogalactan proteins, and some xyloglucans [38]. These polymers are unevenly distributed between cell wall layers. For instance, arabinogalactan proteins are mainly detected at the inner surface of fiber cell wall, close to plasma membrane (**Figure 6**) [37]. The structural peculiarities of these polymers in hemp fibers, same as the possible differences in their structure in fibers of contrast quality are still to be characterized.

Absence or low content of xylan and lignin, axial orientation of cellulose microfibrils, presence of pectic components, processes of cell wall maturation with the involvement of tissue- and stage-specific galactosidase make to consider that the overall structure of hemp fibers resembles that of tension wood fibers and of flax fibers [48, 49], and that the inner layers of thickened cell wall may be viewed as tertiary cell wall. Tertiary cell wall, also named G-layer, is a fiber-specific cell wall type [19]. Its structure is based on the entrapment of RG-I by laterally interacting cellulose microfibrils. Tertiary cell walls are formed in fibers of various plant species and in many ecophysiological situations [50]. Secondary and tertiary cell walls have different mechanical properties, such as the lignified secondary cell wall provides rigidity, while tertiary cell wall adds flexibility due to the tension of cellulose microfibrils. The selection of fiber crops, like hemp, flax, ramie, has led to the extreme development of tertiary cell walls in fibers of their stems.

Summarizing, the major revealed differences between thickened cell wall of primary and secondary hemp fibers lay in the total cell wall width and in the proportion of S1 layer relative to the rest of cell wall. In secondary fibers, the cell wall width is considerably lower than in primary ones, while the proportion of S1 layer is higher. Since it is mainly S1 layer that gets lignified, secondary fibers have higher lignin content and due to that they are coarser than primary fibers. Importantly, the nanomechanical properties of tertiary cell wall as such are similar in primary and secondary fibers, as revealed by peak-force quantitative nanomechanical property mapping (PF-QNM) and micro tomography [31]. The major parameters of cell wall thickening that influence the yield and quality of hemp fibers are (1) the amount of the

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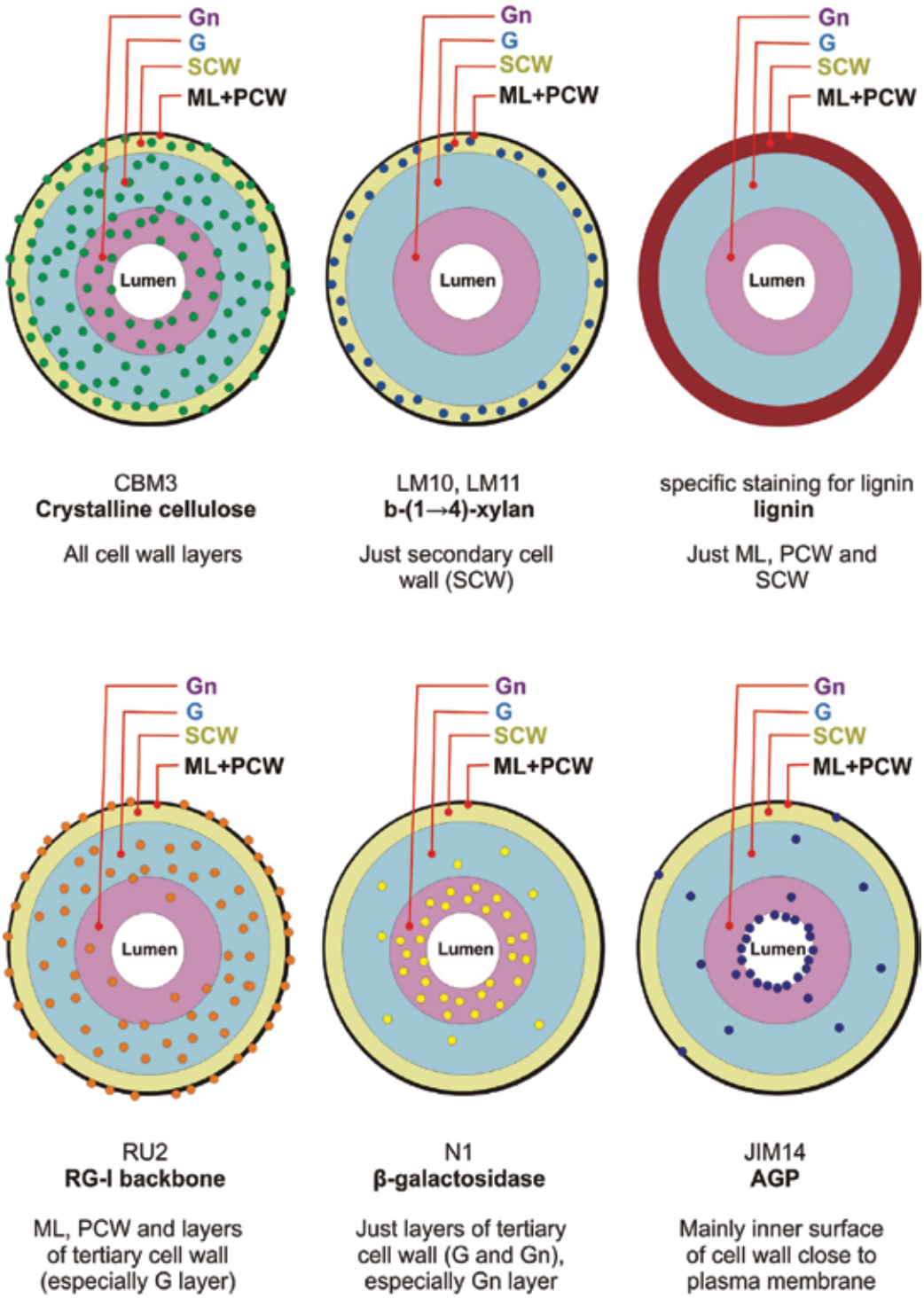

Figure 6. Scheme illustrating the differences in distribution of polymers between cell wall layers in hemp fiber. Occurrence of cell wall epitopes for carbohydrate-binding module CBM3, antibodies LM10, LM11, RU2, N1, and JIM14, and localization of lignin deposition in various layers of phloem fiber cell wall in hemp stem.

deposited cell wall (cell wall thickness), (2) the ratio between the cell wall and cell lumen on the fiber cross-section, (3) the proportion of S1 layer in total cell wall thickness, and (4) the set and peculiarities of structure of cell wall polymers.

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Chapter 4

Effect of Fiber Waviness on Tensile Properties of Sliver-Based Natural Fiber Composites

Taweesak Piyatuchsananon, Baosheng Ren and Koichi Goda

Additional information is available at the end of the chapter

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Abstract

Glass and carbon fiber-reinforced composite materials have been applied for the high demand in industrial use to date, because their advantages are light weight, high strength, and corrosion resistance. However, the disposal problem after the use of these materials has also surfaced as a serious environmental problem. As a measure to solve this problem, many researchers have tried to investigate the potential of plant-based natural fibers instead of artificial fibers. When we use natural fibers as a long fiber-reinforcement, the negative point is *irregular* fiber waviness inherent in a sliver form. This is because such fiber waviness often decreases the mechanical properties. The purpose of this study is thus to clarify the relation between irregular fiber waviness and the composite's tensile strength. The clarification was performed from two points of view: One is quantification of irregular fiber waviness, based on *spatial analysis* such as Local Moran's *I* and Geary's *c*. Result shows that quantified parameters were correlated well with tensile strengths of sliver-based natural fiber composites. Another is a 3-D finite element analysis in which the fiber waviness was treated as an orthotropic body. Finally, the relation of the tensile strengths with maximum stress and Tsai-Hill criterions was discussed.

Keywords: plant-based natural fiber, fiber waviness, spatial analysis, tensile strength, finite element method, failure criterion

1. Introduction

To date, fiber-reinforced composite materials have been used in many industries such as automotive and aerospace, because their advantages are high strength and low specific gravity. Especially, carbon and glass fibers are typical reinforcements of the composite materials.

On the other hand, it is known that production of these fibers needs a large quantity of electric energy [1]. After the use of glass fiber composites, as is also known, serious environmental problems have been caused at the process of disposal [2, 3]. Although several recycle techniques have been developed for used glass fiber composites [3, 4], scientists and engineers have tried to use plant-based natural fibers such as flax, kenaf, curaua, and ramie, which are environmentally friendly materials as an alternative [5–9]. Composite materials reinforced with the natural fibers are a nonexpensive and fast-growing material, and therefore expected to replace in whole or partially artificial fibers. Materials composed of natural fibers and biodegradable or thermoplastic resin are often called "green composites," and have been applied for car interiors [10]. Normally, automotive makers often apply short fiber composites to their interior or exterior parts, which are produced by injection molding, but mechanical properties of short fiber composites are less than long fiber composites. However, there is a problem in the usage of long fiber, which is *fiber waviness* that reduces the stiffness and strength [9]. Hsiao and Daniel [11] explored the relation between fiber waviness and mechanical properties, and proposed a theoretical model of elastic constants on a unidirectional carbon/epoxy composite with fiber waviness by the assumption of sine function. The shape of waviness by sine function was also developed by numerical analysis method [12, 13]. The results show that the ratio of amplitudes denoted as waviness parameter has an effect on the mechanical properties. Allison and Evans [14] inserted a single waviness part in a unidirectional composite and discussed its strength by identifying its wavy shape as a notch. Karami and Garnich [12] applied a failure criterion to a laminate in which a fiber wavy layer is included, using a finite element analysis. As a key-point in these papers, the fiber wavy shape was all given as a sinusoid [15]. On the other hand, the effect of *irregular* fiber waviness on tensile strength has also been studied. Ren et al. [16, 17] used Pearson's type of one- and two-dimensional autocorrelation to analyze the randomness in fiber waviness of a curaua- and flax sliver-reinforced composite, respectively. However, this method does not necessarily classify local size and intensity in irregular fiber waviness, which has the tendency to give risky areas on specimen.

Thus, the first purpose of this study is to evaluate quantitatively the randomness in fiber waviness of a flax sliver-reinforced composite, based on the *spatial analysis* such as Local Moran's I and Local Geary's c [18]. Such analyses are known as an effective method to evaluate spatial autocorrelation of point patterns, of which the object in this study is a fiber orientation angle in a small segment on the composite specimen surface. The analyzed autocorrelation value is recognized as an intensity of fiber waviness on each segment. Finally, we discuss if total size of the high intensity segments, so-called *area ratio*, affects the tensile strength of a flax sliver-reinforced composite or not.

The second purpose of this study is to explore the effect of the fiber waviness on a flax sliver-reinforced composite strength, using three-dimensional finite element analysis (3-D FEA). An orthotropic body was assumed for each finite element, which is corresponding to above-mentioned each segment. Through major stress components, maximum stress and reduced Tsai-Hill criterions [19] were applied for the prediction of risky areas in the composite. Results show that these criterions can predict the degree of damage for each element, but are not a decisive factor inducing the composite fracture. It was estimated that, finally, the specimen was fractured by mechanical unbalance in tensile stress distribution between laminae, brought from irregular fiber waviness.

2. Experimental and analysis methods

2.1. Material preparation

To prepare the composite specimen, a flax sliver (TEIKOK SEN-I Co., Ltd.) was used as a reinforcement and shown in **Figure 1(a)**. A biodegradable resin (Randy PL-1000; Miyoshi Oil and Fat Co. Ltd., Japan) was used as a matrix resin in **Figure 1(b)**. This resin was supplied in a water emulsion containing micro-order fine particles of approximately 5.0 μm diameter. The Randy PL-1000 is made from plant-derived resins. Some physical and mechanical properties were shown in **Table 1**.

2.2. Production method and the measurement of fiber orientation angle

In this research, two preparation methods were used to clarify the difference between the composites without and with fiber waviness. First method is the sheet laminate method (denoted as SLM) in which the sliver was combed to form unidirectionally oriented fibers before resin pasting. Another method is the direct method (denoted as DM), in which the sliver was used as a supply state. Thus, SLM is a composite without fiber waviness, whereas DM is a composite influenced with fiber waviness.

For the molding process, first, the emulsion type resin was painted on the sliver, and the emulsion-immersed sliver was dried for 24 hours. The semi-material obtained is called the *pre-preg*. Next, the pre-preg was cut into the size 100 × 100 mm, and two pre-pregs were put into the mold in a compression molding machine. The temperature was set at 150°C for 40 min, and the hydraulic pressure was set at 3 MPa. Subsequently, the temperature was reduced to room

Figure 1. Materials used in this study: (a) as-supplied flax sliver and (b) biodegradable resin.

Material	Density (Mg/m^3)	Fiber width (μm)	Tensile strength (MPa)	Fracture strain (%)	Young's modulus (GPa)
Randy PL-1000	1.2	–	32.5	–	3.8
Flax fiber	1.5	10–30	600–1100	1.5–2.4	40–100

Table 1. Properties of the matrix resin and fiber used in this study.

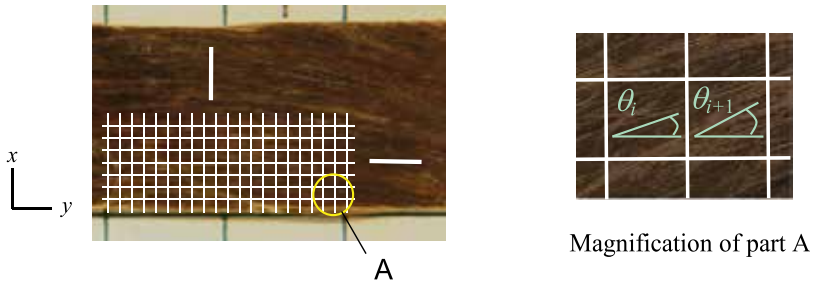

Figure 2. Division into segments on a DM specimen surface.

temperature at 3 MPa pressure for 24 hours. The resultant composite is a laminate consisting of two laminae. The thickness of the laminate is approximately 0.8 mm.

In order to quantify the irregular fiber waviness, fiber orientation angles on the specimen surface were measured by image analysis software (Asahi Kasei Corp., Japan). On the surfaces, y-axis and x-axis were respectively placed along the longitudinal and transverse directions of the specimen. The specimen was divided into small segments of 1.0 × 1.0 mm square size, as shown in **Figure 2**, which are composed of 50 segments along y-axis and 15 segments along x-axis. Thus, the total number of segments is 750 for each surface on the specimen.

2.3. Tensile test of composite specimens with and without fiber waviness

Specimens of DM and SLM composites with the size of 100 × 100 mm square were cut to 15 mm width. Before tensile test, the fiber volume fractions of all composite specimens were calculated as:

$$V_f = 1 - \frac{W - W_f}{\rho_m V} \tag{1}$$

where W is the composite specimen weight, W_f is the flax weight in the composite, V is the composite specimen volume, and ρ_m is the density of the biodegradable resin. After that, aluminum plates were attached with epoxy adhesive to both ends, which shaved 45° for preventing the stress concentration. The shape and dimension is shown in **Figure 3**. The thickness of the specimens was 0.4–0.8 mm. A strain gage was attached on the center of the

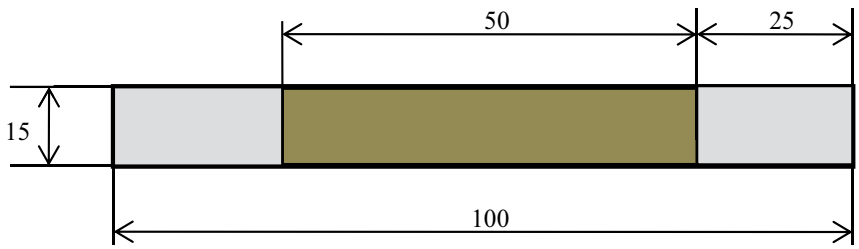

Figure 3. Shape and dimension of the tensile specimen.

specimen surface to measure uniaxial strain. Tensile test was carried out using an Instron-type testing machine (Autograph IS-500; Shimadzu Corp.) at the cross-head speed of 1 mm/min.

When conducting 3-D FEA, as mentioned later, elastic constants are needed. In this study, average Young's modulus and Poisson's ratio of SLM specimens were respectively regarded as the elastic constants, E_2, and Poisson's ratio, ν_{12}. To obtain E_1, SLM specimen with 100 × 100 mm was cut perpendicularly to the fiber axis, and the tensile specimen with 90° direction was produced with the same shape and dimension as **Figure 3**. Regarding E_3, the composite was assumed to be transversely isotropic. In addition to elastic constants, tensile strengths of SLM specimens with 0° and 90° directions were adopted as S_2 and S_1, respectively, and were used for failure criterions in Section 2.6.

2.4. Spatial autocorrelation analysis

Spatial autocorrelation analysis is a statistical tool to interpret the degree of dependence among observations in a space, based on the feature locations and the feature values [18]. From this point of view, the degree of random fiber waviness on DM composite specimen was quantified using two types of spatial autocorrelation analysis methods, Local Moran's *I* and Local Geary's *c*.

2.4.1. Local Moran's I

A spatial measure called Local Moran's *I* was created by P. A. P. Moran [18]. This measure is a typical tool to analyze data point patterns of which the concept is based on a deviation from the average. Local Moran's *I* is given for each segment as:

$$I_i = \frac{\left(\theta_i - \overline{\theta}\right)}{\frac{1}{n}\sum_{i=1}^{n}\left(\theta_i - \overline{\theta}\right)^2} \sum_{\substack{j=1 \\ j \neq i}}^{n} w_{ij}(d)\left(\theta_j - \overline{\theta}\right) \tag{2}$$

where θ_i is the measured angle of *i*-th segment, and $\overline{\theta}$ is the average of all measured angles. $w_{\mathrm{ij}}(d)$ is the weight function of the pair samples in distance class *d* at Eq. (3), given as:

$$w_{ij}(d) = \frac{1}{\sqrt{d}} = \frac{1}{\sqrt{\left(x_i - x_j\right)^2 + \left(y_i - y_j\right)^2}} \tag{3}$$

In Eq. (3), *d* is given as a distance between the central positions of *i*-th and *j*-th segments. Hence, x_i and y_i are central positions of *i*-th segment along the *x*-axis and *y*-axis in the ranges of 1–15 mm and 1–50 mm, respectively. From Eqs. (2) and (3), it is understood that I_i (*d*) is more susceptible for segments closer to *i*-th segment. Local Moran's *I* varies between −1 and +1. If Local Moran's *I* approaches +1, then the angle at this location is more largely far from the average, but relatively close to the neighbor's angles in their deviation from the average. On the other hand, if Local Moran's *I* tends to approach −1, then the angle at this location is also higher or lower than the average. In this case, the sign is different from the neighbor angles. When Local Moran's *I* tends to approach 0, the angle at this location is similar to the average.

Theoretically, when Local Moran's I is either much higher or lower than 0, then the fiber orientation angle is significantly different from the average. Consequently, such Local Moran's I points, if gathered locally, could form a large disordered area in fiber orientation. Hereinafter, Local Moran's I is denoted as LM-I.

2.4.2. Local Geary's c

Local Geary's c is another typical spatial measure, which avoids the effect of average data by using a deviation around i-th position. Local Geary's c is also given for each segment as:

$$c_i = \frac{1}{\frac{1}{n}\sum_{i=1}^{n}\left(\theta_i - \overline{\theta}\right)^2} \sum_{\substack{j=1 \\ j \neq i}}^{n} w_{ij}(d)\left(\theta_i - \theta_j\right)^2 \tag{4}$$

Local Geary's c varies between 0 and 1. When Local Geary's c tends to approach 0, the angle at this location is similar to the neighbor angles. In contrast, when Local Geary's c tends to approach 1, the angle at this location differs from the sign of neighbor angles or much higher than the neighbor's angles in absolute value. Consequently, such points can be disordered parts in fiber orientation. Local Geary's c is hereinafter denoted as LG-c.

2.5. Finite element analysis for composite specimen with fiber waviness

To obtain stress distributions on DM specimens, three-dimensional finite element analysis (3-D FEA) was used on Cartesian coordinate system. In this analysis, we assumed that the material was an orthotropic property, of which the stress $\{\sigma\}$–strain $\{\varepsilon\}$ relation was given as follows [20]:

$$\{\boldsymbol{\sigma}\} = \left[T_{ij}\right]^{-1}[C]\left[T'_{ij}\right]\{\boldsymbol{\varepsilon}\},$$

$$[C] = \begin{bmatrix} C_{11} & C_{12} & C_{13} & 0 & 0 & C_{16} \\ C_{12} & C_{22} & C_{23} & 0 & 0 & C_{26} \\ C_{13} & C_{23} & C_{33} & 0 & 0 & C_{36} \\ 0 & 0 & 0 & C_{44} & C_{45} & 0 \\ 0 & 0 & 0 & C_{45} & C_{55} & 0 \\ C_{16} & C_{26} & C_{36} & 0 & 0 & C_{66} \end{bmatrix} \tag{5}$$

where $\{\sigma\} = \{\sigma_x\ \sigma_y\ \sigma_z\ \tau_{yz}\ \tau_{zx}\ \tau_{xy}\}^T$ and $\{\varepsilon\} = \{\varepsilon_x\ \varepsilon_y\ \varepsilon_z\ \gamma_{yz}\ \gamma_{zx}\ \gamma_{xy}\}^T$. $[T_{ij}]$ is the coordinate transformation matrix and $\left[T'_{ij}\right]$ is the transposed matrix of $[T_{ij}]^{-1}$ ($i,\ j = 1, \ldots, 6$). The components of the stiffness matrix [C] for an orthotropic material in terms of the engineering constants are shown as:

$$C_{11} = \frac{1 - v_{23}v_{32}}{E_2E_3\Delta}, C_{22} = \frac{1 - v_{13}v_{31}}{E_1E_3\Delta}, C_{12} = \frac{v_{21} + v_{31}v_{23}}{E_2E_3\Delta} = \frac{v_{12} + v_{32}v_{13}}{E_1E_3\Delta},$$

$$C_{23} = \frac{v_{32} + v_{12}v_{31}}{E_1E_3\Delta} = \frac{v_{23} + v_{21}v_{13}}{E_1E_2\Delta}, C_{13} = \frac{v_{31} + v_{21}v_{32}}{E_2E_3\Delta} = \frac{v_{13} + v_{12}v_{23}}{E_1E_2\Delta},$$

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$$C_{33} = \frac{1 - v_{12}v_{21}}{E_1E_2\Delta}, C_{44} = G_{23}, \; C_{55} = G_{31}, C_{66} = G_{12},$$

$$\Delta = \frac{1 - v_{12}v_{21} - v_{23}v_{32} - v_{31}v_{13} - 2v_{21}v_{32}v_{13}}{E_1E_2E_3} \tag{6}$$

where, E is Young's modulus, v is Poisson's ratio, and G is shear modulus. The fibers are aligned on the 2-axis (on-axis), and 1- and 3-axes are perpendicular to the 2-axis at right angles to each other. When y-axis is placed along the longitudinal direction of the specimen, a fiber alignment has an angle θ to the y-axis on the x-y plane, and the stress components on the fiber axis are changed through $[T_{ij}]$ as follows:

$$\begin{bmatrix} \sigma_1 \\ \sigma_2 \\ \sigma_3 \\ \tau_{23} \\ \tau_{31} \\ \tau_{12} \end{bmatrix} = [T_{ij}] \begin{bmatrix} \sigma_x \\ \sigma_y \\ \sigma_z \\ \tau_{yz} \\ \tau_{zx} \\ \tau_{xy} \end{bmatrix}, \quad [T_{ij}] = \begin{bmatrix} m^2 & n^2 & 0 & 0 & 0 & 2mn \\ n^2 & m^2 & 0 & 0 & 0 & -2mn \\ 0 & 0 & 1 & 0 & 0 & 0 \\ 0 & 0 & 0 & m & -n & 0 \\ 0 & 0 & 0 & n & m & 0 \\ -mn & mn & 0 & 0 & 0 & m^2 - n^2 \end{bmatrix} \tag{7}$$

where $m = \cos\theta$ and $n = \sin\theta$. In this formulation, no inclination of fibers occurs in the z-x plane. Finite element mesh used in this analysis is shown in **Figure 4**, in which then isoparametric

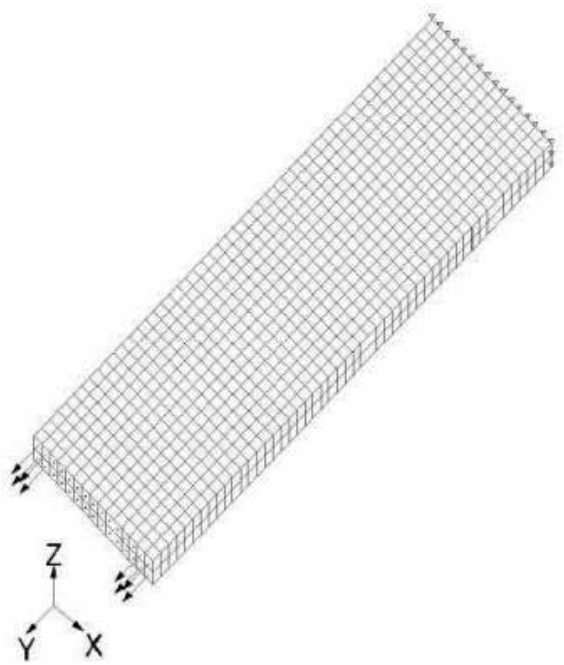


Figure 4. Three-dimensional representation of finite element mesh.

8-node hexahedron element was used. This mesh consists of two laminae, and it is assumed that no delamination occurs during deformation. Thus, each contact point at the interface between the laminae was bonded through one-nodal point. The number of element was 1500, and the number of nodal points was 7344. Each lamina consists of 750 elements, of which the meshing was constructed in the same way as the segments at angle measurement described in Section 2.2. The angle measured in each segment was substituted for θ in Eq. (7).

Elastic constants used here are as follows: when we set the fiber volume faction, V_f, as 0.70, E_1 and E_3 = 3210 MPa, E_2 = 39,500 MPa, ν_{21} and ν_{23} = 0.401, ν_{12} and $\nu_{32} = \nu_{21}E_1/E_2$. These original constants were obtained experimentally, as mentioned in Section 2.3, and estimated to the constant values at V_f = 0.7 through the rule of mixture and Reuss rule. G_{12} and G_{23} = 1610 MPa. These shear moduli was assumed as $E_1/2$ through Ref. [16]. G_{31} was also assumed as being $G_{12}/2$ from Ref. [21].

The boundary condition was a forced displacement, which was applied at one end of the finite element mesh along y-axis. Another end was fixed along y-axis.

2.6. Failure criterion

To evaluate risky area in the composites with fiber waviness, maximum stress and Tsai-Hill failure criterions are applied for the stresses on the specimen.

2.6.1. Tsai-Hill criterion

Tsai-Hill criterion is extended from the distortional energy criterion of von-Mises to anisotropic materials. According to this criterion, the general equation is written as:

$$(G+H)\sigma_1^2 + (F+H)\sigma_2^2 + (F+G)\sigma_3^2 - 2H\sigma_1\sigma_2 - 2G\sigma_1\sigma_3 - 2F\sigma_2\sigma_3 + 2L\sigma_4^2 + 2M\sigma_5^2 + 2N\sigma_6^2 = 1 \tag{8}$$

where F, G, H, L, M, and N are the material strength parameters. For the plane stress in 1–2 plane of a unidirectional lamina with fiber in the two direction, $\sigma_3 = \tau_{13} = \tau_{23} = 0$. Eq. (8) was reduced and written as:

$$\left(\frac{\sigma_1}{S_1}\right)^2 - \frac{\sigma_1\sigma_2}{{S_2}^2} + \left(\frac{\sigma_2}{S_2}\right)^2 + \left(\frac{\tau_{12}}{S_{12}}\right)^2 = 1 \tag{9}$$

Then, we applied Tsai-Hill criterion for the definition of risky areas in failure on the specimen. The stresses at each element are influenced by the interaction between elements. Especially, as mentioned later, σ_2 and τ_{12} were the dominant stress components, so in this study the reduced Tsai-Hill criterion was used as shown in the following: where, S_1 is the transverse strength of the lamina, S_2 is the tensile strength along the fiber axis, and S_{12} is the shear strength. The strength values are given as S_1 = 6.44 MPa, S_2 = 232 MPa, which were obtained by normalizing the SLM experimental values to the fiber volume fraction of 0.70. S_{12} was estimated as a double value of S_1 referring to the data of conventional fibrous composites [22].

The values of Tsai-Hill criterion vary between 0 and 1. When the value tends to approach 0, in general, this location is interpreted as a nonrisky area. In contrast, when it tends to approach 1, this location is recognized as a risky area.

2.6.2. Maximum stress criterion

Maximum stress criterion is given on the assumption that, when the ratio of stress to strength achieves to 1, the failure occurs. For this criterion, we used the longitudinal, transverse strength, and shear strength to predict the risky areas on specimens. Maximum stress criterion was hereinafter denoted as MS. When the MS trends to 1, this is the risky point on the specimen. In contract, MS trends to 0 when the value on the specimen is not the risky point. The MS is described as the below equation.

$$\frac{\sigma_1}{S_1} = 1, \frac{\sigma_2}{S_2} = 1, \text{ and } \frac{\tau_{12}}{S_{12}} = 1 \tag{10}$$

where, S_1, S_2, and S_{12} are transverse strength, longitudinal strength, and shear strength, respectively.

3. Results and discussion

3.1. Tensile test results

Fiber orientation angles and tensile properties of SLM and DM are shown in **Table 2**. The results show that tensile strengths of DM specimens accompanied with the fiber waviness are lower than those of SLM. Young's moduli of DM are also lower. It should be noted that the fiber volume fraction of DM specimens are lower in whole than that of SLM. This is attributed to the fact that the fibers cannot be packed compactly, as compared to SLM specimens, because of irregular fiber waviness. However, the lower volume fraction does not necessarily cause lower tensile strength of DM specimens. Here, we estimate the tensile strength of SLM specimen at 61%, the average fiber volume fraction, using $\sigma_c' = (V_f'/V_f)\sigma_c = (0.61/0.72) \times 238$, which is the lower bound of the law of mixture. Where, σ_c is the composite's tensile strength, V_f is the fiber volume fraction, and a symbol "dash" means a predicted value, such that σ_c' is a value at the fiber volume fraction V_f' predicted from σ_c. The calculated tensile strength was 202 MPa. This value is clearly higher than 175 MPa, the average strength of DM specimens, despite of the value derived from the lower bound. It is considered that, therefore, the waviness is a major factor to reduce the tensile properties in strength of the sliver-based natural fiber composite, whereas several low V_f specimens show relatively large strengths, as mentioned above, and therefore the effect of irregular fiber waviness is needed to be discussed with unified V_f.

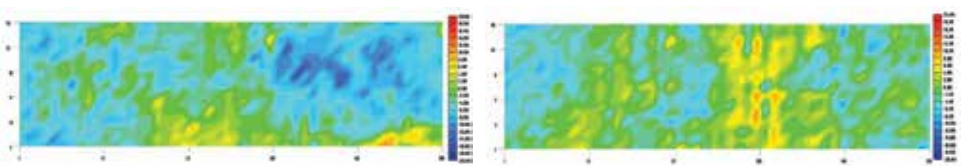
3.2. Angle distribution and analytical results of special autocorrelation

In order to make contour maps of the fiber orientation angle distribution, each measured angle was assigned to the center point of each segment. **Figure 5(a)** and **(b)** show examples of the contour color maps. The blue (upper right portion in **5(a)**) and red (center portion slightly to

Production method	Specimens						Tensile strength (MPa)	Young's modulus (GPa)
	Sample no.	Fiber volume fraction	Side A (lower angle)		Side B (higher angle)			
			Avg. angle (°)	S.D. of angles (°)	Avg. angle (°)	S.D. of angles (°)		
SLM*	—	0.72	0	—	0	—	238	40.5
DM	<1>	0.55	1.99	3.54	3.30	4.28	132	22.7
	<2>	0.55	2.62	5.22	4.19	4.03	224	26.7
	<3>	0.55	5.14	4.06	5.55	3.18	158	29.6
	<4>	0.55	3.05	3.22	8.76	4.18	211	27.6
	<5>	0.65	1.74	3.73	2.85	2.66	158	23.3
	<6>	0.65	2.10	2.62	3.07	3.48	203	26.4
	<7>	0.65	1.61	2.33	2.20	2.32	153	27.5
	<8>	0.65	2.08	3.47	3.76	2.95	170	28.0
	<9>	0.65	1.45	3.72	5.26	4.77	173	26.8
	<10>	0.65	0.76	3.50	4.55	3.90	168	28.8
	Avg.	0.61	2.35	3.50	4.35	3.60	175	26.8

*The number of SLM specimens was seven, and their average values are shown.

Table 2. Fiber orientation angles and mechanical properties of flax sliver-reinforced composite laminates [17].



(a) (b)

Figure 5. Contour map of fiber orientation angle distribution. (a) Specimen 2A (avg. angle: 4.19°, standard deviation: 4.03°) and (b) specimen 5A (avg. angle: 4.55°, standard deviation: 3.90°).

the right in **5(b)**) color areas show the high angle on the negative and positive sides respectively on specimens 2A and 5A, of which the fiber orientation angles vary with approximately 4.0 degree standard deviation. Such contour mapping results and statistical aspect mean that the fiber waviness occurs locally and randomly.

Figure 6(a) and **(b)** shows typical examples of analytical results of LM-*I*. As shown in both the figures, positive LM-*I* areas higher than 0.5 are locally distributed with agglomeration of about 5–10 mm scale, whereas several negative LM-*I* areas are dispersed with a smaller scale. In comparison with **Figure 5**, it is recognized that Specimen 5A of **Figure 6(b)** shows quite a similar distribution. Specimen 2A of **Figure 6(a)** shows that, on the other hand, both positive and negative orientation angles are evaluated with similar color levels. In other words, LM-*I* is

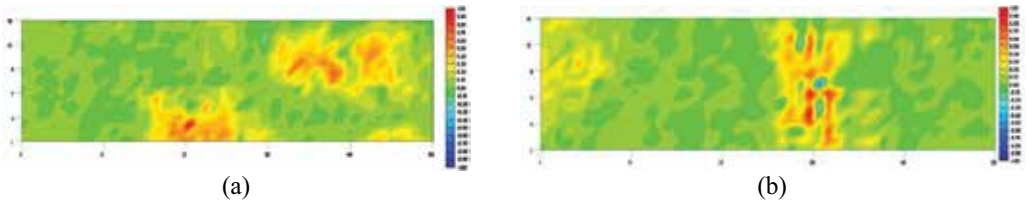


Figure 6. Contour maps of LM-*I* distribution. (a) Specimen 2A and (b) specimen 5A.

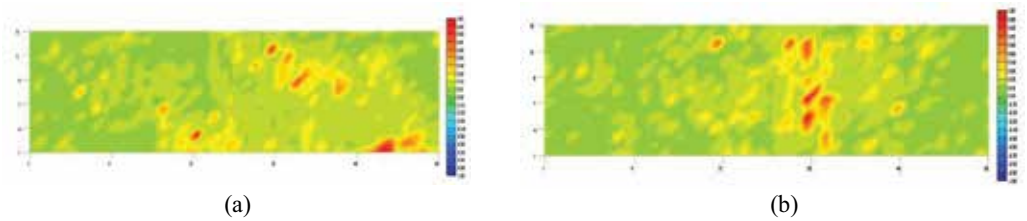


Figure 7. Contour maps of LG-*c* distribution. (a) Specimen 2A and (b) specimen 5A.

characterized by giving an even evaluation, irrespective of the difference between positive and negative angles. We consider, the intensity and scale of such disorders in fiber orientation is related with the tensile strength to some degree.

Figure 7(a) and **(b)** shows typical examples of analytical results of LG-*c*. In these figures, the range from −1 to 1 is intentionally employed to compare them with LM-*I* distribution. As shown in the both figures, LG-*c* areas higher than 0.5 are locally dispersed with about 1–3 mm scale, quite smaller than LM-*I*, whereas major parts of the specimens are occupied by low LG-*c* values. Thus, LG-*c* is a measure accompanied with locally smaller scale.

3.3. Threshold levels of LM-*I* and LG-*c* values: definition of area ratio

In this section, we tried to quantify the results of the above spatial analyses by using the concept of "area ratio" characterized by threshold levels. If the area ratios are well correlated with tensile strength data, the strength could be estimated from the fiber orientation angle distribution measured in advance. In this quantification, we used two imaging programs; first is "Graph R221," which was used for plotting a contour map, and the next is "Azo R235," which was used for calculation of area ratio.

For instance, when we choose temporarily threshold levels higher than 0.3 or less than −0.1, the corresponding LM-*I* areas can be obtained, as shown in black areas of **Figure 8(a)**, **(b)**. In these cases, the area ratios of specimens were calculated as 12.6% (positive side = 11.1% and negative side = 1.5%) and 9.1% (positive side = 6.8% and negative side = 2.3%) for the specimens 2A and 5A, respectively. In **Figure 9(a)**, **(b)**, the black areas of LG-*c* are higher than 0.2 were selected. The area ratios of specimens were calculated as 16.5 and 13.5% for specimens 2A and 5A, respectively.

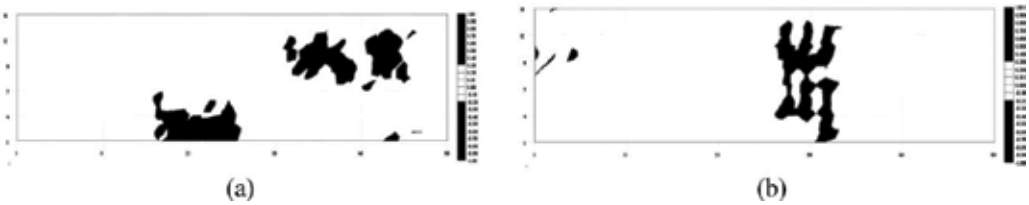


Figure 8. Binary images of Local Moran's *I* distribution: (a) Specimen 2A and (b) specimen 5A (threshold levels: positive LM-*I* = 0.30 and negative LM-*I* = −0.10).

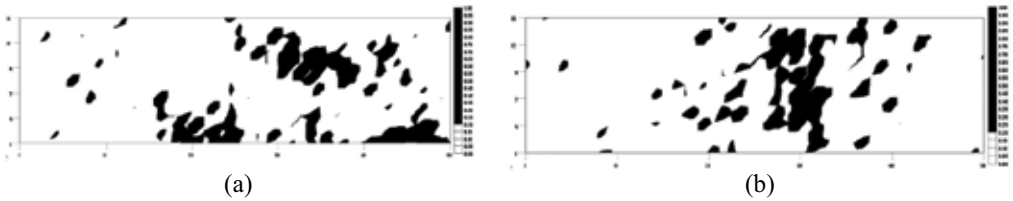


Figure 9. Binary images of local Geary's *c* distribution: (a) Specimen 2A and (b) specimen 5A (threshold level: LG-*c* = 0.20).

Here, we consider the change in area ratio with change in threshold level. Regarding LM-*I*, we take an account of the threshold levels of negative value with a relatively wide range, similarly to that of positive values. This is because the segments with negative LM-*I* values have a possibility of causing premature damage in shear, although these are not so major. We consider, if appropriate threshold levels are given, there is an optimal area ratio at each specimen, which is well correlated with tensile strength. In other words, the ratio of segments suffering damage during tensile loading should be related closely with the area ratio. In the next segment, thus, the relation between area ratio and tensile strength is investigated.

3.4. Relation between area ratio and tensile strength

To investigate the correlation between the area ratio and tensile strength, normalized tensile strength data were plotted as a function of area ratio, as shown in **Figures 10** and **11**. In these plots, each tensile strength value was normalized by dividing the measured strength by the fiber volume fraction V_f and then multiplying it by 0.72, which is V_f of SLM specimen. In **Figure 10**, it is confirmed that the normalized strength is correlated with the area ratio to some extent. The correlation coefficients of LM-*I* were respectively calculated as −0.44 and −0.65 when setting threshold levels at LM-*I* > 0.6 or LM-*I* < −0.1 in **Figure 10(a)** and LM-*I* > 0.8 or LM-*I* < −0.1 in **Figure 10(b)**. The value of −0.65 is not so strong but presents an intermediate strong correlation. This means, if many segments in a specimen are distributed with LM-*I* values higher or lower than the above threshold levels, its tensile strength tends to be lowered. It also means that a rough value of tensile strength can be estimated through the least-squares regression line between area ratio and tensile strength.

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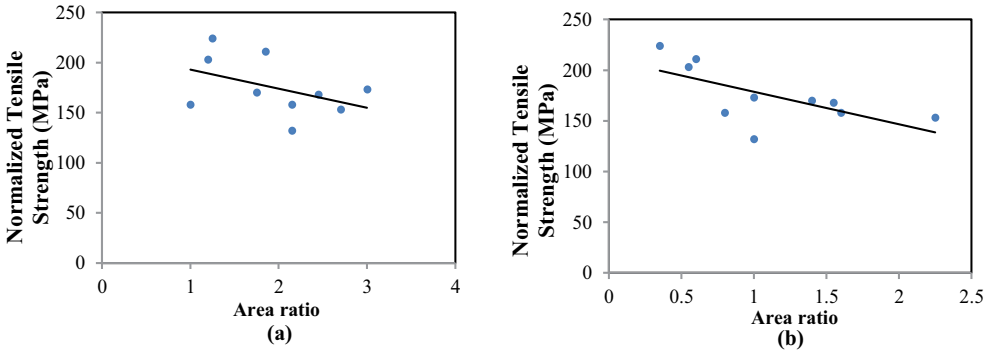

Figure 10. LM-*I* area ratio dependence on normal tensile strength. (a) Threshold levels: LM-*I* > 0.6 or LM-*I* < −0.1 and (b) threshold levels: LM-*I* > 0.8 or LM-*I* < −0.1.

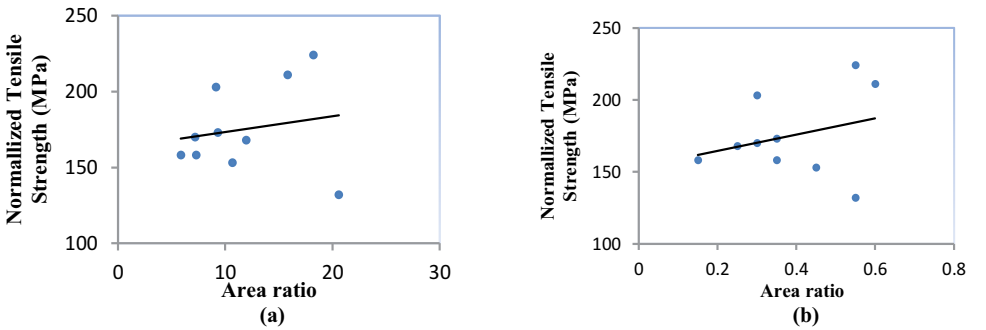

Figure 11. LG-*c* area ratio dependence on normal tensile strength. (a) Threshold levels: LG-*c* > 0.2 and (b) threshold levels: LG-*c* > 0.5.

In contrast, the LG-*c* area ratio plots are not so correlated with tensile strength data, as shown in **Figure 11(a)** and **(b)**. The correlation coefficients between the area ratio and tensile strength were obtained as 0.18 and 0.29, when setting threshold levels at 0.2 and 0.5, respectively. The both values of 0.18 and 0.29 obviously signify weak correlation. This result implies that the values of 0.2 and 0.5 are inappropriate as a threshold level, or that LG-*c* is a measure not to match correlation with tensile strength.

Figure 12(a) shows the change in correlation coefficient with change in positive threshold level of LM-*I*. In this case, the negative threshold level was fixed at −0.1. It is found that the correlation coefficient decreases gradually approximately from 0.25, and shows the lowest value around 0.70, which is approximately −0.80, the highest negative coefficient meaning a strong correlation. The correlation coefficient is also sensitive for the change in negative threshold level, as shown in **Figure 12(b)**. Three positive threshold levels, 0.69, 0.75, and 0.77, were chosen for simplicity. It is confirmed that the optimal threshold levels of LM-*I* are 0.69 at the positive level and −0.10 at the negative level. These optimal levels brought the correlation coefficient of −0.832, from which it is concluded that LM-*I* area ratio yields a strong correlation

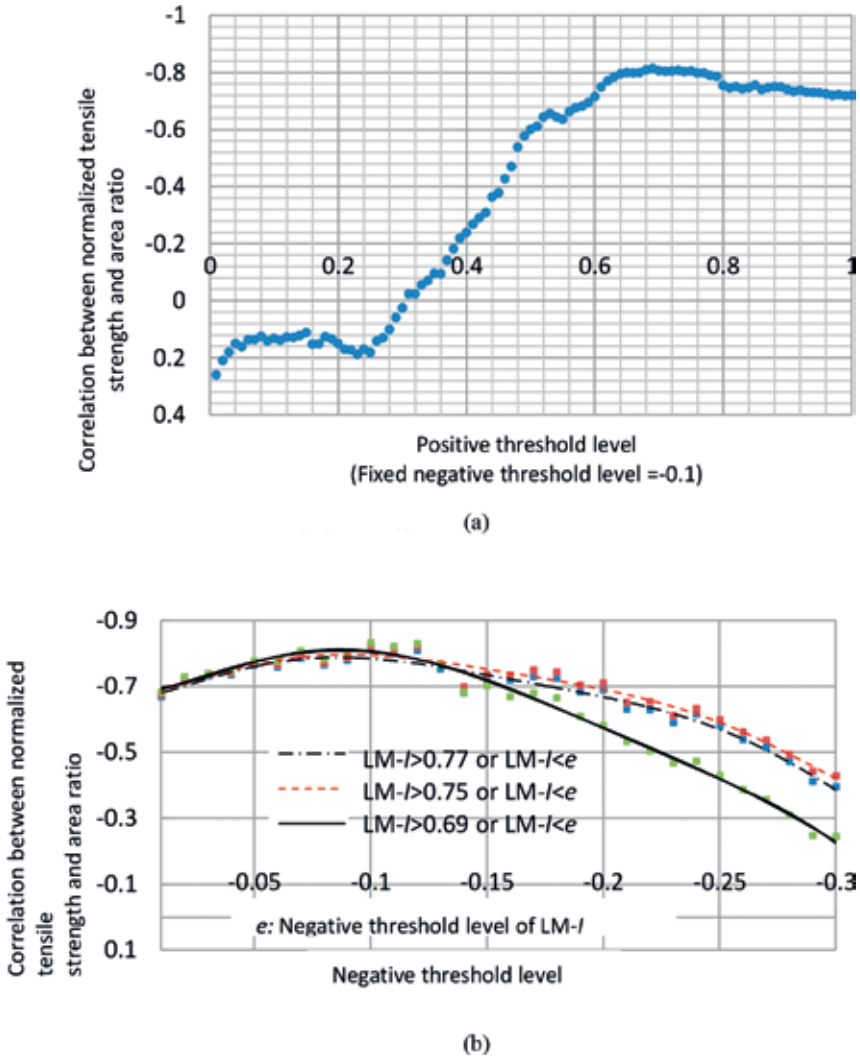

Figure 12. Correlation coefficients between LM-*I* area ratio and normalized tensile strength. (a) Correlation coefficient vs. positive threshold level. (b) Correlation coefficient vs. negative threshold level.

with tensile strength. In other words, tensile strength can be estimated to some degree through the least-squares regression line when setting the optimal threshold levels of LM-*I*. It is also expected that the present procedure is applied as an effective screening method extracting low quality pre-pregs at quality inspection.

Figure 13 shows the relation between correlation coefficient and threshold level of LG-*c*. Although the coefficient varies with change in the threshold level of LG-*c*, the highest coefficient is only −0.14 at threshold level of 0.36. This coefficient value is quite a weak correlation, and therefore it is concluded that LG-*c* area ratio is not a parameter to match correlation with tensile strength.

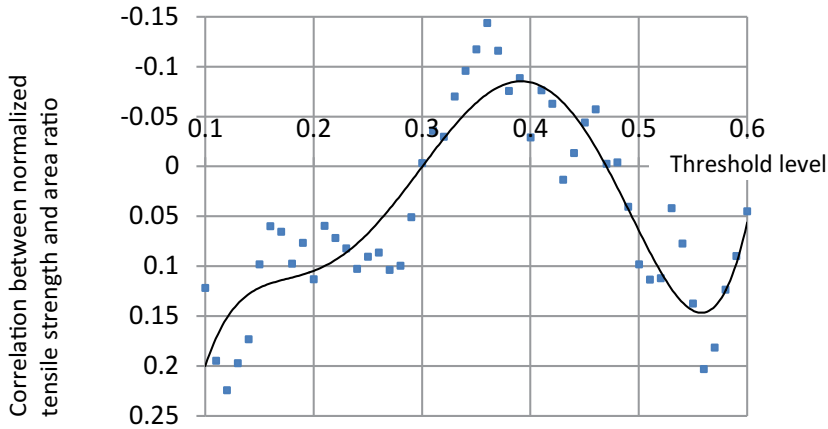

Figure 13. Correlation coefficients between LG-*c* area ratio and normalized tensile strength vs. threshold level.

3.5. Normalized stress distributions and maximum stress criterion

Figure 14 shows typical computed stress distributions. **Figure 14(a)** and **(b)** shows σ_x and σ_y contour maps, respectively, which were divided by the maximum value of all normal stress components. σ_z contour map is omitted in **Figure 14** because it is quite the same as σ_x contour map. **Figure 14(c–e)** are τ_{xy}, τ_{yz}, and τ_{zx} contour maps, respectively, which are divided by the

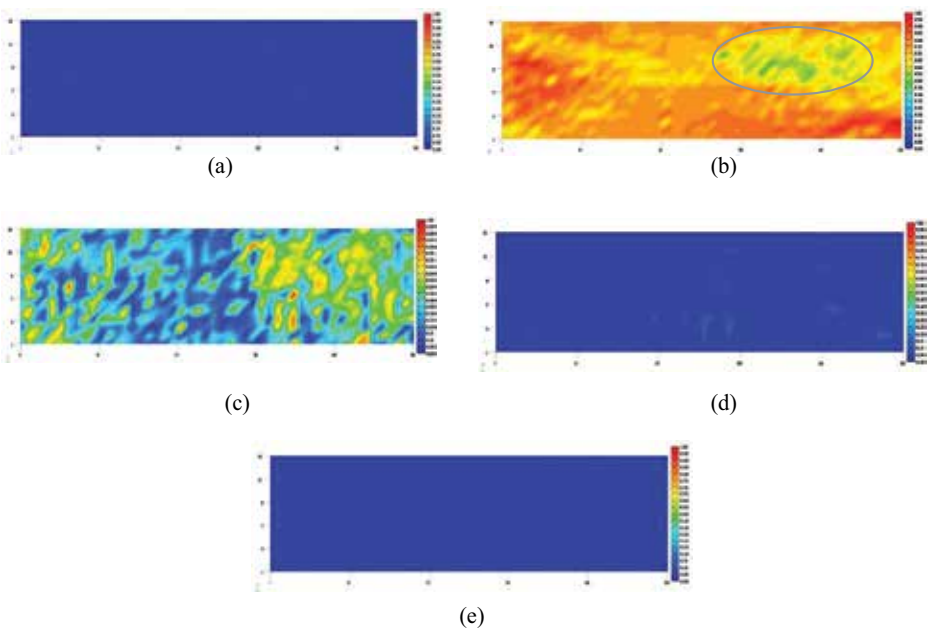

Figure 14. Contour map of normalized stress distributions (Specimen 2A). (a) Contour map of σ_x distribution; (b) contour map of σ_y distribution; (c) contour map of τ_{xy} distribution; (d) contour map of τ_{yz} distribution and (e) contour map of τ_{zx} distribution.

maximum value of all shear stress components. The shear stress components were replaced by absolute values. It is proved that σ_y distribution is much larger than σ_x and σ_z, because the specimen is reinforced along the loading axis (y-axis). Regarding the case of shear stress, τ_{xy} distribution working in the plane is also much higher than the others. Shear stress τ_{yz} slightly occurs in the specimen, while τ_{zx} components are negligibly small.

To evaluate the degree of contribution of on-axis stress components to damage occurrence, the maximum stress criterion was used, in which the on-axis stresses, $\sigma_1, \sigma_2, \sigma_3, \tau_{12}, \tau_{23}$, and τ_{31}, were divided by the failure stresses, respectively.

$$\tilde{\sigma}_1 = \frac{\sigma_1}{S_1}, \tilde{\sigma}_2 = \frac{\sigma_2}{S_2}, \tilde{\sigma}_3 = \frac{\sigma_3}{S_1}, \tilde{\tau}_{12} = \frac{\tau_{12}}{S_{12}}, \tilde{\tau}_{23} = \frac{\tau_{23}}{S_{12}}, \text{and } \tilde{\tau}_{31} = \frac{\tau_{31}}{S_{12}} \tag{11}$$

The normalized stresses in Eq. (11) were furthermore unified by dividing them by α (α: the maximum value of $\tilde{\sigma}_1, \tilde{\sigma}_2, \tilde{\sigma}_3, \tilde{\tau}_{12}, \tilde{\tau}_{23}$, and $\tilde{\tau}_{31}$) in order to deal with the maximum as 1.0. Contour map of $\tilde{\sigma}_3$ is again omitted because of the less stress values. In comparison between **Figure 15(a–e)**, the maximum normalized stress was found in **Figure 15(c)**. It means that the damage or failure may occur in $\tilde{\tau}_{12}$, although the area scale is small. The second risky normalized stress was found in $\tilde{\sigma}_2$ of **Figure 15(b)**. $\tilde{\sigma}_1$ in **Figure 15(a)** are still affecting on the specimens but they do not have influence so much. Regarding $\tilde{\tau}_{23}$ and $\tilde{\tau}_{31}$, they are also quite small, and therefore we can neglect them from the fracture criterion. The same tendency was

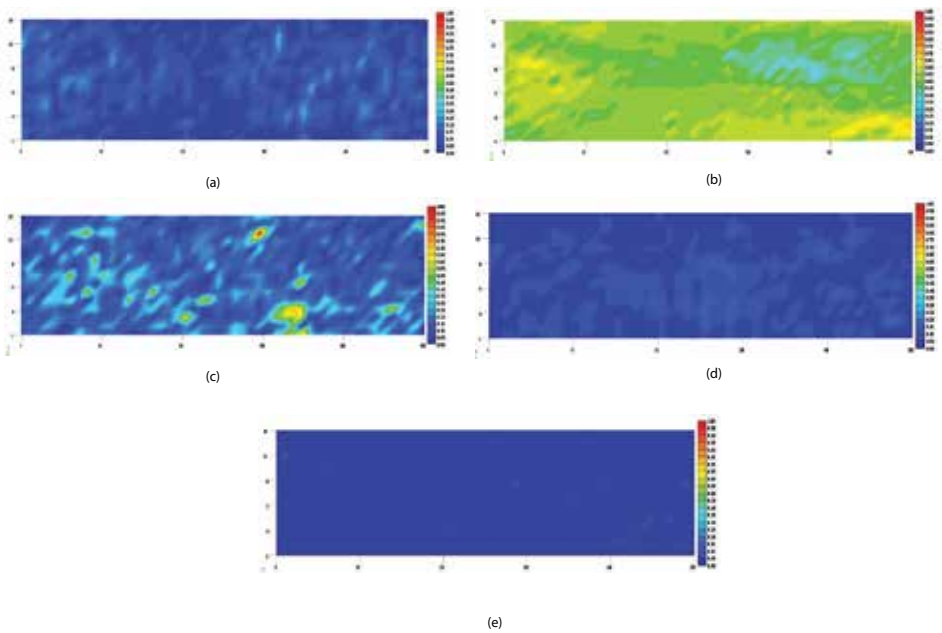

Figure 15. Contour maps of unified normalized stress-distribution based on the maximum stress criterion (Specimen 2A). (a) Contour map of unified $\tilde{\sigma}_1$ distribution; (b) contour map of unified $\tilde{\sigma}_2$ distribution; (c) contour map of unified $\tilde{\tau}_{12}$ distribution; (d) contour map of unified $\tilde{\tau}_{23}$ distribution and (e) contour map of unified $\tilde{\tau}_{31}$ distribution.

also confirmed in the other specimens. Thus, tensile strength of the specimens may be affected by local shear damage mode, according to the maximum stress criterion. But, we should also focus on the possibility of tensile fracture, because the size of the risky area is larger than the shear damage area, as seen from comparison between **Figure 15(b)** and **(c)**.

3.6. Comparison between spatial autocorrelation analyses, maximum stress criterion and Tsai-Hill criterion with the fracture specimen

Figure 16(a) and **(b)** shows the fractured specimen (Sample No.2) and the contour map of Tsai-Hill criterion, respectively. As is seen, the latter resembles **Figure 15(c)**. This means, the obtained result from Tsai-Hill criterion is occupied by in-plane shear stress component. In comparison between **Figure 16(a)** and **(b)**, the both shear fracture portions of the upper and lower sides (see, arrows) in **Figure 16(a)** may be slightly different from those of **Figure 16(b)**, but the locations are quite close. It is considered that, on the other hand, it may be severe to cause the whole fracture from such small damage areas. It also looks in **Figure 16(a)** that tensile fracture occurs between the two shear damage areas. **Figure 16(c)** shows σ_y stress distribution of specimen 2B. It is confirmed that the tensile fracture portion is loaded more largely, whereas σ_y stress distribution of specimen 2A in **Figure 14(b)** is reduced (see both regions surrounded by an ellipse). It is estimated that such an unbalanced σ_y stress distribution between laminae 2A and 2B causes the final tensile fracture. In other specimens, the similar unbalanced σ_y stress distributions were confirmed (it is omitted because of limited space). It is predicted that, thus, although the flax sliver-based composite with irregular fiber waviness may receive some small damages firstly, finally this fractures due to the unbalanced normal stress distribution between laminae.

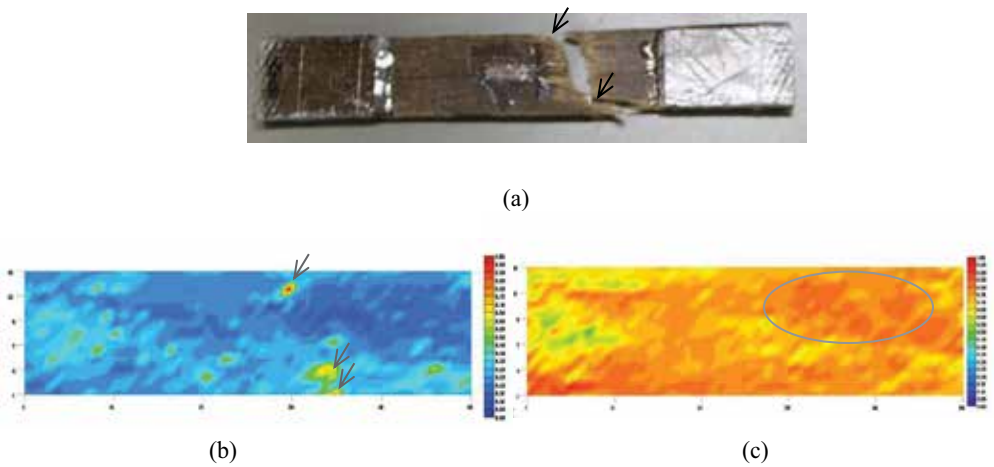

Figure 16. Comparison between fractured specimen, Tsai-Hill criterion and normal stress distribution. (a) Fractured specimen; (b) contour map of Tsai-Hill criterion (specimen 2A) and (c) contour map of σ_y stress distribution (specimen 2B) divided by the same maximum value as (specimen 2A).

4. Conclusions

The effect of irregular fiber waviness on tensile strength of a flax sliver-based biodegradable thermoplastic resin composite laminate was clarified. The fiber orientation angles were measured at fine segments on the both surfaces of the composite specimen. As a first approach, two representative spatial analyses, Local Moran's I and Local Geary's c, were carried out for the quantification of the fiber waviness. Then, we calculated correlation coefficients between tensile strength and various area ratios obtained by changing the ranges of Local Moran's I and Local Geary's c. The results showed that Local Moran's I was correlated well with tensile strength of the composite specimens when appropriate ranges were selected. On the other hand, Local Geary's c was not well correlated with tensile strength. Thus, it is concluded that the analysis method used in this study is an effective tool of predicting roughly the tensile strength of natural fiber-sliver-based composites.

As a next approach, we evaluated stress distributions in the composite specimen using a three-dimensional finite element analysis (3-D FEA) based on the orthotropic theory, in which measured fiber orientation angles were substituted for the finite elements. Results showed that σ_y distribution was much larger than σ_x and σ_z, which means that the specimen is reinforced along the loading axis (y-axis) despite of irregular fiber orientation. Regarding the other stresses, in-plane shear stress, τ_{xy}, was much higher than the others. Shear stress τ_{yz} slightly occurred in the specimen, whereas τ_{zx} components were negligibly small. For these results, the maximum stress criterion was firstly applied, in which the on-axis stresses, σ_1, σ_2, σ_3, τ_{12}, τ_{23}, and τ_{31}, (where 1 and 3 are the transverse direction of the fiber axis, and 2 is the fiber axis) were divided by the failure stresses, respectively. As a result, the normalized maximum stress was found on τ_{12} distribution map. It means that a small damage may occur in τ_{12}. The fiber axis stress, σ_2, occupied a relatively large stress area, although the stress itself is smaller than the normalized maximum stress of τ_{12}. Tsai-Hill criterion, was also applied to predict more dangerous damage areas in the composite specimen. These results were compared with fracture paths of the actual specimens. It was estimated that, finally, fracture occurred by enhanced tensile stress occurring in the counterpart of the composite laminate, which was caused by partially off-axial fiber area in a lamina.

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Chapter 5

Mechanical and Fracture Surface Analysis of Higher Viscous Epoxy/Multiwalled Carbon Nanotube Nanocomposites Subjected to Flexural Loading

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Ahmad H. Muhammad Ismail

Additional information is available at the end of the chapter

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Abstract

This study experimentally characterizes the effect of multiwalled carbon nanotubes (MWNTs) reinforced to higher viscous aircraft thermoset polymer epoxy. The effects of MWNTs weight percentage (wt%) to flexural and fracture toughness properties were investigated via Mode I fracture behaviour. This experiment found that the average increment in fracture toughness of 0.1 and 0.3 wt% MWNTs reinforced to epoxy is 62.7 and 31.8%, respectively. However, shifting to a higher viscosity epoxy lead to some difficulties like to remove void formed in matrix and harder to achieve appropriate carbon nanotubes (CNTs) dispersion due to limited pot life and working time. Morphological study analysis on fracture surface using field emission scanning electron microscopic (FESEM) shows that the mechanical properties enhancement was attributed to crack pinning, crack path deflection and localized inelastic matrix deformation due to agglomerated CNTs. The study concluded that the key important to the extent the strength and fracture toughness is by finding the appropriate processing method to achieve adequate state of CNTs dispersion within the matrix.

Keywords: fracture, impact, carbon nanotube, nanocomposites

1. Introduction

Thermosetting polymers have been extensively used in aviation industry as a replacement for historic manufactured materials such as steel and aluminium due to the remarkable advantages in strength and stiffness properties. Today, the incorporation of composites has become a common practice in aircraft design and manufacturing [1–7]. In the next generation

of aircraft, the use of polymer composites is expected to hit more than 50% of the overall aircraft's structures. This transformation was developed due to the desire of reducing costs and weight while not losing the strength and stiffness of the materials. However, polymer composites have deprived in fracture toughness compared to steel and aluminium, and this became an impediment to polymer composites to become the so-called dominant materials on aircraft structures. Over the years, tremendous efforts [1–13] have been conducted to alter epoxy system by adding either micron-sized soft (rubber or thermoplastic) or rigid (glass or ceramic) particles into the polymer matrix. These filler particles are then expected to provide extrinsic toughening mechanism through crack bridging, crack pinning, crack path deflection, localized inelastic matrix deformation and void nucleation [8].

In recent years, carbon nanotubes (CNTs) have attracted a great interest for nanoparticle-reinforcing candidates instead of those particles on polymer matrices due to proven superior mechanical, electronic and thermal properties [9]. Number of researches has been conducted to ascertain the mechanical characteristic of these nanopolymer composites. Certain properties pertaining to the aircraft structural composite, which are flexural strength and fracture toughness, have become an important basic property to predict the life of the composite structure. In the works of evaluating the effect of CNTs incorporation to polymer epoxy's fracture toughness, Thostenson et al. [10] reported adding 0.1 and 2.0 wt% into epoxy resin shows an increase in fracture toughness from 0.66 to 0.73 and 0.84 $MPa.m^{1/2}$, respectively. In the work of Yu et al. [11], the mode I fracture toughness of 1.0–3.0 wt% of multiwalled carbon nanotubes (MWNTs) reinforced epoxy-matrix composites is experimentally determined via compact test, and it is in the range of 0.53–0.73 $MPa.m^{1/2}$. Other work by Zhou et al. [12] conducted flexural and fracture toughness tests and reported that the strength and fracture toughness of MWNTs reinforced epoxy are 120 MPa and 150 $MPa.mm^{1/2}$, respectively, for 0.3 wt% MWNTs. These variations of results reported are due to the different types of epoxy system and nanocomposite processing techniques used.

Basically, CNTs improve high mechanical properties at low volume fractions due to the high aspect ratio to volume ratio of the nanosized particles. However, high specific ratio has a strong tendency to agglomerate and has an adverse effect that decreases the strength of the nanocomposite due to the stress concentration effect [12]. Thus, the significant enhancement in polymer-CNTs composites is generally dependent to the degree of dispersion and interfacial adhesion conditions between CNTs and matrix. Srivasta and Singh [13] and Gkikas et al. [14] reported that the strength and fracture toughness enhancement in 0.5 and 1.0 wt% MWNTs inclusion have a wide range of enhancement or detraction value, which is very sensitive to the dispersion technique applied. This study is an attempt to apply CNTs into the epoxy resin, Hysol EA956 A/B that has been practically used for modern aircrafts. The primary objective is to determine the effect of adding CNTs to strength and fracture toughness properties of epoxy, in which the viscosity is higher than any resin previously studied. The fracture behaviour and mechanisms of reinforced materials will be inspected using FESEM. FESEM is able to provide morphological evidence of CNTs in epoxy resin that helps to assess the dispersion of CNTs.

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2. Materials and methods

An industrial grade multiwalled carbon nanotubes (MWCNTs) produced by Timesnano, China with >90% purity, outer diameter of 10–30 nm and length of 10–30 μm were used in this experiment. The morphology of the product was inspected with Nova NanoSEM 240 (**Figure 1**). The Hysol EA956 A/B, an aviation grade epoxy system, obtained from Henkel Corporation Aerospace Group, USA. It is a two-component adhesive, Part A (epoxy resin) and Part B (hardener) with the viscosity of 35 Pa.S (Pascal-second @ kg/ms) and 2 Pa.S, respectively. The mix ratio is 100:58.

For the preparation of control samples (0% CNTs), neat resin specimen was prepared by mixing Part A epoxy resin and Part B hardener thoroughly. The slurry was then placed inside a vacuum container to remove the entrapped air for 20 min before transferring into a mould for further curing processes. For the CNT-based polymer composites, MWCNTs (0.1% and 0.3%) were dispersed into Part B via ultrasonication process by Qsonica Q700 for 30 min at 10% of power amplitude. After sonication, Part A epoxy resin was added to the CNT-Part B slurry and stirred for 10 min at 550 rpm using a mechanical stirrer. Air bubbles were removed by placing the mixture inside a vacuum container for 20 min.

Three samples were prepared for each sample types in according with ASTM 5045–99. After the samples cured and de-mould, 2 mm notch tip was generated mechanically using Ray-ran Motorspeed Notching Cutter. The tests were conducted in a 50 kN Instron 5569A servo-hydraulic testing machine with crosshead speed of 1.0 mm/min. A crack opening displacement (COD) gauge was fixed to the samples to measure the initial fracture displacement during the testing. The morphology of MWNTs in epoxy was observed under field emission

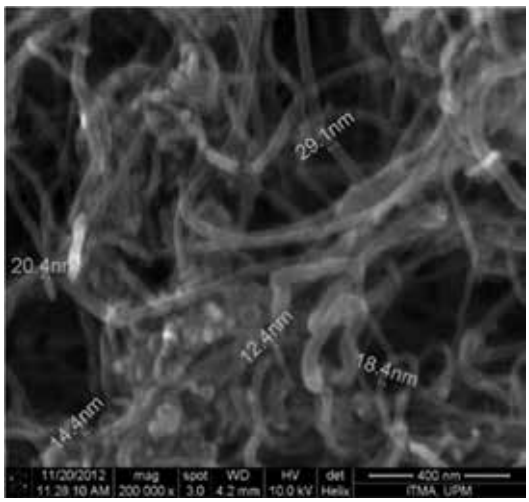

Figure 1. SEM picture of as received MWNTs.

scanning electron microscopy (FESEM) using Nova NanoSEM 240. After mechanical test, all the sample fracture surfaces were coated with a thin gold layer by sputtering process to make them conductive for SEM analysis.

3. Results and discussion

Table 1 and **Figure 2** show that by addition of MWNTs to epoxy system, the flexural strength of MWNT-based composites was found to exhibit non-linear behaviour with respect to MWNTs wt% inclusion. The optimal improvement was found at 0.1 wt% MWNTs, whereas a slight reduction was found at 0.3 wt% MWNTs. This trend was also observed for the flexural modulus but a significant reduction was observed at 0.3 wt% MWNTs content. The mode I fracture critical stress intensity factor (K_{IC}) of the specimen is listed in **Table 2**. As shown in **Figure 3**, the fracture toughness of MWNTs reinforced epoxy was improved by 62.7% at 0.1 wt% CNTs and 31.8% at 0.3 wt%. This result showed a different trend compared to the works reported by Zhou et al. [12] using Epon 862 epoxy with viscosity 4.5 Pa.S, Li et al. [15] using Epoxy 828 with viscosity 15 Pa.S and Gojny et al. [8] using L135i epoxy with viscosity 0.25 Pa.S (**Figure 3**).

CNT weight percentage (%)	Flexural Strength (Mpa)	Modulus (Mpa)
0.0	14.659	917.058
0.1	23.783	952.441
0.3	19.273	857.787

Table 1. Flexural strength and modulus properties of epoxy system.

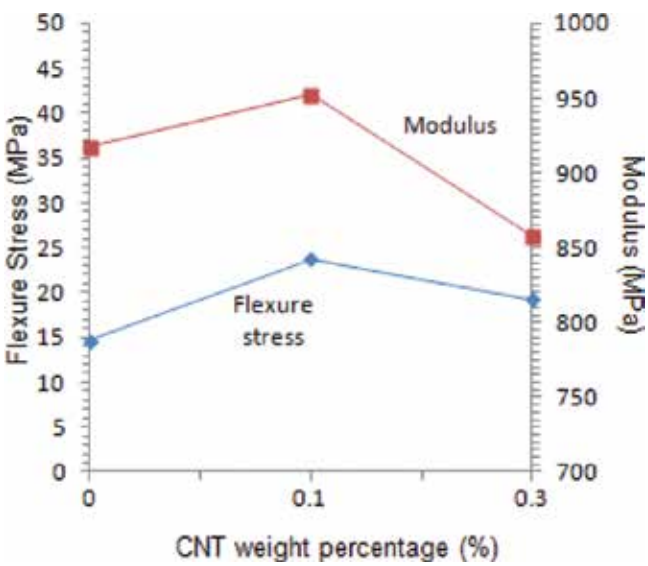

Figure 2. Effect of CNTs content on flexural strength and modulus.

Load, P (N)	Stress intensity factor, K_{IC} (Mpa.$m^{1/2}$)	Increase in K_{IC} (%)
175.915	1.10	0.00
285.392	1.79	62.7
231.268	1.45	31.8

Table 2. Fracture toughness of epoxy system.

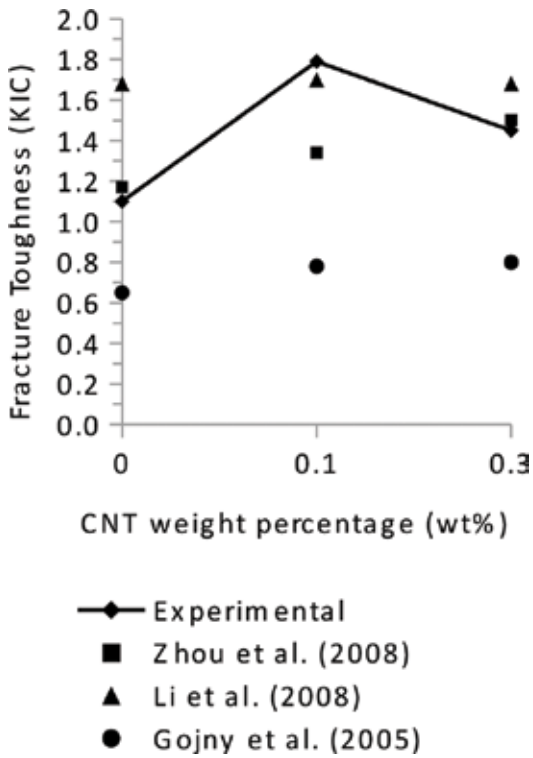

Figure 3. Effect of CNTs content on fracture toughness.

Figure 4 shows SEM images of the fracture surfaces of neat epoxy and MWNTs reinforced composites. **Figure 4a** shows a fractography feature of neat epoxy. The distance between two cleavage steps is about 2.64–7.84 μm, and the cleavage plane between them is flat and featureless. **Figures 4b** and **3c** show the fracture surface of composites with MWNTs inclusion. The fracture surfaces of the nanocomposites show a different fractographic features. The surface roughness increased with higher CNTs content. **Figure 4b** and **4c** also indicates that the size of the cleavage plane decreased with higher CNTs content. The SEM picture in **Figure 4c** shows that the size of the cleavage plane decreased to 0.77–2.74 μm after the infusion of the 0.3 wt% CNTs.

According to Zhou et al. [12] and Srivasta and Singh [13], the decrease of cleavage plane and the increase of surface roughness were correspond to the number of isolated and dispersed

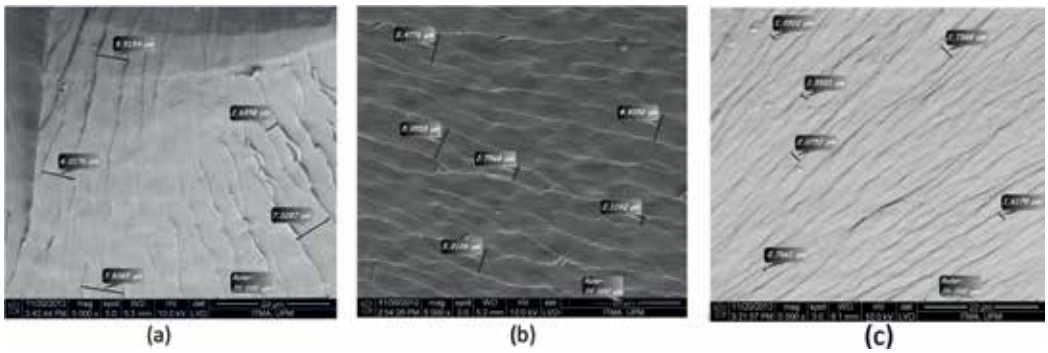

Figure 4. Cleavage plane at 5000X magnification of (a) neat epoxy, (b) epoxy with 0.1 wt% MWNTs, and (c) epoxy with 0.3 wt% MWNTs.

MWNTs, which force the crack to propagate, bypassing the MWNTs and distorted the crack tip. This phenomenon indicates that the good adhesion has taken place between MWNTs and epoxy resin and fractured in different planes. The cracks were spanned by the MWNTs causing enhanced resistance to the crack propagation process. Here, MWNTs have played a role for pinning crack and carry the external force. They have created a firm connection and good interaction with the matrix. Therefore, the MWNTs have prevented the expansion of microcracks resulted from the stress concentration and thus improved the toughness of the composites. Also, some deep cleavages are seen in both neat and modified epoxy surfaces but in modified epoxy, instead of deep cleavage, the crack tip is more bifurcate and also leads to a rough surface features. The bifurcation mechanism probably has caused a dissipation of more fracture energy throughout the failure process, promotes plastic deformation and leads to the improvement of toughness and ductility [16]. Here, MWNTs have played a role of crack path deflection.

In this experiment, the fracture toughness of epoxy with 0.3 wt% MWNTs content was lower than 0.1 wt% MWNTs. A possible explanation can be given for this behaviour is the not-well dispersion state in 0.3 wt% MWNTs. This condition was in agreement with the work reported by Gkikas et al. [14], where at constant processing of sonication power and time, the degree of dispersion of less CNT concentration (0.1 wt% MWNTs) is higher than the higher concentration (0.3 wt% MWNTs). Also, samples with lower CNT concentration exhibited larger modulus than samples with higher concentration. This is because at higher contents of CNTs inclusion, the amount of aggregated particles is higher; thus, the processing power and time should be increased to ensure a good state of dispersion within the matrix. Aggregated particles usually do not have the load bearing ability as individual particles and they may act as defects [14]. Therefore, the state of dispersion is crucial to the extent the strengthening effect of CNT particles as the concentration becomes larger. This means that to the extent of improvement at higher CNTs content, the processing parameters play a key important role to improve the dispersion state.

The presence of a partly agglomerated dispersion of MWNTs that found in epoxy with 0.3 wt% MWNTs can be seen in **Figure 5**. The agglomeration in turn leads to the localized

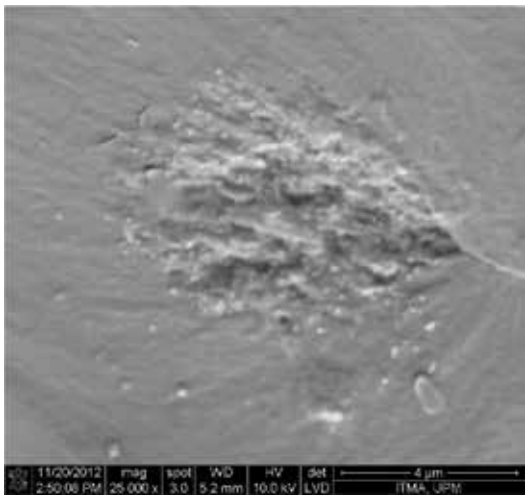

Figure 5. Agglomeration at 0.3 wt% MWNTs-epoxy.

inelastic matrix deformation and void nucleation. The localized deformation converted the elastic strain energy to surface energy [8]. When the epoxy resin is subjected to mode I loading, shear stress is generated between the CNTs wall and the surrounding matrix resin due to the difference in their elastic properties [17]. If they are well bonded, CNTs are deformed together with the matrix. However, if the mode I loading exceeded to some critical value that leads to debonding of CNTs from the matrix, the CNTs will stop elongating together with the matrix and a further increase of the load applied can only result in the deformation of the matrix. Thus, the polymer starts to flow over the surface of CNTs, and deformation energy will be dissipated through the slippage between the CNTs and matrix [17]. Other related explanation by Greef et al. [18] is that CNTs have a high stiffness in comparison with the epoxy, and their presence creates an abrupt stiffness variation in the matrix. Therefore, this stiffness mismatch introduces local disturbances in the stress distribution around the CNTs and establishing a weak link to the material and increases the number of potentials for the material to have such a weak link. Thus, multiple "weak" sites are identified, and they compete with each other for becoming a crack path. Some of these sites fail earlier which made the sample fail quickly as occurred in 0.3 wt% MWNTs content. But, if they are not part of a larger chain, they do not grow into larger cracks and remain isolated (**Figure 6**).

However, in the authors' opinion, this experiment has not yet been fully answered all the possible toughening mechanism occurred in the polymer matrix. There are further mechanisms participate in the toughening process which can be seen through other method than FESEM. Based on those uncertainties enhancement and unpredicted void existence, it is believed that there are such mechanisms lead to stress relation in the materials and decrease the stress intensity at the crack front which consumes energy rather than increasing the total fracture surface area which cannot be seen through FESEM (Wetzel [19]). As a whole, former researchers proposed that energy absorption by crack bridging and fibre pull-out of CNTs

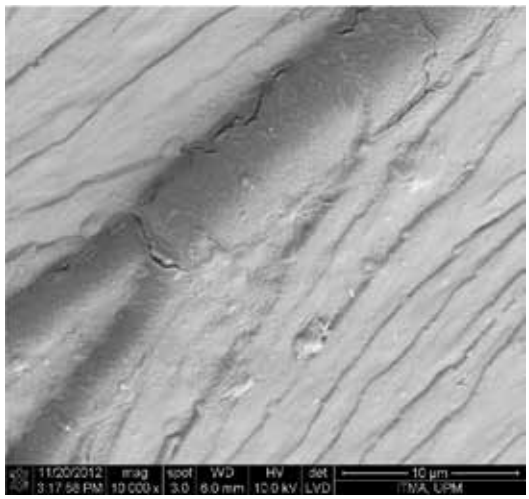

Figure 6. Isolated microcracks found at 0.3 wt% MWNTs.

was responsible for the major increase in fracture toughness. But according to this research work, we found that crack pinning, crack path deflection, localized inelastic plastic deformation and void nucleation mechanisms were the most attribute toughening mechanism in CNTs' modified epoxy.

4. Conclusions

CNTs are qualified for the toughening of the higher viscosity epoxy resin for aircraft application because they are able to enhance damage tolerance of the composite structure. The key important factor for significant enhancement is achieving the optimum state of dispersion and interfacial adhesion by manipulating and controlling the processing method. Well-dispersed MWNTs can easily alleviate the stress concentration of the matrix and eliminate the adverse effect of voids. In this study, the flexural strength and the fracture toughness at the break were clearly improved with CNTs additives. The increase of performance is because CNTs resist the matrix deformation through several toughening mechanisms. No crack bridging mechanism by MWNTs is observed in present study, and no obvious CNTs are detected on the fracture surface during microscopic analysis. However, a more thorough study on the evaluation of surfaces is necessary, and the toughening mechanisms should be further investigated in future works.

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Section 2

Bio-based Composites and Recycled Products

Chapter 6

Waste and Recycled Textiles as Reinforcements of Building Materials

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Abstract

Currently, the use of composite materials in the construction areas has had a great impact on the society; mainly, those related with sustainability and environment aspects. Daily proposals aimed at overcoming the properties of traditional materials that arise, which include emergent materials either from waste or recycled products. One of them is related to the textile materials, which include fibers such as wool, hemp, linen, and cotton. In the past decade, special attention has been focused on the used clothes, which represent a source of raw materials environmentally responsible and economically profitable. Textile materials are discarded daily around the world, representing approximately 1.5% of the generated waste. Blue jeans are the most used clothing in the world, and they are elaborated by one of the most commonly used natural textile fibers—cotton. Textile materials have been reused in different applications, for example, in the production of poor-quality wires, crushed to manufacture noise and temperature insulation materials, and as fillers or reinforcements of concrete. In this chapter, different topics are described that include: (a) environmental impact of textile waste—a result of massive consumption of clothing, (b) recycling and reuse of textile waste, and (c) waste and recycled textile materials used as building materials.

Keywords: recycling, waste, textiles, cotton, cellulose, composites, polymer concrete, gamma irradiation, mechanical properties

1. Introduction

As a consequence of the technology boom and global population growth, the environment is being seriously damaged by different types of waste. Large amounts of wastewater, polluting gases, and solid waste are being disposed of worldwide, which have degraded the ecosystem

to an alarming extent. Concern about the environmental deterioration is leading science to design strategies to remedy the damages caused to some extent, which seek to generate in the population an ecological awareness, aimed at the reduction of waste, recycling of materials that can be reprocessed, and the reuse of objects or materials before proceeding to final disposal.

The use of waste materials or recycled materials for the creation of raw materials for the construction area is a topical issue, with a promising future and primarily aimed at environmental preservation. Concrete, wood, and steel have been the most used materials in the infrastructure of houses and buildings, but the high production costs of these are leading science to develop research aimed at developing composite materials that recycled materials, such as PET, polycarbonate, recycled tires, wood, textile fibers, etc. The use of plastics in the form of small particles to reinforce hydraulic concrete has been applied in several investigations, but the problem lies in the poor interface between the matrix and the reinforcement; which causes diminution of mechanical properties of the concrete, such as flexural and compressive strength.

The use of waste PET fibers, from bottles, as reinforcement in concrete has allowed improvement in mechanical properties. Other reinforcement materials in the processing of concrete are waste tyre particles, which contribute to diminution of the crack propagation effect and improvement of resistance to deformation of the concrete.

Some investigations are focused on the use of natural materials as reinforcements in Portland cement concrete. In special, natural fibers, such as jute, flax, coconut fiber, henequen, and cotton, as a reinforcement in building materials, which have great interest for their advantages when compared with synthetic materials, and one of their biggest benefits are the low environmental impact, low cost, and wide range of applications. Cellulose fibers mixed into the concrete improve the thermal and acoustic insulation.

In this regard, during the last decade, the construction industry is being innovated with a new material known as textile reinforced concrete (TRC): a combination of fine-grained concrete and multiaxially oriented textiles whose structural functionality, ease of production, applicability, and design are investigated [1], as a way to take advantage of the high amount of waste from the industry textile.

2. Environmental impact of textile wastes

At present, there is a growing need for textiles to make yarns, fabrics, and garments of different types, shapes, and colors. Demand increases with population and trends in international fashion being the reason why the rational exploitation of the necessary inputs for this purpose has become one of the main challenges for scientists and industrialists. Wastes from the production of textiles are undesirable but are inevitable byproducts in many manufacturing processes, which are often not given the recognition and economic value that they actually have [2].

In developed countries, each person produces on average 1.5–2 kg of solid waste per day and in Latin America 1 kg per day [3]. The urban waste percentages in Mexico are shown in **Figure 1**, in which textile wastes represent 1.2%, and this means 1100 tons are produced daily.

Actually, garments are discarded more quickly; which causes the industry to streamline its production and generate more textile waste. The production of textile fibers, both natural and synthetic, has been showing sustained growth, as a result of the increase in demand and population growth. The quantities of textile fibers used worldwide, between 1980 and 2000 are shown in **Figure 2**.

The different environmental aspects caused by the production, use, and disposal of garments are shown in **Table 1** [6]. In the textile production of garments, different impacts on health, land, water, and air are detected.

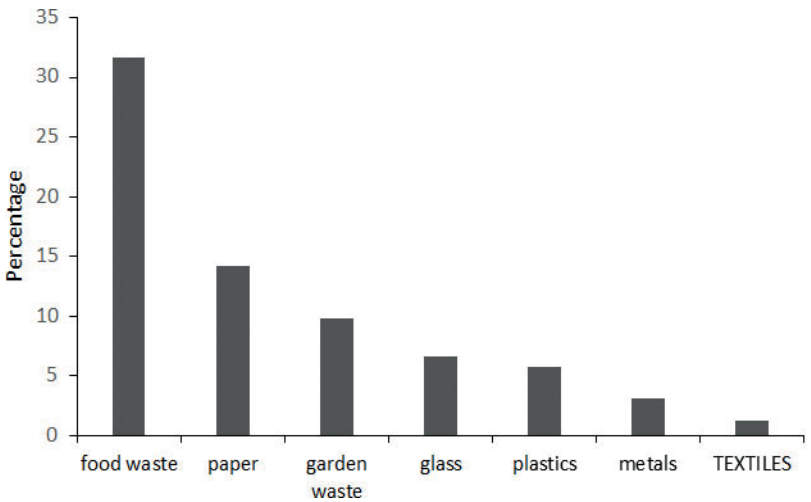

Figure 1. Solid urban waste in Mexico [4].

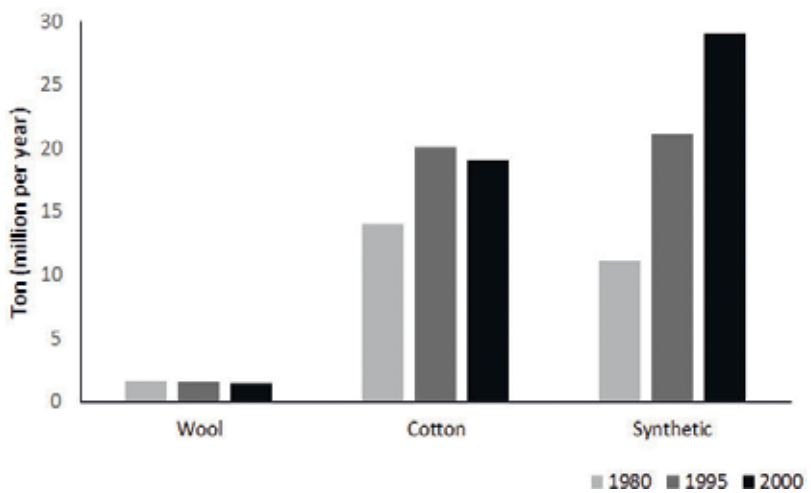

Figure 2. Worldwide production of textile fibers [5].

Fabric and cloth production		Impact		
Activity	**Environmental aspects**	**Health**	**Water**	**Air**
Ironing	Consumption of electricity and occupational diseases	X	X	X
Clothing sewing	Consumption of human energy, and textile and metal waste	X		
Cloth cut	Noise generation	X		X
Bleaching of tissues	Generation of liquid waste		X	
Cloth dyeing	Generation of liquid waste		X	
Cloth wash (Denim)	Water consumption		X	

Table 1. Environmental impacts generated by textiles (adapted from [6]).

Water contamination carried out through the dyeing of fabrics is produced by using synthetic dyes, which has become a common practice. Such toxic nature is a cause of concern for environmentalists [7]. It has a high content of sulfur, nitrates, acetic acid, soaps, enzymes, chromium compounds, and heavy metals such as copper, arsenic, lead, cadmium, mercury, nickel, and cobalt. Also used dyes are fixing agents that are based on formaldehyde, chlorinated stain removers, hydrocarbon softeners, and nonbiodegradable chemicals, which react with many disinfectants, especially chlorine, and form carcinogenic products.

In the research work carried out [8] was evaluated physicochemical parameters in waters coming from a textile industry, as well as the soil where they are arranged. The parameters such as pH, electrical conductivity, temperature, dissolved oxygen (DO), chemical oxygen demand (COD), biological oxygen demand (BOD), and total dissolved solids (TDS), as well as N, P, and K in soils were determined. The salinity and alkalinity of the soils changed; moreover, the food crops that were in these lands reported a very low yield and negative hygienic conditions.

The cotton cultivation requires large amounts of water. The amount of cotton fibers used for textiles is only one-third of the total production of raw cotton and the remainder consists of cotton seed (which are used for the extraction of oil) and weed which is used as food for livestock. The part of the fibers that does not have the necessary quality to enter into textile production processes is used for viscose raw material and cleaning cloths [9].

Requirements for the production of garments made from virgin materials were quantified [10]. They showed that for every kilogram of virgin cotton that is replaced by second-hand clothing, electric energy is saved. The reuse and recycling of used clothing reduces the environmental burden compared with the purchase of clothing obtained from virgin fibers. Rojas et al. [11] indicated the importance of minimizing waste produced in the textile industry.

The textile industry is one of the four most representative and polluting items in Latin America, because its processing employ a great amount of chemical compounds, water, and energy [11]. The indexes of pollutants from the cotton industry include yarns and cotton cloth (**Table 2**).

Types of pollution	Index
Air pollution	
Production of particles during the process	14 kg/ton cotton
Production of particles by incineration of waste	7.5 kg/ton residue
Production of SO_2 by incineration of waste	1.25 kg/ton residue
Production of nitrogen oxides by incineration of waste	1.0 kg/ton residue
Hydrocarbon production by incineration of waste	7.5 kg/ton residue
Water contamination	
Volume of wastewater	317 m^3/ton product
DBO5	155 kg/ton product
Suspended solids	70 kg/ton product
Total dissolved solids	205 kg/ton product
Solid waste	
Preparation of fibers and cotton thread	32 kg/ton product
Fiber fabric, thread, cloth	11 kg/ton product
Dyeing and finishing fabrics	7 kg/ton product
Dried fibers trapped in grids	0.8 kg/ton product
Natural fibers trapped in grids	2.8 kg/ton product
Residual sludge (not dehydrated or treated)	2300 kg/ton product

Table 2. Index of contamination for the cotton industry (Adapted from [11]).

The fact is undeniable that almost all human activities have an environmental impact; the textile industry is no exception, but as in many other productive processes, the population is increasingly acquiring an ecological awareness for recycling, reusing, and reducing (RRR).

At present, the consumption trend is due to the use of natural textile fibers, this as a response to the conservation of the environment, and that fashion is increasingly pronounced by comfortable, light, and friendly with the skin.

World cotton production in 2014 was 25.8 million tons and global production is expected to grow by 2.1% per year over the next 10 years. An important part of cotton produced worldwide is part of the textile fibers used for the production of Denim garments, which was created in 1873 by Jacob Davis, a friend of Levi Strauss, for the hard work of the mining time during the so-called gold rush in the USA. In the case of Mexico, 9000 million tons of Denim were produced in 2010. Jeans are the best-selling item in Mexico, representing 30% of the entire clothing market.

3. Recycling and reuse of textile waste

Textile waste is part of the solid waste that is generated by the population and also contains used clothing, footwear, curtains, sheets, etc. The theme of textile recycling should be taken as

a means to obtain economic and environmental benefits for various reasons, as it contributes to the reduction of spaces required for landfills, reduces the need to produce virgin materials and reduces problems of water pollution. The textile recycling involves the reuse of used clothing, fibrous materials, and production losses of the manufacturing process of clothing.

Some products made from synthetic fibers do not decompose naturally, which causes soil and surface water contamination problems. Although cotton fibers decompose naturally, in doing so they produce methane, which contributes to the global warming.

Textile recycling strategies are based on the knowledge of their classification: (a) post-production materials, such as yarns, textiles, fibers, which come from the loss of the production process and (b) post-consumption materials, such as clothing, carpets, shoes, and furniture, which are discarded once its useful life expires.

The traditional recycling of textiles is based on introducing them back to a productive process that contemplates: (a) the classification is made according to the conditions and type of fiber; at this stage, it is decided whether garments can be reused, crushed, or used for reprocessing, and (b) re-classification, where the granulated material is separated according to its color, to be later crushed, carded, and finally spun.

The idea of using textile waste in applications that do not involve a new industrial process results in a novel research panorama, due to the economic and environmental benefits that can be achieved, as a novel investigation about home-use products obtained from waste textile materials [12]. **Table 3** shows the proposals and results of the recycling.

The results showed that eco-design is an effective tool, involving creativity and innovation, with environmental objectives. Recycling textiles are an excellent opportunity to reduce the environmental impact of commonly used products.

The use of textile waste to obtain ethanol was studied. Two types of textiles were used, one containing viscose, polyester, cotton, and modal and the other containing the cellulose contents that were between 70 and 94%. The textiles were cut from 2 to 5 mm in length and subjected to an alkaline pretreatment based on sodium hydroxide, and subsequently to an enzymatic hydrolysis process. The yields of ethanol obtained at 72 h were between 15 and 36% [13].

In a study carried out by Liang and Hota concerning to fiber-reinforced polymers and their use in engineering, they show a wide range of possibilities for their use within construction industry; due their characteristics, as: lower cost for maintenance (no paint, no decay, no insect infestation), lower heating and cooling costs and high structural strength [14].

Other researches focused on the use of recycled textile fibers in composites, as reinforcing materials [15], i.e., used carpet waste as a soil reinforcement material. The reinforcing fibrous strips were 5 × 5 mm and lengths of 5–45 mm. They were added to the soil in concentrations from 0.4 to 1.2%. The mechanical properties of shear forces were improved, is to say, composites without fibers have a value of 300 kPa, which was improved 25% when adding 1.2% of fibers (375 MPa). Moreover, the axial strain at yield point was improved, covering from 1% for composites without fibers to 4% for those with 1.2% of fibers.

Design alternatives matter virgin	Proposals reuse matter
1. Agglomerates	The fibers of fabrics that are considered as debris are separated by rippers, and the threads are joined together and under pressure and heat in the form of vapor to create textiles as felt.
2. Art objects	Textile waste considered to be waste is subjected to different procedures such as ripping or cut, to create artistic works such as paintings, sculptures, plastic art, etc.
3. Promotional objects	Fabric dolls consisting of materials of waste that promote institutions or campaigns around the type: felt bags made of textile waste that they bring logos, labels promoting congresses, or associations
4. Carpets made with T-shirts	Carpets made of T-shirts, second-hand clothing or scrap, which are cut into strips and woven by hand-form carpets for homes and offices
5. Bags made with pants	Wearing denim jeans that are no longer donated like second-hand clothes, their legs are cut off, sew holes, and debris are used to make the handle
6. Insulation	The same procedure is followed to make felt of but in thicker layers and then subjected to a cut for its adaptation in walls, in cars like acoustic and thermal insulation
7. Cleaning cloths	Used clothing, considered scrap made from cotton mainly, is cut or torn to be used like rags or tows for cleaning. Some fabric synthetics by their oleophilic and hydrophobic characteristics can be used for industrial cleaning
8. Bedspreads or bedding patchwork	Large textile fabric is selected for be subjected to cuts with patterns and form that will be joined with the help of a sewing machine to construct canvases based on pieces.

Table 3. Proposals for replacement of virgin materials by waste textiles (adapted from [13]).

Improvement on the reinforcement-matrix interface is one of the most important challenges for using textile fibers as reinforcements in composite materials. Some studies are concerning to the modification of reinforcing fibers, by means of chemical or physical techniques, that look for changes in the chemical structure and in consequence to improve the interface with the matrix.

There are several physical methods applied on fibers, as temperature plasma treatment, corona treatment, and a novel based on the use of gamma radiation. Physical methods of interface modification involve fibrillation on the fibers, causing structural and surface modification, and in consequence mechanical changes are obtained.

In an investigation about the effects of chemical treatment (with alkali) and gamma radiation on cotton fibers, structural modification of cellulose component was evidenced by a decrease in the degree of polymerization and an increase in carbonyl content [16]. In an other investigation, irradiated cellulose fibers (by using gamma rays) showed degradation from 6 to 12% up to an applied dose of 31.6 kGy; moreover, crystallinity decrease for dosages higher than 300 kGy and finally diminution of 1% in the crystallinity at dosages up to 1 MGy are observed. In general, gamma radiation causes degradation of cellulose in shorter chains and leads to "opening of microcracks" that are easily penetrable by water molecules. The complete degradation of cellulose is given up to 6.55 MGy, where amorphous zones along the length of the microfibrils are seen, which allows penetration of chemical substances in the microfibrils [17].

Industrial development and the need to protect the environment are placing escalating demands on the natural resources and improved technologies. Radiation technology has been used to produce high performance polymeric materials with unique physical and chemical properties. Now, this technology can successfully be utilized to upgrade natural polymers as well, yielding value-added products for diverse applications.

Cellulose is the most abundant natural polymer around the world, and its high abundance and the presence of reactive hydroxyl groups in the chain promise an array of potential applications. The modified celluloses like carboxymethyl cellulose (CMC) or hydroxyethyl cellulose (HEC) are more useful in producing specialized polymers due to their solubility in water. CMC is the most promising derivative of cellulose that can be used for the production of hydrogels, since it has an ionizable carboxyl group.

The natural polymers are presently being exploited for industrial use. However, by judicious use of radiation, these polymers can yield high value products with interesting applications. Possibility of blends of polymers with natural polymers opens up a new avenue for novel applications.

4. Waste and recycled textile materials used in building materials

Fibers recovered from various waste streams are suitable for concrete reinforcement. The advantages of using such recycled fibers generally include lower cost to process than virgin fibers and the elimination of the need for waste disposal in landfills.

Recent studies about composites with textile fibers as reinforcement materials have opened great chances of success. For example, textile cutting waste has been mixed with epoxy resin and foundry sand for producing a unique composite material that can be used for lightweight construction. In general, textile fibers do not increase flexural and compressive strength of polymer concrete, but their addition to the mixture eliminates the signs of brittleness behavior. The use of textile fibers, in specific applications, may solve two problems, namely, elimination of an environmental pollutant and provision of an alternative material for the construction industry.

A significant amount of fibrous waste from the textile industry and postconsumer product is disposed worldwide. This is not only a cause for environmental concern but also represents a waste of useful resources. The textile cuttings are usually disposed of as a waste product that become an environmental nuisance because of its nonbiodegradability or burned in heaps thus releasing highly toxic fumes in the surrounding air. Turning them into useful materials serves a dual function: elimination of wastes and introduction of a new product.

Polymer concrete is elaborated by combining polymers and minerals. The most important parameters in its elaboration include type and size of the minerals, as well as percentages of the components, in order to obtain improved properties. In general, polymer composite materials are brittle in nature but show an increase in both ductility and strength when adding fibers. Nevertheless, fibers have not been widely used in polymer concretes. Moreover, interface between fibers

and polymer matrix influences the strength and toughness of composite materials. When an interfacial failure happens, fibers are pulled out from the polymer matrix and bridging forces are developed on the crack surface. The bridging forces shield the crack and hence reduce the stress intensity factor at the crack tip. Interfacial shear strength plays a dominant role since the bridging pressure from fiber pull-out is governed mainly by shear stress resistance between fibers and the polymer matrix.

Mechanical behavior of polymer concrete with textile residues was investigated. Textile wastes were recovered from lingeries, which are elaborated with cotton, polyester, silk, and rayon fibers. Two sets of resin, fine aggregate and textile fibers were elaborated, and 1 and 2 wt.% of recycled textile fibers were added. The results show diminution on the flexural strength of polymer concrete when increasing the fiber concentration. However, flexural strength values are higher than those obtained for cement concrete. Polymer concrete without fibers failed and was broken into pieces; in contrast, all fiber-reinforced specimens after reaching the maximum load remained as an integral piece, with hold fibers to polymer matrix [18].

Polymer concrete with olive oil from vegetable solid wastes was elaborated; for its use in the construction industry or in the manufacture of furniture. Polymer concrete specimens had from 10 to 60 wt.% of residues, and a silanized process was used to improve the interface characteristics. Silanized was made with untreated alpha-mercaptopropyltrimethoxysilane. The results show that olive oil improves the mechanical properties, and even more silanized process allows improvements on interface and in consequence higher mechanical values [19].

In the search to include textile waste materials in construction materials, an investigation was carried out in which we evaluated the feasibility of using residuals and subresidues of fabrics, for thermal insulation in the construction industry [20]. This was done by making an outer double wall with an air box filled with such debris by placing two heat flow meters and four surface temperature sensors on the wall surface to determine the thermal conductivity of the waste. The results show that the application of fabrics in the external double wall increases its thermal behavior between 30 and 56%.

In the construction industry, one of the most demanding items is the construction of floors for roads and highways, which must be able to withstand the constant impact of the traffic of people and transports. In an investigation, we used recycled carpet waste fibers to make composites of lightweight cements in a 20% fiber ratio. The flexural, tenacity, and impact properties were characterized. The results show that in the three-point flexural test, a ductile behavior and an increase in flexural strength were observed. Nevertheless, density decreases with increasing fiber concentration. Energy absorption was also measured by the weight drop impact test, but this was not very significant due to the total absorption of the impact energy of the specimens [21].

The importance of the concentration of the load of reinforcing material in a composite is evidenced in an investigation in which it developed a polymer concrete elaborated with polyester resin, sand, and textile glass fibers (1, 2, and 3 wt.%). The properties of fibers were weight (160 g/m^2), thickness (0.47 mm), mesh (3.5 $\times$ 3.5 mm), and tensile strength (1200 N/cm^2). Load-displacement curves for different reinforcement contents were obtained. The results show that

after to reach the maximum load this suddenly decreases the matrix cracking completely. Two important factors affect the fracture toughness, fiber pull-out and the bridging effect [22].

In the next section, we show a study concerning to polymer concrete elaborated with polyester resin, marble particles, and waste cotton fibers carried out by our research team. The cotton fibers were obtained from waste blue jeans (Denim).

5. Polymer concrete: experimental results

5.1. Polymer concrete without textile fibers

In a first stage, test specimens were made with different concentrations of polyester resin and marble to determine those with better compressive and flexural strength. The compressive strength results are shown in **Figure 3**, as it can be seen, the values increase when the resin concentration increases too (or marble particles diminish).

In **Figure 4**, it is observed a similar behavior as compressive strength, is to say, flexural strength values increase when increasing resin concentration. Thus, it was decided to work with the specimens with 30% of polyester resin and 70% of marble particles. Results that are in agreement with other investigations where using marble residues as an aggregate in concrete, in order to improve the durability [23].

5.2. Polymer concrete with waste textile fibers

In a second stage, textiles were cut in pieces of approximately 1.0 × 1.0 cm, and then they were crushed in a windmill. The obtained fibers were added to the blend of polyester resin and

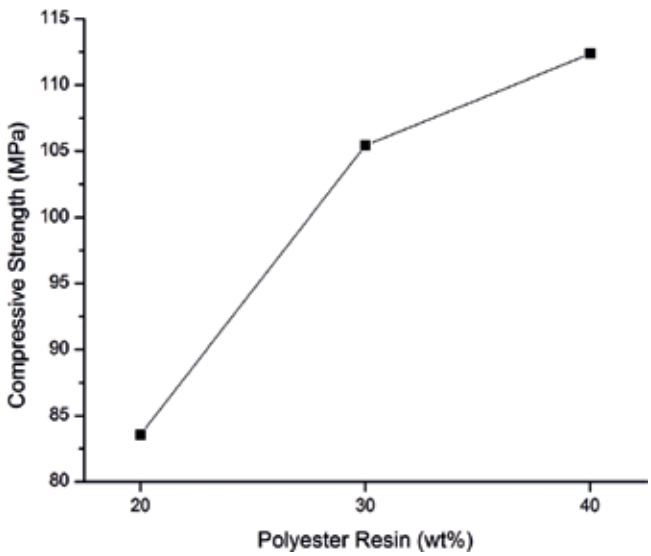

Figure 3. Compressive strength of polymer concrete.

marble particles (without applying them any treatment). The concentrations of cotton fibers were 0.5, 1.0, and 1.4% by weight. The specimens were obtained through the casting method according to the European norm EN-196-1, with dimensions of 4 × 4 × 16 cm. The results are shown in **Figure 5**.

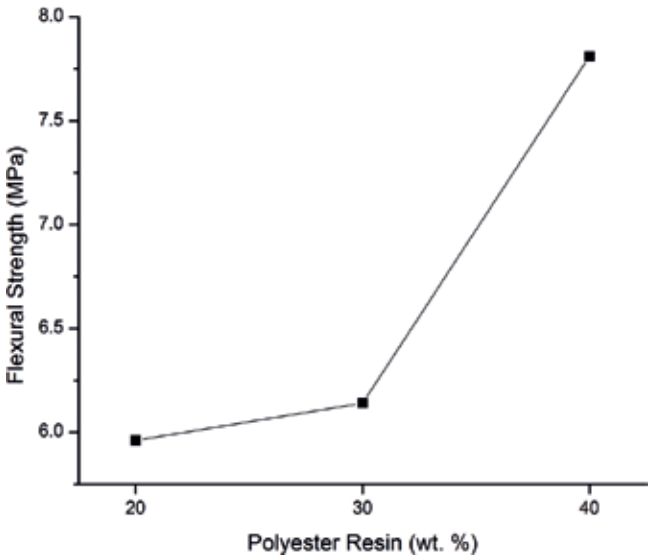

Figure 4. Flexural strength of polymer concrete.

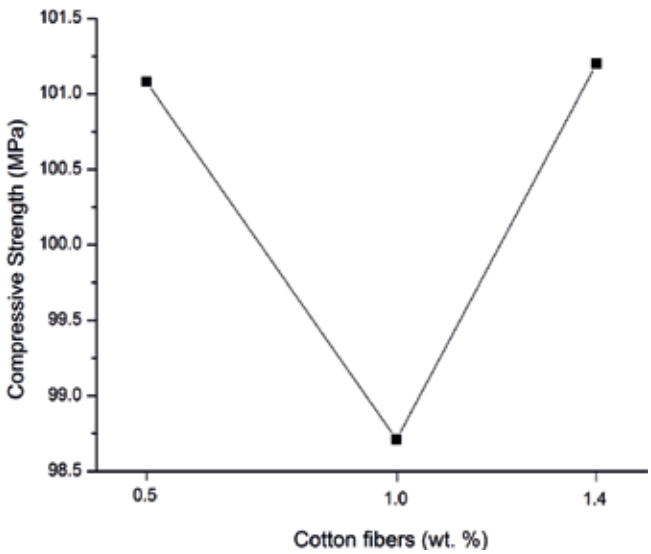

Figure 5. Compressive strength of polymer concrete with recycled cotton fibers.

Compressive strength values show a peculiar behavior; since at a lower concentration of cotton fiber (0.4%), a value of 101 MPa is obtained; however, for a higher fiber concentration (1.0%), the value decreases up to 98.7 MPa, and finally they increase again up to 101 MPa for 1.4% of cotton fibers; such behaviors can be due to the inherent properties of the fibers, i.e., whether they agglomerate or interact with the polymer matrix.

The results agree with early investigations, concerning to mix recycle fibers with the concrete in order to improve their mechanical properties and concomitantly reduce wastes, while minimizing the cost of the raw material. Environmentally, the benefit is also considerable, since natural resources are conserved and the presence of these residues in landfills is reduced [24].

Such compressive strength behaviors of the composites depend on the cotton fiber concentrations, as we know at 1.0 wt.%, lowest values were found. Nevertheless, other important parameter is fiber sizes; cotton fibers were analyzed by using scanning electron microscopy (SEM), as shown in **Figure 6**, where fibers have 10 μm diameter in average and lengths of some millimeters. Then, the mechanical performance of composites depends on the combination of concentration and size of the fibers.

Based on the previous results for composites with different concentrations of cotton fibers, it was determined the proportion ratio of 30% polyester resin/69% marble/1.0% cotton fiber, for the next stage.

5.3. Polymer concrete irradiated with gamma rays

According to the concentrations, composite specimens were elaborated and after they were submitted to different radiation dosages from 100 to 1000 kGy. After irradiating mechanical tests (including compressive and flexural strength) were carried out. The compressive strength results are shown in **Figure 7**; it can be seen that compressive strength results are almost constant, ranging between 120 and 140 MPa, even at highest dose of radiation. The lowest value is obtained at 200 kGy.

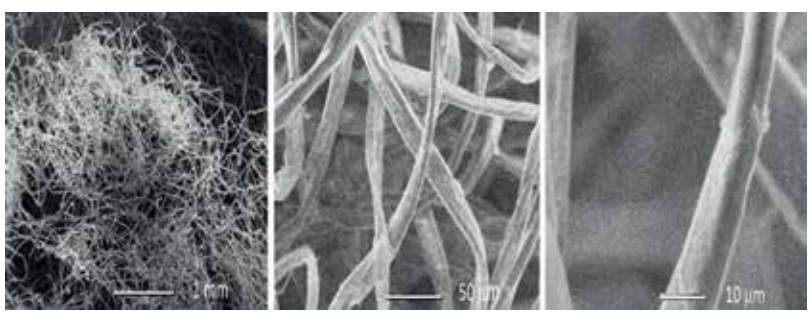

Figure 6. SEM images of cotton fibers, at different amplifications.

Structural modifications of gamma-irradiated cotton fibers were analyzed by using scanning electron microscopy, as it is shown in **Figure 8**. Gamma irradiation causes modifications on the composite as a whole and in each component (polyester resin, marble particles, and cotton fibers). Diminution of the compressive strength at 200 kGy is due in part to surface modifications of the fibers, as it is observed in **Figure 8**. For higher doses, at 600 kGy, fiber surfaces are rougher and the presence of detached particles on them is observed, such characteristics give to composite higher compressive strength values, which holds for the highest dose, at 1000 kGy, where cotton fibers show more deterioration.

In contrast, the flexural strength increases to 200 kGy (7.76 MPa), which can be explained by two causes: an improvement in the interface due to the superficial alterations caused by the

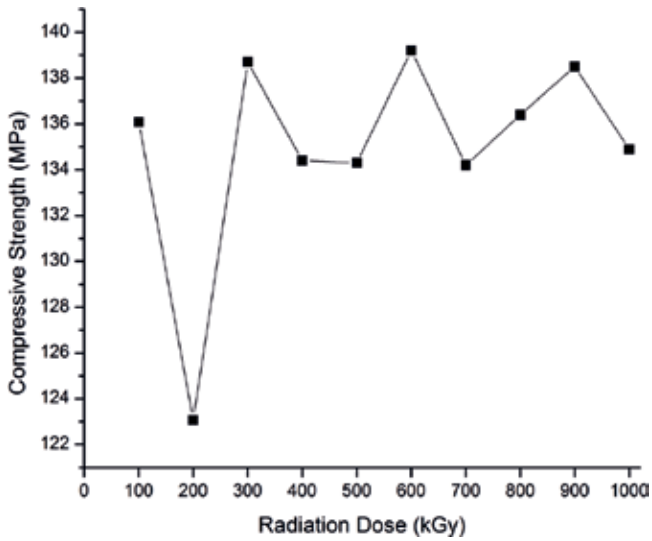

Figure 7. Compressive strength of irradiated polymer concrete at different doses.

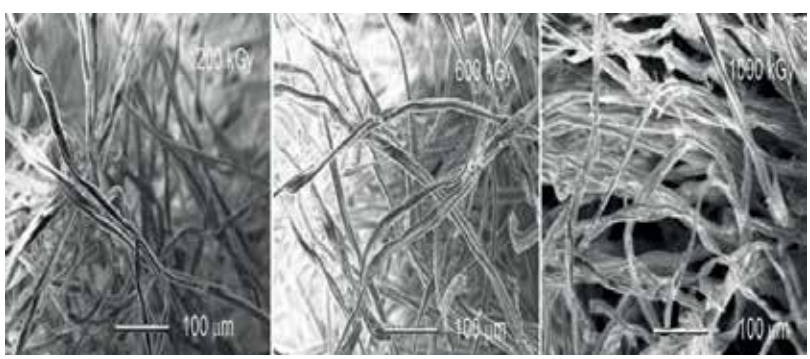

Figure 8. SEM images of irradiated cotton fibers.

irradiation in the cotton fibers, and on the other hand, as a consequence of the way the textile fibers are distributed within the polymeric concrete. At values greater than 200 kGy, the changes were minimal (**Figure 9**).

The behavior of flexural strength and the compressive strength, as we say before, the structural modifications are caused by gamma irradiation, which are observed by scanning electron microscopy (SEM) (as shown in **Figure 10**). In the images, smooth surfaces of nonirradiated fibers can be clearly seen, but when applying 400 kGy, the fibers exhibit rough surfaces and some detachment of particles, such characteristics allow lower flexural strength values. For higher irradiation dose, at 700 kGy, appearance of cracks is observed and, in consequence, diminution of the flexural strength.

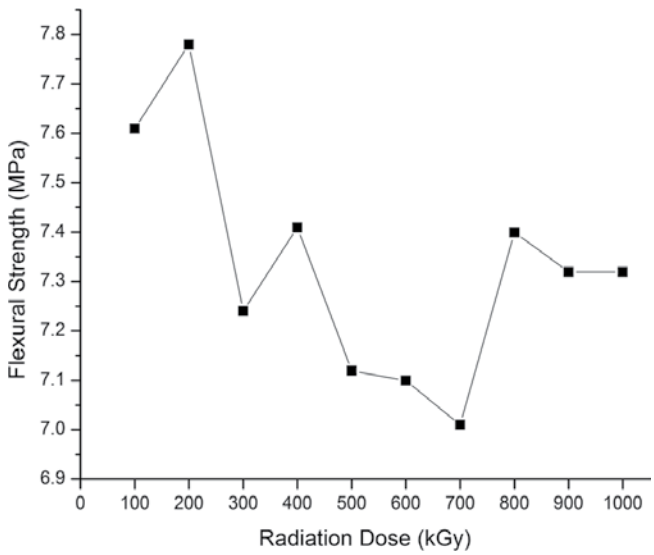

Figure 9. Flexural strength of irradiated polymer concrete at different doses.

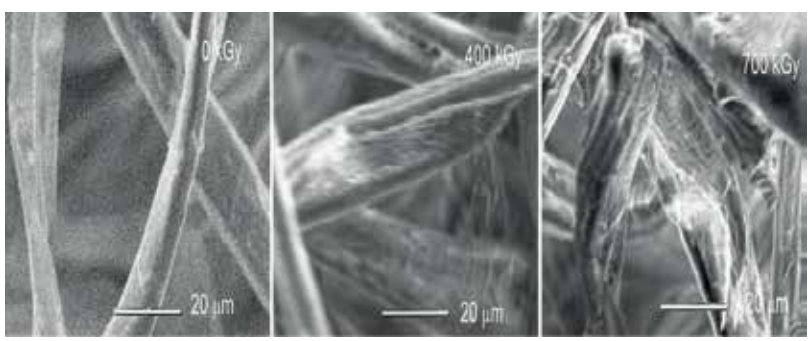

Figure 10. SEM images of irradiated cotton fibers.

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Chapter 7

Natural Fibers for Sustainable Bio-Composites

Tri-Dung Ngo

Additional information is available at the end of the chapter

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Abstract

Over the past decade, the concept of utilizing green materials has become more mainstream. With considerable awareness of preserving the environment, sincere efforts across the globe can be cited in looking for bio-degradable and bio-based sources. Applications of bio-based materials from renewable and bio-degradable sources for preparation of higher valued green chemicals and bio-based products have forced many scientists to investigate the potential use of natural fibers as reinforcement materials for green bio-composites. Cellulosic fibers are becoming very interesting for bio-based material development as they possess advantages with their mechanical properties, low density, environmental benefits, renewability, and economic feasibility. Recently, natural-fiber polymer composites have received much attention for different industrial applications because of their low density and renewability. The bio-composites with natural fiber components are derivatives of depleting resources and can be considered to have substantial environmental and economic benefits. This chapter addresses the potential utilization of natural fiber for the development of green polymer composite materials, with the objective to elucidate the possibility of using these bio-based materials for various industrial applications.

Keywords: natural fiber composite, cellulosic fiber, green-product, bio-composite, thermoset, thermoplastic

1. Introduction

The advantages of composite materials over conventional materials stem largely from their higher specific strength, stiffness, and fatigue characteristics. Over the past few decades, one finds that polymer composites have replaced many of the conventional metals/materials in various applications. The composite materials are finding applications in diverse fields ranging

from household, office appliances, power tools, and business equipment to space crafts. A fiber-reinforced composite is a composite building material that consists of three components: (i) the matrix as the continuous phase, (ii) the fibers as the discontinuous or dispersed phase, and (iii) the fine interphase region, also known as the interface [1, 2]. Many different fibers have been utilized to reinforce polymer matrix composites. The most common are carbon fibers (AS4, IM7, etc.), glass fiber (E-glass, S-glass, etc.), aramid fibers (Kevlar® and Twaron®), and boron fibers. Recently, with advantages of reasonable mechanical properties, low density, environmental benefits, renewability, and economic feasibility, natural fibers have been paid more attention in composite applications. Natural fibers are one such proficient material which would be utilized to replace the synthetic materials and their related products for the applications requiring less weight and energy conservation.

Historical textiles are made from natural fibers and serve to create a special link between the natural environment and the social environment that underlies all our lives, from the everyday textile to patriotic to ceremonial [3]. Natural plant fiber composites as environmentally attractive materials have been proven and emerged as an alternative to the glass-reinforced composites used in many applications since the 1990s [4]. In recent years, there has been an increasing environmental consciousness and awareness of the need for sustainable development, which has raised interest in using natural fibers as reinforcements in polymer composites to replace synthetic fibers [5, 6]. In addition, weight saving opportunities could possibly be obtained by replacing traditional fiber composites with natural fiber composites. The use of low-density renewable natural contents in thermoset and thermoplastic composite materials is a viable means to reduce environmental impact and support sustainability development in the transport industry. The utilization of natural fiber composites has expanded considerably in the shopper merchandise for developing industry sectors throughout the last few years. High specific properties, renewability with lower prices, and natural fiber polymer composites have received much attention for development of different industrial applications. Different kinds of natural fibers mainly flax, hemp, and sisal with bio-resins systems have been actively developing for various interior components in automotive and aircraft industries [7, 8]. This chapter seeks to provide an overview of the science and technology in relation to the potential of natural fiber utilization for bio-composites.

2. Natural fiber

Natural fibers in simple definition are fibers that are not synthetic or manmade and are categorized based on their origin from animals, mineral, or plants sources as shown in **Figure 1** [9]. Some of the natural fibers are in readymade form such as vegetable, cellulose (cotton and linen), and mineral (asbestos) fibers. The fibers are produced and provided by nature from various parts of the plants, trees, and geographies.

The plants which produce cellulose fibers can be classified into bast fibers (flax, hemp, jute, kenaf, and ramie), leaf fibers (abaca, banana, pineapple, and sisal), seed fibers (coir, cotton, and kapok), as well as all other kinds (roots and wood). The photographs of some natural

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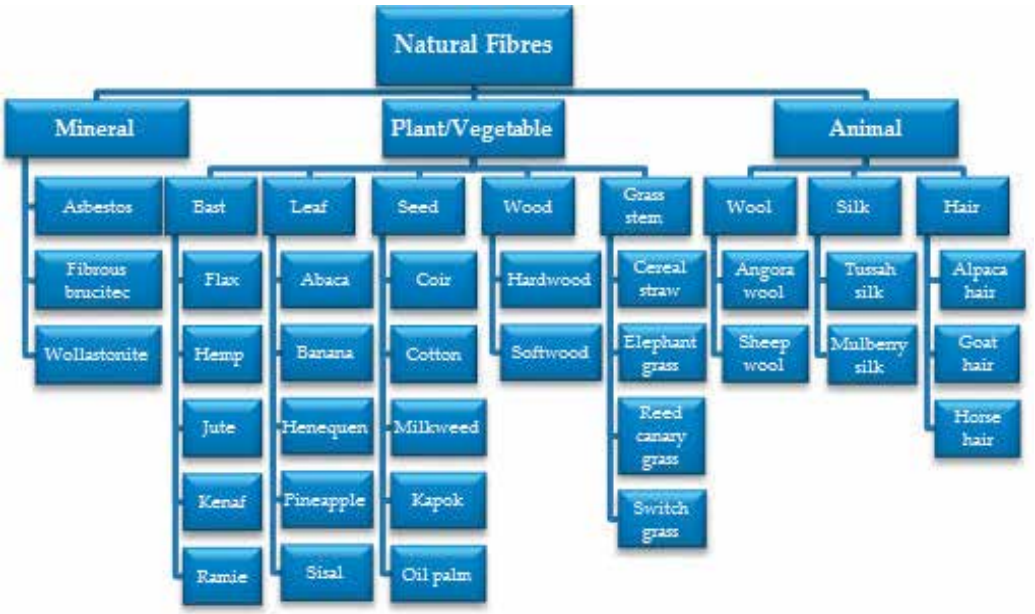

Figure 1. Natural fibers classifications.

Figure 2. Photographs of sources of some natural fibers.

fiber sources are shown in **Figure 2**. Natural plant fibers are entirely derived from vegetative sources and are fully biodegradable in nature. The most common and commercially available natural plant fibers and their global production are shown in **Figure 3**.

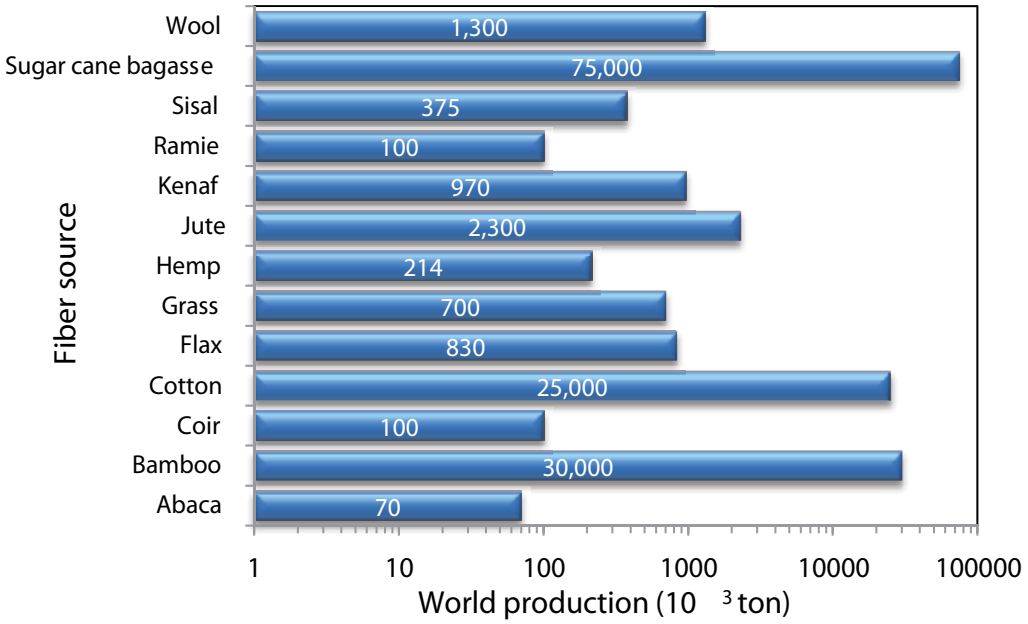

Figure 3. Natural plant fibers and their world production [18, 19].

Many of the plant fibers such as banana, coir, flax, hemp, jute, pineapple, and sisal find applications as resource for industrial materials. The main chemical components of natural plant fibers are cellulose, lignin, hemicelluloses, pectin, and wax. The components and their percentages vary depending on the type of natural plant fibers as shown in **Table 1** [10–17].

Fiber	Cellulose (wt%)	Hemicellulose (wt%)	Lignin (wt%)	Waxes (wt%)
Abaca	56.0–63.0	20.0–25.0	7.0–9.0	3.0
Bagasse	55.2	16.8	25.3	—
Bamboo	26.0–43.0	30.0	21.0–31.0	—
Birch	41.0	32.0	22.0	—
Cedar	44.0	21.0	30.0	—
Coir	32.0–43.0	0.15–0.25	40.0–45.0	<2.0
Corn cobs	33.7–41.2	31.9–36.0	6.1–15.9	—
Corn stalks	35.0–39.6	16.8–35.0	7.0–18.4	—
Cotton	80.0–95 .0	5.0–20.0	0	—
Curaua	73.6	9.9	7.5	—
Douglas fir	44.0	11.0	27.0	—
Eucalyptus	54.1	18.4	21.5	—
Flax	71.0	18.6–20.6	2.2	1.5
Grasses	25.0–40.0	25.0–50.0	10.0–30.0	—

Fiber	Cellulose (wt%)	Hemicellulose (wt%)	Lignin (wt%)	Waxes (wt%)
Hemp	68.0	15.0	10.0	0.8
Jute	61.0–71.0	14.0–20.0	12.0–13.0	0.5
Kenaf	72.0	20.3	9.0	<1.0
Oat straw	31.0–35.0	20.0–26.0	10.0–15.0	—
Oil palm	65.0	—	29.0	—
Pineapple	81.0	—	12.7	—
Ramie	68.6–76.2	13.0–16.0	0.6–0.7	0.3
Rice husk	35.0–45.0	19.0–25.0	20.0	—
Rice straw	41.0–57.0	33.0	8.0–19.0	8.0–38.0
Sisal	65.0	12.0	9.9	2.0
Sugarcane bagasse	25.0–45.0	28.0–32.0	15.0–25.0	—
Sorghum straw	32.0–35.0	24.0–27.0	15.0–21.0	—
Switchgrass	35.0–40.0	25.0–30.0	15.0–20.0	—
Wheat straw	38.0–45.0	15.0–31.0	12.0–20.0	—
Deciduous wood	38.0–50.0	23.0–33.0	23.0–34.0	—

Table 1. Natural plant fibers and their chemical composition.

Fiber-reinforced polymer matrix got considerable attention in numerous applications because of its good properties. Current indicators are that interest in natural fiber composites by industry will keep on growing quickly around the world. The application of natural fiber–reinforced polymer composites and natural-based resins for replacing existing synthetic polymer– or glass fiber–reinforced materials is huge. However, natural fiber quality is influenced significantly by the age of the plant, species, growing environment, harvesting, humidity, quality of soil, temperature, and processing steps, and there is a move to reduce the on-field processing to improve consistency and reduce costs. Properties of several natural fibers and commonly used synthetic fibers are shown in **Table 2** [20–23].

Fiber	Density (g/cm^3)	Elongation (%)	Tensile strength (MPa)	Young's modulus (GPa)
Abaca	1.5	—	980.0	—
Bagasse	1.2	1.1	20.0–290.0	19.7–27.1
Banana	1.3–1.4	2.0–7.0	54.0–789.0	3.4–32.0
Coconut	1.4–3.8	—	120.0–200.0	19.0–26.0
Coir	1.2	15.0–30.0	175.0–220.0	4.0–6.0
Cotton	1.5–1.6	3.0–10.0	287.0–597.0	5.5–12.6
Flax	1.4–1.5	1.2–3.2	345.0–1500.0	27.6–80.0
Hemp	1.4–1.5	1.6	550.0–900.0	70.0

Fiber	Density (g/cm^3)	Elongation (%)	Tensile strength (MPa)	Young's modulus (GPa)
Henequen	1.4	3.0–4.7	430.0–580.0	—
Jute	1.3–1.5	1.5–1.8	393.0–800.0	10.0–30.0
Kenaf	1.2	2.7–6.9	295.0	—
Palf	1.4	3.0	170.0–635.0	6.2–24.6
Pineapple	1.5	1.0–3.0	170.0–1672.0	82.0
Ramie	1.5	2.0–3.8	220.0–938.0	44.0–128.0
Sisal	1.3–1.5	2.0–14.0	400.0–700.0	9.0–38.0
Softwood kraft	1.5	—	1000.0	40.0
Carbon	1.4	1.4–1.8	1500.0–5500.0	230.0–240.0
E-glass	2.5	2.5–3.0	2000.0–3500.0	70.0
S-glass	2.5	2.8	4570.0	86.0
Kevlar	1.4	3.3–3.7	3000.0–3150.0	63.0–67.0

Table 2. Properties of several natural fibers and commonly used synthetic fibers.

3. Natural fibers bio-composites

Bio-composite materials are defined as composite materials in which at least one of the constituents is derived from natural resources. Generally, the term bio-composites cover composite materials made from the combination of:

- Bio-polymers-reinforced synthetic fibers such as carbon fibers, glass, Kevlar, etc.;
- Natural fibers–reinforced petroleum-derived polymers such as polyethylene (PE), polypropylene (PP), epoxy, unsaturated polyester (UPE), vinyl ester (VE); and;
- Bio-polymers reinforced by natural fibers.

The advantages of natural fiber over synthetic fiber in terms of its relatively renewable resources are its abundance, less damage to processing equipment, low weight, low cost, good relative mechanical properties such as tensile modulus and flexural modulus and improved surface finish of molded parts composite. The bio-composite materials made from the combination of natural fibers–reinforced petroleum-derived polymers and bio-polymers–reinforced synthetic fibers such as glass and carbon are nonbiodegradable and are not fully environmentally friendly.

Polymers can be categorized into thermoplastic and thermoset. Though thermoset and thermoplastic sound similar, they have very different properties and applications [24]. Thermosets are materials that undergo a chemical reaction (curing reaction) and normally transform from a liquid to a solid. In its uncured form, the material has small, unlinked molecules (known as

monomers). The addition of a second material (catalyst, cross-linker, and curing agent) and/or the presence of heat or some other activating influences will initiate the chemical reaction. During this reaction, the molecules cross-link and form significantly longer molecular chains and a cross-link network, causing the material to solidify. Subsequently, exposure to high heat will cause the material to degrade, not melt. On the other hand, thermoplastics are melt-processable plastics. When enough heat is added to bring the temperature of the thermoplastic above its melting point (softens enough to be processed), the thermoplastic liquefies. The thermoplastic solidifies back into glass-like solid state, when the heat source is removed and the processing temperature drops below its melting point. This behavior allows melting and re-shaping the thermoplastic as the temperature increases above and reduces below the melting temperature, respectively. Thermoset and thermoplastic materials each have their unique set of properties and position in the market. In general, the market for the thermosets has been well-established, and they tend to have been around for a long time. Frequently, the costs of raw materials for producing thermoset are lower as compared to thermoplastic. In addition, thermoset is often easy for wetting the reinforcing fibers and forming final composites products. Thermoplastics tend to be tougher than thermoset, can have better chemical resistance, do not need refrigeration as uncured thermosets frequently do, and can be more easily recycled and repaired. Elastomers are usually thermosets (requiring vulcanization) but may also be thermoplastics (thermoplastics elastomer). Elastomer is extreme flexible and possesses a huge rate of elasticity which means that when these types of polymers are submitted to a stress, even a small one, they deform significantly. Elastomers can reversibly extend from 5 to 700%. This deformation is reversible, and when the stress ends, the polymer returns to its original shape.

Bio-polymers are polymers obtained from natural resources and are consisting of monomeric units that are covalently bonded to form larger structures. Bio-polymers vary among their melt flow indices, impact properties, hardness, vapor transmission characteristics, coefficient of friction, and decomposition. Bio-polymers can be thermoset, thermoplastic, and elastomer. One can find vast range of applications of bio-polymers in different fields such as agricultural films, automotive, medical and pharmaceutical, food packaging, hygiene, and protective clothing. There are several types of polymers in the market. The most common thermoplastic, thermoset, and bio-polymers are summarized in **Table 3** [25–27].

Natural plant fiber polymer composites are a composite material consisting of a polymer matrix embedded with natural fibers [28]. Loose fiber, nonwoven mats, aligned yarns, and woven fabrics are possible forms of natural fiber for composites, with aligned variants offering the best mechanical properties. Natural plant fiber–derived bio-composites are renewable, lightweight, energy efficient, and environmentally friendly as compared to other binder fabric composites [29]. Utilizing natural plant fibers in particular would decrease waste disposal problems and reduce environmental pollution. The techniques utilized to fabricate bio-composites are based largely on existing techniques for processing plastics and conventional composite materials. Depending on the types and form of natural fiber–reinforced composites, the processing techniques would be chosen accordingly. For instance, compression molding, extrusion, hand lay-up, injection molding, resin transfer molding, and sheet molding compound can be utilized for short natural fibers, while compression molding, filament winding,

	Polymers	Density (g/cm^3)	Elongation (%)	Tensile strength (MPa)	Young's modulus (GPa)
Thermoplastic	Acrylonitrile butadiene styrene (ABS)	1.0–1.1	270.0	47.0	2.1
	Cross-linked polyethylene (PE)	0.9	350.0	18.0	0.5
	Ethylene vinyl acetate (EVA)	0.9–1.0	750.0	17.0	0.02
	High-density polyethylene (HDPE)	0.9–1.0	150.0	32.0–38.2	1.3
	High-impact polystyrene (HIPS)	1.0	2.5	42.0	2.1
	Low-density polyethylene (LDPE)	0.9	400.0	10.0–11.6	0.2–0.3
	Nylon 6 (PA 6)	1.1	60.0	81.4	2.8
	Nylon 6,6 (PA 6,6)	1.1	60.0	82.7	2.8
	Polycarbonate (PC)	1.2	200.0	69.0	2.3
	Polyethylene terephthalate (PET)	1.5–1.6	300.0	55.0–159.0	2.3–9.0
	Polyether ether ketone (PEEK)	1.3–1.5	1.6–50.0	92.0–95.0	3.7–24.0
	Poly ether ketone (PEK)	1.2–1.4	20.0	100.0–110.0	3.5
	Poly methyl methacrylate (PMMA)	1.1–1.2	2.5	72.4	3.0
	Polypropylene (PP)	0.9–1.3	80.0	35.8	1.6
	Polystyrene (PS)	1.04	1.6	34.0	3.0
	Polyvinyl chloride (PVC)	1.3–1.5	50.0–80.0	52.0–90.0	3.0–4.0
	Rigid thermoplastic polyurethane (RTPU, PUR-RT)	1.1	5.0	75.0	4.0
Thermoset	Epoxy (EP)	1.2–1.3	1.3	600.0	80.0
	Melamine formaldehyde (MF)	1.5–1.6	0.6	65.0	12.0
	Phenol formaldehyde (PF)	1.2	1.2	45.0	6.5
	Rigid thermoset polyurethane (RPU)	1.2	90.0	60.0	2.2
	Unsaturated polyester (UPE)	1.1	2.0	60.0	3.4
	Urea formaldehyde (UF)	1.5–1.6	0.8	65.0	9.0
	Polyurethane rubber	1.2–1.3	300.0–580.0	39.0	2.0–10.0
Biopolymers	Polylactic acid (PLA)	1.2–1.3	2.1–30.7	5.9–72.0	1.1–3.6
	Polyhydroxyalkanoates (PHA)	1.2–1.3	2.0–1200.0	10.0–39.0	0.3–3.8
	Polyhydroxybutyrate (PHB)	1.2	1.56–6.0	24.0–40.0	3.5–7.7
	Poly-3-hydroxybutyrate (P-3-HB)	1.3	0.4–6.0	40.0	3.5
	Poly-3-hydroxybutyrate-co-3-hydroxyvalerate (P-3-HB-3 HV)	0.2–0.3	1.6–20.0	23.0–40.0	3.5
	Poly-3-hydroxybutyrate (P-3-HB)	1.2	1000.0	104.0	—
	Polycaprolactone (PCL)	1.1	700.0	16.0–23.0	0.4

Table 3. Properties of polymers and bio-polymers.

hand lay-up, pultrusion, and resin transfer molding can be appalled for long fibers. The properties and performance of products made from natural fiber composites depend upon processing techniques, the properties of their individual components, as well as their compatibility and interfacial bonding between polymer and fiber. A number of drawbacks of natural fiber composites like higher water absorption, inferior fire resistance, and lower mechanical properties as compared to synthetic fibers may limit their applications. The physical, mechanical properties, and fire resistance of the natural fibers can be further enhanced through the treatment, while their moisture absorption can be reduced through surface modification and addition of coupling agents. The bonding strength between fiber and polymer matrix in the composite is considered a major factor in order to get superior fiber reinforcement composite properties. An essential requirement for good fiber matrix adhesion is optimized impregnation of the reinforcing system. Because of pendant hydroxyl and polar groups in natural plant fibers, this leads to extremely high moisture absorption of natural plant fiber resulting in weak interfacial bonding between the fiber and the hydrophobic matrix polymers. In addition, the coupling between natural plant fiber and polymer is considered a challenge because of the various chemical structures of both fibers and matrix. This results in ineffectual stress transfer at the interface of the produced composites. To develop composites with good mechanical properties, chemical modification of fiber is carried out to reduce the hydrophilic behavior of fibers and their moisture absorption [30, 31]. Many attempts have been made to modify the natural fiber surface in order to enhance their adhesion with the matrix through acetylation, acrylation, alkaline treatment, benzoylation, corona treatment, graft copolymerization, heat treatment, plasma treatment, silane treatment, stearic acid treatment, and other chemical modifications [32–41]. The surface chemical modifications of natural fibers have achieved various levels of success in improving adhesion with polymer. Coupling agents have also been utilized in the composite formulations. A coupling is defined as a compound which provides a chemical bond between two dissimilar materials. Coupling agents act as the bridge between polymer and natural fibers and improve their bonding. Mechanical properties of several biocomposites are presented in **Table 4**.

Bio-composites	Fiber content (%)	Tensile strength (MPa)	Young's modulus (GPa)	References
PHB/kenaf	40.0	70.1	—	[44]
PLA/abaca	30.0	74.0	8.0	[45]
PLA/bamboo	20.0	90.0	1.8	[46]
PLA/flax	30.0	53.0, 100.0	8.0	[47, 48]
PLA/hemp	45.0	65.0	—	[49]
PLA/jute	35.0	50.0	—	[50]
PLA/kenaf	40.0	52.9	7.1	[51]
PP/cotton	30.0	58.5	4.1	[52, 53]
PP/coir-alkaline, silane	10.0–30.0	42.1–47.8	2.0	[54]
PP/curaua	5.0–20.0	29.5–31.2	1.4–3.5	[55]
PP/flax Flax-MAgPP, CaO	40.0	96.9–109.0	7.9–10.1	[56]
PP/flax	30.0	52.0	5.0	[57]

Bio-composites	Fiber content (%)	Tensile strength (MPa)	Young's modulus (GPa)	References
PP/flax-acetylation	30.0	39.0–40.0	4.9–5.0	[58]
PP/hemp	30.0	44.0	3.9	[52, 53]
PP/hemp-MAgPP	40.0	52.0	4.0	[59]
PP/jute-non woven mat	32.0	39.0	8.4	[60]
PP/jute	35.0–50.0	76.0–87.0	4.0–5.3	[61]
PP/jute	30.0–45.0	52.0–54.0	4.2–5.1	[62]
PP/kenaf	30.0	46.0	5.0	[63]
PP/kenaf	50.0	53.0	7.5	[64]
PP/rice husk	30.0	32.1	27.5	[52, 53]
PP/sisal	30.0	45.5	3.9	[52, 53]
PP/wood pulp	27.0	28.0	4.2	[58]
PP/wood BKP-MAgPP	40.0	50.0	3.0	[65]
PP/glass-chopped strand mat	20.0	77.0	5.4	[66]
Epoxy/bamboo	57.0	392.0	29.0	[67]
Epoxy/coconut coir-sisal	40.0	56.0	—	[68]
Epoxy/flax yarn-aligned	45.0	133.0	28.0	[69]
Epoxy/hemp	65.0	165.0	17.0	[70]
Epoxy/jute	52.0	216.0	31.0	[69]
Epoxy/rice husk	25.0	117.1	6.76	[71]
Epoxy/sisal	73.0	410.0	6.0	[72]
Epoxy/sisal	48.0	210.0	20.0	[73]
Epoxy/glass	40.0	335.0		[74]
PF/banana	45.0	7.0–23.0	175.0–398.0	[75]
PF/grewia optiva	30.0	37.9	0.8	[76]
PF/glass	25.0	7.0–17.0	175.0–316.0	[75]
UPE/flax	34.0	143.0	14.0	[77]
UPE/jute	35.0	50.0	8.0	[78]
UPE/rice husk	25.0	73.4	6.0	[71]
UPE/glass	47.0	201.0	13.0	[78]
VE/flax	24.0	248.0	24.0	[77]
VE/glass-woven	59.0	483.0	33.0	[77]

Table 4. Mechanical properties of bio-composites.

Eco-friendly bio-composites from crop-derived plastics and plant-derived fibers would be the materials for near future not only as a solution to the growing environmental threat but also as a solution to alleviating the uncertainty of the petroleum supply. The market size of natural fiber composites is projected to reach USD 6.50 Billion by 2021, at a compound annual growth

	Manufacturer	Model	Application
Automotive Industry	Audi	A2, A3,A4, A4 Avant, A6, A8, Coupe, Roadstar	Boot-liner, hat rack, seat back, side and back door panels, spare tire-lining
	BMW	3, 5, and 7 series	Boot lining, door panels, headliner panels, molded foot well linings, noise insulation panels, seat back
	Citroen	C5	Interior paneling
	Chrysler	Chrysler Sebring	Interior door panel
	Daimler Chrysler	A, C, E, and S class, EvoBus	Business table, dashboard, door panels, pillar cover panel, windshield
	Fiat	Alfa Romeo 146, 156, 159, Brava, Marea, Punto	Door panels
	Ford	Mondeo CD 162, Focus Ford fusion and Lincoln MKZ	Boot liner, B-pillar, door panels Seating headrests
	General Motors	Cadillac De Ville, Chevrolet Trail Blazer	Cargo area floor mat, seat backs
	Lotus	Eco Elise	Body panels, interior carpets, seats, spoiler
	Mercedes-Benz	C, S, E, and A classes Trucks	Door panels, glove box, instrument panel support, insulation, molding rod/apertures, seat backrest panel, seat surface/backrest, trunk panel Bumper, engine insulation, internal engine cover, interior insulation, roof cover, sun visor, wheel box
	Nissan	Nissan Leaf	Floor mats
	Peugeot	406	Front and rear door panels, parcel shelf, seat backs
	Renault	Clio, Twingo	Rear parcel shelf
	Rover	2000 and others	Insulation, rear storage shelf/panel
	Saab	9S	Door panels
	Saturn	L300	Door panel, package trays
	Toyota	Brevis, Camry, Celsior, Harrier, Raum	Door panels, floor mats, radiator end tank, seat backs, spare tire cover
	Vauxhall	Astra, Corsa, Vectra, Zafira	Headliner panel, interior door panels, instrument panel, pillar cover panel
	Volkswagen	Bora, Golf, Passat	Boot lid finish panel, boot liner, door panel, seat back
	Volvo	C70, V70	Cargo floor tray, natural foams, seat padding

	Manufacturer	Model	Application
Constructions and other Industries	Bcomp	AmpliTex®, bCores®, powerRibs. Natural fiber (flax, balsa wood) composite reinforcement material	Automotive, summer and winter sports
	Enkev	Cocoform. Coir (coconut) fiber and natural latex rubber	Containers, boxes, trays, packaging
	FlexForm Technologies	Blends of sustainable natural fibers (hemp, jute, and kenaf) and fiberized thermoplastic polymers	Containers for shipping and storage, interior panels, load floors and underbody shields for cars and trucks, workspace panels and furnishings for offices and homes, structural support for agricultural seedlings
	GAF materials corp	DuraLife™. PP hardwood	Composite decking
	GreenCore	GreenCore NCell™. PP or PE matrix reinforced with up to 40% natural cellulosic microfibers	Containers, home & office furniture, housewares, lawn & garden products, power tools, sporting goods, storage, transportation, and toys Automotive, consoles, door handles, instrument panel components, load floors
	Green line	Plastics (PLA and PP) and additives, natural fibers (hemp and flax)	Cases for musical instruments
	Innovation in green composites technology	50% recycled resin, reinforced with flax (25%) and E-glass (25%) roving	BRP green wall panel
	Lingrove	Lingrove pre-impregnated/ thermoplastic composites, linen fabric, cores + resins	Furniture, music, sport, transportation
	Tech-wood International	Wood plastic composites	Modular house construction
	Trex company	Trex Transcend™. Wood plastic composites and PVC	High-performance decking and railing line
	UFP technologies	Mix of 50% natural and 50% PP	Bolsters, door panels, load floors and packaging trays using natural fiber composites, seat backs
	UPM-Kymmene	Wood plastic composites	ProFi decking products

Table 5. The applications of natural fiber composites.

rate (CAGR) of 11.68%, between 2016 and 2021 [42]. This growth is attributed to the high demand for natural fiber composites in the construction and automotive industries and driven by regulatory requirements and superior product performance. Lightweight, high stiffness-to-weight ratio, consumer awareness regarding recyclable, and bio-degradable materials are the advantages of using natural fiber composites in the composites market [43]. The wide applications of natural fiber composites are growing rapidly in several engineering fields.

Natural fiber–reinforced polymer composite use is of great importance in numerous applications including automotive, building and construction industries, sports, aerospace, and others, such as, decking, panels, window frame, and bicycle frame [79, 80]. Several automotive

components and construction materials are already produced with natural fibers composites with various polymers. The most common polymers used for the applications are polyester or polypropylene, and the natural fibers are flax, hemp, and sisal. Natural fiber composites have received considerable attention by many automotive and construction companies. However, the price, weight reduction, and marketing rather than technical demands will drive the application of natural fiber composites in this industry [81]. A summary of the utilization of natural fiber composites in automotive applications, building and construction industries, as well as others is shown in **Table 5** [82–98].

4. Summary

Natural fiber bio-composites have many advantages; they are relatively cost effective, exhibit good thermal and dimensional stability, low coefficient of friction, and low density and are more environmentally friendly. For these reasons, the popularity of these bio-composites is increasing, and a significant amount of scientific knowledge is already generated. To facilitate the utilization and applications of the bio-composites, all aspects must be present in society: (a) concept development, one of the steps in new bio-composite materials development to better address the future needs of applications, (b) material design, select the bio-composites for applications and to model designs to ensure that a material has the necessary performance capabilities, (c) material fabrication, select the right fabrication methods for the designed bio-composite materials, (d) product manufacturing, improve the way bio-composites, components, and systems are manufactured, as well as the final appearance and functionality of the product, (e) market, understand the market's need and size, and (f) regulations act to promote the utilization of the bio-composites.

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Nano- and micro-sized natural fibers of vegetable origin are fully biodegradable in nature. However, the nano- and micro-sized synthetic fibers are fully man-made. Fiber-reinforced composites composed of stiffened fiber and matrix are well-known engineering materials. Fiber-reinforced materials have been used in industrial production. Natural fibers can be obtained from many sources in nature such as wool, sisal, ramie, kenaf, jute, hemp, grass, flax, cotton, coir, bamboo and abaca, banana, and sugarcane bagasse. Artificial fibers have been produced from more stiff materials such as glass, single-walled carbon nanotubes, double-walled carbon nanotubes, carbon, aramid, boron and polyethylene (PE). The cyclic reusability of materials is an important qualification in protecting the environment from waste pollution. Three important factors can be mentioned in terms of material properties in the recycling process. The first factor is "the rate of cyclic usage," the second one is "less material loss in each recycle," and the last one is "the role of waste products in the self-renewal of ecosystem." In engineering area, the usage of waste materials has taken into account in production of composite materials. The use of waste materials as particulate-type composite production is also possible in the industry. Fiber-reinforced materials can be grouped into two categories: "the natural fiber-reinforced materials" and "the artificially produced fiber-reinforced materials." Finally, we conclude that this book consists of mainly summarized three subject headings within the two specific book subsections : The first group contains the main subjects related to the natural and artificial fibers obtained by literature review; second, experimental and numerical studies are made in order to perform the necessary arrangements in the production stages and to establish a decision mechanism on the specification of the technical properties of the fiber-reinforced composites. The third group of studies focused on the use of sustainable bio-composites and recycled textile wastes as reinforcements in construction.

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