Materials Horizons: From Nature to Nanomaterials

Pawan Kumar Rakesh
Inderdeep Singh *Editors*

Processing of Green Composites

Springer

Materials Horizons: From Nature to Nanomaterials

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ISSN 2524-5384 ISSN 2524-5392 (electronic)
Materials Horizons: From Nature to Nanomaterials
ISBN 978-981-13-6018-3 ISBN 978-981-13-6019-0 (eBook)
https://doi.org/10.1007/978-981-13-6019-0

Library of Congress Control Number: 2018968527

This Springer imprint is published by the registered company Springer Nature Singapore Pte Ltd.
The registered company address is: 152 Beach Road, #21-01/04 Gateway East, Singapore 189721, Singapore

Preface

This book presents state-of-the-art techniques in the field of green composites. The objective of the book is to deliberate over the improved manufacturing processes of green composites and how their applications in different industrial sectors such as automotive, aerospace, and sports can be increased. This book consists of a total of eight chapters. Chapter 1 discusses the different types of natural fibers and biodegradable polymers for the manufacturing of green composites. Chapter 2 deals with the lignocellulosic polymer composites, their processing, and the challenges involved in the present scenario. Chapter 3 focuses on the banana fiber-reinforced hybrid composites, their processing techniques, and mechanical properties. Chapter 4 is dedicated to the hygrothermal aging behavior of fiber-reinforced polymer composites. Chapter 5 discusses the chemical treatments of natural fiber and their effects on the mechanical properties of developed composites. All types of chemical treatments cannot be applied to the natural fibers. The selection of matrix materials and chemically treated natural fibers are very important for the development of green composites. The different types of testing methods for green composites are discussed in Chapter 6. Chapter 7 focuses on the silk fiber-reinforced polymer composites. Finally, in Chapter 8, the different types of machining processes suited for thermoplastic composites are discussed.

This book can be a valuable reference for postgraduate students and researchers from different disciplines working in polymeric composites. The editors acknowledge Springer Nature for this opportunity and for their professional support. Finally, we would like to thank all the chapter authors for their valuable contributions to this book.

Roorkee, India — Dr. Inderdeep Singh
Srinagar, India — Dr. Pawan Kumar Rakesh

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Abbreviations

ABS	Acrylonitrile butadiene styrene
AFRP	Aramid fiber-reinforced plastic
APO	Antiparallel orientation
ASTM	American Society for Testing and Materials
AWJ	Abrasive water jet
BFRP	Banana fiber-reinforced polyester
BPS	Banana pseudostem
CFRP	Carbon fiber-reinforced plastic
DEMEP	Diethyl-2-(methacryloyloxyethyl) phosphate
DMA	Dynamic mechanical analysis
FRP	Fiber-reinforced plastics
FTIR	Fourier-transform infrared
GFRP	Glass fiber-reinforced plastic
GPa	Gigapascal
H_2O_2	Hydrogen peroxide
HAZ	Heat-affected zone
HMM	Hexakis(methoxymethyle)melamine
HSS	High-speed steel
ILSS	Interlaminar shear strength
ISO	International Organization for Standardization
KOH	Potassium hydroxide
MEKP	Methyl ethyl ketone peroxide
MPa	Megapascal
NaOH	Sodium hydroxide
NFRPC	Natural fiber-reinforced polymer composites
NO	Normal orientation
PA	Polyamide
PBAH	Polycyclic benzenoid aromatic hydrocarbons
PC	Polycarbonate
PE	Polyethylene

PEEK	Polyether ether ketone
PHAs	Polyhydroxyalkanoates
PHB	Polyhydroxybutyrate
PHBV	Polyhydroxybutyrate-co-hydroxyvalerate
PLA	Polylactic acid
PLLA	Poly-L-lactide
PMC	Polymer matrix composites
PO	Parallel orientation
PP	Polypropylene
PS	Polystyrene
PVC	Polyvinyl chloride
RH	Relative humidity
SEM	Scanning electron microscope
SOD	Stand-off distance
TAP	Thermally assisted piercing
UFFD	Ultrafast feed rate drilling
UPVC	Un-plasticized polyvinyl chloride
UTM	Universal testing machine
UV	Ultraviolet rays

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Chapter 1
Introduction to Green Composites

Hitesh Sharma, Ujendra Kumar Komal, Inderdeep Singh, Joy Prakash Misra and Pawan Kumar Rakesh

Abstract This chapter provides an insight into the comprehensive arena of green composites, looking for materials with a perspective to be useful in the near future in various structural and non-structural applications, by replacing composites made by synthetic fibers and matrices. At this point, materials originating from Mother Nature (renewable resources) are favored as compared to the products obtained from non-renewable resources of energy. From the technical statistics of bio-polymers and natural reinforcements, a databank was formed providing insight into the mechanical performance of numerous possible components for the futuristic green composite products. Following the analysis, an assessment is accomplished where facets of suitability for the aspiring essentials in terms of mechanical properties are examined. In that segment, green materials for matrix and reinforcement are placed in order to recognize them as materials which embrace both ample strength and stiffness besides providing with the reasonable cost so as to be encouraging materials for the green composite-based products.

Keywords Green composites · Renewable resources · Bio-polymers · Environmental aging

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P. K. Rakesh and I. Singh (eds.), *Processing of Green Composites*, Materials Horizons: From Nature to Nanomaterials, https://doi.org/10.1007/978-981-13-6019-0_1

1 Introduction

Ever since the advent of lightweight materials, their application in various industries like aerospace, automobiles, sports equipment, and structural components has increased manifold. Composite materials are one such family of lightweight materials. The composite material is a heterogeneous mixture of two different phase materials which are combined in a certain proportion leading to improved properties than the parent materials. In the composite material, there is a continuous phase called matrix and a discontinuous phase called reinforcement. The reinforcement generally bears the load, whereas the matrix holds the composite together in addition to transferring the load between the reinforcements. Among the different class of composites, polymer matrix composites (PMCs) have proved to be a vital material in both structural and non-structural applications.

The polymer matrix composites are being used widely as a replacement for traditional materials due to their numerous advantages like high strength-to-weight ratio, high tensile strength, and low thermal expansion to name a few. In polymer matrix composites, the matrix is a polymer ingredient, which is mostly hydrocarbon based. The reinforcement materials mostly are synthetic fibers, generally glass, carbon, aramid, etc. [1–3]. The classification of various synthetic fibers, as well as matrix, is shown in Fig. 1.

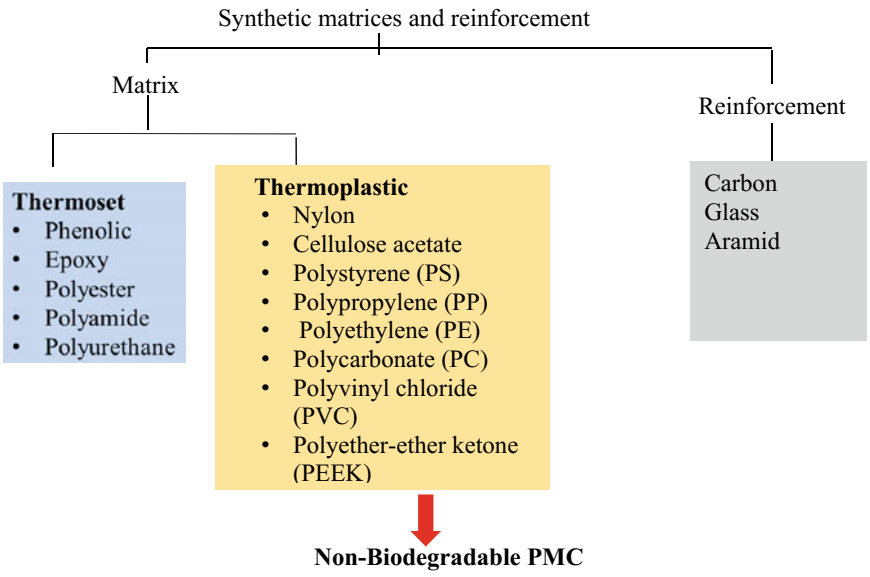

Fig. 1 Classification of synthetic matrices and fibers

The major problems associated with synthetic fibers and synthetic matrices are as follows:

1. They are derived from petroleum-based products. These petroleum-based products are already dwindling with each passing day, and the human race cannot rely on them for too long.
2. The synthetic polymers in practice are non-biodegradable in nature, i.e., once the life of the product is over, their disposal becomes a prime concern.
3. Their fabrication is an energy-intensive process, i.e., most of them requires the application of the high amount of heat and pressure.
4. Their processing is not easy as they may pose certain health problems like itching of skin, respiratory problems, etc.
5. Recyclability of synthetic matrices and fibers also presents a lot of challenge.

The above-mentioned factors leave no option but to go for such fibers and matrices which are biodegradable in nature and pose no threat to non-renewable resources. The composite developed from such raw materials are cheap, lightweight, and biodegradable with properties comparable to composites developed from synthetic raw materials. Green composites are the ones in which various natural fibers like jute, kapok, basalt, banana, henequen, sisal, hemp, flax, cotton, etc., are used in combination with matrices derived either naturally like PLA, polycyclic benzenoid aromatic hydrocarbons (PBAH), etc., or synthetically like polypropylene (PP), ABS, polyethylene (PE), PVC, etc. [4–6].

2 Green Composites: Constituents and Types

The main constituents in the green composites are matrix and reinforcement similar to that of polymer matrix composites (PMCs). In green composites, also the function of the matrix is to bind and transfer the load whereas the reinforcement provides strength by bearing the load. The green composites are classified based on the type of matrix and reinforcement materials.

2.1 Reinforcing Element

Natural fibers are basically renewable fibers that grow in agricultural fields or can be obtained through various food wastes. These can be used as reinforcements in the form of fibers, flakes, particulates, etc., for processing of composites in the similar mode as the synthetic ones like glass for instance. The various natural fibers in practice are shown in Fig. 2. The main advantage associated with natural fibers is the low density in comparison to synthetic fibers. The low density of natural fibers results in making them lighter in weight. The problem associated with most of the natural fibers is that they are hydrophilic in nature which means the problem of absorbing

Fig. 2 Typical natural fibers

Table 1 Chemical compositions (wt%) of natural fibers [13, 15–17]

Types of fibers	Cellulose	Hemicellulose	Lignin	Pectin	Ash content
Jute	45–71.5	12–20.4	12–25	4–10	8
Hemp	70–74.4	17.9–22.4	3.7–5.7	0.9	8
Kenaf	31–57	21.5	8–19	3–5	–
Flax	65–85	16	1–4	5–12	1–2
Sisal	50–64	6	10–14	10	7
Ramie	68–76	–	0.6–0.7	1.9	5
Cotton	85–90	26.9	–	0–1	1
Hardwood	40–50	21–36	20–30	0–1	–
Softwood	40–45	25–30	34–36	0–1	–
Abaca	56–63	–	12–13	1	1
Rice straw	41–57	33	8–20	8	7
Bamboo	42.3–49.1	24.1–27.7	23.8–26.1	–	1.3–2.0
Bagasse	40–46	24.5–29	12.5–20	–	1.5–2.4

moisture and swelling is common. This hydrophilic nature of natural fibers can be curbed by treating then with various reagents like sodium hydroxide, NaCl solution, etc. Chemical treatments such as alkali acetylation and bleaching have been found to enhance the matrix–fiber adhesion by disrupting the moisture absorption process through a coat of OH groups in fiber and by altering surface roughness of the fiber by cleaning surface from impurities [7, 8]. In addition to these treatments, of late there is a lot of focus on eco-friendly treatment of natural fibers. Treatment with sodium bicarbonate results in removing hemicellulose content and surface impurities, but due to the mild alkaline nature of sodium bicarbonate, the time taken for treatment is more. The chemical composition of some natural fibers, both plant and animal, is shown in Table 1.

2.1.1 Mechanical Properties of Natural Fibers

In order to have a wider assessment of the physical and mechanical properties of diverse natural fibers, existing data from several research papers has been summarized in Table 2.

2.1.2 Key Challenges Concerning the Use of Natural Fibers as Reinforcement

In spite of various advantages that the natural fibers bring along with them with their usage in composites, they also have few drawbacks concerning their performance, their processing, and their behavior in various matrix systems. The properties of

Table 2 Physical and mechanical properties of common natural fibers [18–23]

Type of natural fiber	Density (g/cm^3)	Tensile strength (MPa)	Young's modulus (GPa)	Elongation at break (%)
Abaca	1.5	400	12	3–10
Alfa	0.89	350	22	5.8
Bagasse	1.25	290	17	–
Bamboo	0.6–1.1	140–230	11–17	–
Banana	1.35	500	12	5.9
Coir	1.2	175	4–6	30
Cotton	1.5–1.6	287–597	5.5–12.6	7–8
Curaua	1.4	500–1150	11.8	3.7–4.3
Date palm	1–1.2	97–196	2.5–5.4	2–4.5
Flax	1.5	345–1035	27.6	2.7–3.2
Hemp	1.48	690	70	1.6
Henequen	1.2	500 ± 70	13.2 ± 3.1	4.8 ± 1.1
Isora	1.2–1.3	500–600	–	5–6
Jute	1.3	393–773	26.5	1.5–1.8
Kenaf	–	930	53	1.6
Nettle	–	650	38	1.7
Oil palm	0.7–1.55	248	3.2	25
Piassava	1.4	134–143	1.07–4.59	21.9–7.8
Pineapple	0.8–1.6	400–627	1.44	14.5
Ramie	1.5	560	24.5	2.5
Sisal	1.5	511–635	9.4–22	2.0–2.5
Silk	1.3	1300–2000	30	28–30

natural fibers can vary from every harvesting region and/or from harvesting season based on various factors like the intensity of sun, rain, and soil conditions. In addition to these factors, like part of the plant from where they are harvested, how mature the plant is, production route and whether there is any preconditioning done on the fiber or not. All the above-mentioned factors affect properties of natural fibers significantly compared to their synthetic fiber counterparts. These problems can be addressed by mixing fibers from different batches together in order to get more uniform properties out of these natural fibers. Such mixing of natural fibers reduces the variation in properties. One of the important concerns regarding natural fibers is their poor adhesion properties with various matrices due to the hydrophilic nature of fiber and hydrophobic nature of the matrix. This may result in non-uniform mixing of fibers and matrix and agglomeration of fibers at one place. The limitation in the usage of natural fibers at high temperature can also not be ruled out. The use of various compatibilizer agents like maleic anhydride, bio-amides, etc., helps in

achieving better affinity and adhesion between matrix and the fiber. The use of thermal compatibilizers helps natural fibers to get processed at higher temperatures.

2.2 The Matrix Material

Quite a few matrix materials originating from renewable resources might represent themselves as capable contenders for application in green composites being either biodegradable or non-biodegradable. The evolving question henceforward is the recyclability and biodegradability of composites when they are disposed in nature. For a suppositious fully bio-based composite, there are methods to be chosen to burn the composite for energy recovery, even if the material could not be recycled directly. In the case of burning, there are no emissions of lethal gases and by decomposition, there are no gases at all. The classification of bio-polymers on the basis of the resources is shown in Fig. 3.

2.2.1 Mechanical Performance of Natural Resins

For processing of green composites, information from several studies on bio-resins is presented in Table 3. The values obtained after studies from various researchers were found to be different; hence, only extreme values are shown in Table 3. In contrast to the natural fibers, these natural resins can provide with reproducible properties, which make them usable in the ever-growing materials market.

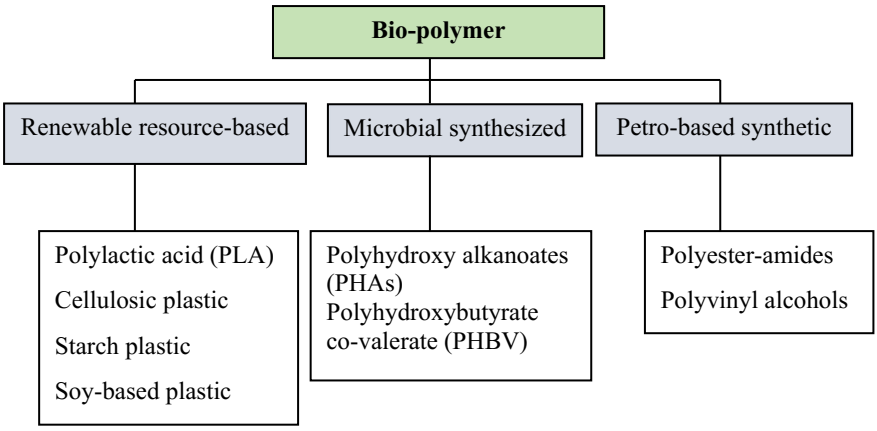

Fig. 3 Classification of bio-based polymers

Table 3 Properties of natural resins [24–27]

Polymer	Density (g/cm^3)	Melting point (T_m °C)	Tensile strength (MPa)	Young's modulus (GPa)	Elongation at break (%)
Thermoplastic starch	1–1.39	110–115	5–6	0.125–0.85	31–44
PLA	1.21–1.25	150–162	21–60	0.35–3.5	2.5–6
PLLA	1.25–1.29	170–190	15.5–65.5	0.83–2.7	3–4
PHB	1.18–1.26	168–182	24–40	3.5–4	5–8
PHBV	1.23–1.25	144–172	20–25	0.5–1.5	17.5–25

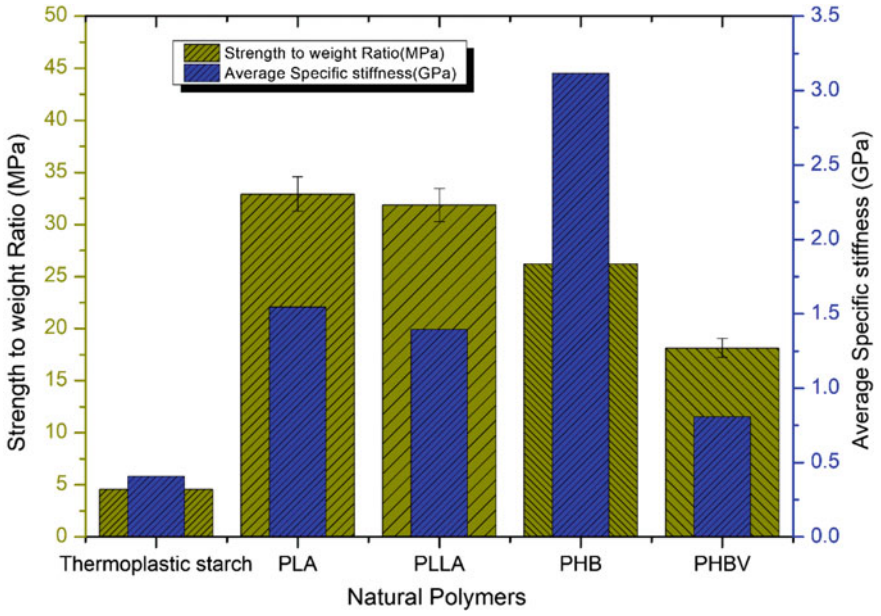

Fig. 4 Mechanical properties of various natural polymers

The abbreviations enumerated in Table 3 are as follows: PHB stands for polyhydroxybutyrate—another aliphatic polyester, and PHBV is the copolymer poly(3-hydroxybutyrate-co-3-hydroxy valerate). PLA denotes the poly(lactic acid) and PLLA is the poly-L-lactide; they are both thermoplastic aliphatic polyesters. PLA is considered a man-made polymer, as it does not originate in nature but it is fully biodegradable [9]. From Table 3, two graphs are designed showing the average stiffness and strength-to-weight ratio of various natural resins as shown in Fig. 4.

2.2.2 Key Challenges Concerning the Use of Resins from Natural Resources as a Matrix

Resins derived from natural resources are often also termed as bio-resins. They can either be biodegradable or compostable in nature. The problem that can be faced with bio-resins is their brittle nature, low melt viscosity, high gas permeability, low heat distortion temperature, low hydrolysis resistance, and low impact strength [10]. These factors affect further processing because of which their use gets restricted in a wide variety of applications. Another vital factor is the cost of natural resins; the cost of PLA is two to three times higher than the cost of PP on an average. The question comes to mind, that, by going for naturally derived resins, are we helping or destroying the Mother Nature. Another question that surrounds the environmentalists is the source from where these bio-resins are being developed. The bio-resins developed from edible sources like edible crops should be avoided as they may hinder with the food chain. The other problem to address is that where to decompose these bio-resin-based products when their life will be over. This problem may lead to encroachment of fertile land for decomposition purposes, or it may lead to the cutting of forests to get arable land [11]. Moreover, these bio-resins lack proper adhesion and binding properties which can further be enhanced using bio-amides, which further stabilizes its melt viscosity, increases service life, and also slows down polymer degradation.

3 Mechanical Performance of Green Composites

Green composites made up from cellulosic plant fibers and various resins are already been widely used in the interiors of automobiles. Unique green composites have been developed and tested in several studies with an effort to discover their performance in numerous applications. A number of studies have been reported by various researchers globally that tested numerous types of natural fibers reinforcing different kinds of bio-resins, and the results of the same have been summarized in Table 4.

Hence, the information regarding the mechanical properties of the established composites is not adequate for evaluating the complete in-service performance of the developed composites. The fabrication technique, type of fiber treatment (physical, chemical, and surface modification) involved as well as various additives used play a vital role in determining the true properties of green composites. It will be impractical to compare all these results without considering the above factors. The studies reported in the field of green composites are very limited, and the work is still going on in various industries and academic institutes.

Table 4 Mechanical properties of green composites [28–34]

Type of composite	Processing technique	Tensile strength (MPa)	Young's modulus (GPa)	Elongation at break (%)
Starch + 30% jute	Thermoplastic injection molding	26.3 ± 0.55	2.5 ±0.23	2 ± 0.2
PLA + 30% ramie	Hot pressing sheet molding	66.8 ± 1.7	–	4.8 ± 0.2
PLA + 30% jute	Thermoplastic injection molding	81.9 ± 2.9	9.6 ± 0.36	1.8
PHBV + 30% jute	Thermoplastic injection molding	35.2 ± 1.3	7 ± 0.26	0.8
PLLA + 30% flax	Film stacking compression molding	98 ±12	9.5 ± 0.5	2.3 ± 0.2
PHB + 30% flax	Film stacking compression molding	40 ±2.5	4.7 ± 0.3	7 ± 1.5
PLA + 30% flax	Twin-screw extruder + compression molding	53 ±3.1	8.3 ± 0.6	1 ± 0.2
PLA + 30% abaca	–	74	5.84	–
PLA + 40% jute	Film stacking	100.5	9.4	1.6
PLA + 30% man-made cellulose	Injection molding	92	8.032	–
PLA + 30 vol.% denim fabric	Hand layup	75.76	4.65	–
PLA + lyocell	–	81.8	6.78	4.09
PLA + 5% silk	Injection molding	62	4.2	–
PLA + 10% sugar beet pulp	Compression molding	37.5 ±0.5	1.043 ±0.0395	6.7 ±0.5

4 Environmental Aging

In spite of several advantages of green composites over synthetic fiber-based composites, there are certain drawbacks such as moisture absorption and degradability which limit the applications of these composites in various engineering applications. Natural fibers are prone to moisture absorption and may degrade under different environments such as water, acids, bases, and UV radiation. The green composites may be exposed to different environments during their service life. In order to overcome

these challenges and to study the degradation behavior of these composites, several researchers have studied the environmental aging behavior of these composites. In a study, the moisture absorption behavior of short roselle and sisal fiber-reinforced polyester composites was investigated. It was concluded that the exposure to moisture significantly deteriorated the mechanical properties of the composites due to degradation of interfacial properties between the fibers and matrix [12]. In the similar type of study, the effect of river water, diesel, sunlight, and freezing condition on the tensile properties of nettle fiber-based polypropylene composites was investigated. The maximum degradation in tensile properties was reported for the composites exposed to river water and sunlight [13]. In another investigation, the rice starch-based PLA composites were exposed to water and enzymes. A significant reduction in tensile properties was observed in both the environments [14].

5 Applications of Green Composites

Nowadays, the green composites are being extensively used in various sectors such as automobile, aerospace, sports, and furniture. It has been reported that the various parts of the luxury car like Mercedes Benz are made of natural fiber-based composites [13]. Table 5 depicts the various engineering parts/components made of natural fiber-based composites.

Table 5 Engineering parts/components made of natural fiber-based composites [13, 35, 36]

Engineering parts/components	Materials used
Damping and insulation parts, C-pillar trim, center console trim, rear parcel shelf, door panels, seat cushion	Plant fiber-based composites
Floor panels	Flax fiber-based polypropylene composites
Door panel	Flax/sisal fiber-based thermosets
Trunk floor	Cotton fiber-based polypropylene composites
Seat backs, trunk liner, dashboard	Jute fiber-based composites
Ceiling, floor, window	Jute fiber-based composites
Window frames, docks, molded panel	Wood fiber-based polymer composites
Circuit boards, spare tire covers	Kenaf fiber-based PLA composites
Dais-deck assembly	Jute fiber-based polyester composites
Dummy cards in personal computers	Kenaf fiber-based PLA composites

6 Considerations and Conclusions

The green composites have been found to be comparable in mechanical properties in comparison to synthetic fiber-based composites. Their applications have also been found in numerous fields like automobile, aerospace, sports, and other structural and non-structural components. On the other hand, the biodegradability issue has to be addressed in order to propose for fully 100% bio-based products. Moreover, the compatibility issue between natural fibers with synthetic resins or synthetic fibers with bio-based natural resins also needs to be addressed. Another key hindrance is related to development of such composites at the lowest possible cost without much compromise in the properties. Unluckily, the current major obstacle for generalized use of fully biodegradable polymers in developing products is a cost, but it is anticipated that soon reasonable solutions will be given by the producers of these bio-based materials as their applications in industries will tend to reduce their costs to more reasonable levels.

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Chapter 2
Lignocellulosic Polymer Composites: Processing, Challenges, and Opportunities

Ujendra Kumar Komal, Hitesh Sharma and Inderdeep Singh

Abstract The use of lignocellulosic fibers such as pineapple, banana, jute, and sisal as a reinforcement for developing the biocomposites is an emerging area of research in the field of polymer-based composites. Biocomposites have replaced the traditional fiber-reinforced polymer composites in various non-structural applications. The number of processes has been developed and commercialized for near-net-shape manufacturing of biocomposite components. However, complex composite products necessitate the secondary operations such as hole-making as an essential step for ascertaining the assembly operations. The hole-making operations lead to the damage in the biocomposite components in the form of delamination and fiber pullout. The researchers and engineers worldwide have tried to investigate the various issues, challenges, and opportunities in the primary and secondary processing of biocomposites. The current chapter highlights the fundamental issues, the challenges, and the existing opportunities which can help in formulating a road map for research and development in the field of primary and secondary processing of biocomposites.

Keywords Biocomposites · Primary processing · Secondary processing · Delamination

1 Introduction

Composite materials have been developed in response to the need for man-made materials for various applications. These have proved to be an effective tailor-made material for the present-day industry, due to their unique combination of properties, which are not available in traditional materials. The salient advantages (such

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P. K. Rakesh and I. Singh (eds.), *Processing of Green Composites*, Materials Horizons: From Nature to Nanomaterials, https://doi.org/10.1007/978-981-13-6019-0_2

as lightweight, high strength-to-weight ratio, chemical resistance, and design flexibility) of composite material have led them to replace the conventional materials in many engineering applications. Synthetic fiber-reinforced polymer matrix composites (PMCs) are the most common and widely used composite materials. PMCs offer several advantages over conventional materials such as high strength-to-weight ratio, corrosion resistance, lower maintenance costs, ease of processing, and the ability to produce near-net-shape products. Currently, synthetic fibers (glass, aramid, and carbon) are extensively being used as reinforcement in the thermoset- and thermoplastic-based composites in structural and non-structural applications. These composites have shown the durability in all types of interior and exterior applications [1]. Although these composites possess excellent mechanical properties, their major drawbacks are their non-renewable, non-biodegradable, and non-recyclable nature, which is a major threat to the ecological system. The requirement of lighter products and strict environmental policies and rules in many engineering applications has led to the development of natural fiber-reinforced polymer composites.

In the last decade, the feasibility of using natural fibers as reinforcement for the polymer composites has been extensively explored. The biocomposites have been identified as a potential substitute to the synthetic fiber-reinforced polymer composites in many engineering applications due to their exceptional properties (Table 1) such as low density, high modulus, non-abrasive nature, ease of fiber surface modification, abundant availability, and most importantly, environment friendliness. The broad classification of natural fibers is shown in Fig. 1

Natural fiber-reinforced polymer composites (NFRPCs) sometimes also called as biocomposites or lignocellulosic polymer composites have shown comparative or even better characteristics than synthetic fiber-reinforced polymer composites, like synthetic fiber-reinforced polymer composites; biocomposites can also be engineered easily to meet the specific requirements of products for different applications [3] (Table 2).

These have encouraged the various industries to use natural fibers as an alternative reinforcement material in the polymer-based products. The natural fiber-reinforced polymer composites can be classified based on their constituents. Figure 2 depicts

Table 1 Properties of natural fibers and glass fibers [2]

Properties	Natural fibers	Glass fibers
Density	Low	High
Mechanical properties	Comparable	High
Environment friendliness	Yes	No
Renewability	Yes	No
Energy consumption	Low	High
Health risk associated	No	Yes
Abrasion to machine	No	Yes
Recyclability	Yes	No

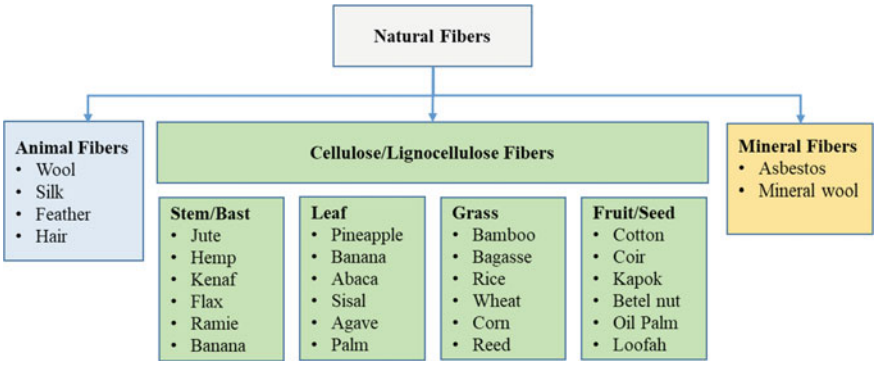

Fig. 1 Classification of natural fibers

Table 2 Comparison of properties of natural fibers and synthetic fibers [4, 5]

Fiber	Density (g/cm^3)	Tensile strength (MPa)	*E*-modulus (GPa)	Elongation at break (%)	Water absorption (%)
Coir	1.2–1.5	180–220	6	15–25	10
Jute	1.3–1.5	200–800	10–30	1.8	12
Ramie	1.5	500	44	2	12–17
Hemp	1.4–1.6	550–900	70	1.6	8
Sisal	1.3–1.5	600–700	38	2–3	11
Cotton	1.5–1.6	290–490	12	3–10	8–25
Flax	1.4–1.5	800–1500	60–80	1.2–1.6	7
Glass	2.5–2.6	2200–3600	65–75	3	–
Carbon	1.4–1.8	3000–4000	250–500	1–1.5	–

the classification of natural fiber-reinforced polymer composite based on their composition.

Natural fiber-based polymer composites are either partially or fully biodegradable, depending upon their constituents. When natural fibers are used as reinforcement for the non-biodegradable petroleum-based polymeric matrices, then these can be termed as partially biodegradable composites. When the natural fibers are used to reinforce the biodegradable polymeric matrices, these are called as fully biodegradable composites. Figure 3 depicts the classification of polymer composites based on their disposal characteristics.

The properties and performance of biocomposites depend on the various aspects such as the properties of their constituents, processing methods, processing parameters, the orientation of fibers, and the interfacial properties between the fibers and matrix [6–8]. The processing of polymer composites can be divided into two stages, primary and secondary processing.

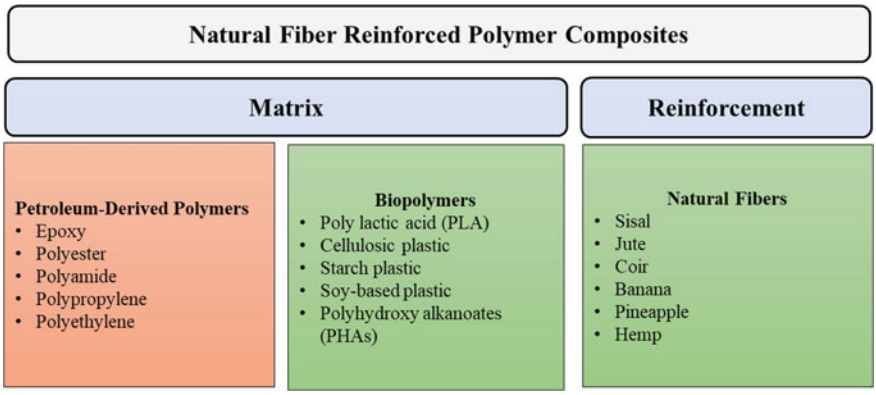

Fig. 2 Composition of natural fiber-reinforced polymer composites

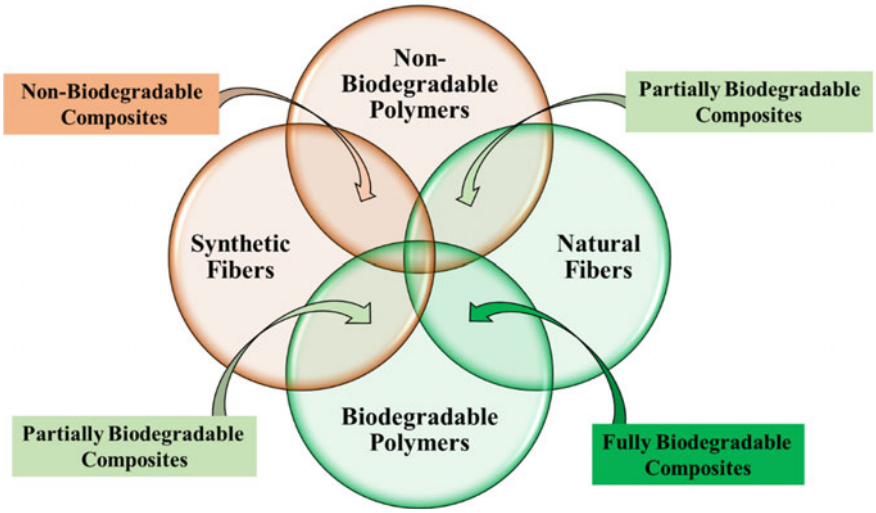

Fig. 3 Classification of polymer composites

Primary processing deals with providing the initial or structural shape to the composite materials. In the case of polymer matrix composites, generally, thermoplastics are available in granular and thermosets in liquid forms. Various techniques are used to convert the matrix into the final product. The primary processing techniques used for the manufacturing of biocomposite parts are hand layup, extrusion–injection molding, compression molding, and resin transfer molding. Each process has its own advantages and limitations based on the materials, accuracy, precision, tolerance, wastage, and cost.

Secondary processing is the essential and the second step to fabricate the final composite products. It deals with the machining, drilling, and joining of the composite

products fabricated by primary processing. The secondary manufacturing processes include drilling, edge trimming, contouring, adhesive joining, and mechanical fastening. In order to fabricate high-performance composite products, it becomes necessary to focus on various aspects of the primary and secondary processing techniques. This chapter provides a brief discussion on various techniques, issues, challenges, and opportunities in the primary and secondary processing of lignocellulosic polymer composites.

2 Primary Processing of Lignocellulosic Polymer Composites: Issues and Challenges

Primary processing is the crucial and the first step to fabricate the biocomposite parts. The performance of the developed composite parts mainly depends on the processing techniques. Although biocomposites possess required properties such as lightweight, high strength-to-weight ratio, corrosion resistance, and biodegradability, the fabrication of biocomposites according to the requirement is not an easy task. There are several issues and challenges associated with the primary processing of biocomposites. Some of these are discussed here:

- Although biocomposites are used in numerous engineering applications, the selection of appropriate processing technique and the processing parameters is difficult and challenging task.
- The natural fibers vary in terms of the properties, such as mechanical, thermal, and structural properties. The properties also depend upon the topographical region where the plants are grown from which the fibers are extracted. The properties of natural fibers and polymers are completely different. The optimum proportion and proper blending of the constituents are the key factors which govern the properties of the biocomposites.
- In the case of unidirectional and bidirectional fiber-reinforced composites, the selection of appropriate orientation of fiber is a challenging task. During processing of short fiber-reinforced composites, the control of distribution and alignment of fibers in the direction of flow is a difficult task. Also, the damage and degradation of fibers in terms of attrition, bending, and burning during mechanical compounding and processing of biocomposite are difficult to control.
- The most severe problem associated with natural fibers is their hydrophilic nature. Generally, the interfacial bonding between the hydrophilic natural fibers and the hydrophobic polymer matrix is poor. There are several methods which can be used to improve the interfacial bonding between the fibers and matrix. The bonding can be improved by physical as well as enzymatic and chemical treatment of the fibers. The addition of fillers, compatibilizer, additives, and catalyst to the matrix can also be done for the improvement of interfacial bonding.
- Compared to traditional materials, the constituents of polymer composites are entirely different. Hence, the machines and tooling requirements are completely

different from that for traditional materials. The properties of constituents (reinforcement and matrix) are entirely different with each other. Hence, the tooling and operating parameters for the matrix may not be suitable for the reinforcement or vice versa.

Hence, the judicious selection of appropriate processing technique is required to achieve the exceptional properties in the biocomposites.

3 Selection of Processing Techniques

The ideal processing technique should be able to convert the input materials to the tangible product according to the desired shape, size, and properties without any defect. The selection of appropriate processing technique for the processing of biocomposite depends on:

- The properties of the biocomposites
- Shape and size of the biocomposites
- Properties of the matrix and reinforcing materials
- Manufacturing cost.

Biocomposites are tailor-made materials, and these can be manufactured as per the required properties by varying the weight percentage of the reinforcement, by the addition of additives and compatibilizer into the polymer matrix. The selection of appropriate processing technique depends upon the size, shape, and the orientation of the fibers in the developed composites. The properties, size, and shape of the desired biocomposite components also play a crucial role in selecting the processing technique. For the fabrication of large-size biocomposite components, usually open mold processes such as hand layup and spray layup are preferred while the closed mold processes such as compression and injection molding are preferred for the fabrication of small-to-medium-size biocomposite components. The intricacy of the design of a component also plays a crucial role in selecting the processing technique. Usually, complex components which require close tolerance, accuracy, and precision are made by injection molding process. During processing of biocomposites, the polymers and natural fibers may be subjected to high temperature and pressure; both the polymers and natural fibers have the tendency to degrade at elevated temperatures. Therefore, the appropriate raw materials and manufacturing technique should be selected according to the processing requirement and desired properties of the biocomposite product.

4 Lignocellulosic Polymer Composites: Processing Techniques

The primary processing techniques can be broadly categorized as open mold and closed mold processes. Hand layup, spray up, and filament winding are open mold processes while compression molding, injection molding, and resin transfer molding are closed mold processes. Some of these processes are discussed in the following sections:

4.1 Hand Layup

The hand layup (Fig. 4) is a simple and most widely used technique for the processing of thermoset-based polymer composites. In this technique, there are two molds: a top mold and a bottom mold. The mold release gel is sprayed on top of the bottom mold, or thin polyester sheets can also be used to get the good surface finish and for ease of removal. The thermosetting resin is mixed thoroughly with a suitable proportion of hardener to get the desired matrix in the form of resin. A layer of the matrix is coated on the mold, and then, reinforcement in the form of woven mat or chopped strand mat is placed over it. The number of layers for stacking depends upon the desired thickness of the composite laminate. A cylindrical roller can be used to initially press the layers to remove any air entrapment while fabricating the composites and the excessive resins. Then, the second mold plate is placed over the stacked layers, and the load is applied on top of the mold assembly, left for the curing. The curing time of the composite mainly depends on the type of matrix. Sometime, a catalyst can also be used to accelerate the curing of the composites. The curing can be done at room or at elevated temperature depending upon the requirement. After the curing, the mold is opened and the composite plate/laminate is taken out and processed further.

4.2 Compression Molding

Compression molding (Fig. 5) is the most common and one of the oldest techniques for the fabrication of composite parts. It is a high-pressure closed mold process. It can be used for the processing of both thermoplastic- and thermosetting-based composite products. The reinforcement in the variety of forms such as unidirectional, bidirectional, mat of randomly oriented fibers and short fibers can be easily used. Two matched plate-type metal molds are used to fabricate the composite product: the upper mold and the lower mold; the lower mold is generally fixed while the upper mold is movable. The reinforcement and the matrix are kept in between the metallic molds of the desired shape at elevated temperature and pressure. The molds are

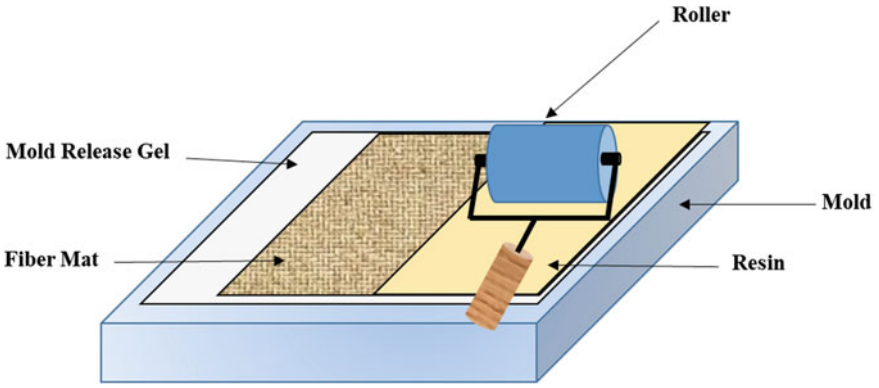

Fig. 4 Hand layup

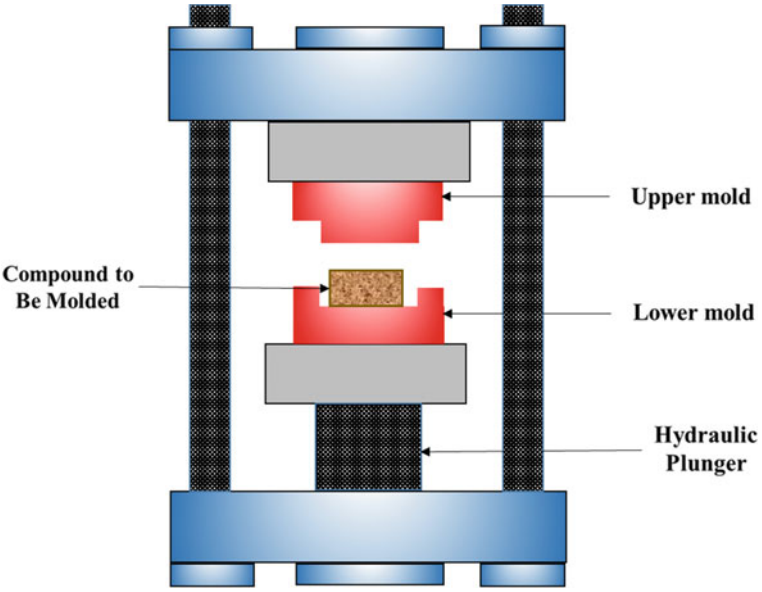

Fig. 5 Compression molding machine

kept in closed position for the predefined period of time as per the requirement. The material placed in between the upper and lower molds takes the shape of the mold due to the application of pressure and heat. The shape, size, accuracy, and design of the composite products depend upon the mold design. The polymerization can be done either at room temperature or at elevated temperature as per the processing guidelines of the materials. After polymerization, the molds are opened and the composite product is removed for further processing. The most important processing parameters which need to be controlled to get good-quality composite products are:

- Mold heating rate
- Mold cooling rate
- Applied pressure
- Compression rate
- Curing time.

4.3 Injection Molding

It is one of the most widely used closed mold processing techniques, and it is applicable for all types of polymers and their composites. Generally, the reinforcement in the form of short fibers is used. In the case of extrusion–injection molding, the short fibers and polymer are fed into the hopper of the extrusion machine. The mixture of polymer pellets and fibers goes into the barrel where the melting of the polymeric material takes place due to heating. The heat is generated due to direct heating from the heating elements and due to the shearing action of materials inside the barrel. The rotation of screw facilitates the mixing of fiber with the melted polymers. The molten compound is then forced through the opening of the die. Thus, the output in the form of composite strands is obtained; this composite strand is passed through the water bath for cooling. After cooling, the composite strand is converted into the composite pellets by pelletizing. The composite pellets are then dried in the oven at suitable temperature prior to injection molding machine. These composite pellets are used as a raw material and fed into the hopper of the injection molding. As the pellets go into the barrel, softening of materials takes place due to heating. As the screw rotates, mixing of pre-blended fibers and polymers takes place and at the same time, the melted composite compound is forced toward the converging section of the barrel. At the end of the barrel, the nozzle is connected which is used to inject the material into the mold cavity with high pressure. The cooling or heating arrangement can be made to control the temperature of the mold; it can be air cooled or water cooled. The cooling rate is an important process parameter which can be optimized to get the desired properties of the product. The clamping unit is provided to clamp the mold together under high pressure to prevent the defects during injection. Once the curing is completed, the composite part is taken out with the help of ejector pins. The schematic of the injection molding machine is shown in Fig. 6.

In the direct injection molding, the short natural fibers and polymer pellets are mixed manually and fed directly into the hopper of the injection molding machine. After melting and mixing, the blend is injected into the mold cavity with high pressure. After cooling, the composite product is taken out. In order to avoid the damage of fiber due to high temperature and shearing action, sometimes an additional hopper can be provided to feed the natural fibers near the injection end of the screw. During injection molding, the major operating parameters are:

- Speed of the screw
- Injection pressure

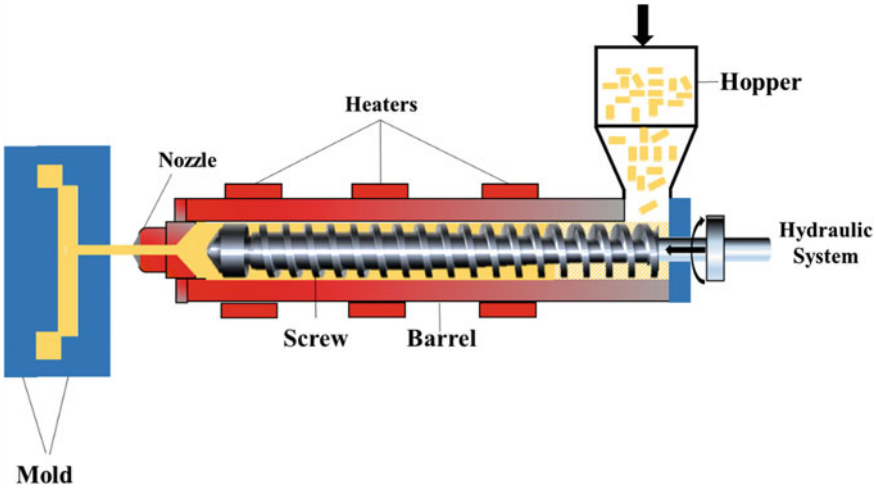

Fig. 6 Injection molding machine

- Injection speed
- Temperature profile
- Holding pressure
- Cooling rate.

Injection molding produces composite parts with high accuracy in desired size and shape. It is mainly suitable and profitable for mass production of identical products in larger volume.

5 Primary Processing of Lignocellulosic Polymer Composites: Opportunities

The preliminary investigations in the area of lignocellulosic polymer composites have established themselves as a potential candidate for replacing the synthetic fiber-reinforced composites. A brief summary of the area of applications and research directions in the field of lignocellulosic polymer composites is presented in the following discussion.

The behavior of magnesium-hydroxide-impregnated natural fiber-reinforced composites was compared with automotive glass fiber sheet molding compound. It was reported that the natural fiber-based composites had great potential to be used as a replacement for glass fiber sheet molding compound for automotive applications [9]. In another work, the mechanical properties of the flax fiber-reinforced poly-lactic acid (PLA) composites were compared with automotive flax fiber-reinforced polypropylene composites. It was reported that the strength of the PLA-based com-

posites was found to be 50% higher as compared to flax fiber-reinforced polypropylene composites. Also an increase in the stiffness from 3.4 to 8.4 GPa was observed in the composites [10]. In another work, the mechanical behavior of kenaf fiber-based PLA composites was studied. It was concluded that the tensile and flexural strength increases linearly when fiber loading is increased up to 50%, which proved that kenaf fiber exhibits higher mechanical properties as compared to other natural fibers, when used as reinforcement in PLA matrix [11]. The researchers also investigated the feasibility of using recycled fiber from disposable chopsticks as reinforcement with PLA matrix to fabricate green composites by the melt-mixing process. It was reported that the tensile strength of the composites increases with an increase in fiber loading. At 40% of fiber loading, the improvement was reported as 3 times higher than the pure PLA [12]. Studies show that natural fibers such as cotton, jute, flax, and kenaf have the ability to reinforce the thermoplastic- and thermosetting-based composites and the resulting composites can be used for numerous engineering applications [13].

6 Secondary Processing of Lignocellulosic Polymer Composites: Issues and Challenges

The primary processing techniques for the manufacturing of polymer-based composites have been discussed in the previous section. Though the most of the polymer-based composite components are manufactured to a near net shape, in order to manufacture the products having the complex geometry for certain applications, several composite components have to be joined together to get the final product. The techniques for joining of components made of polymer composites are entirely different from those of traditional materials. The joining of polymer composites has always been a challenging task among the researchers and manufacturing engineers. The variety of joining techniques is now available for the joining of polymer-based composites. Some of these are adhesive bonding, mechanical fastening, and microwave joining. Each technique has its own advantages and limitations. The adhesive bonding technique cannot be used where the temporary joining of components is required. Generally, the novel microwave joining is also limited to thermoplastic-based composite parts. All these problems can be overcome by adopting the mechanical fastening for the joining of composite parts. For mechanical fastening, certain machining operations like drilling, trimming, finishing are required. Among these machining processes, drilling is the most commonly and frequently used method for making holes in order to assemble the composite parts.

Although composite materials offer numerous advantages over traditional materials, their inhomogeneous structure makes the drilling of these materials difficult. Therefore, the drilling techniques of polymer composites have now become the major area of research. There are several issues and challenges involved in the drilling of composite materials; some crucial observations are discussed here:

- For mechanical fastening, the drilling of composite parts is required. It has been reported that the drilling of parts made of polymer composite by traditional techniques is a highly challenging task [14]. It was reported that the most of the manufactured composite components are rejected by manufacturing industry, due to the drilling-induced damage generated around the drilled holes.
- This damage is mainly caused by the heat generated during the interaction of the drilling tool and the composite parts. The thrust force, torque, and sometimes the poor interfacial bonding between the fibers and polymers were observed as the major causes for the drilling-induced damage.
- Drilling-induced damage has now become the prime area of research in the field of machining of composites. The damage can be in the form of delamination, fiber pullout, and hole ovality around the drilled hole. This damage is responsible for the failure of the composite parts during their service life.
- Delamination in composite laminates is basically the separation of layers. It creates potential points of origin for failure under loading. There are two types of delamination; peel-up delamination and pushdown delamination. Peel-up delamination generally occurs when the cutting edge comes in contact with the composite laminates, the separation and bending of layers take place resulting into the fracture of the composite laminate. Peel-up delamination is generally observed at the entry of the drilled hole.
- When the drill approaches toward the exit point, the drill point employs compressive force on the uncut layers, resulting into bending of these layers. This leads to fracture of material under the drill point. The damage around the drilled hole at the exit side is termed as pushdown delamination.
- Another major challenge in the drilling of composite parts is fiber pullout; this type of damage also deteriorates the quality of drilled holes. The poor adhesion between the fibers and matrix is responsible for such type of damage.
- During drilling, when the orientation of reinforcement is unidirectional and bidirectional, then the cutting angle varies with the rotation of the drill. This results in the form of an inaccurate hole in terms of circularity. This is generally termed as the hole ovality.

7 Drilling of Lignocellulosic Polymer Composites

The drilling of lignocellulosic polymer composites is entirely different from those of conventional materials. In order to meet the specific requirement (such as bolted and riveted joints) for structural applications, the drilling of a hole in composite part is required. The drilling can be done by different techniques. Usually, the drilling is performed on the typical drilling machine. Figure 7 shows the schematic of a traditional drilling setup; the setup for drilling mainly depends on the outputs required. In order to measure the thrust force and torque generated during the drilling operation, the dynamometer can be used, and an amplifier can be used to amplify the signal

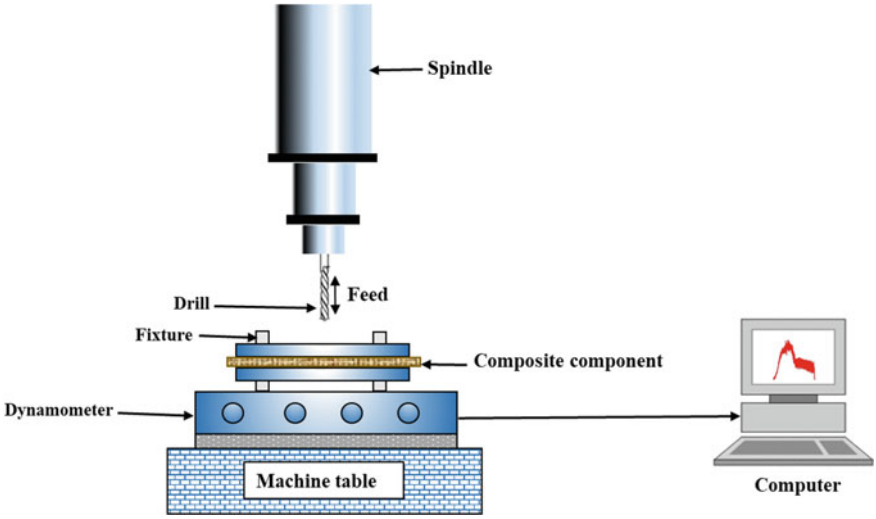

Fig. 7 Schematic of traditional drilling setup

received from dynamometer. The data acquisition system and specific software are required to analyze the generated thrust force and torque.

8 Secondary Processing of Lignocellulosic Polymer Composites: Opportunities

The drilling techniques and drilling-induced damage of the parts made of polymer composites have now become the major areas of research. In order to get the damage-free hole, there are important process parameters which need to be optimized. These process parameters are shown in Fig. 8.

The damage of holes can be reduced by optimizing the process parameters as well as drill point geometry. The thrust force, torque, and heat generated during drilling operation mainly depend on these parameters.

For drilling operation, a variety of modified drill point geometries were developed and the effect of these geometries on damage was investigated. Drilling behavior of nettle fiber-reinforced polypropylene composites was investigated. The effect of three drill geometries (4-facet, step, and parabolic) on the drilling behavior was investigated. In case of 4-facet and step drill, the thrust force increases linearly with feed while the nonlinear increase was observed for the parabolic drill. It was also reported that the torque increases linearly with feed for all three types of drill geometries. As compared to 4-facet and step drill, the thrust force and torque generated with the parabolic drill were found to be lower [14]. The same author has conducted the similar type of investigation on sisal fiber-reinforced epoxy laminates and sisal fiber-

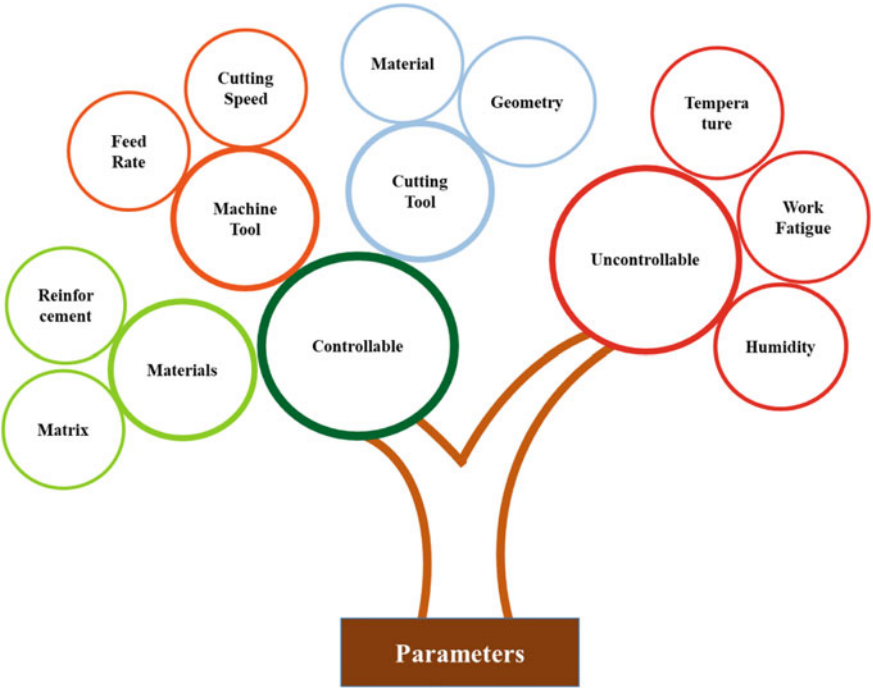

Fig. 8 Process parameters affecting the drilling of composite parts

reinforced polypropylene laminates. It was concluded that, for parabolic drill, the thrust force and torque were found to be higher for sisal epoxy composite laminate. Whereas, in the case of step drill, higher thrust force was reported for sisal polypropylene composite laminates [15]. In another investigation, the drilling behavior of jute and polypropylene-based composite laminates with three different drill geometries (parabolic drill, jo-drill and twist drill) was investigated. The cutting behavior was reported when the drilling was conducted with the parabolic drill. Both types of delamination (peel-up and pushdown) were observed during the investigation. The thrust force was found to be the main reason for the delamination [16]. In the similar type of experiments on sisal fiber-reinforced polypropylene composites, two different drills (twist and trepanning) were used. Visual inspection confirms the better cutting behavior, and damage-free holes were generated with trepanning tool. The thrust force was found to be lower in case of trepanning tool, but the torque was reported higher for the same [17]. The effect of various drill tool materials on the thrust force during vibration drilling of fiber-reinforced plastics was also conducted. The study recommended that carbide drill is appropriate for drilling of fiber-reinforced plastics. Also, the thrust force generated with carbide drill is lower than the high-speed steel (HSS) drills [18].

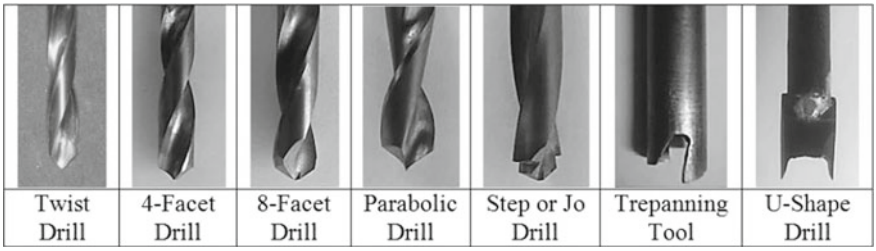

Fig. 9 Drill point geometries [19]

Figure 9 depicts the different drill point geometries which are specifically designed for the drilling of polymer-based composites. These drills have been developed to minimize the drilling-induced damage during the drilling operation.

In order to minimize the drilling-induced damage, apart from the drill geometries, the researchers have attempted modifications in the existing methods of drilling. Some of these methods are backup plate, helical method, and ultrasonic-assisted drilling. A researcher has suggested the use of the helical method of drilling. In this method, the drill moves helically with respect to drilling axis instead of straight movement in traditional drilling. The decrease in thrust force, drilling-induced damage, better chip removal, and flow of coolant are some of the advantages associated with the modified method [20]. Another innovative drilling approach suggested is drilling by the ultrasonic mechanism which is called as ultrasonic-assisted drilling. In this approach, the ultrasonic vibration can be applied to the tool or workpiece. In a study, the ultrasonic vibration was applied to the workpiece. As the outcome, the reduction in thrust force, torque, and burr formation was reported. The better quality holes with improved material removal rate and chip removal were also reported [21].

9 Conclusions

With growing concern for the limited petroleum resources, environment, and ecosystem, there is now a challenge for researchers and engineers to develop the sustainable and environmentally friendly composite materials. The most of the investigation available on biocomposites has shown that the biocomposites have the huge potential to replace the synthetic fiber-reinforced polymer composites in many engineering applications. The use of biocomposites is increasing rapidly ranging from automobile to household applications. As the demand is expected to increase further; rapid, easy, economical, precise, and accurate processing techniques are required for the fabrication of biocomposites. The present chapter highlights the fundamental issues, the challenges, and the existing opportunities which can help in formulating a road map for research and development in the field of primary and secondary processing of biocomposites.

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Chapter 3
Feasibility Study of Fly Ash as Filler in Banana Fiber-Reinforced Hybrid Composites

N. Venkateshwaran, V. Santhanam and A. Alavudeen

Abstract Natural fiber-reinforced biodegradable composites have evolved as excellent alternatives to glass fiber-reinforced plastic (GFRP) materials. Due to improved physical and mechanical properties, they have the potential for a wide range of applications. Moreover, natural fibers are cheaper in cost, environmentally friendly, and biodegradable. The properties of some natural fibers have been investigated, and the reported results showed the promising utilization of them as an alternative to glass fiber in many applications. This work is to study the practical use of fly ash as a filler material in banana fiber-reinforced polyester (BFRP) composite. The fly ash in various percentages (5, 10, 15, and 20) was added in the composites and tested for mechanical properties like tensile, flexural, impact viscoelasticity, chemical resistance, density, and water absorption properties. The effect of fly ash content on the fiber–matrix interface was studied using scanning electron microscope. From the results, it is found that the addition of fly ash as filler material improves the mechanical properties and decreases the moisture absorption resistance of banana fiber-reinforced polyester composites.

Keywords Natural fibers · Composite · Polyester · Banana fiber · Fly ash

1 Introduction

The research in the field of polymer reinforced composites has gained widespread recognition owing to their appealing strength–weight property; however, their biodegradability is still a matter of concern. Further, they can also be engineered

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P. K. Rakesh and I. Singh (eds.), *Processing of Green Composites*, Materials Horizons: From Nature to Nanomaterials, https://doi.org/10.1007/978-981-13-6019-0_3

to get the desired properties [1]. Many types of natural fibers were investigated for use in polymer resins including flax, hemp, jute, straw, wood fiber, bagasse, bamboo, sisal, coir, etc. Natural fibers have the advantage that they are renewable resources and have marketing appeal. Especially in India, natural fibers have been in use for many years, e.g., jute. For the past decade, natural fibers, as reinforcement, have attracted the attention of researchers, because of their advantages over other established reinforcing materials. The main reason for the use of plant fibers as reinforcement is their biodegradability, which contributes to a better ecosystem, along with low cost and acceptable properties [2]. Although natural fibers and their composites are environmentally friendly and renewable, they have the disadvantages of poor wettability, incompatibility with some polymeric matrices, and high moisture absorption capability [3]. Joshi et al. [4] reviewed the life cycle assessment of natural fiber and glass fiber-reinforced composites and found that natural fibers are environmentally superior to glass fiber, and also natural fibers reduce the polymer content as reinforcement. Wotzel et al. [5] concluded that hemp fiber-reinforced epoxy composite will replace glass fiber acrylonitrile butadiene–styrene (ABS) for usage in auto-side panels.

Venkateshwaran et al. [6] studied the effect of fiber parameters on mechanical properties of banana fiber-reinforced composites. The results showed that maximum tensile strength and modulus is obtained when fiber length is 15 mm and weight ratio of 12%.

Pothan et al. [7] studied the effect of fiber length and content on the mechanical properties of the short banana/polyester composites. A study shows that 30–40 mm fiber length and 40% fiber loading provides better mechanical properties. Idicula et al. [8] investigated the mechanical performance of banana/sisal hybrid composite, and the positive hybrid effect for tensile strength was found in the ratio of 4:1 (banana:sisal). Further, the tensile strength of the composite is better when banana fiber is used as skin and sisal as a core material. Venkateshwaran et al. [9] observation on mechanical and water absorption behavior of banana–sisal fiber epoxy composite showed that addition of sisal fiber in banana/epoxy composites up to 50% by weight results in increasing the mechanical properties and decreasing the moisture absorption property of the hybrid composites. Pothan et al. [10] concluded that the hybridization of banana fiber with glass fiber will affect the dynamic mechanical response of the composites. They also revealed that an intimate mixture of banana and glass fibers or layering pattern with an even distribution of both the fibers will give better properties. Pothan and Thomas [11] studied the effect of various chemical treatments on glass and banana fiber hybrid composites. The results showed that the fibers treated with silane A1100 have the lowest water uptake capability. The prediction of the optimum percentage of alkali (NaOH) treatment on banana fiber was carried out by Venkateshwaran et al. [12]. The results showed that 1% NaOH-treated fiber-reinforced composites behave superiorly than other percentages of treated and untreated fiber composites. Sapuan and Maleque [13] designed and fabricated the household telephone stand using woven banana fabric and epoxy resin. Tensile test result of woven banana fiber-reinforced epoxy showed that the woven type of reinforcement has better strength, and the results were compared using ANOVA technique [14]. Zainudin et al. [15] studied the thermal stability of

banana pseudo-stem (BPS)-filled unplasticized polyvinyl chloride (UPVC) composites using thermogravimetric analysis. The study revealed that the incorporation of banana fiber decreases the thermal stability of the composites. Zainudin et al. [16] investigated the effect of banana filler content in the UPVC matrix. The insertion of filler increases the modulus of the composite and not the tensile and flexural strength. Singh et al. [17] developed the banana fiber- and silica powder-reinforced composite materials. The observation using a scanning electron microscope showed that banana fibers and silica powder are well dispersed in the epoxy resin. The investigation also found that the addition of fiber increases the modulus of elasticity but decreases the ultimate tensile strength of the epoxy resin. Chaowasakoo and Sombatsompop [18] evaluated the mechanical and morphological properties of fly ash/epoxy composites cured by thermal and microwave methods. The study suggested that higher tensile and flexural properties will be achieved by thermal curing method when compared with microwave curing. Guimaraes et al. [19] carried out X-ray powder diffraction and thermal analysis study on starch–banana composites. Thermal analysis showed excellent thermal stability, while fractography studies show that the sample is ductile. Garbacz and Sokolowska [20] carried out a work based on polymer concrete containing fly ashes of various types, origin, combustion technology, and chemical composition. Different mechanical properties (such as compressive strength, flexural strength, and tensile strength) and chemical resistance were analyzed. It was found that the use of fly ash has reduced the cost of polymer concrete without compromising the properties. Srivatsava and Pawar [21] analyzed the effects of fly ash filler, impingement angle, and particle velocity on the solid particle erosion behavior of glass fiber-reinforced epoxy (GFRP) composites. The investigation revealed that composites with fly ash filler have the lowest wear due to the resistance offered by the filler against fiber–matrix debonding. Manoj and Vikas [22] studied the compressive and impact strength of fly ash-filled epoxy composites. The result shows that the addition of fly ash increases the mechanical properties due to the hollowness of fly ash particle and better interfacial adhesion. Gu et al. [23] investigated the damping properties of various percentages of fly ash-filled low-density epoxy composites. The results show that the damping property of 30–50% fly ash-filled composite is better than that for other volume fractions. From the above literature, it is found that banana fiber and fly ash were used as reinforcement and filler particles separately with the polymer matrix to form composites with improved mechanical properties. Hence, in this work, banana fiber and fly ash at various percentages are used, and the effect of fly ash on mechanical properties of banana fiber-reinforced polyester composite was investigated.

2 Materials and Methods

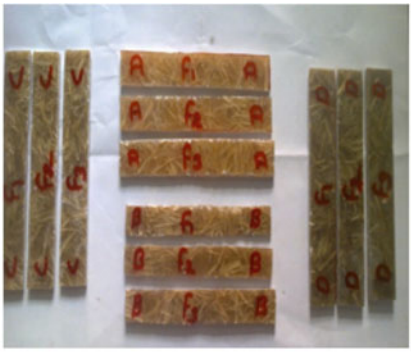
Banana fiber, fly ash, and polyester with hardener are chosen as the materials for this research work. Initially, fiber lengths of 15 mm along with fiber weight of 25 wt% and the weight percent of fly ash of 5, 10, 15, and 20 were chosen for the fabrication

of the composites using hand layup method. Composite samples are designated as follows:

A: 100% banana fiber-reinforced polyester composite.
B: 95% banana fiber- and 5% fly ash-reinforced polyester composite.
C: 90% banana fiber- and 10% fly ash-reinforced polyester composite.
D: 85% banana fiber- and 15% fly ash-reinforced polyester composite.
E: 80% banana fiber- and 20% fly ash-reinforced polyester composite.

Mechanical properties—tensile strength, flexural strength, impact strength, and water absorption behavior—of the developed composites were tested as per the ASTM D3039, D790, D256, and D570, and chemical resistance tests (ASTM D543), respectively. The fractography study of the composite was carried out using the scanning electron microscope to analyze the effect of fly ash on fiber–matrix bonding. Various samples cut as per ASTM standards are shown in Fig. 1.

(a) Tensile test specimens



(b) Flexural test specimen

(C) Water absorption test specimen

(d) Water absorption and acid test specimen

Fig. 1 Various test specimens

3 Results and Discussion

3.1 Tensile Testing

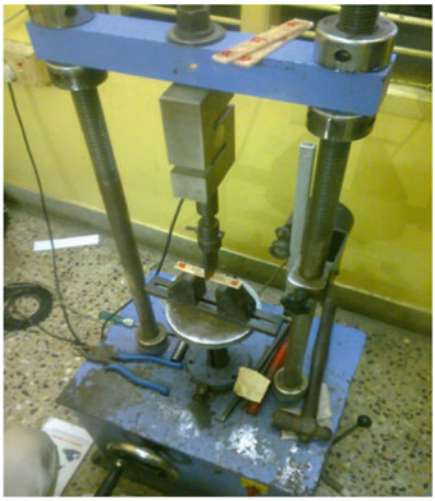
Tensile testing of the developed composites was carried out as per the ASTM 3039 standard with the test speed of 2 mm/min using computerized UTM as shown in Fig. 2a. Five specimens were tested to obtain the average value. The results of tensile tests are shown in Fig. 3. For specimen A, i.e., without fly ash, tensile strength is 14 MPa. For specimen B, i.e., with 5% fly ash, the tensile strength slightly increases to 14.55 MPa. For specimen C, 10% fly ash, tensile strength increases to 15.1 MPa. For specimen D, 15% fly ash, tensile strength further increases to 18 MPa. For specimen E, with 20% fly ash, the tensile strength starts to decrease to 17.2 MPa. Similarly, Fig. 3 clearly shows that the tensile modulus starts with 0.536 GPa and continuously increases 0.628, 0.661, and 0.712 for 5, 10, and 15% fly ash content. Further, the addition of fly ash content (20%) results in decreasing the modulus value to 0.68 GPa. Hence, the maximum fly ash content to be used along with banana fiber is 15% by weight of total reinforcement. This decrease in the value is due to the agglomeration of filler particle which reduces the interfacial adhesion between polymer and reinforcement. The significant amount of addition of filler also creates crack on the matrix surface which tends to propagate at the higher rate and causes early failure.

3.2 Flexural Testing

Flexural properties of the composites were tested under three-point bending mode, as per the ASTM D790 standard using the computerized UTM as shown in Fig. 2b. In each case, five specimens were tested, and the average value is reported.

From Fig. 4, it is clear that by adding the fly ash, flexural properties were improved. For specimen A, i.e., without fly ash, flexural strength is 45.67 MPa. For specimen B, with 5% fly ash, the flexural strength increases to 53.28 MPa. For specimen C, i.e., with 10% fly ash, flexural strength increases to 59.37 MPa. For specimen D, 15% fly ash, flexural strength increases to 72.3 MPa. For specimen E, i.e., with 20% fly ash, the flexural strength decreases to 62.11 MPa. Also, Fig. 4 shows the flexural modulus variation for various composites. For specimens A, B, C, D, and E, the flexural moduli are 4.806, 5.986, 6.67, 8.123, and 7.307 GPa, respectively. The flexural properties continuously increase with the addition of fly ash content up to 15 wt% of reinforcement. Further addition (20%) of fly ash results in decreasing the flexural properties of the composite. The decrease in the flexural property is due to the fact that in three-point bending, the accumulated filler material comes out from the interface and causes damage to the fibers which in turn decrease the load carrying capability of the composites.

(a) Specimen tested under tensile mode



(b) Specimen tested under bending mode.

(c) Equipment used for DMA test.

Fig. 2 Equipment used to obtain test data

3.3 Impact Testing

Impact testing of the composite was carried out in the Izod mode as per the ASTM D256 standard using the impact testing equipment. The results of the impact test are shown in Fig. 5. The impact strengths of the various composites, A, B, C, D, and E, are 16, 16.28, 17.6, 17.63, and 16.7 J/mm^2, respectively. The addition of fly ash continuously increases the impact strength of the composite from 16 to 17.63 J/mm^2. The

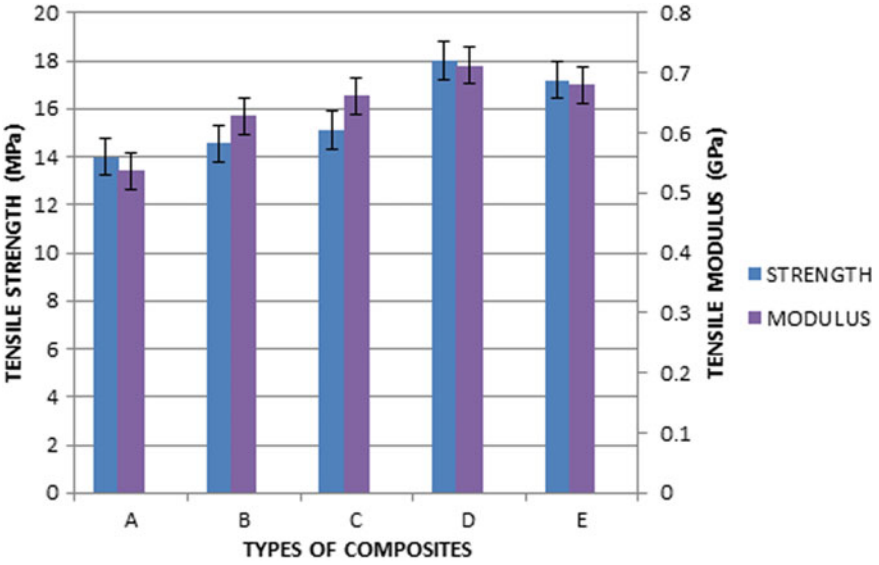


Fig. 3 Tensile strength and tensile modulus of various composite samples

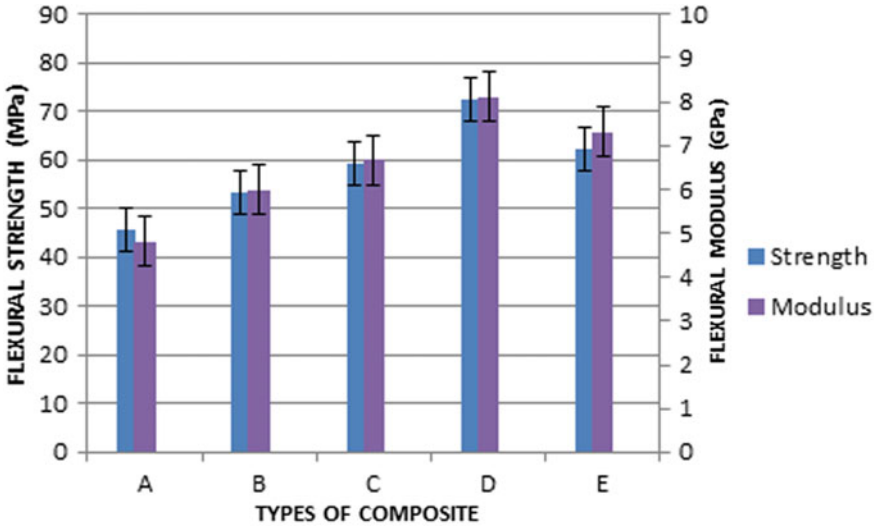


Fig. 4 Flexural strength and flexural modulus of various samples

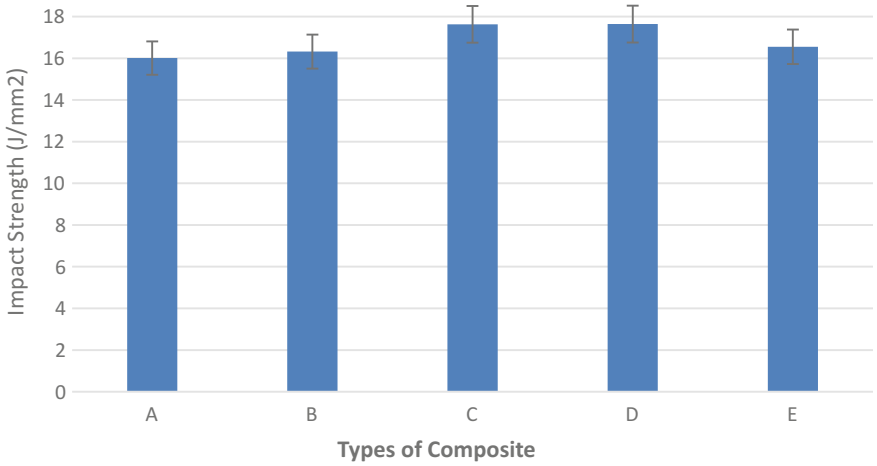


Fig. 5 Impact strength of various samples

variation in impact strength is not as predominant as tensile and flexural properties due to higher strain rate experienced during impact testing.

3.4 Density and Void

Density is the most important physical property of the composite materials. Composites are preferred due to their strength-to-weight ratio in most of the applications. The density of the composites was determined using a Mettler densimeter. In each case, four samples were tested, and the average value is tabulated. The theoretical density (ρ_T) was evaluated using Eq. (1). The difference in the experimental and theoretical density determines the void content.

$$\rho_T = \frac{100}{\left(\frac{R}{D}\right) + \frac{r}{d}} \tag{1}$$

where R is the weight percentage of fiber in the composite, D is the density of the fiber, r is the weight percentage of the matrix, and d is the density of the matrix.

The density values of banana fiber- and fly ash-reinforced polyester composite are presented in Table 1. It is clear that by adding the fly ash, density of the composite changes. The densities of the specimens A, B, C, D, and E are 1.24, 1.357, 1.396, 1.355, and 1.154 g/cc, respectively. Initially, the fly ash increases the density of the composite by 9 and 11% for 5 and 10% addition. Further addition by 15 and 20% shows the reverse trend. This is due to the decreasing percentage of banana fibers which are higher in density than fly ash. Further, Table 1 also shows the comparison of

Table 1 Percentage of void content

Composite sample	A	B	C	D	E
Exp. density (g/cc)	1.24	1.357	1.296	1.355	1.154
Theoretical density (g/cc)	1.261	1.373	1.311	1.368	1.163
Void (%)	2.1	1.6	1.5	1.3	0.9

experimental and theoretical density to measure the void presence in the composite fabricated. It shows that the addition of fly ash decreases the void content in the composite. According to the survey, the void content should be less than 3% for a good composite [24]. The lower void content indicates good bonding between matrix and reinforcement, which in turn improves the properties of the composites.

3.5 Water and Chemical Absorption Study

The moisture absorption rate of the composite was carried out as per ASTM D570. The specimens were initially weighed using a digital weighing machine of 1 mg accuracy. After initial weighing, the specimens were immersed in a container of water maintained at room temperature. At the end of every 24 h, the specimens were removed from the water one at a time, and their surfaces were wiped off with a dry cloth and weighed again. This procedure was repeated until equilibrium/saturation was reached. Figure 6 shows the percentage of water absorption of the composites at specified time intervals. Composite E has the maximum moisture absorption percentage of 6.79. Similarly, the composites D, C, B, and A have the maximum moisture absorption percentage of 6.3, 6.11, 5.45, and 5.269%, respectively. The minimum moisture absorption is 5.269% for the composite consisting of 100% banana fiber, i.e., composite A. The result shows that the addition of fly ash increases the moisture absorption rate of the composites. This is due to the fact that the fly ash has the strong affinity toward moisture. Almost for all the types of composite, the saturation state reaches after six days of exposure.

The chemical resistance of banana fiber- and fly ash-reinforced composite specimens was studied as per ASTM D 543-87 standard with 10% concentration of hydrochloric acid, sulfuric acid, and acetic acid. In each case, three samples were preweighed in a precision electronic balance of 0.001 mg accuracy, and the samples were immersed in the respective chemical reagents for 1 h. They were then removed, immediately washed in distilled water, and dried by pressing them on both sides with a filter paper at room temperature. The immersed samples were then reweighed, and the percentage of weight loss/gain was determined using Eq. (2).

$$\%\ \text{Weight loss or gain of the sample} = \frac{W_f - W_i}{W_i} * 100 \tag{2}$$

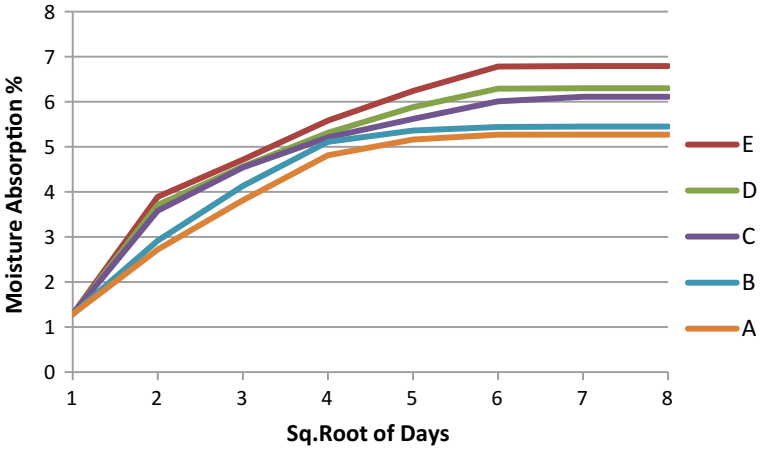


Fig. 6 Water absorption of composite sample

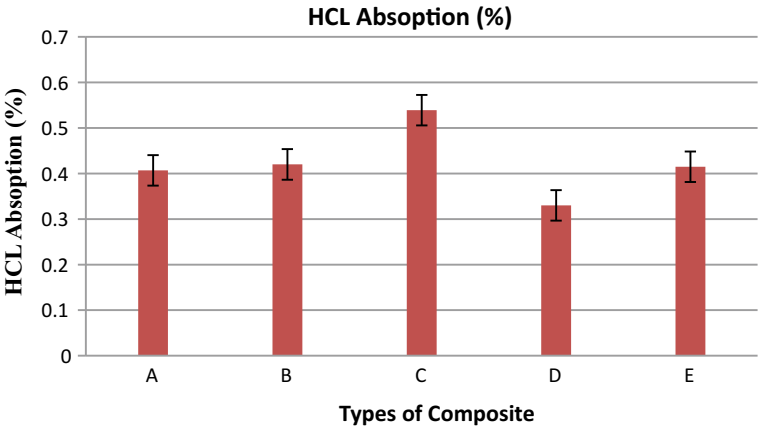


Fig. 7 HCl absorption of composite sample

where W_f = final weight and W_i = original weight.

Figure 7 shows the absorption effects of hydrochloric acid on the composites. Composite A has the absorption percentage of 0.407%. Similarly, the composites B, C, D, and E have the absorption percentage of 0.42, 0.539, 0.33, and 0.415%, respectively. It is clear that composite D is least (0.33%) affected by the hydrochloric acid when compared with other samples tested for four hours.

From Fig. 8, the absorption effects of sulfuric acid on the composites are shown. Composite A has the absorption percentage of 0.141%. Composites B, C, D, and E have the absorption percentage of 0.168, 0.266, 0.062, and 0.44%, respectively. Hence, it is clear that the composite D is the least (0.062%) affected by the sulfuric acid when compared with other samples tested for the four-hour duration.

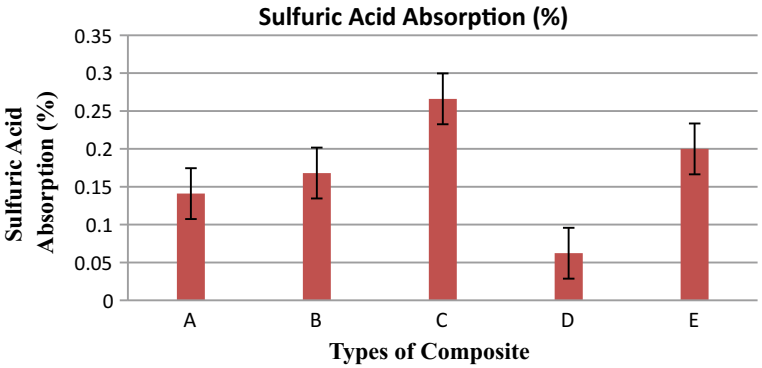


Fig. 8 Sulfuric acid absorption of composite sample

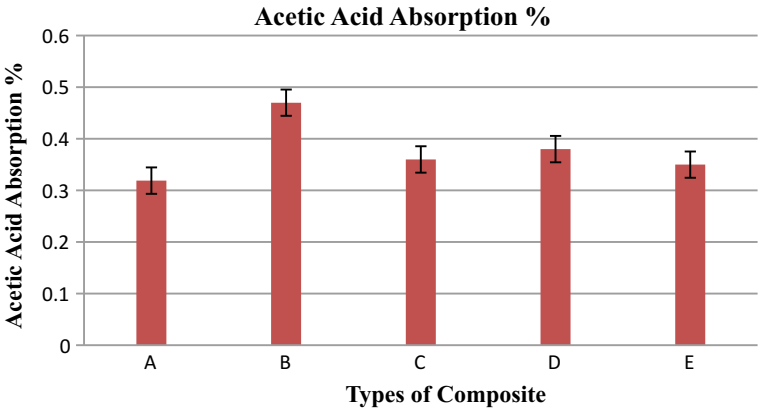


Fig. 9 Acetic acid absorption of composite sample

Figure 9 shows the absorption effects of acetic acid (CH_3COOH) by the composites. Composite A has the absorption percentage of 0.319%. For composite B, C, D, and E, the absorption percentages are 0.476, 0.366, 0.38, and 0.35%, respectively. It is clear that composite A is least (0.319%) affected by the acetic acid when compared with other samples tested for four hours.

3.6 Dynamic Mechanical Analysis

Dynamic mechanical analysis (DMA) yields information about the mechanical properties of a specimen subjected usually to sinusoidal, oscillation force as a function of time and temperature. Viscoelasticity refers to time and temperature dependence of mechanical behavior.

Figure 10a–c shows the results of storage modulus (E') evaluated at 0.5, 1, and 2 Hz, respectively. The storage modulus value refers to stiffness of the material. It indicates that the addition of filler increased the storage modulus values at all measured temperatures. This increase is due to better fiber/matrix adhesion and the greater degree of stress transfer at the interface [25]. Similarly, as the excitation frequency increases, the storage modulus decreases. This is because as the excitation frequency increases, time for the mobilization of molecules becomes lesser to cause localized stress concentration.

The effects of temperature and frequency on the damping property of the various composites are shown in Fig. 11a–c. Since damping is one of the critical parameters associated with the vibration characteristic of composites, its evaluation plays a vital role in application viewpoint. The damping property or tan δ value is the ratio of the amount of energy released (E'') to absorbed (E'). Figure 11a shows the damping property at 0.5 Hz frequency. At this frequency, the glass transition (T_g) of 20% addition of fly ash as filler shifts the T_g value from 80 to 90 °C. Similar observation of shift in T_g values occurs in other frequency (1 and 2 Hz) range also. Of these low frequencies tested, the maximum shift in glass transition temperature occurs at 2 Hz from the temperature of 80–100 °C. This shows that at this temperature the thermal expansion coefficient undergoes discontinuity and hence transition in T_g value occurs. This increase in thermal expansion is due to an increase in temperature and vibration of molecules. In Fig. 11a–c, after glass transition temperature, the modulus becomes almost constant in the rubbery plateau. In the rubbery plateau, polymers exhibit a long-range rubber elasticity, which means that the elastomers can be stretched further.

3.7 Fractography Study

The fracture surfaces of the specimens were used to analyze the fiber–matrix adhesion using a scanning electron microscope (SEM). The images of the samples were scanned with a beam of electrons in a raster scan pattern. The electrons interact with the atoms that make up the sample producing signals that contain information about the sample's surface topography, composition, and other properties. Since SEM utilizes the vacuum condition and electrons to form an image, special preparations must be done to the sample for analysis.

Figure 12a, b shows that the fiber bundles are completely removed from the matrix during fracture of 100% banana fiber composite. This is attributed due to weak interface adhesion between fiber and matrix as it was evident in Fig. 12b.

Figure 13a, b shows the SEM image of 10% fly ash-filled banana fiber-reinforced polyester composite. It shows that the closer presence of fly ash improves the bonding between reinforcement and matrix and hence improves the mechanical properties of the composite. Figure 14 shows the agglomeration or cluster formation of fibers takes place when 20% fly ash by weight was added as the filler in BFRP composites.

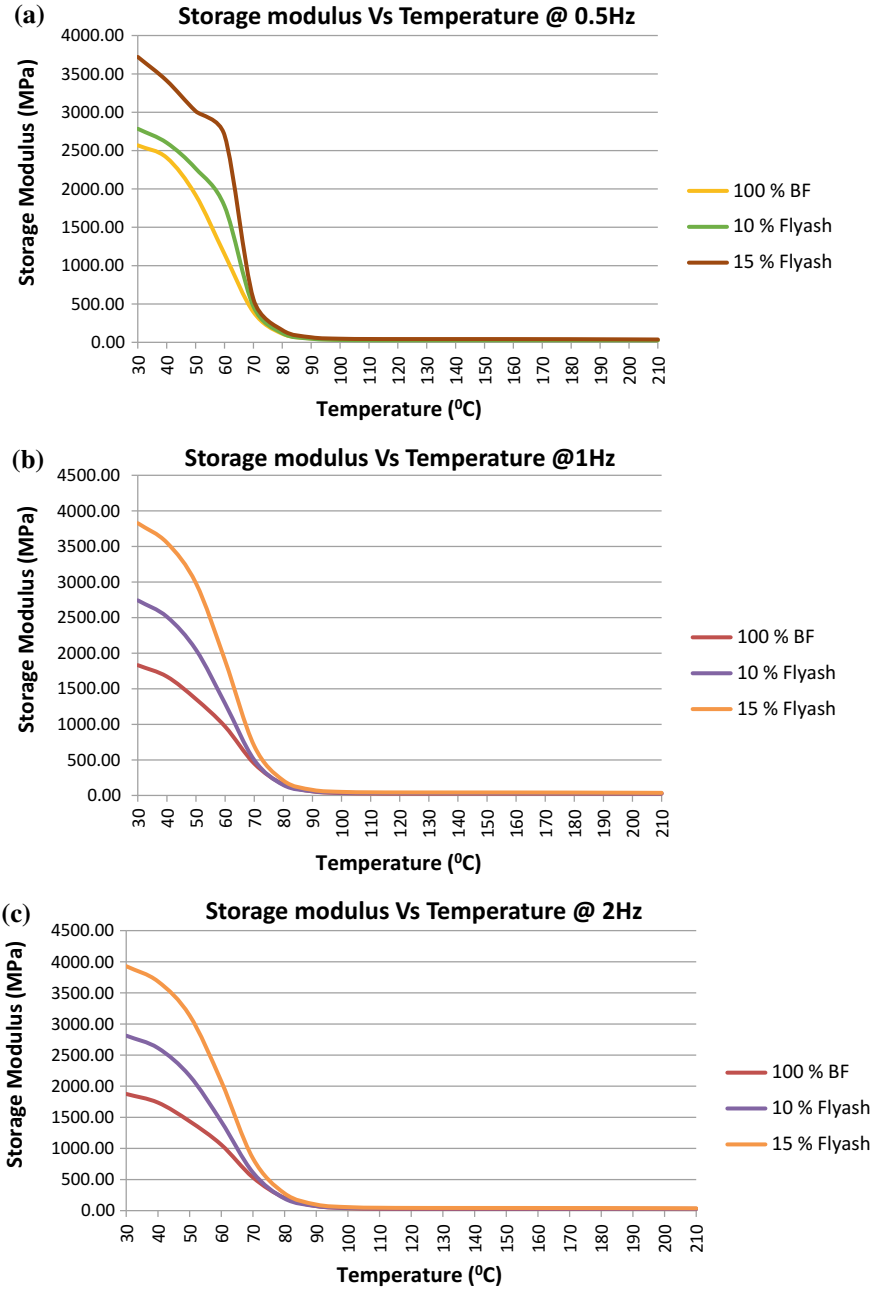


Fig. 10 Effect of temperature on storage modulus at **a** 0.5 Hz, **b** 1 Hz, **c** 2 Hz frequency

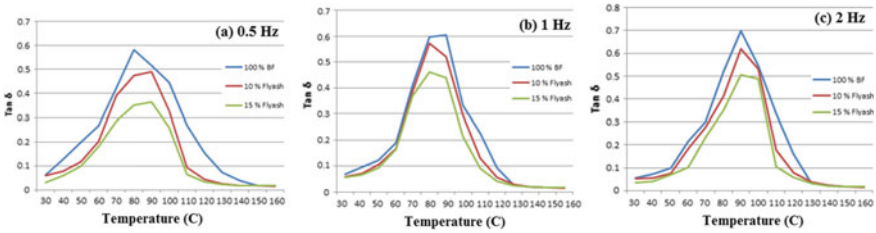


Fig. 11 Effect of temperature on damping factor at **a** 0.5 Hz, **b** 1 Hz, **c** 2 Hz frequency

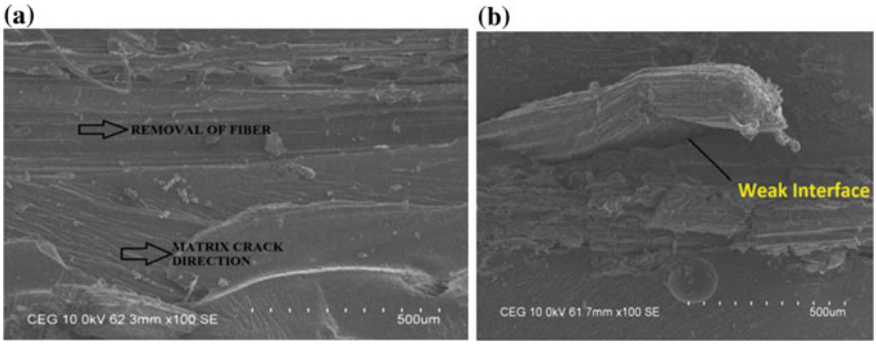


Fig. 12 SEM micrograph of the fracture surface of 100% banana fiber-reinforced composite

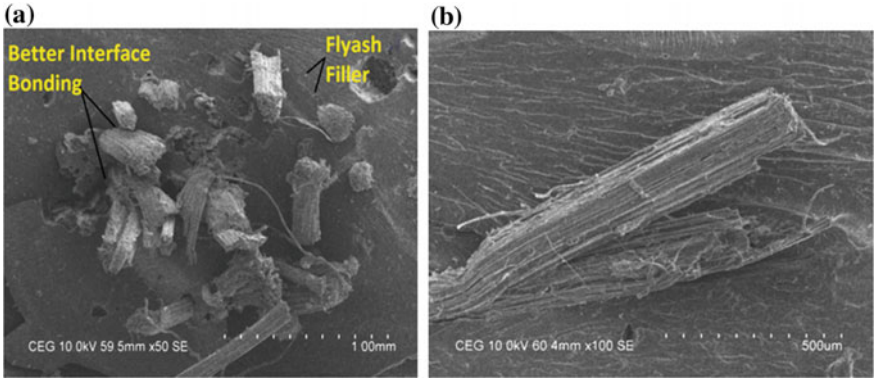


Fig. 13 SEM micrograph of the fracture surface of 15% fly ash-filled banana fiber composite

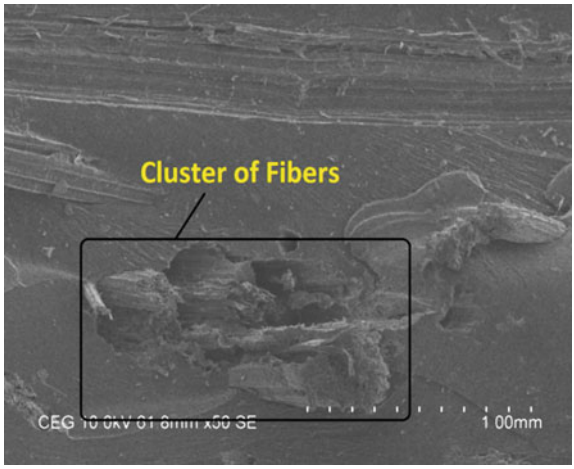


Fig. 14 SEM micrograph of the fracture surface for the 20% fly ash-filled banana fiber-reinforced composite

4 Conclusions

Based on the findings of this investigation, the following conclusions can be drawn:

1. The composite of 80% banana fiber and 15% fly ash results in the maximum tensile, flexural, and impact properties.
2. The minimum moisture absorption for the composite is around 5.269% for the composite consisting of 100% banana fibers by weight. The addition of filler increases the moisture absorption due to its hydrophilic nature.
3. The density of all the composites prepared is almost the same and is around 1.5 g/cc.
4. From the chemical resistance test, it is found that composite D is least affected by hydrochloric acid and sulfuric acid, whereas composite A is least affected by acetic acid.
5. From DMA analysis, it is clear that damping property for 15% fly ash-filled banana fiber composite is better than other composites. It also aids in shifting the glass transition temperature as the frequency surges.
6. The SEM analysis shows that the addition of fly ash enhanced the bonding between fiber and matrix and thereby attributed to the improved mechanical behavior.

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Chapter 4
Hygrothermal Aging Behavior of Fiber-Reinforced Composites

Shishir Kumar Sahu and Manoj Kumar Rath

Abstract Composite materials are ideal for engineering applications such as aircraft, spacecraft, automobiles, ship structures, roofs, wall panels, fishing rods, tennis rackets, and even retrofitting of buildings and bridges due to their high strength-to-weight and stiffness-to-weight ratios. The environmental hygrothermal conditions significantly affect the strength and stiffness of composites. Hygrothermal aging occurs due to temperature and moisture. In particular, deflection and stresses are significantly affected by environmental factors. Thus, it is necessary to consider such environmental factors in the computation of stresses and manufacture of structures. Hence, the effects of hygrothermal factors on the static characteristics of composites are of practical interest and technical significance. The present study deals with the experimental investigation on the effects of different parameters including the type of constituent materials, the percentage of different material constituents, and loading speed on the static strength of composite specimens under different hygrothermal conditions. With the same fiber-to-matrix ratio, glass epoxy composites show higher interlaminar shear strength (ILSS) in all rate of loading as compared to glass polyester. The changes in ILSS in accordance with temperature show greater value for all compositions in glass fiber epoxy as that of glass fiber polyesters. It is observed that due to hygrothermal aging, the bonding behavior between glass/epoxy and glass/polyester composites is considerably affected at a higher degree of temperature when exposed for a longer duration.

Keywords Hygrothermal environment · ILSS · Composites · Rate of loading · Fiber · Matrix

1 Introduction

There has been an incredible progression in the field of science and technology of fiber-reinforced composites (FRCs) in preceding years. The higher specific

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P. K. Rakesh and I. Singh (eds.), *Processing of Green Composites*, Materials Horizons: From Nature to Nanomaterials, https://doi.org/10.1007/978-981-13-6019-0_4

strength and specific stiffness, tremendous durability, and design tailorability of fiber-reinforced polymers (FRPs) are the major reasons for their utility in various structural components in the aerospace, vehicles, naval, and other areas of engineering. Nowadays, FRPs are employed in applications reaching from space frames to ladder rails, from aircraft wings to automobile doors, from rocket motor cases to oxygen tanks, and from printed circuit boards to tennis rackets. With the substantial increase in temperature and moisture, the elastic moduli and internal stresses get reduced. This may adversely affect the safety and stability of the structures. The temperature variations during flying and landing conditions of aircraft are the most prominent example. Hence, the variation in stresses due to the hygrothermal effect seems to be a vital concern in the analysis and design of composite materials. An elaborate study of earlier research work on composite mechanics was investigated by Chamis [1]. Shen and Springer [2] conducted numerous tests on moisture absorption and desorption of composite materials. The influences of hygrothermal parameters on residual strength of laminated composites were studied by Ishai and Arnon [3]. Aditya and Sinha [4] elaborately discussed the evaluation of diffusion coefficients for continuous expose of laminates under hygrothermal environment.

Harding and Li [5] developed a new technique to study the ILSS behavior of glass/epoxy and carbon/epoxy laminates with the strain rate being increased using double-lap shear specimen. Govindarajan et al. [6] explained the reason for low interlaminar strength and the resulting prospect of failure in laminated composites. Melvin et al. [7] discussed the deformation behavior of carbon fiber/epoxy resin laminated composites hypothetically and checked with the experimental observations. It was noticed that the surrounding temperature on surface area is in a great relationship with the near-surface layup. Harding and Dong [8] experimentally investigated the influence of the rate of strain on the ILSS of carbon fiber-reinforced laminates. Selzer and Friedrich [9] investigated the mechanical characteristics and failure performance of carbon fiber-reinforced polymer (CFRP) composites under moisture environment. Shibasaki and Somiya [10] explored the time dependency of the degradation phenomena of woven aramid fiber-reinforced plastic (AFRP) in hot, wet environmental exposure. Woven AFRP specimens were dipped in hot water at 80 °C and exposed to humid air at 80 °C, 80% RH for 300 days, and they were tested by means of tensile setup to examine time dependence of their degradation. Naik et al. [11] studied interlaminar rupture characterization of laminated E-glass/epoxy. The test setup was followed by doing the double cantilever beam test and the end notch flexure test.

Patel and Case [12] studied the changes in the durability of graphite/epoxy woven composites when exposed to the hygrothermal environment. It was observed from the experimental studies that the initial and residual tensile characteristics of hygrothermal aged materials were unaffected by imposed environmental conditions. Baley et al. [13] explained the connection between interlaminar properties and glass fiber/polymer interfaces of marine laminated composites. Karbhari [14] examined the behavior of E-glass/vinyl ester composites in a moist environment. In this study, E-glass composites, made up by the resin infusion method (unidirectional and bidirectional), were tested to evaluate the influence of temperature on the short beam shear strength. Botelho et al. [15] described the hygrothermal effects on the

interlaminar shear properties of carbon fiber/epoxy composites. ILSS was determined by using a short beam shear test and Iosipescu shear strength. Ray [16] performed various tests on laminated unidirectional carbon composites at room temperature to evaluate the ecologically induced damage in composites. Lua et al. [17] developed a forecast tool to study the multiscale dynamic failure for marine composite structures. Zenasni and Bachir [18] measured the interlaminar fracture behavior of woven fabric-reinforced composite materials subjected to hygro-thermomechanical aging. In order to determine the loss in crack propagation resistance, a test setup for interlaminar fracture and double cantilever beam were carried out. Chan et al. [19] have suggested a new technique to govern the elastic interlaminar shear modulus of laminated composites. Gigliotti et al. [20] studied the various considerations of modeling and the simulation of hygrothermal deformation of laminated composites. The developed model can handle any type of environmental loading.

Sereir and Boualem [21] presented the damage of hybrid composites under long-term hygrothermal loading. Fu et al. [22] developed a constitutive model with interfacial damage to investigate the interlaminar stresses for the laminated composite plate. Bergeret et al. [23] studied the effect of the fiber/matrix interface on the aging mechanism of glass fiber-reinforced thermoplastic composites in a hygrothermal environment. The study comprises the properties of short glass fiber-reinforced thermoplastic composites based on poly (ethylene terephthalate), poly (butylenes terephthalate), and polyamide-6, 6 in an aggressive environment. Pilli et al. [24] examined the effect of moisture on the behavior of mechanical properties and durability of composites. The moisture ingression was achieved by doing an accelerated humidity test where the pressure was increased in the test chamber. Tsai et al. [25] examined the absorption and diffusion of water in carbon fiber/glass fiber hybrid composites. After immersion of the test specimens at various temperatures for 32 weeks of mechanical property test, water absorption tests and dynamic mechanical analysis were conducted. However, most of the available literature deals with the static behavior of unidirectional composite structures under ambient temperature and moisture. With an increased application of bidirectional woven fiber composites, a good amount of research work is essential for better understanding of composite material behavior in different environmental conditions.

To the best of author's knowledge, the static behavior of woven fiber-laminated composites in hygrothermal environments is not reported in the literature. The objective of the present study deals with the failure behavior of industry-driven bidirectional woven fiber composites under different loading speeds and weight fractions of constituents under hygrothermal conditions. The investigation was also carried out on the variation of ILSS with different types of composite matrices with a different combination of their weight fractions (55:45, 60:40, 65:35) and different exposure time of industry-driven woven fiber-laminated composites in hygrothermal environments.

2 Experimental Procedure

2.1 Materials and Fabrication

The constituent materials used for fabricating the glass/epoxy fiber plate specimens are:

- Glass woven roving as reinforcement (FGP, RP-10)
- Hardener as catalyst (Ciba-Geigy, Araldite HY951)
- Epoxy as resin (Ciba-Geigy, Araldite LY556)
- Polyester resin (MEKP-methyl ethyl ketone peroxide)
- Polyvinyl alcohol as a releasing agent.

2.2 Fabrication Procedure for Specimens

In this present work, two different types of fiber composite matrix specimens were prepared. They are (i) glass/epoxy and (ii) glass/polyester (E-glass fibers). Each type of composites is laminated by using three different types of weight fractions, i.e., 55:45, 60:40, and 65:35. Woven roving E-glass fibers (FGP, RP-10) were cut into the desired size and shape according to the number of specimens required for testing. Each composite laminate consists of 16 plies of fiber in balanced form as per ASTM D5687/D5687M-07 specification [26]. The epoxy resin matrix was prepared by taking hardener 8% (Ciba-Geigy, Araldite LY556, and Hardener HY951) of the weight of epoxy. For the preparation of polyester matrix, cobalt octane 2% was added to the polyester resin, and after that, 1.5% of catalyst (MEKP-methyl ethyl ketone peroxide) was added to the mixture and mixed thoroughly to get polyester matrix as per ASTM D5687/D5687M-07. After that, the subsequent fiber layers were arranged one over another to attain the desired configuration as shown in Fig. 1. A hand roller was employed for uniform distribution of resin, ply compaction, and removal of entrapped air to avoid the voids as shown in Fig. 2. The mold and layup were covered with a release sheet to prevent the layup from bonding to the mold surface. Then, the fiber composite specimens were placed in the mold for curing. The laminates were cured at normal temperature (25 °C and 55% relative humidity) under a pressure of 0.2 MPa for 3 days. After proper curing of laminates, the release sheet was separated, and the composite plates are ready for hygrothermal treatment as shown in Fig. 3.

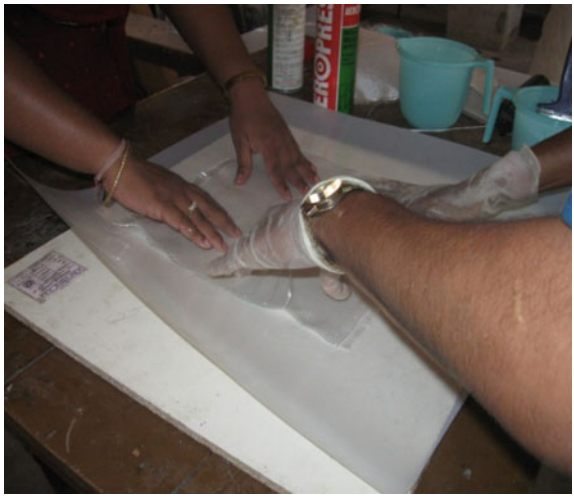

Fig. 1 Placing of woven glass fiber layer

Fig. 2 Removal of entrapped air using a steel roller

2.3 Hygrothermal Treatment

The fiber-laminated samples were hygrothermally treated in a humidity chamber as shown in Fig. 4, where the conditions were maintained at a temperature of 323 K and relative humidity (RH) ranging from 0 to 1% for moisture concentration as per ASTM D5229/D5229M-04 [27]. The humidity chamber had built-in hygrometer and thermometer for measurement of relative humidity and temperature. The variation in temperature was maintained between 300 and 425 K, whereas the RH was 0 in temperature bath as shown in Fig. 5. The composite laminates were placed on perforated trays.

Fig. 3 Composite plates

Fig. 4 Humidity chamber

Fig. 5 Temperature bath

The duration of hygrothermal treating of samples was six hours over a period of 36 h. The specimens were cut for three-point bend test by brick cutting machine into 45 mm × 6 mm × 4.8 mm (length × breadth × thickness) size as per specification.

2.4 Static Testing of Woven Fiber Composites in Hygrothermal Environment

The present work comprises substantial experimental work to examine the hygrothermal effects on the mechanical behavior of glass/epoxy and glass/polyester composites. For the static test, three-point bend test was carried out and it appears that the interlaminar shear strength (ILSS) has noteworthy importance for the evaluation of the static capacity of composite structures in the hygrothermal environment [28].

The present study deals with the failure behavior of woven fiber composites under the different rate of loading and change in weight fraction of constituents under hygrothermal conditions. Further studies were carried out on various types of composites with different constituents and their weight fractions (55:45, 60:40,

and 65:35) at a constant speed of 200 mm/min in a hygrothermal environment with different exposure time.

Most commonly used test for shear strength is the short beam strength test for composite laminates under three-point bending. The shear is the dominant applied loading in this test method. The internal stresses are complex, and the specimens may fail either in interlaminar shear or flexure or inelastic deformations or in combinations as per Cl 11.8 of ASTM D2344/D2344M-06 [29]. As most of the specimens have failed either interlaminar shear or shear–flexure combined mode, the short beam strength or interlaminar shear strength are calculated using this test (Cl 3.2.2 of ASTM D2344/D2344M-06).

A three-point short beam strength bending test was conducted on the specimens on a material testing machine (make: Instron 1195) as shown in Fig. 6 with the various rates of loadings to achieve ILSS and to analyze the influence of rate of loading for various kinds of laminates. The test was performed with different crosshead velocities, viz. 1, 10, 100, 200, and 500 mm/min with fixed span of 34 mm. To obtain max load of each laminate, at least five samples were tested. The details of glass/epoxy and glass/polyester specimen tested for different loading speeds are given in Tables 1 and 2. A humidity chamber was utilized for conditioning of moisture concentration, and a temperature bath was used for temperature conditioning of the sample at a constant crosshead velocity of 200 mm/min according to ASTM D5229/D5229M-04. For temperature conditioning, the results of glass/epoxy and glass/polyester samples are given in Tables 3 and 4. The results of glass/epoxy and glass/polyester samples are given in Tables 5 and 6 for moisture concentration conditioning. For hygrothermal treating of the sample at a 59 °C temperature and at 95% relative humidity environment, a humidity chamber was used. The results of glass/epoxy and glass/polyester samples are presented in Tables 7 and 8 for different exposure times. Before the experimentation, the width and thickness of the samples were computed precisely at the midsection. The test samples were arranged in the testing machine in such a way that its long axis was orthogonal to the loading nose and midpoint was centered. For all the samples, the aforementioned procedure was repeated. The experiments on the test specimens were carried out at a regular interval with a constant rate of loading 200 mm/min for each case, at least five test samples were tested, and the mean value was recorded in desired proforma. The ILSS value was evaluated according to ASTM D2344/D2344M-06 standard. The interlaminar shear strength is calculated using Eq. (1), in which b is the width of the specimen in mm, P_b is the breaking load in N, and t is the thickness of specimen in mm.

$$\text{ILSS} = \frac{0.75 \times P_b}{b \times t} \tag{1}$$

Fig. 6 Complete setup of Instron 1195 machine

Table 1 Variation of ILSS with different loading speeds of glass fiber/epoxy composites

Loading speed (mm/min)	ILSS (MPa) 55:45	ILSS (MPa) 60:40	ILSS (MPa) 65:35
1	31	36.7	39.4
10	34.7	31.6	28.4
100	22.9	21.5	19.1
200	28.4	29	25.2
500	27.1	27.1	27

Table 2 Effect of different loading speeds of glass fiber/polyester composites with ILSS

Loading speed (mm/min)	ILSS (MPa) 55:45	ILSS (MPa) 60:40	ILSS (MPa) 65:35
1	18.7	18.9	21.5
10	18.9	18.7	18.4
100	14.3	14.7	13.7
200	22	21.7	21.8
500	19.7	17.2	18

3 Results and Discussion

The variation of interlaminar shear strength (ILSS) with a rate of loading for glass/epoxy samples for three different proportions is given in Table 1. The ILSS of three weight fractions such as 55:45, 60:40 and 65:35 are considerd for crosshead velocity from 1 to 500 mm/min. With the increase of crosshead velocity, the ILSS decreased up to 100 mm/min for all the three weight fractions as epoxy and polyester are highly sensitive to the rate of loading. In general, the variation of interlaminar

Table 3 ILSS variation of glass/epoxy composites at various temperature (rate of loading: 200 mm/min)

Temperature (°C)	ILSS (MPa) 55:45	ILSS (MPa) 60:40	ILSS (MPa) 65:35
27	18.6	18.5	23
52	30.4	29.8	34.4
77	20.5	30.2	30.2
102	32.8	39.9	31.5
127	30.7	35	31.5
152	31.2	36.2	35.1
177	41	28.3	31.8
202	35.9	25	27.2

Table 4 Variation of ILSS of glass fiber/polyester composites at different temperatures (loading speed: 200 mm/min)

Temperature (°C)	ILSS (MPa) 55:45	ILSS (MPa) 60:40	ILSS (MPa) 65:35
27	5.1	12	8.5
52	24.1	20	23.7
77	19.7	20.9	23.5
102	27	21.2	23.7
127	21.3	23.2	27.4
152	19.3	19.4	20.5
177	25.1	20.6	21.4
202	13	20.2	20.5

Table 5 Variation of ILSS of glass fiber/epoxy composites at different moisture concentrations (loading speed: 200 mm/min)

Moisture concentration (C %)	ILSS (MPa) 55:45	ILSS (MPa) 60:40	ILSS (MPa) 65:35
0	12.7	14.5	12
0.25	27.2	23	27.3
0.50	18.4	13.9	11.5
0.75	32.6	14.5	33.2
1	28.1	23	22.5

Table 6 Variation of ILSS of glass fiber/polyester composites at different moisture concentrations (loading speed: 200 mm/min)

Moisture concentration (C %)	ILSS (MPa) 55:45	ILSS (MPa) 60:40	ILSS (MPa) 65:35
0	9.4	15.4	18.7
0.25	26.6	16	25
0.50	8.9	10.6	10.4
0.75	24	24	26
1	18.9	19.3	21.9

Table 7 Variation of ILSS of glass fiber/epoxy composites at different exposure times (loading speed: 200 mm/min)

Exposure time (h)	ILSS (MPa) 55:45	ILSS (MPa) 60:40	ILSS (MPa) 65:35
5	32.3	33.8	33.9
10	31.4	26.8	32.5
15	31.2	25.2	32.3
20	31.1	24.8	32.1
25	16.5	16.5	26.3

Table 8 Variation of ILSS of glass fiber/polyester composites at different exposure times (loading speed: 200 mm/min)

Exposure time (h)	ILSS (MPa) 55:45	ILSS (MPa) 60:40	ILSS (MPa) 65:35
5	23.7	20.7	25.4
10	21.7	20.4	25
15	18.6	17	20.4
20	16.6	15.4	15.7
25	10.4	13.2	10.1

shear strength was found with increasing loading speed. The ILSS for glass/epoxy sample is increasing with the rate of loading up to 200 mm/min, and after that, the interlaminar shear strength remains nearly steady with an increase in the rate of loading. Besides this, the ILSS increases with the increase of fiber content of fiber/epoxy composites.

The variation of interlaminar shear strength with different proportion of glass/polyester composites is given in Table 2. However, in the case of glass: polyester fiber matrix of three weight fractions, lower ILSS is observed in comparison with glass/epoxy mixes as given in Table 2. The strength is then decreased for the rate of loading up to 100 mm/min. The ILSS is then increased with the increase of crosshead velocity up to 200 mm/min. After that, the ILSS decreased for all weight fraction of

glass: polyester matrix resin, unlike glass/epoxy mixes. Woven roving glass fiber-reinforced polymer shows substantial rate sensitivity.

The significant variation in interlaminar shear strengths is observed with increasing crosshead velocity for this group. As observed, it is essential to note that a change in crosshead velocity can result in a variation in failure loads and subsequently the shear strength. When the specimen is subjected to an increasingly higher impact velocity, the laminate acts as a more rigid beam or plate, which is less prone to bending behavior. This changes its behavior from that of a flexible rigid beam with very low impact velocity, and the failure initiated from the rear surface to that occurs near the point of contact in the case of much higher impact rate of loading.

At an intermediate rate of loadings, the complex behavior of mixed fracture modes is observed. For all weight fractions, the woven glass fiber laminates made with epoxy matrix show higher interlaminar shear strengths than the polyester matrix, because the epoxy resin has relatively low molecular weight than polyester and is capable of being processed under different conditions. The main benefits of epoxy resins are having more viscosity and display low shrinkage during curing over polyester resins. The variation of ILSS with a temperature of glass fiber/epoxy composites for three different proportions is given in Table 3 for a fixed loading speed of 200 mm/min.

In the present investigation, the values of ILSS for glass/epoxy samples are increasing progressively when the temperature is increased from 27 to 52 °C and then decreased up to 77 °C. The ILSS for glass/epoxy samples increased further with the increase of temperature up to 152 °C and thereafter decreased with the increase of temperature for glass/epoxy composite in 65:35 and 60:40 proportions. The variation of shear strength with a temperature of glass fiber/polyester composites for three different proportions is presented in Table 4 for the same loading speed of 200 mm/min. As observed, for glass/polyester sample of 65:35 proportion, higher the value of temperature, more will be the values of ILSS, and it remains constant from 52 to 152 °C. After this value of temperature, the values of ILSS are decreasing with the increase of temperature. For fiber/polyester composite of 60:40 and 65:35 proportions, the results show the ILSS values are increased gradually with the increased temperature and the values remain constant from 52 to 102 °C. The ILSS for glass/polyester sample decreased beyond 152 °C temperature and remains constant with increase in temperature except 55:45 proportion. As given in Table 4, the variations are observed for the ILSS values for glass/polyester sample in every temperature range of nearly 25 °C up to 177 °C. Thereafter, the temperature decreased at the constant rate of loading s of 200 mm/min.

When the specimen is exposed to a higher temperature, the degradation of mechanical properties, cracking, and flaking of polymers can occur. The primary damage in the laminated composite is generally matrix microcracks. Matrix microcracks can cause degradation of material properties in laminated composite and also act as predecessors to other forms of failures in laminates. The moisture absorption capacity is more when the specimen is exposed to elevated temperatures. At higher temperature, the thermal stresses are more which leads to a higher mismatch in the thermal strain of constituents.

The variation of ILSS with the moisture of glass fiber/epoxy composites for three different proportions is given in Table 5 for a fixed loading speed of 200 mm/min. As observed, the ILSS for glass/epoxy specimen increased and decreased in every 0.25% moisture concentration gradually up to 1 for all types of composites. The ILSS has increased from 12.7 to 14.5 with an increase of fiber content in the composite. However, on a further increase of fiber content, the ILSS reduces for glass fiber/epoxy composites at different moisture concentrations for loading speed: 200 mm/min.

In the glass/polyester composites, as given in Table 6, all the fabricated composites behave almost the same as glass/epoxy composites at the constant crosshead velocity of 200 mm/min. When exposed to the moist environment, the positive concentration gradient between the specimen and the environment, the higher pressure outside the specimen favors moisture absorption. Again the same sample is exposed to a comparatively dry environment, the situations get reversed resulting in moisture desorption. The comparative rates of absorption and desorption control the net moisture pickup by the specimen.

The ILSS values are varying significantly with the moisture concentration. Amount of moisture absorbed by the epoxy matrix is much greater than fibers which absorb little or no moisture. This results in a significant mismatch in moisture-induced volumetric expansion between matrix and fibers leading to the evolution of localized stress and strain fields in the composite at the interfacial region.

The humidity affects all constituents of the laminated composites, mainly the matrix, the fiber and the fiber matrix interface. The plasticization method involves the intrusion of the Van der Waals bonds between ethers, secondary amines, and hydroxyl groups. Polymers which are having imides and ketones are highly resistant to hydrolysis; they have some polar groups which can decrease their moisture sensitivity. Plasticization lessens the residual stresses and rises the viscoelasticity. Hence, the impact of an increase in moisture concentration is the same as that of increase in temperature as given in Tables 3, 4, 5, and 6.

The variation of ILSS with a various exposure time of glass fiber/epoxy composites for three different proportions is given in Table 7 for a fixed loading speed of 200 mm/min. As given in Table 7, it is observed that the decrement in ILSS with the increase of exposure time is due to the significant amount of absorbed moisture. The variation of ILSS with a different exposure time of glass fiber/polyester composites for three different proportions is given in Table 8 for a fixed loading speed of 200 mm/min.

The variation of ILSS with exposure time is similar to that of glass/epoxy composites. As given in Table 8, it is observed that the decrement in ILSS with the increase of exposure time is due to the significant amount of absorbed moisture. It was noted that the moisture absorption kinetic increases with more conditioning time in glass/epoxy and glass/polyester composites for different weight fractions at the constant crosshead velocity of 200 mm/min. The failure of the specimen occurs due to continuous exposure to the environment may be attributed to the reduced interfacial stress transmissibility. This happens because of matrix plasticization and chemical degradation. The elastic modulus of the matrix may be reduced due to matrix plasticization. The hydrolysis of interfacial bonds results in chemical degradation.

Table 9 Variation of moduli of glass fiber: epoxy composites (55:45) at different temperatures (loading speed: 200 mm/min)

Temperature (°C)	E_1 (GPa)	E_2 (GPa)	G_{12} (GPa)
27	7.9	7.4	2.9
52	7.6	6.8	2.6
77	7.1	6.4	2.3
102	6.7	6.2	2.1
127	6.5	5.9	1.8
152	6.3	5.7	1.6

Table 10 Variation of moduli of glass fiber: polyester composites (55:45) at different moisture concentrations (loading speed: 200 mm/min)

Moisture concentration (C %)	E_1 (Units) (GPa)	E_2 (Units) (GPa)	G_{12} (Units) (GPa)
0	7.9	7.4	2.9
0.25	7.6	7.3	2.8
0.50	7.4	6.9	2.7
0.75	6.6	6	2.2
1	6.2	5.5	1.8

The tensile tests are performed with coupons as per ASTM D3039/D3039M-2008 [30] after temperature and moisture treatments. The tensile test outcomes of glass fiber/epoxy composites (55:45) at various temperature and dampness are recorded in Tables 9 and 10. The modulus of elasticity and modulus of rigidity diminished considerably with increment in temperature and damped condition. The temperature and dampness influences all the constituents of laminated composites including the matrix, the fiber and the fiber matrix interface. Accordingly, the tensile strength and compressive strength diminish. Because of increment in temperature and dampness, the longitudinal and transverse tensile strength proportionately varies with temperature and moisture.

Also, the comparative linear variation occurs in case of the modulus of rigidity subjected to the hygrothermal environment. The rate of degradation for Young's modulus of elasticity appears differently because of the adhesion among the fiber and matrix.

4 Conclusions

Based on the test results and discussion on the variation of ILSS with different parameters, the following conclusions were drawn:

- For the same weight fraction of the fiber matrix, glass/epoxy laminated composites show higher interlaminar shear strength for all values of loading rate.
- The variation of the interlaminar shear strength of woven fiber-laminated composites is significant for low loading speed and is not so prominent for high values of speed.
- The variation of ILSS with respect to temperature is more for all weight fraction combinations in glass/epoxy than glass/polyester composites.
- The ILSS value with respect to moisture concentration is more for glass fiber/polyester composites as compared to glass fiber/epoxy composites for all weight fraction combinations.
- The interfacial bond for glass/epoxy and glass/polyester composites is considerably affected by hygrothermal aging at higher conditioning temperature and for longer exposure time.
- Polyester and epoxy, the two matrix resins, are extremely loading rate sensitive.

Finally, it is concluded that the static behavior of woven fiber-laminated composites is significantly influenced by the materials, loading speed, and hygrothermal conditions. The experimental data showing a variation of the ILSS is recommended as a design aid for structural members in hygrothermal environment. The above recommendations for the design of composites are valid within the range of materials, temperature, and moisture conditions considered in this study. The designer has to be cautious while dealing with structures subjected to hygrothermal loading. The results can be utilized to the advantage of designers during the design of laminated composite structures subjected to the hygrothermal environment.

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Chapter 5
A Study of Chemical Treatment of Natural Fibers and Its Effect on the Mechanical Properties of Developed Composites

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Abstract In this chapter, three different types of natural fibers namely jute, banana, and bamboo fibers were used as reinforcing material to fabricate the three different types of composite laminates. The composites were prepared by taking 50–60% weight fraction of the fiber. Two different types of chemical treatments of natural fibers were carried out to study the effect of chemical treatment of natural fibers on the mechanical properties (tensile and flexural) of the developed composites. The natural fibers were treated with (i) 5% of sodium hydroxide (NaOH) for 4 h and (ii) 5% NaOH for 4 h followed by 1% of potassium hydroxide (KOH) and 1% of hydrogen peroxide (H_2O_2) for 2 h. The mechanical properties such as (i) tensile strength, modulus, and percentage elongation and (ii) flexural strength, modulus, and percentage elongation of the developed composites were evaluated. The results indicate that natural fibers treated with 5% NaOH, 1% KOH, and 1% H_2O_2 resulted in better mechanical properties than the 5% NaOH treated natural fiber-based composites.

Keywords Natural fiber · Composites · Chemical treatment · Tensile test · Flexure test

1 Introduction

Composites are known as the combination of reinforcement and matrix where the former is much stronger and act as a load-carrying member, while the latter is considered to be the weaker that holds the reinforcement together. Reinforcement provides strength and rigidity and helps in supporting the structural load. The composite material was first used by the ancient Egyptians about 3000 years ago for constructing walls using a mixture of straw and clay. Over the years, composites have been used with various natural fibers. The various industries and academic institutions have

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P. K. Rakesh and I. Singh (eds.), *Processing of Green Composites*, Materials Horizons: From Nature to Nanomaterials, https://doi.org/10.1007/978-981-13-6019-0_5

understood the significance of this material and made use of it for the development of the society. Composite materials are used in various fields related to design and manufacturing technologies. These materials are also widely used in aerospace and automobile industries due to their multifunctional properties such as (i) low weight to strength ratio, (ii) low thermal expansion, (iii) good acoustic properties, (iv) low wear on tools, (v) better toughness, and (vi) low-cost. However, composites also have some drawbacks such as poor compatibility between the hydrophobic polymer and hydrophilic fiber. The natural fibers are highly sensitive to water and have relatively poor thermal stability. Subsequently, the swelling and dimensional instability of the natural fiber may lead to reduced mechanical properties. In ancient times, Chinese paper makers used to select almost all types of plants such as hemp, silk, cotton, etc., for papermaking. Some of the natural fibers used in composite materials are jute, bamboo, and banana. Jute is an important fiber and is commonly used for its high specific strength, low-density, less abrasive, and excellent dimensional stability. It is the second most common natural fiber cultivated after cotton. Moreover, it is abundantly available and has moisture retention and drapability characteristics. Also, jute does not produce any toxic gases on combustion. The stem of the jute plant is covered with thick bark which contains fiber. The plants need 2–3 months to grow fully. Fully grown plants are then cut, tied up in bundles, and kept under water for fermentation. These fibers are then pulled out by the cultivators, washed very carefully, and dried under the sun. Banana fiber has the good specific strength and lower density as compared to those of conventional glass fiber. The pseudostem of the banana plant is a cylindrical, clustered aggregation of leaf stalk bases. Banana fiber is a waste product of banana cultivation. It is either not correctly utilized or is partially done. It has high-strength, lightweight, fire resistance, strong moisture absorption, and biodegradable characteristics. It is used for making products like filter paper, paper bags, lamp stands, pen stands, decorative papers, rope, mats, and composite materials. It is used in the printing of currency notes in Germany. Banana fiber-reinforced polypropylene composite is used in the automobile sector for making of underfloor protection panels in luxurious cars. Banana fiber is mostly used in making decorative products. Composite materials based on banana fiber are used in buildings boards and fire resistant boards. Bamboo is the fastest growing woody plant, and it grows 30% faster than the fastest growing tree. The growth of bamboo is approximately 3 ft overnight. The bamboo plants are of global interest because of their distinctive life form, a wide range of uses, and their ecological concern. Bamboo fiber is a cellulose fiber extracted from natural bamboo. It possesses certain superior characteristics like high breaking tenacity and better moisture wicking properties.

2 Literature Survey

The application spectrum of natural fibers has been increasing due to their multifunctional properties like biodegradability, flexibility, low-cost, and lightweight. These are also easily available and widely used as reinforcement the material in compos-

ites without much processing as compared one-on-one with synthetic fibers. Karus et al. [1] surveyed the use of natural fibers in automotive sector. It was stated that the use of natural fiber is increasing unprecedentedly in various manufacturing sectors. Memon and Nakai [2] developed jute fiber-reinforced composite using compression molding. It was observed that the molding temperature exerts a significant influence on the impregnation quality and dispersion of the fibers. Both impregnation quality and the fiber dispersion were increased with the molding temperature. The tensile strength was also reduced with the molding temperature due to the deterioration of natural fibers. This indicates that the molding temperature is an important parameter that controls the mechanical properties of natural fiber-based composites. Ticoalu et al. [3] stated that glass fiber-based composites can be used as roof materials if the tensile strength of the composites is more than 50 MPa. Thus, the natural fiber-based composites can be a potential substitute for infrastructural applications where the use of synthetic fiber-reinforced composites is not suitable. Mishra and Biswas [4] reported that the void content is minimum for the neat epoxy specimen and maximum for composite specimen having fiber loading of 12% by weight, respectively. It was also found that the void content decreases with an increase in the fiber loading. Both the tensile strength and hardness of the jute fiber-reinforced epoxy composites also increased with the fiber loading. Gopinath et al. [5] compared the mechanical properties of woven jute fiber-reinforced epoxy and polyester composites. It was concluded that jute/epoxy composite exhibited better mechanical properties than the jute/polyester composites. Bledzki et al. [6] compared the various properties of natural fibers with glass fiber. The strength and modulus of the synthetic fibers were much higher than that of the natural fibers. However, specific values of synthetic and natural fiber are quite close to each other. It was also seen that jute and flax fibers have similar and better mechanical properties than the other natural fibers. Abilash and Sivapragash [7] stated that jute fiber is a versatile reinforcement which can be used to develop composites for application in rural areas. Processing of natural fiber consumes less energy as compared to synthetic fibers. Huang and Sun [8] investigated the influence of water absorption on the mechanical properties of glass fiber-reinforced polyester composites. It was found that the tensile strength and breaking strength of the developed composites decrease with the water immersion time because of the weakening of fiber–matrix interfacial bonding. Gowthami et al. [9] investigated the effects of silica on thermal and mechanical properties (tensile strength and modulus) of sisal fiber-reinforced polyester composites. The composites with and without silica have been made by incorporating biodegradable sisal fiber. Sathish et al. [10] studied the effect of volume fraction on the tensile and flexural properties of natural fiber-reinforced hybrid composites. Dhal and Mishra [11] developed a low-cost composite using bloom of brown grass. The various physio-mechanical properties of the developed composites showed that the developed composites are light in weight and pose high strength. Sanjay et al. [12] exhibited an outline of various natural fibers and their composites which are being utilized in engineering as well as commercial applications. Ashik and Sharma [13] summarized the history of natural fibers and their applications along with properties of various natural fibers which are being used as a substitute to glass fiber. Gon et al. [14] studied the various properties of

natural fibers and concluded that jute-reinforced composites used as a substitute to currently used thermoplastics have great potential in terms of high-stiffness, low-density, and high-strength. Sait and Subramaniam [15] concluded that the failure strain is more consistent failure criterion for natural fiber-reinforced composites. It was recommended to use failure strain as the key design criterion to improve the reliability. Reddy and Hussain [16] studied the mechanical behavior of natural fiber-based polyester composites. It was found that the mechanical properties of hemp and sisal fiber-reinforced polyester composites improved with increasing the fiber weight fraction. Chandramohan and Marimuthu [17] put forward an effort to utilize the renewable resources for the development of bio-composite materials using bio-epoxy resin and natural fibers. It was concluded that the developed composites coated with calcium phosphate and hydroxyapatite can be efficiently used for both external and internal fixation of fractured bone. Sakthivel and Rajendran [18] found that hybrid composite laminates (glass/banana/glass and banana/glass/banana) exhibited superior mechanical properties and thus can be used in various applications in naval, automobile, and transportation sector. Dubey and Agnihotri [19] determined the interfacial strength between midrib and polyester resin and the tensile properties of midribs. They also determined the properties of critical embedded length by tensile test and fiber pull-out test. The results showed that midrib of coconut palm leaves has tremendous potential for development of natural fiber-reinforced composites. Anand [20] reported the development and designing of novel natural fiber geotextiles with various behavioral patterns with soil and environmental conditions. These developed fibers were found to be much more environment friendly, biodegradable, and renewable than their synthetic counterparts.

From the literature, it is evident that handful of work has been conducted on jute, bamboo, and banana fiber-reinforced composites. The effect of chemical and heat treatment on the mechanical properties is also rarely investigated for these types of natural fibers. Thus, the mechanical properties like tensile strength, percentage elongation, tensile modulus, flexural strength, and flexural modulus of the jute, bamboo, and banana fiber-reinforced composites have been evaluated for different types of chemical treatment of natural fibers used to develop the composites.

3 Materials and Methods

3.1 Treatment of Natural Fiber

Three different types of natural fibers, namely jute, bamboo, and banana fiber mats have been used to develop three different types of composites. The natural fiber mats were cut into the dimension of 25 cm $\times$ 25 cm, as shown in Fig. 1. These fiber mats were then treated chemically to improve their surface characteristics. Two types of chemical treatment of natural fibers have been carried out (i) alkaline treatment using NaOH and (ii) alkaline treatment using NaOH, KOH, and H_2O_2. In the first type of

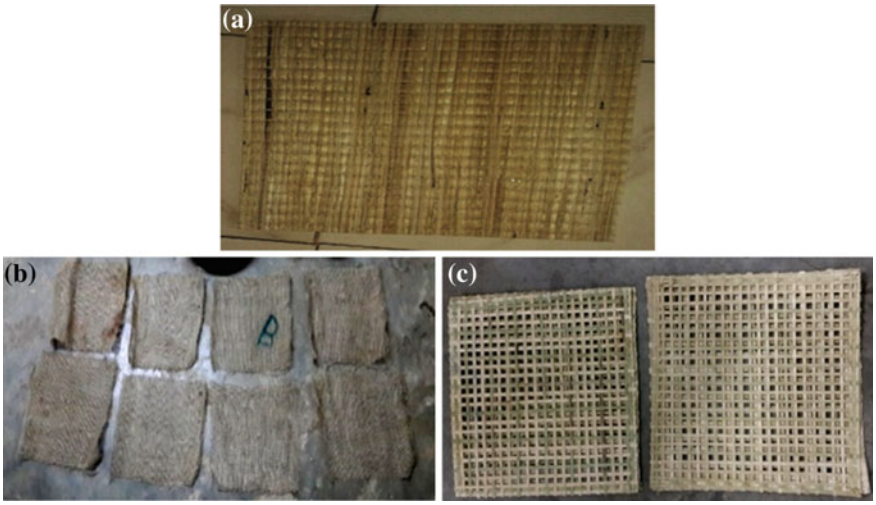

Fig. 1 Natural fiber mats **a** banana fiber, **b** jute fiber, and **c** bamboo fiber

treatment, 50 g of NaOH is mixed with 1 L of distilled water and the solution is stirred until NaOH is fully dissolved. Fiber mats are then dipped into the solution and are kept at room temperature for 4 h. The fibers were taken out and dried at room temperature. In the second type of treatment, 20 g of both KOH and H_2O_2 and 50 g of NaOH is mixed in 2 L of water at room temperature. Fibers are then dipped into the solution and are kept at room temperature for 2 h. The process of alkali treatment is shown in Fig. 2. The chemically treated natural fibers are kept in an electric oven for a specific period of time at a particular temperature, as shown in Fig. 3. The specimen treated with 5% NaOH is kept in the electric oven at a temperature of 80 °C for 24 h. The other specimen treated with 5% NaOH, 1% KOH, and 1% H_2O_2 is kept in the electric oven at a temperature of 40 °C for approximately 24 h. Proper care was taken while putting the chemically treated fibers in the oven by keeping them in a special metallic vessel so that heat could be easily conducted to the fibers thereby heating it to the desired amount.

3.2 Fabrication of Composite Laminates

Hand layup process was employed to develop the composites as it is one of the simplest molding techniques. Initially, the top surface of the mold is cleaned. Then a polymer film is placed on the top of the mold surface so that the epoxy resin will not stick to the mold surface. The sticking of resin with the mold surface may lead to difficulty in removing the final composite laminate from the mold. A layer of epoxy is then applied over the polymer film using a paintbrush to obtain a smooth surface.

Fig. 2 Chemical treatment of natural fiber **a** preparation of solution, **b** NaOH solution, **c** alkaline treatment of fiber with NaOH, and **d** alkaline treatment of fiber with NaOH, KOH, and H_2O_2

Fig. 3 Heat treatment of natural fiber **a** chemically treated jute fiber in oven and **b** fiber taken out of oven

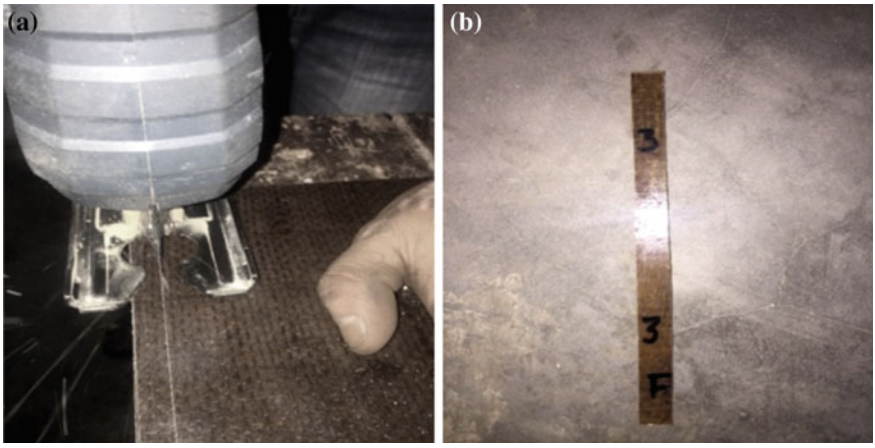

Fig. 4 Preparation of composite specimens **a** cutting of composite laminate and **b** composite specimen

The resin which was prepared is a mixture of epoxy and hardener. The mixing ratio of epoxy and hardener is 10:08. Then the natural fiber mat is placed over the epoxy layer. Again epoxy is applied over the fiber mat and one more natural fiber mat is placed over it. The same process is repeated several times until the required thickness of the composite is achieved. Finally, another polymer film is placed to cover the final layer of resin. A roller was used to prevent the formation of any bubble between fiber layer and resin. The upper part of the mold is then placed maintaining a gap of 4 mm between the molds. The mold is kept for 24 h at room temperature for curing process. Finally, the mold is opened and composite laminate is taken out. All the plastic films were removed. The laminate is then cut in the required dimensions. The fabricated composite laminates and the preparation of the test samples are shown in Fig. 4.

3.3 Mechanical Testing

The tensile and flexural testing was carried out to evaluate the tensile and flexural properties of the developed composites. Both tensile and flexural properties of the developed composites were evaluated as per ISO 527-1:2012 and ISO 178:2003 standards using the universal testing machine (UTM). All the tests were performed at normal environmental conditions (23 °C and 50 ± 10% relative humidity). Each tensile and flexural test was repeated five times in order to nullify any random variation in the experimental results.

4 Results and Discussion

The tensile strength, percentage elongation, and tensile modulus of the developed natural fiber composites (jute/epoxy, bamboo/epoxy, and banana/epoxy treated with (i) 5% NaOH and (ii) 5% NaOH, 1% KOH, and 1% H_2O_2 are shown in Tables 1 and 2, respectively. The average values of tensile strength, tensile modulus, and percentage elongation for different types of composites were calculated and presented in Fig. 5. The flexural strength, percentage elongation, and flexural modulus for the natural fiber-reinforced composites treated with (i) 5% NaOH and (ii) 5% NaOH, 1% KOH and 1% H_2O_2 are shown in Tables 3 and 4, respectively. Similarly, the comparison plots of the flexural strength, flexural modulus, and percentage elongation are shown in Fig. 6. The tensile strength and tensile modulus of jute/epoxy composites treated with NaOH, KOH, and H_2O_2 were increased by 18 and 15%, respectively, as compared to NaOH treated jute/epoxy composites. Similarly, the tensile strength and tensile modulus of bamboo/epoxy composites were improved by 20.6 and 55.5%, whereas for banana/epoxy composites the tensile strength and modulus were improved by 46.5 and 34.3%, respectively. The figure also indicates that the bamboo/epoxy composites have the highest tensile strength as compared to the other developed composites. However, the percentage elongation at break for banana/epoxy composites is more as compared to jute/epoxy and bamboo/epoxy composites. It was also observed that the brittle behavior of the developed composites (except jute/epoxy composites) was reduced due to the NaOH, KOH, and H_2O_2 treatment of natural fibers. This is a favorable improvement as highly brittle polymer composites may have limited applications. The average flexural strength and modulus of NaOH, KOH, and H_2O_2 treated jute/epoxy composites were increased by 39 and 5%, respectively, when compared one-on-one with NaOH treated jute/epoxy composites. The improvement in flexural strength and modulus for bamboo/epoxy and banana/epoxy are 25, 20.5, 49.6, and 51.5%, respectively. It was also seen that percentage elongation is more when the fibers are treated with NaOH alone. This indicates that the composite material becomes more ductile in nature when fiber is treated with NaOH. On the other hand, when the specimen is treated with additional KOH and H_2O_2, the percentage elongation decreases; i.e., the material becomes more brittle in nature. From the figures, it was also observed that the tensile and flexural properties of the developed composites were significantly increased when the natural fibers were treated with 5% NaOH, 1% KOH, and 1% H_2O_2. The alkali treatment of NaOH can make the fiber surface clean by removing waxes, hemicellulose, pectin, and part of lignin. The removal of these substances results in enhanced surface roughness of the fiber. Therefore, the mechanical interlocking at the interface of the fiber and the matrix material is substantially improved. The adsorption of epoxy resin on the surface of the natural fiber increases after NaOH treatment which is a prerequisite condition for creating the interphase. The interface properties of fiber and matrix were further improved due to the treatment of fiber by KOH and H_2O_2. The peroxide initiated free radicals react with the hydroxyl group of the fiber and also with the matrix. This results in good adhesion between the fiber and matrix at the

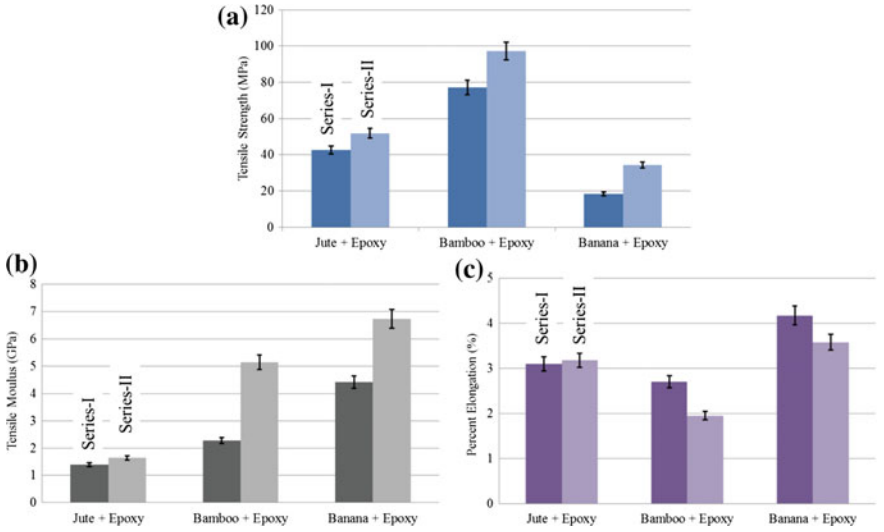

Fig. 5 Comparison of mechanical properties **a** tensile strength, **b** tensile modulus, and **c** percentage elongation (series-I: NaOH treated samples and series-II: NaOH + KOH + H_2O_2 treated samples)

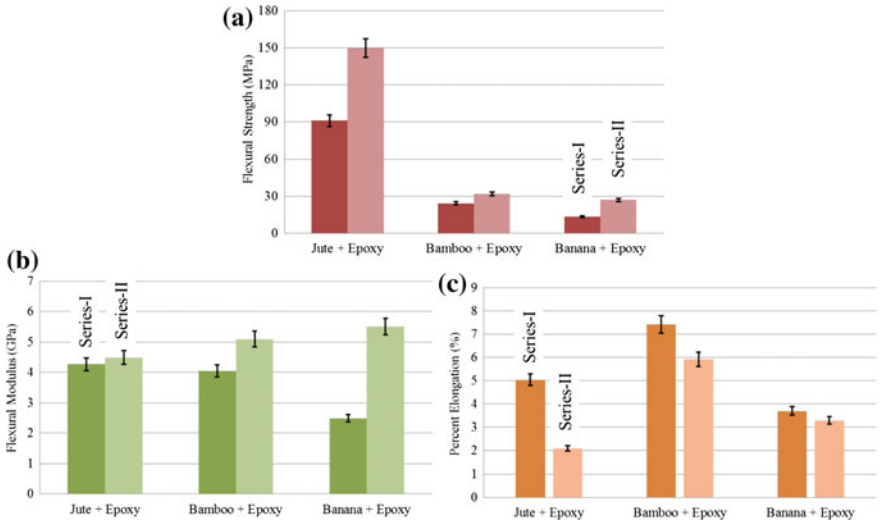

Fig. 6 Comparison of flexural properties **a** flexural strength, **b** flexural modulus, and **c** percentage elongation (series-I: NaOH treated samples and series-II: NaOH + KOH + H_2O_2 treated samples)

interface. It also helps in removing the wax and oil which are generally present over the fiber surface. Thus, the mechanical properties of the composites are enhanced after chemical treatment of fibers.

Table 1 Tensile properties of the developed composites treated with 5% NaOH

Sl. No.	Jute/epoxy			Bamboo/epoxy			Banana/epoxy		
	Tensile strength (MPa)	Percentage elongation (%)	Tensile modulus (GPa)	Tensile strength (MPa)	Percentage elongation (%)	Tensile modulus (GPa)	Tensile strength (MPa)	Percentage elongation (%)	Tensile modulus (GPa)
1	40.0	2.8	1.4	86.1	2.6	1.0	18.7	4.2	4.4
2	45.0	3.4	1.3	88.3	3.3	2.6	18.2	4.1	4.3
3	45.0	3.4	1.3	57.5	1.4	3.9	22.4	4.1	5.4
4	37.5	2.5	1.5	69.5	3.1	2.1	17.1	4.4	3.9
5	46.3	3.4	1.4	85.0	2.9	1.7	15.6	3.9	3.9

Table 2 Tensile properties of the developed composites treated with 5% NaOH, 1% KOH, and 1% H_2O_2

Sl. No.	Jute/epoxy			Bamboo/epoxy			Banana/epoxy		
	Tensile strength (MPa)	Percentage elongation (%)	Tensile modulus (GPa)	Tensile strength (MPa)	Percentage elongation (%)	Tensile modulus (GPa)	Tensile strength (MPa)	Percentage elongation (%)	Tensile modulus (GPa)
1	52.5	3.4	1.5	76.6	1.1	6.7	35.8	3.6	6.3
2	55.0	3.5	1.6	119.1	2.3	5.2	37.5	3.2	8.8
3	47.5	2.7	1.7	113.3	2.6	4.3	34.7	3.5	6.3
4	53.7	3.2	1.7	85.0	1.9	4.5	31.6	3.6	5.6
5	51.0	3.1	1.6	92.7	1.8	5.0	32.5	3.9	6.6

Table 3 Flexural properties of the developed composites treated with 5% NaOH

Sl. No.	Jute/epoxy			Bamboo/epoxy			Banana/epoxy		
	Flexural strength (MPa)	Percentage elongation (%)	Flexural modulus (GPa)	Flexural strength (MPa)	Percentage elongation (%)	Flexural modulus (GPa)	Flexural strength (MPa)	Percentage elongation (%)	Flexural modulus (GPa)
1	92.7	5.1	4.3	24.2	10	4.5	15.1	5.4	3.1
2	92.8	5.8	4.3	12.1	9.4	1.8	17.3	5.3	2.6
3	88.1	5.5	4.1	24.2	5.8	4.3	11.7	2.1	2.6
4	90.0	4.1	4.2	36.2	6.5	6.8	11.7	2.9	1.9
5	91.6	4.7	4.3	24.2	5.4	2.63	11.7	2.7	2.1

Table 4 Flexural properties of the developed composites treated with 5% NaOH, 1% KOH, and 1% H_2O_2

Sl. No.	Jute/epoxy			Bamboo/epoxy			Banana/epoxy		
	Flexural strength (MPa)	Percentage elongation (%)	Flexural modulus (GPa)	Flexural strength (MPa)	Percentage elongation (%)	Flexural modulus (GPa)	Flexural strength (MPa)	Percentage elongation (%)	Flexural modulus (GPa)
1	150.0	2.1	4.5	36.3	5.1	6.0	38.4	3.0	4.3
2	148.5	1.5	4.4	36.2	6.3	2.5	28.8	4.2	6.5
3	153.7	1.9	4.6	40.5	8.1	2.3	19.2	2.2	4.3
4	150.0	2.6	4.5	24.2	5.3	5.0	19.2	3.4	4.3
5	146.2	2.4	4.3	24.2	4.8	9.5	28.8	3.7	6.0

Recognizing the continued research and growing interest in the development of natural fiber-based composites along with its biodegradable and ecofriendly properties, various attempts have been made to prepare some products which can be used in a day-to-day basis. Some of these applications include hurricane resistant housings, automobile components (door panels, instrument panels, armrests, etc.), and entertainment accessories (archery bows, boat hulls, golf clubs, etc.). In the present work, a spoiler used in cars has been designed and developed using the natural fiber-reinforced composites. Spoilers are an automotive aerodynamic device. The main function of the spoiler is to spoil or destroy the unfavorable air movement across the body of the vehicle which is in motion. When vehicles move at high speeds, the increased airflow causes lift which is dangerous when the vehicle takes a turn as this can make it fly-off the road and lose control. Thus, the spoiler is fitted at the back of the cars which help in reducing the lift without adding extra weight. This is mostly used in the racing cars to keep the weight of the cars down by evenly distributing the airflow and reducing the drag forces acting on it. CREO Parametric software was used to design the spoiler as shown in Fig. 7. The analysis work has been carried out in ANSYS. The actual model of the spoiler is shown in Fig. 8. Jute fiber-reinforced epoxy composites treated with 5% NaOH, 1% KOH, and 1% H_2O_2 has been considered among the developed composites to fabricate the spoiler as it has shown superior mechanical properties as compared to the banana and bamboo fiber-reinforced composites. The data of drag force at different velocities have been taken from Ramya et al. [21]. These drag force values have been used in ANSYS simulation to find the equivalent stress and deformation. From the simulation, it was observed that the average equivalent tensile stress values obtained from the experiment (51.75 MPa) are greater than the simulated equivalent stresses (31.61 MPa) obtained at maximum velocity of 120 km/h. Hence, it can withstand the maximum stresses at high velocity. The maximum deformation (1.74 mm) that jute fiber-reinforced composites can withstand is more than the deformation values that are obtained from simulation (1.28 mm). This indicates that it can also withstand maximum deformation at a maximum velocity of 120 km/h.

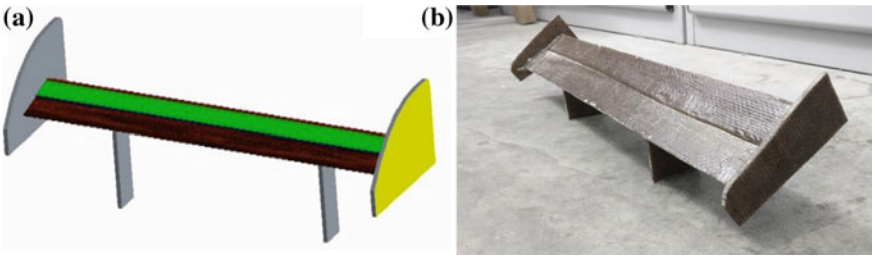

Fig. 7 Spoiler design and fabrication **a** CAD model and **b** fabricated spoiler

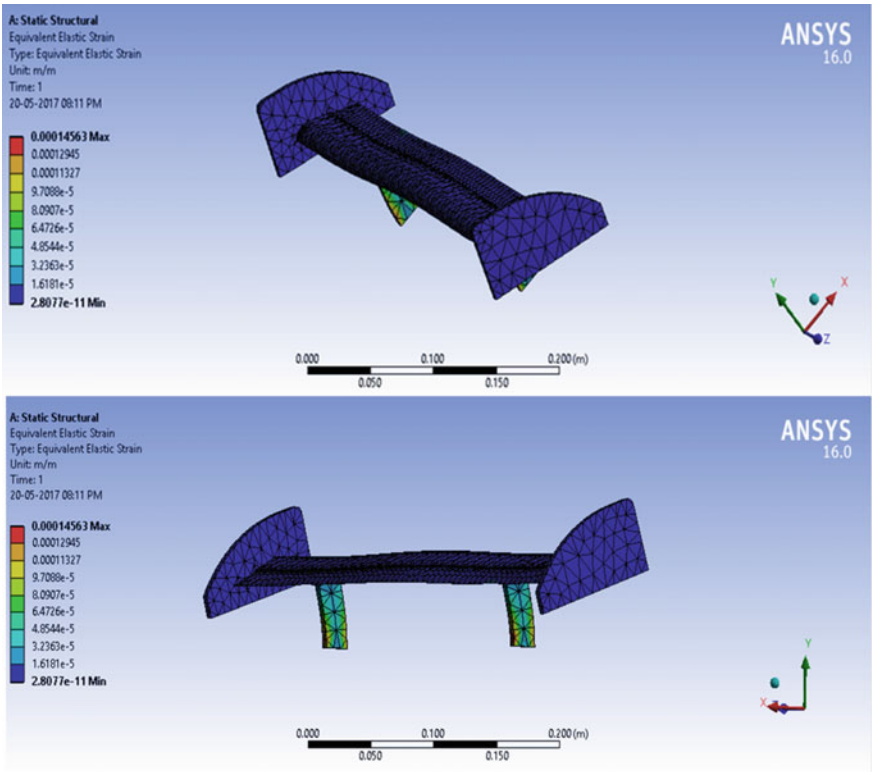

Fig. 8 ANSYS simulation

5 Conclusions

The results of the investigation of the chemical treatment of natural fiber showed that it enhances the various mechanical properties of the natural fiber-reinforced composites. The treatment of natural fiber with NaOH made the fiber surface cleaner due to the removal of waxes, pectin, hemicellulose, and part of lignin. There was an increase in surface roughness thereby increasing mechanical interlocking between fibers and the epoxy matrix. The treatment of the natural fibers with NaOH, KOH, and H_2O_2 enhances chemical interlocking at the interface and provides better adhesion with the matrix. The developed composites have the combined property of being lightweight and excellent mechanical properties than conventional materials. Thus, the developed composites can be used to replace the synthetic fiber-reinforced composites which are non-biodegradable in nature.

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Chapter 6
Mechanical Behavior of Epoxy-Based Green Composites

Divya Zindani, Kaushik Kumar and J. Paulo Davim

Abstract Keeping in view the environmental concerns and depleting petroleum-based resources, the present work proposes to develop epoxy-based green composites based on bagasse filler. The fabricated composite has been investigated for static mechanical properties, i.e., tensile strength, flexural strength, and fracture toughness. Properties have been investigated for varying percentage of filler loading and crosshead speeds. The investigation has revealed an improvement in static mechanical properties, thereby suggesting its application for a wide range of engineering applications.

Keywords Green composites · Bagasse filler · Epoxy · Tensile strength · Flexural strength · Fracture toughness · Void content

1 Introduction

The use of eco-friendly materials has increased in recent times owing to the significant concern toward the environment by society and ever depleting petroleum-based products. Petroleum-based synthetic products are now being replaced with the natural fibers that are not only eco-friendly but also possess better mechanical properties in comparison to their synthetic counterparts [1].

The trend of natural fiber usage indicates that the trend will further increase notably in developing countries. It has been estimated that the natural fiber industry will grow at the rate of 10% around the world [2]. By definition, natural fibers are not man-made and can be derived from plants or animals [3]. The research community has been focusing on the usage of natural fibers to produce composites that could benefit

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P. K. Rakesh and I. Singh (eds.), *Processing of Green Composites*, Materials Horizons: From Nature to Nanomaterials, https://doi.org/10.1007/978-981-13-6019-0_6

the engineering society at large. The plant-derived natural fibers can be categorized into bast fibers, leaf fibers, seed fibers, cotton fibers, coir fibers, and grass and reed fibers [4].

Numerous advantages have been revealed of fiber-reinforced polymeric composites such as low weight, low cost, relatively better mechanical properties, abundance [5], biodegradability, and easy processing. Addition of light and tough fibers to the polymeric matrix has resulted in natural fiber-reinforced composites with high specific strength and stiffness [6]. However, natural fibers associate with many problems as their structure comprises of cellulose, lignin, hemicelluloses, and pectin. The presence of such substances results in higher moisture absorption from the surrounding which in turn leads to weak bonding between fiber and the polymeric matrix. One of the other challenging tasks is to obtain a functional coupling between the natural fiber and the polymeric matrix. A poor bonding may result in ineffectual stress transfer through the interfaces. This calls for chemical treatments that can modify the fibers and reduce moisture-absorbing capacity, and thereby excellent compatibility between fiber and the polymeric matrix can be achieved [7]. A wide range of applications of natural fiber-reinforced composite include automotive, building industry, aerospace, sports, bicycle frame, etc. [8].

Some of the polymeric non-biodegradable matrix materials that are being used with the natural fibers are unsaturated polyester, polypropylene, epoxy, etc. Among the various matrix being used, epoxy resins are the mostly employed matrix owing to their versatile nature. Epoxy matrix is thermosetting polymers that have been employed to a large extent for producing both particulate and fiber-reinforced composite materials. Some of the main advantages possessed by epoxy resins are better strength and modulus, better thermal properties, and excellent dimensional stability.

The growth of sugarcane is abundant in different parts of India. Mills equipped with heavy rollers are used to crush the sugarcane. As a result of crushing, the squeezed cane stalk known as bagasse is left out as a by-product [9]. The disposal cost associated with bagasse is one of the major challenges for the sugarcane mills. It is one of the largest agricultural residues in the world [10–13]. The main constituents of bagasse include water, fibrous material, and small percentage of soluble solids [14]. Their percentage contribution depends on variety, maturity, and method of harvesting. The versatility of sugarcane residue has been delineated in the various literatures [10, 13, 15]. Apart from being used for the production of ethanol [16] and as a fuel for sugarcane mills, bagasse is now being used for the production of composite materials that are widely used in automotive vehicles, buses, and railway coaches. Improved mechanical properties such as flexural strength and tensile strength are obtained on following proper modification and manufacturing processes thereby expanding its potential application base.

Researchers have carried out numerous investigations on biodegradable composites reinforced with bagasse fibers [17–22]. Investigation of mechanical properties of the composite with treated and untreated bagasse fibers was carried out by Cao et al. [23]. The properties of NaOH-treated fibers were found to be far more superior to that of the untreated fibers. Compression and injection molding processes were investigated for the composite's fabrication by Luz et al. [24]. Injection mold-

ing under vacuum resulted in composites with a homogeneous distribution of fibers throughout the matrix, but the adhesiveness between the matrix and the fiber did not show any significant change.

Study on the polypropylene composites with bagasse as reinforcements was performed by Luz et al. [25], and acetylating process was employed to chemically modify the fibers. Presence of acetyl groups and reduction in OH bonds were observed through Fourier transform infrared (FTIR) spectra. The mechanical properties were found to decrease owing to the changed morphological aspects.

Mulinari et al. [26] carried out an investigation on high-density polyethylene and bagasse fiber composites. The thermokinetic mixer was used for the fabrication of composites. The modification of cellulose derived from bagasse with zirconium oxychloride yielded positive results in context of the tensile strength.

Thermal aging behavior of Al–Cu–Mg/bagasse ash particulate composites was investigated by Aigbodion et al. [27] wherein the bagasse ash particles were produced using the double stir-casting method. Percentage weight of bagasse ash was one of the major determining factors for the aging behavior which was followed by aging temperature, the aging behavior being determined using hardness value. Experimental values of hardness were found to be an excellent match with that of the hardness value determined using the mathematical model.

Cellulose whiskers isolated from bagasse were reinforced in natural rubber matrix and the effect of whiskers loading on tensile properties, thermal properties, soil biodegradation, and water vapor permeation was studied by Bras et al. [28]. Young's modulus and tensile strength were found to significantly improve with the whisker loading. Glass transition temperature of the rubber matrix, on the other hand, showed no change. The moisture absorption, on the other hand, increased with the presence of bagasse.

Two-body abrasion wear tester was employed by Mishra et al. [29] to study the wear behavior of bagasse fiber-reinforced epoxy composites. Three directions, i.e., parallel orientation (PO), normal orientation (NO), and anti-parallel orientation (APO) were investigated for comparing the wear behavior of composites. Microplugging was responsible for the abrasion in PO type samples, whereas it was microcutting that was responsible for wear process in APO and NO type samples.

A novel fabrication method for bagasse/bamboo fiber-reinforced composites was proposed by Bolzur et al. [30] and the fabricated composites were studied for their flexural properties. Effect of holding time and fibers content was investigated, and the investigation revealed a strong influence of holding time and fiber content on flexural properties of the specimen. Flexural strength was found to increase for holding time of up to 10 min. However, due to the insufficient resin, the flexural property decreased beyond 10 min. On the other hand, flexural property showed an increasing trend for fiber content up to 50% and decreased beyond 50% due to weak bonding between fiber and matrix.

Solid particle erosion behavior of bagasse fiber-based composites was studied by Mishra et al. [31]. Erosion behavior was evaluated for impingement angle varying between 30° and 90° and for four different velocities of 48, 70, 82, and 109 m/s. Brittle behavior was observed with maximum erosion rate at 90° impingement angle.

Cerquiera et al. [32] investigated the mechanical properties of composites obtained after modification with 10% sulfuric acid solution and delignification with 1% sodium hydroxide solution. Propylene was used as a matrix. The results showed enhanced tensile, flexural, and impact strength.

In the present work, bagasse dust has been used as the reinforcement. It can be obtained in abundance in Asian countries as the waste from sugarcane which is presently being used as a bio-fuel and in the manufacture of pulp and building materials. But the major part is still unutilized. Apart from this, other natural fillers are also being used recently. Among these, some examples are coconut dust and fibers, sisal dust and fibers, polysaccharide, starch, banana, kenaf, agave, pissava, etc. The most widely used and found in abundance is wood dust.

From the above literature, it is clear that the major focus of most of the researchers has been to evaluate experimentally the mechanical characteristics of the natural fiber-reinforced composite materials. The investigation has been done on the mechanical properties with the varying composite composition and the operating conditions. These studies from the time have played a crucial role in the establishment of suitability of composite materials for wide range of engineering application domain.

In the present work, samples have been fabricated as epoxy-based composites strengthened with bagasse dust as filler. The fabricated composite materials have been evaluated for their mechanical behavior.

2 Materials and Composite Fabrication

2.1 Materials

The epoxy-based thermosetting polymer was chosen as matrix material in this work. Resin AW 106 and hardener HV 953IN were the constituents of the thermosetting polymer. Better mechanical strength and bonding strength with fibers in comparison with other polymeric resins are the main reasons behind the selection of epoxy-based thermosetting polymer. High viscosity, excellent thermal stability, and dimensional stability are some of the other important properties of the chosen resin.

Bagasse dust obtained from sugarcane mill has been taken as the filler material. The ball mill was used to grind the bagasse dust. The bagasse particles were then passed through a set of sieves to obtain particle size <75 μm, finally. The magnet was then used to remove any iron particle from the finally obtained particulate. Multistep cleaning process comprising of magnetic stirring and specific water and acetone washes was followed to remove grease, oil, and mixed soil particles. The obtained particulates were then washed multiple times with distilled water to remove any other remaining contaminants and were then heated in an oven at 70 °C for 8 h. The successive drying of fillers has been carried out with due consideration to fiber degradation and decoloration.

The bagasse filler is a hygroscopic material and hence cannot be thoroughly dried by conventional hot air dryers, because the humidity in hot air dryers is dependent on ambient conditions. They are also relatively incompetent in reducing water content. The filler requires a consistent low dew-point dry air (under 32 °C) and constant drying temperature which ensures a lesser moisture content than the parent filler material. If this is not attained, the composites can never show their optimum mechanical performance. For this, the bagasse fillers were dried in a dehumidifier at a specified temperature and period.

2.2 Chemical Treatment

Bagasse filler was surface modified using alkaline treatment. The direct influence of hydroxyl group on the cellulosic content of the fiber and the extraction of lignin and hemicellulosic compounds were the main reasons for selection of alkaline treatment. 5 wt% of aqueous NaOH solution was used for oxidation of bagasse filler and the reaction was carried out in a beaker with magnetic stirring. The filler was washed using distilled water and acetone and the process was carried on until pH was reached. The filler was dried in an oven at 70 °C and then stored in airtight polyethylene bags.

2.3 Composite Fabrication

Hand layup technique was used for the fabrication of composite samples from the alkali-treated bagasse fiber and epoxy matrix. Molds from silicon rubber were prepared for the production of composite specimen. ASTM standards, i.e., ASTM D-638 type V and ASTM D 790 for tensile and flexural tests were, respectively, followed to obtain molds with dimensions 63.5 mm × 10 mm × 3.2 mm and 65 mm × 12.7 mm × 3.2 mm. Resin and hardener were mixed in the ratio of 10:8 and stirred mechanically at 100 rpm for 10 min. Bagasse in desired wt% was also added to the resin and hardener mixture and the entire mix was stirred at 225 rpm for 15 min. A mixture of epoxy matrix and bagasse filler was poured into the silicon rubber mold and the mold with the mixture was placed in the vacuum desiccator. The composite specimen in the mold was cured for 12 h at room temperature. After curing at room temperature, the whole setup was placed in the muffle furnace and heated at 70 °C for 3 h. Samples were then taken out of the mold for the purpose of testing. Composite specimen was prepared with filler loading of 2.5, 5, 7.5, 10, and 12.5% for carrying out the investigation.

3 Testing Procedure

3.1 Density and Void Content

The quality of fabricated composite material can be assessed through measurement of void content. It is one of the important physical properties that can greatly affect the performance characteristics of the fabricated composite material. It is calculated by measuring the difference between theoretical and experimental density. Weight additive principle equation is used for the calculation of theoretical density and experimental calculations are done by immersing the composite sample in water and then using electronic balance. The difference is then calculated between the two densities to arrive at the percentage void content. ASTM D 2734-94 standard is adopted for assessing the void percentage of the fabricated composite samples.

It was observed that the void content of the composite specimen increases with the increase in the filler percentage and reaches its maximum value at 12.5%. Hydrophilic nature of bagasse filler may be attributed as one of the major reasons leading to the increase in the void content since this property allows for maximum moisture absorption which later on evaporates during the curing process leaving behind pores and voids. The air entrapment also increases with the increasing filler content due to the increased surface area. The curing of the composite specimen resulted in generation of air bubbles. Voids are formed when these air bubbles leave the cured specimen. In the fabricated composite specimen, void content was found to be 3.5%.

3.2 Tensile and Flexural Testing

The tensile and flexural behavior of the fabricated composite materials was obtained by employing the Universal testing machine. The testing machine provided for tensile strength and modulus and flexural strength and modulus. ASTM D 638 standard was adopted to obtain the mechanical behavior of the fabricated composite materials. The specimens for mechanical tests would be of the shape of a rectangular bar of size 65 mm $\times$ 12.7 mm $\times$ 3.2 mm thickness. ASTM D 790 standards are also different in two sections. Both the tensile and flexural behavior was obtained for a varying percentage of filler material and for various crosshead speeds.

The acceptance of the composite material for the engineering application could be well judged with the tensile testing of the material. The stress–strain curve helps in the recording of values for ultimate tensile strength, elongation at break, and modulus of elasticity for the composite sample. Effect of variation of filler content and the crosshead speed on the mechanical properties of the composite specimen was obtained. It was established that the addition of filler to the matrix enhanced the tensile strength value. The tensile strength became maximum for 5% of filler weight. However, with the further increase in filler content, tensile strength was found to decrease. The reason can be attributed to the inadequate bonding of the fillers with

the epoxy matrix. Young's modulus, on the other hand, was recorded to be maximum for 7.5 wt% of filler weight. Increased filler content rendered the composite material to develop better resistance to deflection and was one of the reasons that could be attributed to different filler wt% for maximum tensile strength and Young's modulus. The difference has also been highlighted by various researchers [33]. Increased percentage of crystallinity of the composite specimen with the increased filler content could possibly be another reason since the crystals possess higher moduli in comparison with the amorphous region in the composite [34, 35]. Further, the associated strain value of the composite specimen was found to decrease with the filler content. The reinforcement of stiffer bagasse fiber in epoxy matrix could well be one of the reasons that could be attributed to the decrease in a strain of the composite specimen.

The tensile strength value was also affected by the crosshead speed. Fracture initiation rate and propagation were also affected by the crosshead speed. The tensile strength was found to be increasing with the crosshead speed increasing from 1 to 2 mm/min, reaching its highest value at 2 mm/min. Tensile strength then began to fall steadily for all the loading conditions. Due to the lesser amount of time available for stress transfer among the filler and the epoxy matrix, as a consequence, the composite specimen failed at much lesser value of tensile strength.

3.3 Flexural Testing

ASTM D 790 standard was followed to investigate the flexural properties of treated and untreated bagasse filler-based composite specimen at three different crosshead speeds of 1, 2, and 3 mm/min. Flexural strength was found to increase with the increasing filler amount. The increasing trend was observed for up to 10% filler loading. Improved filler–matrix interaction is one of the major reasons that could be attributed to this enhanced flexural strength. However, due to filler agglomeration and trapping of resin resulted in deterioration of flexural strength at higher filler loading of 12.5%. Further, higher flexural strength was reported for alkali-treated bagasse filler-based composites in comparison with the untreated composite specimen. The flexural strength was also found to be affected by the crosshead speed. Flexural strength was revealed to increase with crosshead speed increasing from 1 to 2 mm/min. However at 3 mm/min, the strength started to deteriorate. The reason may be attributed to the less time given to the fillers to orient them in the loading direction.

Flexural strain value decreased with the filler loading. Loss of filler ductility in comparison with the neat epoxy may be attributed as one of the reasons behind falling flexural strain value. Formation of stress concentration spots at higher filler loading may be another reason behind lower strain value. Stress concentration may result in early crack initiation and ultimate failure.

3.4 Fracture Toughness

The fabricated polymeric composites were analyzed for fracture toughness and fracture energy. An initial drop in fracture toughness with the addition of filler particles was observed in comparison with the neat epoxy sample. The drop was observed for 2.5% filler loading. However, with further increase of filler % from 2.5 to 10%, the fracture toughness showed an increasing trend and the value was higher in comparison with the neat epoxy sample. Fracture toughness declines with the further addition of filler to 12.5%.

Addition of filler to epoxy led to increase in fracture energy in comparison with the neat epoxy. The value increased from 95 to 205% for different filler loading in comparison with the base matrix. The maximum value of fracture energy was found for 12.5 wt% filler loading. A drop in fracture energy is observed for filler content varying between 2.5 and 5%, beyond which a consistent increase in fracture energy was observed. Evolution of stress concentration at different locations is the reason that can be attributed to the decreased in the fracture energy.

4 Conclusions

The present work investigates mechanical behavior of novel epoxy–bagasse composite material. Investigations have been done on mechanical behavior of the fabricated composite by taking into consideration the following mechanical properties: tensile strength and modulus, flexural strength and modulus, and fracture toughness. The fabricated composite has improved mechanical properties and can effectively endure various static loading conditions. The fabricated composite has increased void content with the increased fiber loading and the maximum value was recorded as 3.5%. Increased filler content led to increased tensile strength and Young's modulus. The maximum tensile strength was recorded for filler loading of 5%, whereas Young's modulus was maximum for filler loading of 7.5%. No significant changes in mechanical properties were observed with the strain rate. Further, Fracture toughness of the fabricated composite was also revealed to be higher than the neat epoxy sample. Higher flexural strength was reported for alkali-treated bagasse filler-based composites in comparison to the untreated composite specimen. The flexural strength was also found to be affected by the crosshead speed. The effect of crosshead speed on mechanical properties with much wider range can be investigated in future.

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Chapter 7
Silk and Silk-Based Composites: Opportunities and Challenges

Lalit Ranakoti, Manoj Kumar Gupta and Pawan Kumar Rakesh

Abstract Increasing demand for lightweight material has moved the industries to switchover to the polymer-based composite materials. The polymer composite materials mainly use synthetic fibers like carbon fibers and glass fibers, which generally show better mechanical properties. The integration of these composites has increased drastically from aerospace to automobile industries. Further, the drawback of non-biodegradability is always associated with the synthetic fiber-reinforced polymer composites. Natural fibers, on the other hand, are cheap, readily available, and biodegradable, which is thought to be a better alternative to synthetic fibers. Among the natural fibers, silk fiber is one of the most durable fibers and has excellent mechanical properties like stiffness, strength, and ductility, etc. Nowadays, silk fiber is being used extensively for the engineering and medical applications due to its several attractive properties in which environment-friendliness is hugely alluring for the researchers.

Keywords Silk fiber · Biomedical · Sericulture · Degumming process · Scaffolding

1 Introduction

The demand for products based on polymer matrix composites (PMCs) is increasing day by day. The synthetic fibers PMCs have made their place in every sector of the industry such as the automobile, electronics, textile, and medical due to their numerous attractive properties such as strength, durability, lightweight, ease of fabrication, and ease of availability. [1]. Besides all these features, the drawbacks inherent in

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P. K. Rakesh and I. Singh (eds.), *Processing of Green Composites*, Materials Horizons: From Nature to Nanomaterials, https://doi.org/10.1007/978-981-13-6019-0_7

these polymer composites are their non-biodegradability. These polymer composites when dumped after their end uses do not degrade and hence affect the soil and the environmental conditions [2]. To alleviate the effect on the global environment, fully or semi-biodegradable materials are being prepared with the combination of natural fibers and biodegradable polymers. The synthetic fibers are now replaced with the natural fibers for reinforcing the biodegradable polymers to manufacture sustainable composites. Mostly used natural fibers for the composites are plant and animal fibers such as silk, sisal, wool, yak, abaca, agave, alpaca, bamboo, banana, calotropis, camel hair, cashmere, coir fiber, cotton, date palm, flax, hemp, jute, kapok, kenaf, mohair, pashmina, pineapple, rabbit hair, and ramie. Silk fibers are one of the most extended continuous fibers and most active animal fibers known to the humankind, among all the naturally available fibers [3–6]. The properties of silk fibers especially tensile strength and elongation at break are highest among all the natural fibers. This makes the silk fibers as one of the important natural fibers that could be used as reinforcement in the biodegradable polymers. It is biodegradable and hence leaves no harmful traces on earth after dumping. Silk fiber is mostly used in textile industries like handlooms and sarees. Silk is known as the sign of the lavishness in the society. Now it is also being used in the medical field, i.e., scaffolding, tissue generation, nerve regeneration, etc.

2 Types of Silk Fibers

Silk is a natural protein fiber. Caterpillar and mulberry are the two most common silkworms used for the production of silk fiber. Silk fiber is unwound from the cocoon produced by the silkworm. It is mainly consists of fibroin 75% and sericin 23%. The remaining percentage is a mixture of fat, wax, and mineral salt. It also has the essential attraction toward manufacturing of the natural dyes [7]. The different types of silk fibers have been discussed in the following section.

2.1 Mulberry Silk

Mostly cultivated silk in the world is known to be the mulberry silk. It constitutes 90% of the share of all the silk cultivated around the world. Bombyx mori is the silkworm which produces mulberry silk [4]. This silkworm is fed from the mulberry bush. Mulberry silks are abundantly available particularly, in China, Japan, and Korea. Since it is easy to acquire from the cocoon, it is acquired by killing the insect in the cocoon itself to obtain longer fibers. It is exclusively softer, finer, and more lustrous. These silk fibers required intense attention for the maintenance of its smooth texture. Generally, white shade color is obtained in these types of silk fibers. Figure 1 shows the Bombyx silkworm, its cocoon, and silk fibers.

Fig. 1 Silkworm, cocoon, and fiber of mulberry silk

Fig. 2 Silkworm and tasar silk fiber

2.2 Tasar Silk

Tasar silk is produced by tasar silkworm and it is also called as the wild silkworm. The natural color of tasar silk is usually not white; in fact, its color is dark brown to golden. This is due to the fact that the silkworm of tasar is fed the leaves rich with tannin. It is thicker and coarser than other types of silks which makes it suitable for the manufacturing of couches, jackets, and sweaters, etc. Figure 2 shows tasar silkworm and tasar fibers [5].

2.3 Muga Silk

The muga silkworm known as *Antheraea assamensis* is semi-domesticated for the production of muga silk. In other words, muga can be said akin to tasar silk. Muga silk is majorly cultivated in India. Its color has a range from golden brown to glossy texture. Muga is more eco-friendly than any other silk as delicate care is not required for muga silk. One thing to be noted in case of muga silk is that the bleaching process is not straightforward due to the porosity in the fibers. Figure 3 shows the muga silkworm and its fibers [8].

Fig. 3 Silkworm and fiber of muga silk

Fig. 4 Silkworm and eri silk fiber

2.4 Eri Silk

Philosamia ricini is the silkworm for the production of eri silk. Eri silk is very much similar in color with Bombyx mori silk, and it is fine too. Eri is derived from the domesticated silkworm but it is called peace silk because of the caterpillar of the cocoon from which the eri is obtained. It is not killed rather it is allowed to develop as moths and live the full life cycle. Eri silk is spun and not reeled due to the fact that after the development of moth, the cocoon is destroyed after which the eri silk is obtained. The appearance of Eri is in the form of the mat which looks similar to wool or cotton. It has high durability which is the suitable property for the application of clothing and soft furnishings like curtains and couches. One disadvantage associated with eri silk is washing, which takes much labor during the process [9]. Figure 4 shows eri silkworm and eri silk fibers.

3 Silk Fiber Processing

The silk fiber is processed from the silkworm in four stages: (a) sericulture, (b) hatching the eggs, (c) feeding period, and (d) spinning the cocoon.

3.1 Sericulture

Sericulture is known as the cultivation of silk from the silkworm, but commercially cultivated silk is the filament which is produced by Bombyx mori called as mulberry silk. Mulberry silk moth and a few others are of same genes, and remaining all others belong to different genes. Technically, the silkworm is not a worm instead it is a moth pupa [10]. The term silkworm is used for the sake of simple understanding.

3.2 Hatching the Eggs

In the beginning, the eggs of silkworm are kept in a controlled environment to make sure that the eggs are free from any disease. The eggs are deposited by female silkworm which is around 300–400 numbers at a time. The lots of space are required for the deposition of these eggs, i.e., equal to the area of the computer monitor screen. Female and male silkworm both have the different life cycle. The death of female occurs at the instant of deposition of eggs while the male survives for a little time. The male silkworm holds an undeveloped moth pupa. The eggs deposited by the silkworm are reared for about 10 days till they are shaped into larvae, i.e., caterpillars. At this moment, the larvae are developed or hatched to about 60–70 mm [11].

3.3 Feeding Period

After the completion of hatching, the larvae are kept underneath a fine layer of gauze. Here these larvae are fed massive amounts of sliced mulberry leaves. During the process of feeding, the larvae shed their skin around four times. Osage orange and lattice leaves are also eatable food for larvae. The eating of silkworm last for around 6 weeks till it grows to 3 in. which generally occurs in as stated 6 weeks. The eating of silkworm stops and the color also changes after 6 weeks of eating. At this moment, the weight of the larvae becomes 10,000 times heavier than the weight during the time of hatching of the eggs [12].

3.4 Spinning the Cocoon

The spinning of the cocoon is the last step before extracting the silk fiber from the cocoon. In this step, the silkworm is reared and housed for 3–8 days and left it to spin. During spinning, the silkworm attaches itself to tree, twig, or shrub. Sometimes this period is also known as pupating. Sericteries is a pair of modified salivary gland possessed by silkworms which is the vital element that produces fibroin. The sericteries has a characteristic of the clear, viscous, and proteinous fluid. This fluid is made to come out from the openings present at the mouth of larvae known as a spinneret. This spinneret is the only tube for the exit of the present fluid at the head of the silkworm [3]. The silk thread obtained from the spinneret has the diameter equal to the diameter of the spinneret. Usually, the fiber that comes out is in the form of a long and continuous filament. The secretion continues further and gets hard as it comes in contact with air. This hardness of secretion is composed of two forms: One is fibroin which is a protein material; this fibroin is the main constituent of the fiber as 70–80% of fiber consists of protein. Another one is sericin; it is produced from the second pair of the gland. The sericin has a very special role to play. Sericin has a property of gum-like material and it binds the two layers of filament together. To form a single strand of silk fiber, usually 4–8 cocoons can be unreeled or unwound with a single twist. Sericin also protects the fiber during processing. Sericin is generally removed from the fiber which makes it 30% lighter than the raw one. But in the case of yarn and woven fabric, sericin is often left. Cleaning of silk fiber is known as treatment [4]. This treatment can be carried out by various solutions like NaOH, the silane compound, makes the silk fiber soft and lustrous. Silk obtained from the cocoon is very meager, that is why it is very costly. For the production of a pound of raw silk, at least 2500 silkworms would be possibly required [13]. The full silkworm cycle that is from the sericulture to the spinning of the cocoon is shown in Fig. 5.

4 Properties of Silk Fibers

Silk fiber is produced by the insect which classifies it in the category of animal fibers. Being a protein fiber, silk fiber is the only natural fiber which is found in the form of the filament. The main component of the silk fiber is the fibroin which is responsible for the strength of the silk fiber. The physical, chemical, and mechanical properties of the silk fibers are important to select the polymer which has better adhesion properties towards silk fiber. The manufacturing process of silk fiber-reinforced polymer composite has to be selected. Silk fiber is judiciously environmentally stable than any other protein fiber, i.e., found in the earth crust due to the extensive hydrogen bonding, hydrophobic nature, and substantial crystallinity. The continuous filament silk fiber produced by B mori silkworm contains 18 amino acids as shown in Fig. 6. These 18 amino acids are in polypeptide chain which forms the main structure of silk fiber. These acids are alanine (Ala), glycine (Gly), tyrosine (Ty), and sericin (Ser).

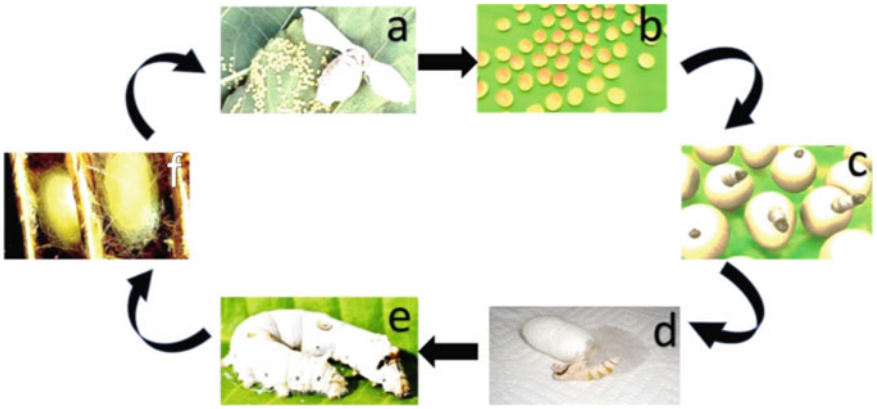
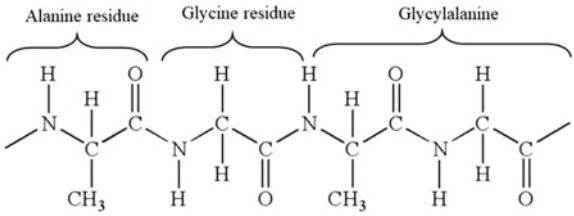


Fig. 5 Life cycles of silkworm: **a** the female moth lays tiny eggs; **b** a tiny black caterpillar hatches out if its egg; **c** after 10 days of hatching the eggs; **d** worm coming out from the cocoon; **e** spinning of cocoon; and **f** the caterpillar changes into the moth pupa

Fig. 6 Molecular structure of silk fiber



These acids together contribute around 80% of the protein of silk fiber [14]. Silk is generally soluble in water due to the content of fibroin and sericin.

Physically, silk is round at the corners having the triangular cross-sectional dimension which is about 5–10 μm of width. Because of its molecular structure and presence of protein, silk perhaps manifest the piezoelectric effect which can be applied in the field of electric-energy generation. Silk has the property to resist many acid compounds, but it encourages the sulfuric acid.

Silk fiber is obtained in yellow, brown, gray, and lusty white color. Its specific gravity is around 1.25–1.34. Moisture regain percentage of silk is around 11%. Silk can withstand high temperature than wool and it can remain unaffected at 140 °C for a prolonged period. The decomposition temperature of silk is 175 °C. Sunlight increases the rate of decomposition of silk fiber by atmospheric oxygen [15]. Some commonly known types of silk fiber and their properties are listed in Table 1.

Table 1 Properties of different types of silk fiber [31–36]

S. No.	Properties	Mulberry	Tasar	Eri	Muga
1.	Moisture regain % at standard atmosphere	11	8.44	11.18	10.9
2.	Ash contents	0.5–0.9	2.75	1.62	2.06
3.	Fatty matter %	2.3	1.8	–	0.08
4.	Solvent	5% NaOH at the boil	12 N NaOH at the boil	12 N NaOH at the boil	12 N NaOH at the boil
5.	Microscopical examination	Triangular cross section no well-defined striations	Wedge-shaped cross section. Flat ribbon with convolutions	Flat rod-structured surface striations	Triangular cross-sectional surface striations
6.	Natural color	White, yellow or yellowish green	Brown	Brick red or creamy white	Light brown or golden
7.	Tenacity (gm/Dn)	3.1–3.6	1.9	3.0	2.5–3.0
8.	Elongation %	18	22–25	28–28	20–22

5 Chemical Treatment of Silk Fiber

Poor interfacial bonding with the thermoplastic matrix is the significant weakness of silk fiber. This interfacial bonding depends on the intermolecular structure of fiber and polymer as interfacial bonding plays a vital role in defining the final strength of the composite. During the manufacturing of silk fiber composites, the interfacial bond formed for the hydrophilic group with the polymer matrices is of inferior nature. This poor bonding leads to overall poor mechanical properties of the composites. Hence, the fibers which are not appropriately bonded weaken the content of matrix and cause the imperfection. Another reason for the poor mechanical strength of composites could be the formation of the inter-fiber hydrogen bonding. The chemical treatment of the silk has been tried to improve the mechanical properties. Generally, the degumming process is employed to increase the adhesion of fiber and matrix materials. Various other surface treatments like a chemical reaction, pre-impregnation, surface modification, and plasma treatment have also been employed to improve the adhesion of the silk fibers with the polymer matrices.

5.1 Degumming Processes

Degumming is a process involving the removal of the upper layer of silk fiber called sericin. This sericin is removed by keeping the fiber in the alkali media. It is found that the elasticity of the fiber increases on the removal of the sericin from the fiber. Various degumming methods have been created, and some are under investigation such as boiling off in soaps or alkali, enzymatic degumming, extraction with water, degumming in boiling acidic solution, and genetic modification [16]. For the removal of sericin from the silk fiber for the preparation of silk yarn/web, it is kept in a vessel having fiber to liquor ratio of 1:40. For example, 7 g soap and 1 g soda per L are added to the solution. The solution is then boiled for 1 h. Now, the fiber is thoroughly dried to remove the moisture. The presence of amino acid group is the main reason behind the solubility and crystallinity of silk fibroin. Hence, distilled water is most commonly used for the degumming process. Generally, soap and soda solution is recommended; however, treatment with enzyme and acid are also in trend [17].

6 Mechanical Properties of Silk Fiber-Reinforced Composites

The method of processing of raw materials plays an essential role during the manufacturing of composites.The specific property required in the composite mostly depends on the process by which it is prepared. It is suggested that the composites of silk fiber are being manufactured using techniques like injection molding, compression molding, casting, extrusion, freeze drying machine, hand lay-up, and vacuum bag molding [18]. In the field of biomedical, polymers like polylactic acid has boundless applications. Improvement in the tensile and elastic properties can be seen in silk-PP composites with an increase in silk fiber proportion. It is found that the impact strength of silk fiber-PP composite is equivalent to glass fiber-reinforced plastics. The mechanical properties of silk fiber-reinforced polymer composites are shown in Table 2.

7 Applications of Silk Fiber-Reinforced Polymer Composites

The characteristics of silk fiber like strength, absorbance, luster, and moisture make it appropriate for the textile industries. The composite of silk fiber is manufactured with various bio and non-biodegradable polymers. These fabricated composites are tested for mechanical, thermal, wear, and electric properties which come out to be fruitful to be used in various engineering applications. Molecular compound conforms that majority of silk fiber contains the presence of protein which makes the silk fiber most

Table 2 Silk fiber composite with various polymers with varying percentage [25–30, 37–46]

Composites	T.M. (GPa)	T.S. (MPa)	F.M. (GPa)	F.S. (MPa)	Remarks
Silk/PLA	2.54	62.08	–	–	5 wt%, long fiber
Silk/gelatin	0.65	44.5	3.7	63	20 wt%, long fiber
Silk/PP	1.8262	54.7	3.5707	58.3	20 wt%, long fiber
Silk/HDPE	0.4184	31.1	–	–	6 wt%, chopped fiber
Silk/PBS	1.6	42	3.9	119	30 wt%, chopped fiber
	1.9	48	5.1	150	40 wt%, chopped fiber
	2.3	50	6.5	155	50 wt%, chopped fiber
Silk/PCR	–	11.45	–	–	20 wt%, chopped fiber
Silk/PP	0.4546	38.9	–	–	6 wt%, chopped fiber
Silk/PC	0.6968	36.7	–	–	3 wt%, chopped fiber
	1.9	48	5.1	150	40 wt%, chopped fiber
	2.3	50	6.5	155	50 wt%, chopped fiber
Silk/PCR	–	11.45	–	–	20 wt%, chopped fiber
Silk/epoxy	0.844	58.35	1.503	60.81	25 wt%, fabric
Silk/EPCNSL	1.067	69.98	1.92	71.21	25 wt%, fabric
Silk/epoxy	–	87.52	–	135.81	25 wt%, fabric
Silk/epoxy	5.4	60	5.2	143	36.2 vol%, nonwoven fabric
Silk/epoxy	6.5	111	6.4	250	45.2 vol%, plain woven fabric
Silk/PBS	0.72	40.6	0.59	49.04	30 wt%, fabric
	0.88	50.1	0.67	56.08	45 wt%, fabric
	1.03	62.5	0.87	65.19	60 wt%, fabric
	0.82	82.6	0.61	33.09	75 wt%, fabric
Silk/wheat protein	1.089	18.8	–	–	1.5 wt%, short fiber

(continued)

Table 2 (continued)

	1.396	20	–	–	2.5 wt%, short fiber
	1.605	28.4	–		5 wt%, short fiber
Silk/epoxy	1.478	31.49	1.98	43.66	5 wt%, short fiber
	1.52	36.5	4.059	90.82	10 wt%, short fiber
	1.649	41.31	8.752	147.04	15 wt%, short fiber
	1.949	48.74	1.119	188.07	20 wt%, short fiber
Silk/epoxy	–	41.1	–	53	10 wt%, long fiber
Silk fiber/PLA	4.08	70.6	4.6	97.41	5 wt%, long fiber

suitable for biomedical applications. It is suggested that the silk fiber is playing the vital role in the field like the suture, wound dressing, tissue scaffolding, cell adhesion improvement, drug delivery, etc. Matter of fact is that silk fiber has dominantly captured the sphere of biomedical where the need of protein fiber is essential due to its biodegradability as silk fiber gets dissolved quickly in the human body [19, 20]. The application-based results of silk fiber-reinforced polymer composites in engineering, medical, and textile field are discussed in the following sections.

7.1 Textile Industries

Ever since the textile industry started making fabrics, silk has remained one of the critical fibers among all the natural fibers. Due to its shiny and luster appearance, its demand in the world has remained unchanged. Fabrics based on silk fiber have undergone various types of mixing routes with several synthetic fibers to fabricate the desired products. These fabrics have shown promising results concerning strength and longevity [21]. Essential characteristics required for a suitable textile material have improved water absorption, antibacterial property, and stable to photo-oxidation. Polyacrylonitrile and its copolymer possess all these properties which are required for the manufacturing of durable fabrics. Silk fiber when mixed with polyacrylonitrile resulted in improved water absorption rate and reduced conducting property [22]. Naturally occurring fibers like cotton, jute, hemp, etc., are rich in cellulose. Silk fiber and cellulosic fibers when combined in the ratio of 30 and 70% resulted in improved elastic modulus but breaking strength decreases. Cellulosic fiber can be used solely where requirement of yield stress is high. These materials can be used

where the requirement of elasticity is high like head and hand band [23]. Coating of silver nanoparticles to the surface of silk fibroin prevents the development of specific kind of bacteria known as Staphylococcus aureus. Formation of this bacteria shows the sign of dullness at the surface of the fabric. Combining silk with wool in the ratio of 60:40 resulted in a product of superior quality. The obtained product has the quality of high strength, warmth, comfort, and mild luster. This material lacks in fire-retardant characteristics which can be improved by the treatment of phytic acid (a plant tissue contains phosphate groups). Various methods have been implemented to increase the hydrophobicity of silk textile. Spraying solution of silica nanoparticles and water called siloxane on to the silk fabric form a protective layer on it. Silver-coated silk fabric can be used in those areas where the environment possesses a lot of moisture. Moreover, a coating of silk does not affect the appearance of silk fiber [24]. The techniques such as Teflon coatings, Carol ZN liquid, and ceranine HSC/HCl liquid are used to make stain-resistant silk fabric. *N*-methylene bisacrylamide, triacryloyl polyethylene glycol methacrylate, etc., are some of the compounds used to treat the silk fiber for the enhancement of antistatic property and hygroscopic property. Silk grafted with vinyl monomers, silk cross-linked with formaldehyde, and non-formaldehyde reagents are some processes that can enhance the creased property of silk fabric. Pyrovatex (CP), diethyl-2-(methacryloyloxyethyl) phosphate (DEMEP), and hexakis (methoxymethyl) melalmine (HMMA) are some of the chemicals which when used for the treatment of silk that increases the flame-retardant property and durability. Ultraviolet rays (UV) are the primary cause of degradation of silk fabric when it is exposed to the environment. Some UV absorbers are namely, *o*-hydroxyl benzophenones, *o*-hydroxyphenyltriazines, *o*-hydroxy hydrazines, etc., are used to treat silk fabric and contain the ortho-hydroxyl group which helps in absorption of UV rays and are released in the form of heat keeping the fabric safe [25].

7.2 Biomedical Applications

Suturing is the area where the silk fiber has dominated for centuries due to strength, biocompatibility, and low immunogenicity. Suturing is known as the process of stitching the wounds. Silk fiber-based thread is used to suture the wounds to achieve the high healing as silk dissolves in the skin due to its pretentious property. The wet spinning of silk fiber with formic acid solution possesses a remarkable breaking strength. This breaking strength is achieved for the loss of inflexibility, low knot strength, and increase in brittleness. Addition of polyvinyl alcohol by 50 wt% resulted in the enhancement of elongation at break and tenacity. Fiber for antimicrobial suturing can be prepared by coating the silk fiber with silver nanoparticles [26]. Silk fiber mats have also been made for the application of wound dressing. Fibroin form of silk has also applications in antibacterial coatings. Body implants like hip replacement are materials requiring a high degree of compatibility. Polyamide coated with silk fibroin proves to be a better alternative for the hip replacement. Composite prepared

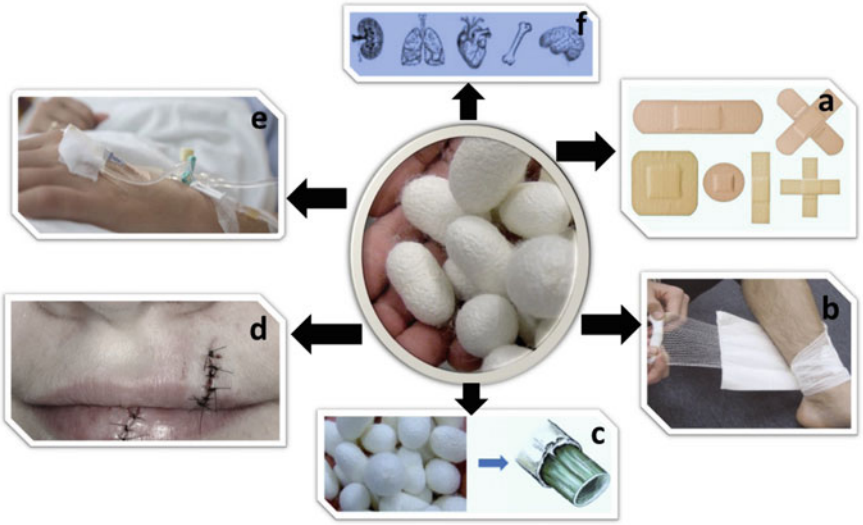


Fig. 7 Silk fiber in the medical field **a** bandaging, **b** wound healing, **c** scaffolding, **d** stitching, **e** drug delivery, and **f** nerve generation

from silk fibroin and polyallylamine leads to a material which reduces the problem of water solubility and results in a more stable material in the water medium.

Degradability property of biomaterial is of great concern. At what rate and for what period, the biomaterial is degrading should be known. Figure 7 shows the typical applications of silk fiber composites which are extensively used in the biomedical field.

7.3 Engineering Applications

Silk fibers-reinforced polymer composites with various polymers have been manufactured with the view of their engineering applications. A unique property that silk fiber possesses is its strength, elongation, and stiffness increase as the loading increases [27]. This makes it suitable for the automobile industry. Reduction in the CO_2 content is also associated with the use of silk fiber composites [28]. Use of silk fiber composites can also be seen in building material. Better insulation of silk fiber has influenced the interest for its use in building industries. Silk fiber-reinforced polypropylene composites possess better mechanical properties than glass fiber-reinforced plastics, and it can be used in household buildings [29]. Besides using cellulosic fibers, using silk fiber composites bring out the interior household more glossy and silky. Silk can also be used in the form of laminates with wood as the core for flexible interior laminated products. These laminates can be used for decorative

purposes. Flowers and radiant shades of lamps can also be manufactured with the use of highly embellished silk papers. For the decoration of plastics, fabrics and silk powder can be used for beautification. Art and craft is another field where silk fiber shows great dominance. Silk waste can be used to carve dolls, pen stand, wreath, clocks, greeting cards, etc. [30]. These composites are prepared from silk fiber with different polymer matrixes. Changes have been made by changing the fiber percentage and by using short silk fiber, long silk fiber, and silk fiber in the form of mat or fabric.

8 Conclusions

In this chapter, the processing of silk fibers, manufacturing techniques and its application in various fields like textiles, engineering, and medical components are discussed. The mechanical properties of silk fibers depend upon the cultivation technique and its processing conditions. The chemical treatment of silk fiber is required to improve the surface adhesion properties of fibers. The selection of the silk fibers with the biodegradable polymer is a big challenge for the researchers. Silk fiber-reinforced polylactic acid composites has the unbound application in medical industry.

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Chapter 8
Machining of Thermoplastic Composites

Furkan Ahmad, Ankit Manral and Pramendra Kumar Bajpai

Abstract Evolution of high-strength thermoplastics has made it possible to replace thermosets in the field of fiber-reinforced polymer composites. Though, near-net-shaped products can be made out of fiber-reinforced thermoplastics using available processing methods, still, in most of the cases, assembling is required to get the desired shape of products. Drilling is the most often required machining process in fiber-reinforced plastics industry in order to assemble various components into a finished product. A number of products get rejected at the stage of joining due to the damage induced in the component at the time of drilling. Thus, it has become necessary to evaluate the parameters which may affect the quality of the drilled hole. Drilling of the thermoplastic-based composites is much different than that of thermoset-based composites in various aspects. The present chapter is an attempt to understand the complexity behind machining of fiber-reinforced thermoplastic components due to their heterogeneous nature. The present chapter also emphases numerous problems occurring while machining thermoplastic composites and their possible solutions. The discussion on the machinability of thermoplastic composites and the effect of machining parameters like feed rate and cutting speed, etc., on the integrity of thermoplastic composites is also included in the present chapter.

Keywords Drilling · Thermoplastics · Composites · Feed rate · Spindle speed · Problems · Twist drill

1 Introduction

Fiber-reinforced plastics (FRPs) are man-made materials used in various products where high strength to weight ratio is required. Recently, thermoplastics have gained a lot of importance as a potential matrix material in polymer-based composites. Even

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P. K. Rakesh and I. Singh (eds.), *Processing of Green Composites*, Materials Horizons: From Nature to Nanomaterials, https://doi.org/10.1007/978-981-13-6019-0_8

though near-net-shaped products are manufactured using various processing techniques available in the market, the machining of thermoplastics is an unavoidable step in the production cycle of composite material-based products. The machining spectrum of material includes orthogonal/oblique cutting, turning, drilling, milling, and finishing operations. Orthogonal/oblique cutting is an operation where the wedge-shaped cutting tool is orthogonal or at an angle to the tool motion, removing the material in the form of continuous or discontinuous chips. Turning is an operation in which the workpiece rotates while the cutting tool moves along the axis of workpiece with some depth of cut.

The drilling operation is used to make holes in components with the help of various types of drill bits available in the market. Milling is also used to remove unwanted material from a workpiece by moving the rotating cutters in a specific direction. Finishing operations are required to obtain the desired surface finish of the component [1]. The surface finish of polymeric composite materials is very good due to the glossy outer layer of the polymer, hence ruling out the requirement of finishing operations for polymer composite materials. Out of all machining operations, drilling is the most practiced machining operation in case of fiber-reinforced polymer composite materials in order to assemble the various components into a useful product [2]. In the present chapter, the emphasis is on the hole-making processes by conventional and non-conventional methods. Machining of FRPs is different from machining of conventional materials due to non-homogeneous nature of FRPs. The phenomenon of the interaction of cutting tool and the composite workpiece is quite complex and still under the research phase. Thermoplastics can be easily melted in comparison with thermosets due to linear chains of molecules connected by secondary bonds (Van der walls forces) which are easier to break. On the other hand, reinforcements are mechanically strong and thermally poor conductive materials [3]. Thermoplastics are usually called linear plastics and thermosets are known as network plastics [4]. Though thermosets dominate in FRPs-based applications, thermoplastics are gaining popularity due to recyclability, shorter curing-time, and appreciable toughness. Diaz-alvarez et al. [5] experimentally analyzed the damage induced while making holes in various bio-composites. Authors used polylactic acid (PLA) as matrix material while cotton (basket weave), flax (plain weave and basket weave), and jute (plain weave) were used as reinforcement. Compression-molding method was used for the fabrication of composite laminates. Authors quantified the induced damage in terms of delamination factor. Delamination factor was defined as the ratio of maximum diameter of drilled hole containing all the damage forms to the nominal diameter of drilling tool. Authors evaluated the damage factor at the entrance side of hole as well exit side of the hole. Authors investigated the effect of drilling speed, feed rate, and twist drill angle on the delamination factor. Authors concluded that higher cutting speed and feed rate can be used to produce the holes with lower delamination factor due to increased brittle nature of thermoplastic matrix. Higher cutting speed seems to cut the fibers very efficiently without fraying. Influence of drill point angle on the delamination factor was higher than the influence of drill diameter. Authors recommended the lower drill point angle and basket weaving pattern to reduce the delamination factor. Debnath et al. [6] investigated the drilling characteristics of uni-

directional sisal fiber-reinforced composites. Authors used epoxy (thermoset) and polypropylene (thermoplastic) as the matrix materials to develop the composites with 50–55 wt% of reinforcement. Three different types of drill geometries (4 facet, parabolic, and step drill) were used to make holes of 8 mm constant diameter in the laminates. Authors selected five levels of feed and three levels of rotational speed of drill on the basis of literature review. Authors concluded that the type of chip produced was mainly affected by the polymer used. For thermoset polymer (epoxy), the chips were shorter and discontinuous in nature while for thermoplastic polymer (PP)-based composites, longer and continuous chips were produced due to large plastic deformation range of thermoplastics. Authors suggested the use of parabolic drill geometry for lower value of thrust force and torque instead of other two geometries. At all levels of parameters, the torque generated was found to be lower in case of thermoplastic polymer-based laminates compared to thermoset polymer-based composites. Authors also established that the value of drilling force decreased while increasing the spindle speed and it increased with increment in feed rate. Brown et al. [7] used thermally assisted piercing (TAP) process to produce holes in polyetheretherketone (PEEK)-based composite laminates and investigated the mechanical performance of laminates after piercing process.

Similarly, unconventional methods have also been investigated by various researchers to improve the quality of holes produced in fiber-reinforced thermoplastic-based composites [8]. In conventional machining, the tool and workpiece are in direct contact with each other and the relative motion between them causes friction which in turn produces a huge amount of heat. The distribution of the developed heat generally depends on the conductivity of the constituents used in the composites. Heterogeneous nature and low thermal conductivity of thermoplastic composites result in the development of heat affected zone (HAZ) which directly affects the performance of composite material [3]. Unconventional machining methods like abrasive water jet machining and ultrasonic machining are useful in producing holes without HAZ. Despite various advantages of non-conventional methods used to produce holes in composites laminates, there are certain limitations of these processes. Low material removal rate and high power consumption are such limitations associated with unconventional methods. Hydrophilic nature of the natural fibers used in the composites as reinforcement may also impose some problems while making holes by using unconventional methods like water jet machining. Aoki et al. [9] produced holes in epoxy-based composites by introducing the ultrasonic vibrations in the conventional drilling tool of 8 mm diameter. The excitation frequency used was fixed at 17.8 kHz. Authors showed that the surface roughness achieved with ultrasonic vibration improved at every feed rate compared to the surface roughness achieved without ultrasonic vibration.

The quality of holes can be compared by quantifying the accuracy of hole diameter achieved, surface finish of hole, delamination and damage factors, thrust force and torque generated during drilling, circularity achieved, and the energy required to make a hole. The parameters of drill machine must be optimized to improve the quality of hole on the basis of above-mentioned factors. The present chapter emphasise on various defects developed during hole-making process of thermoplastic poly-

mer composite laminates along with the techniques used to make holes. Mechanical damage (delamination and cracks formation), thermal damage (HAZ), and chemical damage are associated with conventional and non-conventional methods of hole making. The drilling mechanism of thermoplastic polymer-based fiber-reinforced composite laminates using conventional and unconventional methods has been discussed in the present chapter.

2 Hole Making in Thermoplastic-Based Composites

Along with reinforcement, the matrix material also influences the machinability of composite laminates. Matrix materials used for fabrication of composites are of two types namely, thermoset and thermoplastics. Thermoset polymers are brittle in nature while thermoplastics are comparatively ductile. Thermosets possess higher thermal conductivity than thermoplastics which in turn affects the machining process. The nature of chips produced while machining the thermoplastic-based composites are long and continuous due to the ability of thermoplastic polymers to deform substantially before breaking [10]. The machined surface of thermoplastic-based composites is smoother than thermoset-based composites [11]. Hole making in FRP-based components can be done either before curing or after curing process while fabricating a component. Hole making before curing process involves the cutting of reinforcement/prepregs at the required place before placing then in the mold/layup. Cutting of reinforcement/prepregs can be achieved by either shearing action of reciprocating knife, ultrasonic cutting, or other methods used by textile industries. These methods are beyond the scope of this chapter and our discussion is focused on the hole-making/machining processes after the curing of composites. Conventional and non-conventional methods are being used to make holes in the cured FRP components. Selection of parameters in conventional or unconventional techniques is very critical in order to produce good-quality holes.

3 Conventional Method for Hole Making

Conventional method involves the drilling operation using conventional twist drills on vertical drilling machine tools or milling machines. High-speed steel and carbides mostly used drill tool materials. Primary rotational motion to the twist drill is provided by the spindle of drilling machine. The workpiece is fixed on the table using fixture. A chuck is used to hold the twist drill and a hand wheel is used to give the feed motion to the twist drill. A piezoelectric drill dynamometer coupled with charge amplifier is generally used to measure the forces and torque involved while drilling FRP laminate [2]. Engineers are investigating various drill geometries to make defect-free holes in FRP components. Two flute twist drill consisting of two cutting edges, i.e., is the most common drilling tool used in conventional drilling. The material is removed by

the shearing action provided by cutting edges of the twist drill rotated by the motor. The primary requirement for the material removal through conventional method is to have a cutting tool which is harder than the workpiece. The cutting tool is always in contact with the workpiece while removing the material. The material is removed in the form of continuous or discontinuous chips. Rotational cutting speed of drill, feed rate, and drill geometry and fiber volume fraction in a composite are important parameters affecting the quality of hole. Table 1 shows the parameters used by various researchers while making holes in thermoplastic matrix-based composite materials.

3.1 Effect of Spindle Speed

In drilling operation, cutting tool may induce damage in the workpiece at the entry side as well as exit side of the hole due to direct contact of rotating tool with the workpiece, which are known as peel-up and push-out delamination, respectively. Rotational speed of tool given by motor attached atop the spindle affects the thrust force generated and the temperature developed at the interface. Davim et al. [15] used cemented carbide helical drills of 5 mm diameter to make holes in carbon fiber-reinforced polymer composite laminates of 3 mm thickness. Authors used three cutting speeds of 50, 60, and 70 m/min and investigated the damage at the entry and exit side of the hole. Authors concluded that the damage around the desired hole increased with increment in cutting speed. Bajpai et al. [16] investigated the hole-making process in sisal and grewia-optiva fiber-reinforced PLA-based composites. Authors used three different cutting speeds of 900, 1800, and 2800 RPM. Thrust force and torque were measured at every instant of hole-making process. The thrust force and torque decreased with increment in cutting speed for each type of drilling tool used by the authors in the experimental investigation. In contrast to the drilling of synthetic fiber-based thermoset composites by Davim et al. [15], drilling-induced damage was lower for high-speed drilling of natural fiber-based thermoplastic composites. Chegdani and El-Mansori [17] used flax fiber as reinforcement and polypropylene as matrix to fabricate composites. Authors investigated the effect of cutting speed on specific cutting energy required to make a hole using conventional drilling method. Authors concluded that the specific cutting energy increased with increment in cutting speed irrespective of feed rate. Cong et al. [18] studied the effect of cutting speed on temperature achieved by the workpiece during conventional drilling of CFRP composite laminates. Authors concluded that the maximum temperature achieved by the workpiece first increased with increment in cutting speed keeping other variables constant and after a particular speed, the temperature showed a decreasing trend. Lower contact time of tool cutting edge and the workpiece at higher speeds was the proposed reason behind decreasing temperature after a particular speed. Debnath et al. [19] investigated various drilling parameters like tool geometry and cutting speed to make damage free holes in sisal and nettle fiber-reinforced polymer composite laminates. Authors suggested that thrust force could be minimized while drilling a hole with higher cutting speed if the cutting tool of optimum geometry is selected. Lopez-

Table 1 Drilling parameters used for thermoplastic composites [2, 5, 12–14]

Sr. No.	Reinforcement/matrix	Parameters				Remark
		Spindle speed	Feed rate	Drill parameters	Fiber fraction	
1	Jute/cotton/flax/PLA	15, 20, 25 m/min	0.03, 0.06, 0.12 mm/rev	Two drills with 80° and 118° point angle	0.65 by weight	Damage factor was lower for 80° point angle drill and basket weave
2	Bamboo/polyester	500, 860, 1360 RPM	18, 26, 34 mm/min	4, 6, 8 mm diameter twist drill	–	Lower delamination corresponds to 500 RPM, 18 mm/min and 4 mm drill
3	Sisal/polypropylene	900, 1800, 2800 RPM	0.05, 0.12, 0.19 mm/rev	10 mm core diameter twist drill and trepanning tool	0.2 by weight	Thrust force was lower for trepanning tool as compared to twist drill
4	Glass fiber/polypropylene	1000, 3000, 5000 RPM	100, 200, 300 mm/min	6, 9, 12 mm diameter brad and spur type drill	–	Feed rate was found to be most significant factor for thrust force generation
5	Glass fiber/polyester	400 RPM	0.1–0.7 mm/rev	6 and 10 mm diameter	0.63 by volume	Feed rate was found to be most significant factor for thrust force generation

Arraiza et al. [20] used three types of drill geometry namely reamer, twist drill, and drill-end cutter to investigate the effect of drill geometry while making holes in carbon fiber-reinforced polycyclic butylene terephthalate thermoplastic composites. Authors concluded that the quality of holes produced in terms of diameter accuracy and surface roughness was better achieved with the reamer at lower spindle speed of 3000 RPM than high spindle speed of 15,000 RPM.

3.2 Effect of Feed Rate

Feed rate is that velocity of tool which advances the tool into the workpiece thickness. Feed rate mainly affects the force required to perform the drilling operation and the thrust force generated. Tsao [21] studied the effect of feed rate on thrust force generated while drilling a 16-ply woven CFRP composite laminate. Author varied feed rate from 0.005 to 0.025 mm/rev. Author concluded that thrust force increased with increment in feed rate irrespective of the drill geometry used for conventional drilling. Srinivasan et al. [22] investigated the delamination of thermoplastic-based composite laminates. Authors used glass fiber as reinforcement while polypropylene was selected as matrix material (due to good abrasion resistance) to fabricate 4 mm thick laminate. Authors concluded that feed rate is highly influencing factor, controlling delamination of laminates during conventional drilling. Delamination factor increased at rapid rate with increment in feed rate (from 100 to 300 mm/min) irrespective of the spindle speed. Authors suggested the use of lower feed rate while drilling a composite laminate in order to minimize delamination factor. Srinivasan et al. [23] also studied the effect of feed rate on thrust force generated while drilling thermoplastic-based composite laminates. Authors concluded that thrust force increased with increase in the value of feed rate. Palanikumar et al. [13] studied the drilling behavior of glass fiber-reinforced polypropylene composites. Authors suggested that feed rate is the main factor influencing the delamination. Lower values of feed rate produces lower delamination factor. Anand and Patra [24] produced micro-holes in CFRP laminates using a twist drill of 0.5 mm. Results indicate that thrust force generated depends on feed rate. The value of thrust force increased by increasing the feed rate. Mohan et al. [14] fabricated glass fiber-reinforced thermoplastic composites and investigated the effect of drilling parameters on thrust force and torque required to make holes via conventional drilling method. Authors established that the required torque and thrust force generated are nonlinear in nature and increase with the increase in feed rate. Some other authors also found the similar trends for thermoset composite laminates [25]. Kim et al. [26] studied machinability of graphite fiber-reinforced PIXA-M and PEEK thermoplastic composites. Authors found that thrust force and torque required for making holes increased with increment in feed rate. Authors also found that the surface quality of the holes in terms of R_a value was improved when thermoplastic-based composites were processed and consolidated using induction heat press. Kakinuma et al. [27] attempted ultra-fast feed rate drilling (UFFD) of woven carbon fiber-reinforced thermoplastic Nylon

PA66-based composite laminates. Authors used very high feed rate of 3000, 5000, and 7000 mm/min. Authors concluded that the surface quality of machined hole and drilling efficiency were improved by using UFFD compared to conventional drilling.

3.3 Effect of Drill Geometry

Tool geometry influences thrust force and quality of holes produced by the conventional drilling. Various researchers used different geometries of cutting tool to improve the quality of hole. Rubio et al. [28] used three twist drills with different point angles (85°, 115° and 135°) of 5 mm diameter. Authors used two types of materials to investigate the effect of feed rate on thrust force. First material was pure polyamide (PA6) and the second was glass whiskers-reinforced polyamide (PA66GF30). Authors concluded that tool point angle of 135° was best suited for PA6 while 115° was optimum for PA66GF30 in order to minimize thrust force. Mohan et al. [14] used two twist drills of 6 and 10 mm diameter to investigate the effect of drill size on torque required to make holes in thermoplastic-based laminates. Authors concluded that torque required increased with increase in drill size irrespective of the feed rate. Lopez-Arraiza et al. [20] used three types of drill geometries namely reamer, twist drill, and drill-end cutter to investigate the effect of drill geometry while making holes in carbon fiber-reinforced thermoplastic composites. Authors used bidirectional woven carbon fiber and polycyclic butylene terephthalate thermoplastic matrix to fabricate 3 mm thick laminates. Authors concluded that the quality of hole produced in terms of diameter accuracy and surface roughness was better achieved with the reamer than the other two geometries used for experimentation. Debnath and Singh [29] used three different types of drilling tool geometries namely 4 facet, parabolic, and step drill while making holes using conventional method assisted by low-frequency modulation. Authors found that the value of thrust force generated was minimum for step drill geometry and maximum for parabolic geometry. Authors also established that thrust force and delamination factor were reduced when frequency modulated drilling was performed instead of conventional drilling for all types of tool geometries. Jia et al. [30] proposed a novel intermittent sawtooth drill structure to reduce drilling-induced damage while making holes using conventional method.

4 UnConventional Methods for Hole Making

The demand of high-performance composite materials in high-end industries like aerospace has increased drastically in the past decade. These materials are stronger but lightweight, heterogeneous, highly abrasive, and thermally conductive in nature which makes them very hard to machine by conventional methods. Conventional methods also induce various kinds of defects in the laminates which in turn reduce

the overall performance and reliability of components made of FRPs. Under these circumstances, unconventional machining processes are used to effectively and economically make holes in the FRP components. Unconventional methods do not require the tool to be harder than the workpiece as the tool is never in direct contact with the workpiece. The material removal takes place by transferring energy in the form of mechanical, thermal, electrical, or chemical energy to the workpiece. Chip formation does not take place while machining using unconventional methods. Some of the unconventional methods require the workpiece to be electrically conductive in nature like electric discharge machining. FRPs are not electrically conductive in nature; therefore, some of the unconventional methods are not applicable to machine FRP components. Abrasive water jet machining and laser beam machining are the most common unconventional techniques used to make the holes in FRP composites.

4.1 Abrasive Water Jet Drilling

The high thermal damage and distortion of material induced while machining FRPs using conventional methods lead to the use of abrasive water jet (AWJ) cutting. High-velocity water jets containing the abrasive particles are used as cutting medium in AWJ cutting. The primary advantage of AWJ cutting is the removal of material without any thermal damage of the workpiece. AWJ cutting can be used to make holes in the FRP components by removing the material at the circumference of the hole. When combined with CNC tools, AWJ cutting can be used to cut extremely complex contours. During the operation, no chemical coolant is used and therefore no fumes are generated which makes this process environment-friendly machining process. High operation cost and high initial setup are the limitations of this process. As the accelerated abrasive particles hit the workpiece surface, erosion, and shearing of material takes place [8]. Traverse speed, abrasive flow rate, feed rate, and stand-off distance (SOD) are the most common parameters used by the researchers while machining the composite laminates using AWJ [31]. Ramesha et al. [32] developed green composites using banyan tree saw dust powder as reinforcement and polypropylene as matrix material. Authors performed AWJ machining with abrasive particles of garnet of 80 mesh size. The diameter of the nozzle used for water jet was 0.76 mm. Two levels of water pressure, 1500 and 3000 bar were selected for machining. Traverse speed of the jet was varied from 250 to 850 mm/min with a step of 200 mm/min. Authors found that as the traverse speed of the jet increases, the kerf taper angle increases at both pressure levels. Kerf taper angle is explained with the help of Fig. 1. For ideal case of water jet machining, the value of kerf taper angle should be zero. Wang [33] performed abrasive water jet machining on 3 mm thick sheet of phenolic resin-based composites. Authors used stand-off distance from 2 to 5 mm and traverse speed from 1000 to 1800 mm/min. Authors found that the kerf width and taper angle increased with increment in water jet pressure as well as with stand-off distance.

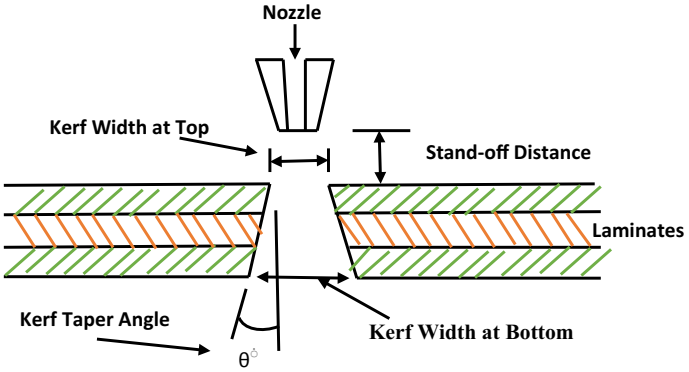

Fig. 1 Schematic of abrasive water jet machining

4.2 Laser Beam Machining for Hole Making

Laser beam is being used in the field of machining since the advent of high-performance aerospace materials. Lasers are very high-energy infrared waves which when strike at very small spot at workpiece transfers energy to debond the material molecules from parent materials by melting and vaporization [8]. Thin kerf width and high material removal rate are the unique features associated with laser beam machining. Lasers can be used for making holes in thermoplastic-based composite materials. Cutting the material from circumference with the help of CNC and removing the internal billet would give an accurate hole. Kerf taper and heat affected zone are very critical limitations of laser beam machining which can be optimized by controlling various parameters associated with lasers. Laser beam machining of polymer-based composites may result in generation of hazardous fumes. It has been observed by the researchers that a huge quantity of hydrogen cyanide was generated by the machining process of aramid fiber-reinforced epoxy-based composites. Solid (Ruby, gas and YAG) and gaseous (CO_2, helium–neon, and argon) type of medium can be used for the generation of laser beams [8]. Akshay et al. [34] compared the machining damage of composite laminates occurred during conventional drilling and laser beam drilling. Authors used fiber laser machining of 400 W power fitted with CNC machining system with a maintained stand-off distance of 1.5 mm. Authors concluded that the exit ply delamination was removed by laser machining system but there was a thermal damage of laminate due to HAZ (heat affected zone). Chouhan et al. [35] performed laser machining of kevlar fiber-reinforced polymer composites. Authors used polyetherimide and polypropylene as matrix materials. Authors used 400 W fiber laser machining system fitted with CNC system. Authors found that there was a recast layer of polymer after machining of both type of composites. It was observed from SEM images that the thickness of recast layer was lower for the polyetherimide-based composite than polypropylene based composites. Kerf width of around 150 μm was obtained when low-energy lines (30–60 J/mm) were used,

but it increased up to 250 μm for high-energy line (>90 J/mm). Certain other authors also performed laser-assisted drilling in fiber-reinforced thermoplastic composite laminates [36].

4.3 Ultrasonic Machining for Hole Making

Ultrasonic machining has been successfully used for making holes in polymer-based composite materials. The main component of ultrasonic machining setup is ultrasonic spindle system which comprises of ultrasonic spindle, power supply system, and electric motor. First, low-frequency power supply is converted to high-frequency electrical energy. A piezoelectric convertor is further used to convert that high-frequency electrical energy into high-frequency vibrations. The feed is given to the spindle by the motor situated above the spindle arm. The amplitude of vibration can be controlled by controlling the power supply [3]. High-frequency ultrasonic vibrations of spindle lead to the indentation of abrasive grits of very small size flowing on the workpiece with some medium. Some authors performed vibration-assisted machining of fiber-reinforced polymer composites and concluded that the quality of machined surface could be improved with the help of high-frequency vibrations [37]. Cong et al. [18] performed rotary ultrasonic machining of fiber-reinforced composite materials and observed the cutting temperature. Cutting tool was forced to vibrate at very high frequency of 20 kHz. Feed rate was found to be the dominating factor playing with the cutting temperature. Authors concluded that the increment in feed rate increased the cutting temperature.

5 Defects and Problems While Hole Making

Thermoplastic polymer-based composite materials are being widely used in household products as well as high-performance components of aerospace industry. Generally, industries prefer those materials which are easy to machine and the quality of machined surface is good. As machining is an inevitable step in the production cycle, the cost of machining should be low in order to reduce the cost of product. Due to non-homogeneous nature of the composites, various problems occur at the time of machining as well as there are several defects that come into picture after machining of composite materials. In the present section, most of drilling related defects and problems are emphasized. Thermoplastic nature of matrix also adds up to certain problems while machining fiber-reinforced composite laminates.

5.1 Damage While Drilling Thermoplastic Composites

Drilling of the thermoplastic composite is normally required to assemble various components to make a product. In the drilling operation, when tool comes in contact with non-homogeneous material, several kinds of defects come into picture like fiber linting, fiber pull-out, cracks, and delamination at entry side and exit side [38]. Delamination is a dominant problem to deal with when making hole via conventional method. Mohan et al. [14] explained that at the starting of drilling operation, lower layers support the layer being cut and provides strength to bear cutting force. But the time when remaining layers are not enough to provide strength to bear cutting force, layers start separating from each other causing push-out delamination. Delamination generally occurs while drilling on both tool entry and exit side of the workpiece but the push-out delamination is greater than peel-up delamination [39]. Figure 2 illustrates the types of delamination occurring while drilling FRP laminates. Excess amount of feed rate given to drilling tool may result in delamination [22]. According to some authors, the primary driver of delamination is axial thrust force [40]. Delamination in thermoplastic-based composite laminates can also be reduced by lowering the thrust force developed during the drilling operation [41].

While machining a fiber-reinforced composite material, a lot of heat is generated due to abrasive nature of the fibers which starts fusion of thermoplastic-based composites [38]. Compared to the thermoset composites, thermoplastic composites are more prone to the thermal damage during drilling operation due to low thermal conductivity and high thermal expansion of the thermoplastic materials [42, 43]. Use of coolant to transport generated heat is avoided because it may lead to swelling of laminates or may start some chemical reactions [44]. Different thermal expansion coefficient of fiber and matrix may lead to the generation of thermal residual stresses after machining which further may generate a crack in the material [45]. In order to transport generated heat, a thermal gradient is required. A drilling tool of high thermal conductivity may solve the problem as explained in Fig. 3.

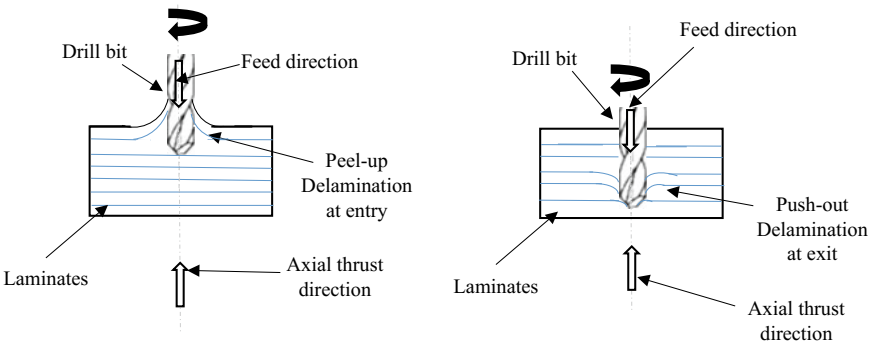

Fig. 2 Peel-up delamination and push-out delamination

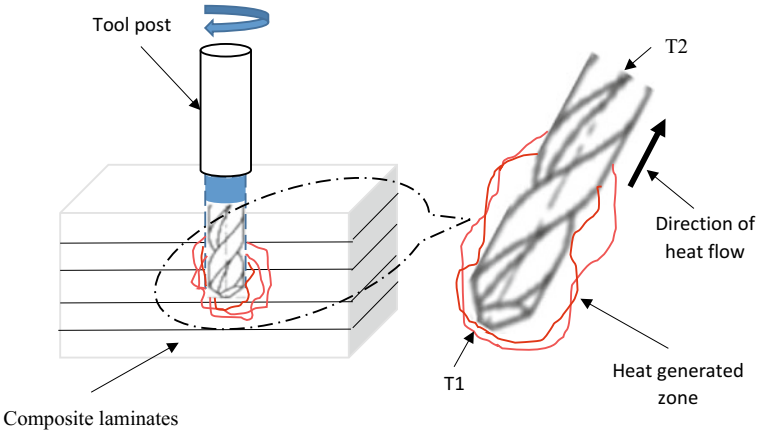

Fig. 3 Heat generated during drilling of thermoplastic composite

Generally, dry condition is used while drilling fibrous composites. There may be some chemical damage to fiber and matrix material if some chemical coolant is used while drilling because it may react with polymer or fiber altering the properties of the constituents of composite material [38].

5.2 Problems While Machining of Thermoplastic Composites

While drilling a thermoplastic-based composite material, several issues come into picture mostly due to non-homogeneous structure of composite and variable thermal conductivity of matrix and reinforcement material [46]. The most severe problem faced during machining of thermoplastic composites is clogging of the tool due to matrix material and damage of cutting tool at the cutting edges due to the abrasive nature of fibrous reinforcement in composite [3].

While machining thermoplastic-based composites, clogging of tool is a more severe problem because of the small gullet (a passage for chip removal) size and tool diameter [3]. Clogging of tool is mostly seen in all thermoplastic composite machining operations such as drilling, milling, and grinding [4]. Main causes of clogging of a tool in thermoplastic composites are low glass transition temperature and low thermal conductivity of matrix material. At the time of machining, tool temperature increases due to friction and when the temperature reaches more than glass transition temperature, the matrix material becomes rubbery and gets deposited on the surface of tool. The solutions to the problem of clogging are to use thermally conductive tool material for quickly reducing temperature of tool, use of compressed air and selecting optimum cutting speed and feed rate [3]. If thermal conductivity of tool material is high, it will increase heat dissipation rate which will further reduce

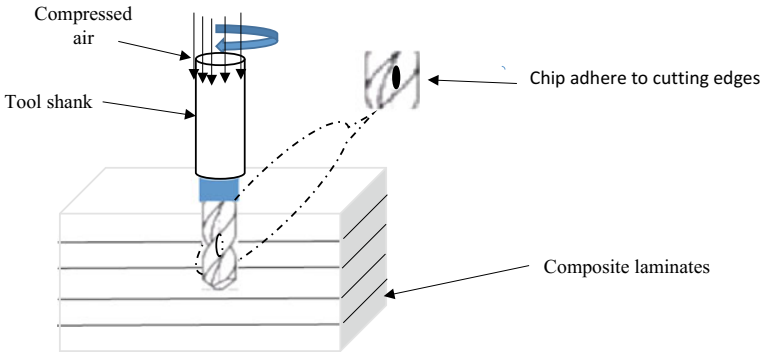

Fig. 4 Drilling of composite laminates with compressed air to avoid clogging of tool

the tool temperature [47]. Heat dissipation from the tool surface reduces the chances of softening of matrix material which directly influences clogging of tool during operation. The chances of clogging of tool in thermoset composites are negligible due to high glass transition temperature of matrix material. Compressed air supplied via flutes separates the rubbery particle of thermoplastic composite from the surface of the tool reducing the chances of clogging during drilling operation. The use of compressed air to solve the problem of clogging of tool during drilling operation is shown in Fig. 4.

Cutting tools generally fail due to one of the many reasons like excessive plastic deformation, mechanical breakage, blunting of edges, and brittle fracture of tool or elevation in the interface temperature [3]. After reaching a certain value of tool wear, increasing cutting speed and cutting temperature causes a deterioration of tool and increases the dimensional error in finished products. Therefore, it is advised to replace or grind the tool whenever required. Main cause of tool wear in thermoplastic composite is abrasive nature of fiber. Synthetic fibers like carbon and glass are more abrasive than natural fibers. Tool materials which are generally preferred for the machining of thermoplastic composites are cemented carbide, cubic boron nitride, and polycrystalline diamond. Because these materials are very hard and resists abrasive action of fiber material and has a satisfactory thermal conductivity for dissipation of heat. The wear of tool during machining is dependent on the abrasive nature of reinforcement, such as, less abrasive (jute, kenaf, hemp etc.) and more abrasives (carbon, glass, aramid etc.) [48].

6 Conclusions

Machining of fiber-reinforced thermoplastic-based composites is an inevitable process, especially hole-making operation. Conventional and non-conventional methods are being successfully used to make holes in thermoplastic-based composite lami-

nates. Still, a number of components get rejected after the drilling operation even after attaining high-quality component during primary processing. Parameters of conventional techniques can be optimized to get high-quality defect-free holes. The present chapter has presented various parameters affecting the quality of holes in detail. Drilling-induced delamination and various other problems like clogging of tool during the hole-making process have also been discussed in the present chapter.

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