Springer Series on Polymer and Composite Materials

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Shishir Sinha · M. R. Sanjay ·
Suchart Siengchin *Editors*

Bast Fibers and Their Composites

Processing, Properties and Applications

Springer

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Contents

Introduction to Bast Fibers

G. Rajeshkumar, T. Vikram Raj, A. Shake Ashik, R. L. Sooraj, and S. Aravindh

Abstract Natural fiber-based composites have made significant progress in recent decades due to their environmental friendliness, lightweight, and low cost. While there are numerous sources of natural fibers, this chapter focuses on bast fibers because they possess desirable characteristics for a variety of applications. These fibers are derived from the phloem that surrounds the stems of fibrous plants, primarily dicotyledonous. Bast fibers' qualities are regulated by environmental conditions, maturity, extraction method, and processing. This chapter discusses various aspects of different types of bast fibers, their physical, chemical, and mechanical properties, and their applications in a variety of fields, intending to promote their use in advanced technology sectors.

Keywords Bast fibers · Mechanical properties · Composite reinforcement · Cellulose

1 Introduction

In recent years, the significance of natural fibers has been appealing for the reinforcement of polymers. These natural fibers have excellent chemical, mechanical, and physical properties with more desirable characteristics. They are also environmentally friendly, renewable, biodegradable, and abundant in nature [1, 2]. These properties have created a great interest in the production of renewable polymers and as well in composite applications. However, there are some limitations to the use of natural fibers such as they are inferior to synthetic fibers in some way and natural fibers possess hydrophilic nature [3]. So, certain physical and chemical methods have been conducted to increase the mechanical strength and make the fiber hydrophobic. Each natural fiber demands a specific type of technique to make them strong and water-resistant [4].

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Natural fibers are acquired from three sources: animals, plants, and minerals. Animal fiber includes animal hair and silk fibers. Mineral fibers predominantly involve asbestos and basalt on the other hand it causes health problems. In general, the most economic and befitting fibers for the production of biopolymers and other bioproducts are plant-based fibers. Plant fibers are generally obtained from different parts such as leaves, seeds, bast, roots, and grass [5, 6]. Among these, fibers obtained from the bast part of the plant have excellent characteristics. The major chemical elements of bast fibers are cellulose, hemicellulose, and lignin. The pectin and proteins are available in minor constituents. Fats, waxes, and other components occur in small numbers. Bast fibers obtained from the phloem contribute to most of the natural fiber production around the world. Some of the bast fibers are kenaf, jute, ramie, hemp, and so on. These fibers are cultivated in various parts of the world, each fiber requires unique climatic conditions for its growth and development. For instance, India is the largest producer of jute followed by Bangladesh, Ramie is another fiber that is cultivated mostly in China and hemp is grown mainly for fiber application in Europe, Canada, and the United States [7, 8].

Approximately, around 2000 plant species can be utilized as a source for natural fibers, but only a few of these are commercially accessible, prominent, and found to be applicable for making biopolymers and composites. In this chapter, basic facts and information about various types of bast fibers are discussed. Furthermore, at the end of this chapter, the mechanical, physical, and chemical constituents of the bast fibers are summarized.

2 Bast Fibers

2.1 Kenaf

Kenaf (botanical name: *Hibiscus cannabinus*, family name: Malvaceae) is an annual fiber crop of the warm season and it has a close relationship with cotton and jute. Kenaf is a natural fiber that is widely used as reinforcement for producing polymer matrix composites. Kenaf is well known for its economic and ecological merits. Its period of growth is 3 months and it can grow in a variety of weather conditions. It grows more than 3 m and its base diameter ranges from 3 to 5 cm. This plant has a fibrous stalk that creates resistance to insect damage, so it demands very less pesticides. Only minimal chemical, herbicide treatment, and some fertilizers are enough to grow kenaf effectively. This plant is adaptable to various soils and 1 kg of kenaf can produce by spending 15 MJ of energy. This fiber possesses better tensile strength and is the material of choice for a variety of extruded, molded, and non-woven products. Some of the applications of kenaf are paper commodities, building supplies, animal feeds, and absorbents [9, 10] (Fig. 1).

Fig. 1 Kenaf: **a** plant, and **b** fiber (adopted from [11] with permission)

2.2 Ramie

Ramie (botanical name: *Boehmeria nivea*, family name: *Urticaceae*) is commonly known as China grass. It is a robust, perennial, and herbaceous plant. It is planted largely in China and other Asian countries such as the Philippines, India, South Korea, and Thailand [12]. This plant species was cultivated in the Mediterranean region in the early 1900s. Since Ramie is a bast fiber, some of the characteristics are great thermal conductivity and tensile strength. Also, it has fine coolness, ventilation function, and moisture absorption. Advantages of Ramie fiber: good antibacterial properties, provides resistance to mildew and insect attacks. Furthermore, this fiber establishes a silk luster appearance and can reduce shrinkage in various kinds of textile material. The ramie plant gets a very high amount of growth in warm and humid climatic conditions. They are commonly used in the fabric industry because of their softness, bleachability, and superior dyeability [13] (Fig. 2).

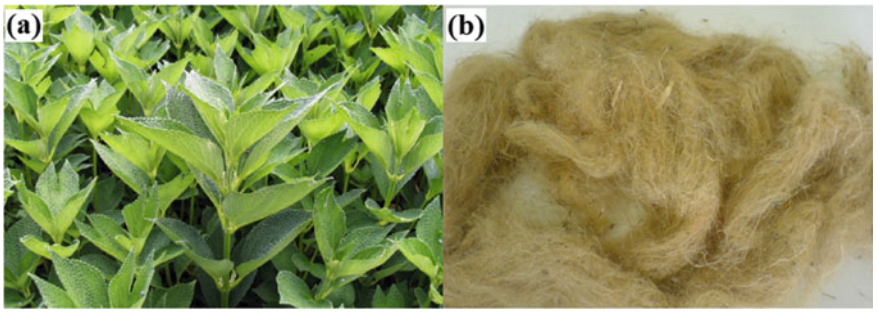

Fig. 2 Ramie: **a** plant and **b** fiber (adopted from [14] with permission)

Fig. 3 Jute: **a** plant and **b** fiber (adopted from [17] with permission)

2.3 Jute

The most commonly cultivated jute fibers are *Corchorus olitorius* (tossa jute) and *Corchorus capsularis* (white jute). Jute belongs to the *Tiliaceae* family. After cotton, jute is the most crucial natural fiber in terms of production, global utilization, availability, and usage. The major constituents that make jute are cellulose, hemicellulose, and lignin. It also comprises pectin, fats, and waxes in small volumes [7, 8]. Jute is a rainy season crop as it is seeded from March to May according to the rainfall and nature of the land. So, the harvest time differs from June to September. Jute cultivation is prominent in the Ganges delta which is shared between Bangladesh and parts of India (mainly West Bengal). Some other major sources of raw jute are China, Myanmar, Nepal, and Thailand. Key positive facts about jute are that they are agro-origin, annual renewability, soil friendly organic properties, and biodegradable in nature. Various applications of jute are in home textiles, agricultural textiles, automobile textiles and jute fibers are used in the manufacture of canvas, carpet, ropes, and sacks [15, 16] (Fig. 3).

2.4 Hemp

Hemp or industrial hemp is from the family *Cannabaceae* and its botanical name is *Cannabis sativa.* More than 30 countries cultivate hemp, among them, China is the largest hemp producing and exporting country. Hemp plants can grow in several types of soil and environmental conditions, making suitable surroundings available in many countries around the world. Temperatures around (semi-humid conditions) 7.8–27 °C are good for the growth of hemp plants. Harvesting of hemp occurs before the flowering, then it is cut down and rolled into large bails ready to be transported for

Fig. 4 Hemp: **a** plant and **b** fiber (adopted from [20, 21] with permission)

extraction and formation of fibers. This fiber shows an economically viable option to cotton, hemp fiber encourages cleaner production through material choice with a lower ecological footprint. Hemp is used in the production of apparel, fabrics, papers, biodegradable plastics, biofuels, and animal feed [18, 19] (Fig. 4).

2.5 Sunn Hemp

Sunn hemp fibers are one of the oldest yielding crops used in textiles. Its botanical name is *Crotalaria juncea* from the family *Fabaceae*. The majority of sunn hemp production is in India, Bangladesh, and Brazil. Due to the presence of high cellulose and low microfibril angle, this fiber has high tensile strength. This fiber is easily recognized by its white color appearance. Sunn hemp ensures good strength-to-weight ratio, corrosion resistance, high toughness, and resistance to fatigue [22]. Also, this fiber is broadly famous for its high aspect ratio, cost-effectiveness, and as a reinforcing material for composites. Various techniques and treatments are performed to improve the tensile strength of the fiber. It is mainly utilized in the manufacturing of pulp and paper [23] (Fig. 5).

2.6 Roselle

Roselle is from the *Malvaceae* family and its botanical name is *Hibiscus sabdariffa.* It is mostly grown in tropical Africa and parts of India. Roselle plants need direct sunlight and sufficient water supply for their growth and are mostly grown in tropical countries. Roselle fiber has a smooth surface, excellent tensile strength, and modulus [26]. To develop fiber from the plant, it is suitable to have a long neat stem free of

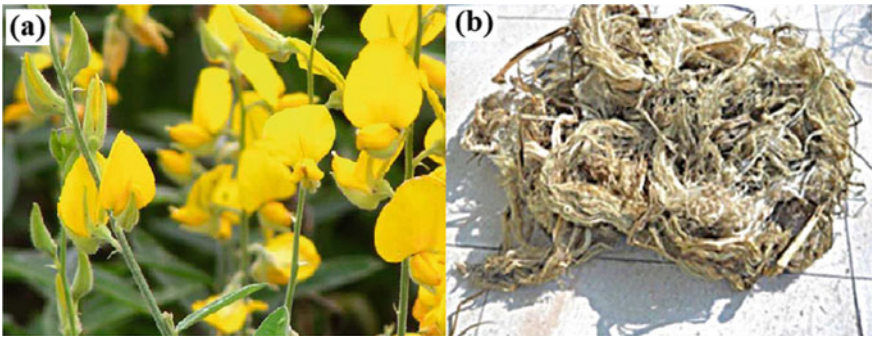

Fig. 5 Sunn hemp: **a** plant and **b** fiber (adopted from [24, 25] with permission)

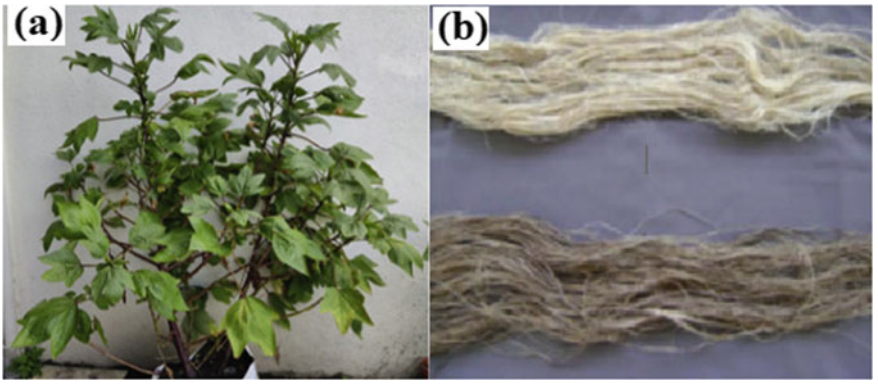

Fig. 6 Roselle: **a** plant and **b** fiber (adopted from [29] with permission)

branches or fruiting stalks. 18–20 inches of rainfall is necessary for roselle plants to grow, it normally takes 3–4 months to fully grow [27]. The primary constituents are cellulose, lignin, and hemicellulose. Polymer composites can be reinforced with the help of roselle fibers. It is further applied in automobile, aerospace, building, and construction fields [28] (Fig. 6).

2.7 Urena Lobata

Its full name is *Urena lobata* from the family *Malvaceae*. Urena plant is commonly known as Caesar weed or Congo jute. The fiber is fine, soft, flexible, and bright [30]. It is easily available and can be broadly grown under various conditions. For the extraction of urena, the stems were cut down and then the retting process takes place in a flowing stream usually for 14–16 days. After which the retted fibers were washed and dried out. It is traditionally used for making coarse textiles, industrially

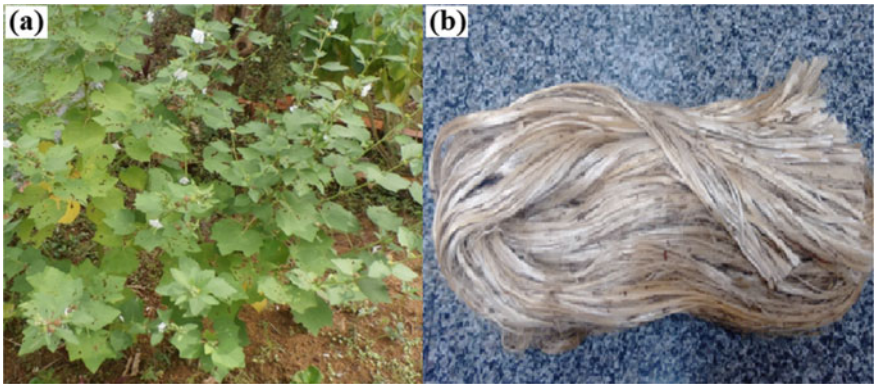

Fig. 7 Urena lobata: **a** plant and **b** fiber (adopted from [33] with permission)

as a substitute for jute, and sometimes mixed with jute. Whole urena plants can be pulped and also made into strong, bank-note quality paper [31, 32] (Fig. 7).

2.8 Flax

This fiber is acquired from the skin or bast surface of the flax plant which is present on its stem surface. The botanical name of the flax plant is *Linum usitatissimum.* These plants are cultivated in subtropical countries and are also available in temperate regions. This is one of the oldest fibers and is stronger than cotton. The length of flax fibers varies from 10 to 100 cm. The diameter of these fibers lies between 40 and 80 μm. Growing worldwide demand for linen makes flax a cash crop. Today flax is a high-end fiber because of its less production and high cost. Since 1994, Canada is playing as the largest producer of flax fiber. During 2005 and 2006, Canada has produced 1.035 million tons of flax fiber and currently supplying 60% to EU and 30% to the US. Fabrics made from flax are called linen. These linens are used as a high-quality textile for household purposes such as bed coverings, furnishing, and household interior decorations. Flax fibers produce thick yarns suitable for tents, kitchen towels, canvas, and sails. Reinforced flax fibers are used in automobile interiors [34, 35] (Fig. 8).

2.9 Nettle

The botanical name for nettle is *Urtica dioica*, which comes under the family Urticaceae. This plant is mostly grown in temperate and tropical areas of the earth, it can height range from 0.6 m to over 2 m [37]. The level of fiber that can be obtained

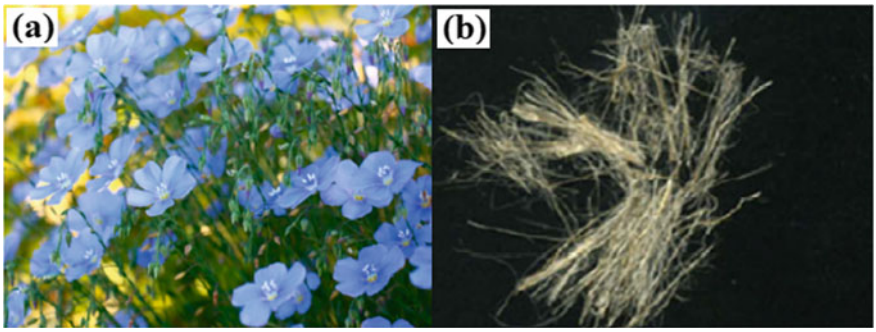

Fig. 8 Flax: **a** plant and **b** fiber (adopted from [34, 36] with permission)

in the stem of the plant differs. Stinging nettle is also a type of fibrous nettle plant with a great quantity of fiber where ever this plant is grown, where the local people procure this fiber and make use of it in textiles or for cordage. This fiber is present around the outside of the plant stem. The nettle fiber is used mostly for its medicinal aspects. The overall plant might be used for uncontrolled menstrual bleeding, diarrhea, diabetes, urinary problems, and respiratory issues. It can also be found in a wide range of pain relief medications for the diseases such as muscular dystrophy, eczema, ulcers, and rheumatism [38, 39]. Another type of nettle called *Girardinia diversifolia* (Himalayan Nettle) is quite similar to flax or hemp, it is used widely in textile industries [40] (Fig. 9).

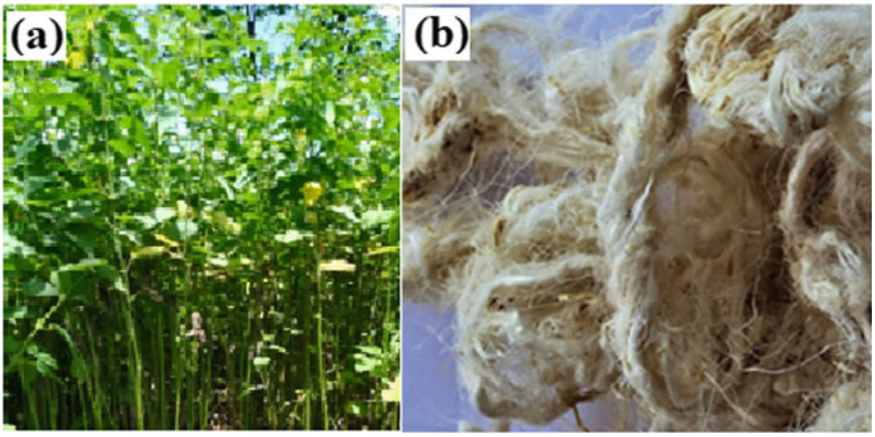

Fig. 9 Nettle: **a** plant and **b** fiber (adopted from [41, 46] with permission)

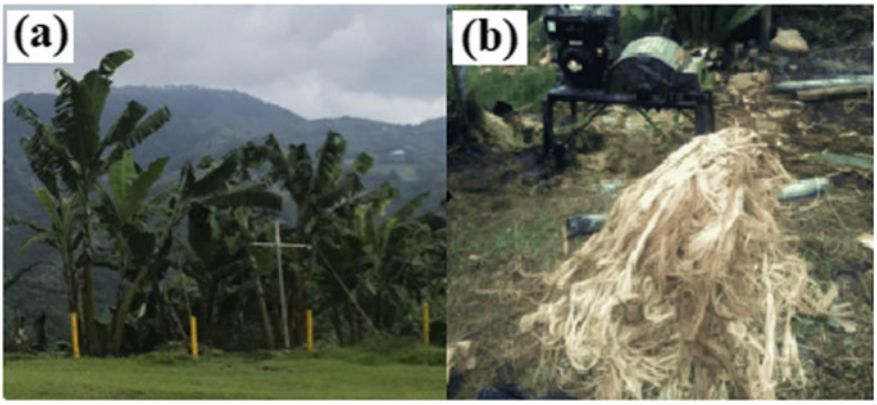

Fig. 10 Banana: **a** plant and **b** fiber (adopted from [44] with permission)

2.10 Banana

The botanical name for banana is *Musa acuminate*. Rather than delicious fruits, banana also provides fibers for textile purposes. India is one of the largest producers of banana fiber. Merely 10% of pseudostem can be utilized to make the fiber and the remaining is waste or applied as fertilizers [42]. They also have low density, high tensile strength, and modulus. This fiber has numerous applications due to the presence of properties like moisture absorption, anti-oxidant, UV protection, and weather resistance. Banana fibers have the potential to be used as reinforcement for composites in non-structural applications [43]. Ropes and yarns are made using banana fibers. Due to its resistance to seawater and natural buoyancy, it is used to make shipping cables (Fig. 10).

3 Properties of Bast Fibers

The various properties of bast fibers are evaluated in order to determine their suitability for use as reinforcement in the manufacture of polymer matrix composites. One significant disadvantage of bast fibers is that the properties of each fiber vary. Additionally, the properties are influenced by the environment in which the plants are grown. The properties of different bast fibers are given in Table 1.

Table 1 Properties of various types of bast fibers

Fibers	Density (g/cm^3)	Diameter (μm)	Cellulose (%)	Hemicellulose (%)	Pectin (%)	Lignin (%)	Wax (%)	Ash (%)	Moisture (%)	Tensile strength (MPa)	Young's modulus (GPa)	% of elongation	References
Flax	1.4–1.5	12–600	62–72	18.6–20.6	2.3	2–5	1.5–1.7	–	5–10	343–2000	27.6–103	1.2–3.3	[45]
Nettle	1.5	20–80	53–82.6	–	0.9–4.8	0.5	–	–	–	650	38	1.7	[46]
Banana	1.35	12–30	63–67.6	10–19	–	5	–	–	8.7–12	500	12	1.5–9	[45]
Kenaf	1.31	65–71	45–57	21.5	3–5	8–13	0.8	2–5	6.2–12	930	53	1.6	[47, 48]
Jute	1.3–1.49	20–200	61–71	13.6–20.4	0.2	12–13	0.5	0.5–2	12.6	320–800	8–78	1–1.8	[16, 45]
Hemp	1.47	25–500	70–74	17.9–22.4	0.9	3.7–5.7	0.8	–	6.2–12	690	70	1.6	[47]
Roselle	1.332	40–100	64.50	20.23	–	6.21	–	1.25	5.8	147–184	2.76	5–8	[26, 28]
Urena	0.94	170	56.42	13.61	–	25.01	–	0.55	–	309.3	16.5	3.07	[32, 49, 50]
Ramie	1.50	20–80	68.6–76.2	13.1–16.2	1.9	0.6–0.7	0.3	–	8	400–100	24.5–128	1.2–4.0	[13, 45]
Sunn	1.53	20–70	70–88	12.2	–	4.3	0.58	0.3	–	272–529	2.686	5.50	[51, 52]

4 Applications of Bast Fibers

The applications of various bast fibers are given in Table 2.

Table 2 Applications of various types of bast fibers

Fibers	Application	References
Kenaf	• Paper commodities, building supplies, and absorbents • Kenaf core fibers are used in animal breeding, summer forage, and potting media	[53]
Ramie	• Composites reinforced with ramie fibers are used in armours and vests because of their better ballistic performance than Kevlar • Ramie with glass fibers is widely applied in the automobile industry to produce hybrid polymer composites	[54]
Jute	• It is primarily utilized in manufacturing ropes, jute sacks, and twines • Jute fibers also find their use in making automobile interior door panels and seat backs	[55, 56]
Hemp	• It is used in the production of apparel, biodegradable plastics, animal feeds, and biofuels • They also find applications in beverage industries and nutraceutical products	[18]
Roselle	• It is widely used in beverage industry • Roselle is commercially viable to be utilized in food, pharmaceuticals, paper, and textile industries	[57]
Urena	• Urena plant can be pulped and used for making paper • This fiber is used for producing coarse textiles and also as a substitute for jute	[50]
Flax	• Flax fibers have the potential to be used as livestock feed, linen in the textile industry, and in the manufacture of decorative fabrics/yarns • Flax composites are employed in wooden fittings, fixtures, and furniture	[58]
Nettle	• Nettle plant is used for its medicinal aspects such as be used for uncontrolled menstrual bleeding, diarrhea, diabetes, urinary and respiratory issues • It is used for making ropes, threads, and other related items	[59]
Banana	• This fiber is a very good raw material for the textile and packaging industry. Also, the remaining waste of pseudo stem is used as fertilizer • It is extensively used in making power transmission ropes, cordage, cables, and fishing nets	[42]
Sunn	• These fibers are utilized to make pulp and papers • It is also used in the manufacturing of twines, nets, canvas, and fancy articles like bags	[60]

5 Conclusions

The chapter describes a few bast fibers, despite the fact that they demonstrate the breadth of their application possibilities, most notably as composite reinforcement. Bast fibers can also be used in a variety of different applications, including nonwovens, filters, geotextiles, and technical textiles. All the bast fibers discussed in this chapter have a high amount of cellulose content and higher mechanical properties. It is possible to modify the qualities of bast fibers by altering their surface through surface modification techniques. Finally, bast fibers demonstrate significant promise as a substitute for inorganic fillers and reinforcements, and their use in the composites industry should be expanded.

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Effect of Extraction Methods on the Properties of Bast Fibres

Sivasubramanian Palanisamy, Mayandi Kalimuthu, Carlo Santulli, Rajini Nagarajan, and Ganesan Karuppiah

Abstract The advantages of using lignocellulosic natural fibres from the plant bast, as compared with synthetic ones, namely in terms of environmental impact and availability for a number of industrial sectors, from textile to wood replacement and even to automotive or aerospace one, are counterbalanced by the need to extract them from the plant. In principle, the number of plants that are suitable to obtain bast fibres is considerable: some of the most popular are, among others, jute, hemp, flax, ramie, kenaf, nettle, etc., although this work aims also at giving a hint on the wider picture of the possible expansion of the market to other species. However, the quality of the fibre obtained depends on the extraction method employed, namely mechanical extraction, decortication or retting in different environments (water, dew, chemical) for the single species. Information on this aspect is not always consistent and very precise, although this chapter would like to underline the importance to consider and thoroughly describe the extraction method for every batch of bast fibre obtained to try to improve the general quality of the products and generally expand further the market for natural fibres.

Keywords Extraction methods · Decortication · Chemical retting · Water retting · Dew retting

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1 Introduction

Natural fibres play an essential role in the emerging "green" economy, which is concerned with the conditions of the environment: their use in materials, such as composites, does represent an important completion of their role in textile industry and, conversely, lignocellulose waste obtained from agro-forestry sector can often have a fibrous form. The use of natural fibres enables coming closer to carbon neutrality, which means absorbing as much as possible the same amount of carbon dioxide (CO_2) that is produced during the manufacturing of products. Another application that is promising is the use of natural fibres to produce environment-friendly building materials, also they are completely biodegradable at the end of their life cycle.

The International Year of Natural Fibres was celebrated in 2009 to raise worldwide awareness regarding the natural fibre's significance; this applied not only to natural fibre producers, consumers and industry but also to businesses relying on natural fibres for efficiency and long-term sustainability. The vast majority of natural fibres are harvested throughout developing countries, coming to a total of 30 million tonnes per year worldwide. The sale and proceeds outsourcing of natural fibres significantly donate to poor farmers' income and food security in many developing countries, giving, therefore, a significant economic importance to them: the diffusion of possible products can span also to highly technological industrial sectors, such as automotive [1]. Numerous parts of the plants are able to produce natural cellulosic fibres, which are then used to make products. Seed fibres (from kapok and cotton, for example), bast or stem fibres (from jute, flax, kenaf, sugarcane and hemp), leaf fibres (from a variety of plants, including several palms, pineapple, and banana) and fruit fibres (from coir or Borassus, for example) are the most commonly encountered types. The main difficulties in using natural fibres are the extraction, which is sometimes problematic, the not always very high repeatability of performance, and the usual local character of most of these [2].

Bast fibres are extracted, with various processes defined collectively as *retting,* from different plants and cultivars of these [3]. These include among others hemp, flax, jute, kenaf and ramie: to be a bast fibre indicates that the fibre is essentially found under the bark stalk plant, also recognized as phloem [4]. The fibres must be extracted and processed differently because of their inherent structure in the first place. A common characteristic of bast fibres is that, as with any natural material, a wide variability of quality is possible depending on the crops. After extraction, even only concentrating on their mechanical properties, they are affected by the variety of plant cultivated conditions for growth and the environment, harvest date, ripeness of harvest, decortication and cleaning processes [5].

2 The Possible Use of Bast Fibres

It is common for bast fibres to be both of sufficient length for use, e.g. in textiles, as well as reasonably strong. As a result, bast fibre, "soft" and derived from the stems of plants, is widely considered the most significant fraction of any plant [6]. In general, the stem of a fibre-producing plant is composed of three layers: the bark layer, the bast and the core stem. The external thin skin is the bark layer, or cuticle, covering the outside of the stem and holding the bast fibres in place while also protecting the entire structure. Between the stem core and the bark layer resides like a fibrous plant layer. The core of the stem consists of two parts: the pith and the tissue of wood (xylem) (Fig. 1). In the main and secondary fibre layers, fibrous layers are invented, paralleling dicotyledonous plants between nodes. Bast, phloem and soft fibres are the terms used to describe these fibre layers, which vary from stem to stem in various stem parts. Bast and phloem are the terms used to describe these fibre layers. There could be as few as 15 bundles or as many as 35 or more bundles. Each fibre bundle comprises 10–40 individual fibre cells, each of which is pointed at the extremities. The number of bundle cells is determined by their very location in the stem, with the most significant number of cells found in the centre of the stem (see figure). The ultimate fibre cell dimensions vary depending on where they are located in the stem, with cells at the stem base approximately three times as thick and long as cells on the upper end of the trunk. Bast fibres have a molecular structure composed of cellulose molecular chains in an amorphous matrix formed by pectin, lignin and hemicellulose [7]. When compared to the total number of plants grown and processed on a commercial basis, the bast fibre group contains a vast number of plants, many of which are possibly used also in textile products. Some of these are reported in Table 1.

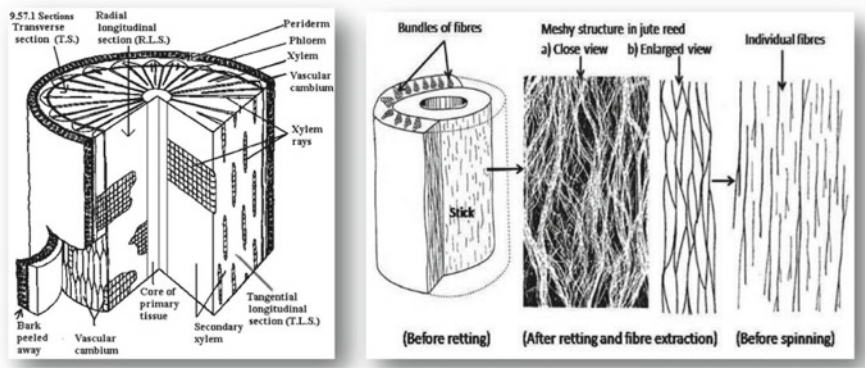

Fig. 1 Microscopical view of bast fibres (cross section and longitudinal) [8]

Table 1 List of potential fibres from bast plant sources

Order	Family	Botanical name (genus and species)
Sapindales	Anacardiaceae	*Rhus typhina*
Myrtales	Onagraceae	*Epilobium angustifolium and E. hirsutum*
	Lecythidaceae	*Couratari tauari*
Geraniales	Euphorbiaceae	*Euphorbia gregaria and E. gummifera*
Poales	Poaceae	*Saccharum officinarum*
Asterales	Compositae	*Eupatorium cannabinum*
Loasales	Datiscaceae	*Datisca cannabina*
Geraniales	Euphorbiaceae	*Euphorbia gregaria and E. gummifera*
	Linaceae	*Linum angustifolium and L. usitatissimum*
Gentianales	Apocynaceae	*Apocynum cannabinum, A. venetum, Chonemorpha macrophylla*
Polygalales	Polygalaceae	*Securidaca longipedunculata*
Laminales	Labiatae	*Phlomis lychnitis*
Malvales	Malvaceae	*Hibiscus cannabinus, Corchorus capsularis*
Rosales	Cannabacae Urticaceae	*Cannabis sativa* *Boehmeria nivea, Urtica dioica*

3 Bast Fibres Structure

Bast fibres can also be intended as a hierarchized material, therefore, including as the core part, giving strength to them, cellulose microfibrils arranged into different layers (S1–S3) internal to the phloem. The differentiation between S1, S2 and S3 is also offered by the different angles for winding of the microfibrils, which is referred to as the microfibrillar angle. The dimension of the microfibrillar angle of the different layers is also depending on their respective dimensions and of course on the species. At the very core of the fibre, an empty space is present, referred to as lumen, which may take different geometries from circular to more elliptical or even bow-shaped, although for ease of representation, as from Fig. 2, the first one is usually reported [9, 10].

Some of the above-mentioned bast fibres are more than a promising possibility, yet they are annually harvested for production purposes. In particular, Table 2 provides information about the five major bast fibre contributors.

The bast fibre is extracted from the plant's trunk, which is the outermost part of the plant. There are primary and secondary layers on each fibre and a hollow lumen within each fibre. The primary walls form an initial network of glycoproteins, lignin and hemicellulose microfibrils. The epidermis (bark) helps avoid wetness evaporating from the plant; also, it allows moderating the mechanical injuries to the plant structure. In particular, two types of transport tissue can be distinguished in the plant: xylem helps in transferring water and nutrients from the root to the whole extension of the plant [11]. The second transport tissue is phloem: phloem fibres

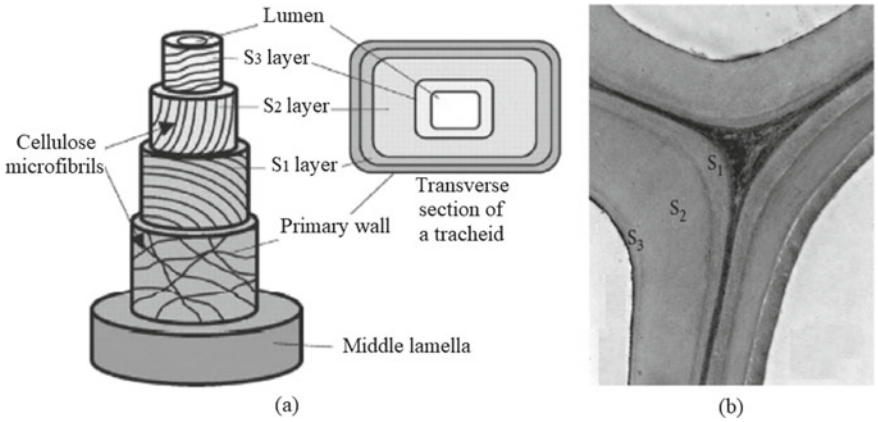

Fig. 2 **a** Single fibre exhibiting the S1, S2 and S3 layers; **b** TEM cell layer micrograph, showing the centre lamella (dark shadowed colour among fibres) (original drawing)

appear as large quasi-aligned bundles and are responsible for the plant's strength and stiffness [12]. Individual fibres are bonded together into bundles via mid of lamella, whose main components are lignin [13] and pectin [14]. The most important task of the retting method is the coherent and uniform removal of the bonding components, which results in the consequent separation of the single fibres from the bundle, hence able to be treated and employed, therefore referred to as "technical".

4 Major Bast Fibres for Use

4.1 Jute

Jute is a natural golden and silky fibre also referred to as the "glorious fibre". That's one of the least expensive bast fibres. In terms of use, worldwide demand, manufacturing and availability, jute fibre is the second largest vegetable fibre, following cotton [15]. Jute is extracted primarily from white jute (*Corchorus capsularis*); also, stem in less quantities are there in tossa jute (*C. olitorius*). This plant can be cultivated and harvested very hard [16]. Currently, Bangladesh and India are the world's primary jute fibre producers, accounting for 93% of total global jute production. Mainly jute fibre is used in the manufacture of twine and yarn, carpet backing cloth, hessian and a variety of many other textile blends. In addition to making imitation silk, very fine jute fibres are used. Blending jute with wool is also a possibility. To enhance the crimp, flexibility, softness and visual appeal of jute and its capacity for wool spun, caustic soda is used in the treatment process. It is possible to weave chair covers,

Table 2 Major bast fibre contributor (kenaf, jute, hemp, flax and ramie)

Plant	Kenaf	Jute	Hemp	Flax	Ramie
Botanical name	*Hibiscus cannabinus*	*Corchorus capsularis* and *C. olitorius*	*Cannabis sativa*	*Linum Usitatissimum*	*Boehmeria nivea*
Growth climate	Tropical and subtropical regions	Hot and wet conditions	625–750 mm/year of precipitation in a mild, damp atmosphere	Humid climatic conditions	–
Country	India, Northern Africa, China and Russia	Bangladesh, India, Uzbekistan and China	Equatorial countries and Eastern Europe	Asia and Europe	Philippines, China, Brazil, Europe and Japan
Plant outlook	With relatively easy yields to grow up to 5 m in 150 days, it provides about 6000–10,000 kg of dry matter per acre over a year	It can rise from 2 to 3.5 m within four months. It can absorb 15,000 kg of carbon dioxide and release 11,000 kg of oxygen per hectare	This plant has soft and hollow stalks, high in lignin, with harsh foliage at the very top	With respect to height, there are few branches on flax plants, producing very little seed	Stalks of plants could grow to 1–2.5 m in height and 0.8–1.6 cm in thickness
Fibre quality	Adapted for the replacement of jute. Preferable to other fibres due to its homogeneity, consistent fibre orientation and high CO_2 absorption capacity	Long jute fibre varies between 1 and 4 m with a polygonal area of varying shape and wide lumen, leading to greater diameter and fibre strength variation, moderate moisture conservation and good resistance to photochemical and chemical attacks	Stiffer and coarser than flax	The long fine flax fibre is usually turned into linen textile threads	Recovery is not possible because of the high content of gum (xylan and arabic up to 35%)

curtains, area rugs and carpets out of the fibres you receive. Jute fibre is widely used in various applications, including rigid packaging material, reinforced plastic and sacking for agricultural goods. Jute and jute composites can also replace wood-like products in the paper and furniture industry [17].

4.2 Flax

When compared to cotton fibres, flax fibre is crispier and more difficult to handle. It can retain and quickly release water, easy to wear linen in hot conditions. In India, linens/flax is grown solely for oil and seed, 20–25% yield flax fibres.

Figure 3 depicts the microstructure of flax fibre. The major countries that can produce flax fibre are France, Belgium, China, Ukraine and Belarus. Seventy per cent of the overall flax production is used in the textile industry. In thermoplastic composites, low grades of fibres are also used as reinforcing and also for fillers. The coarse flax fibres are being used to produce shipping cords, twines, strong ropes, sails, kitchen towels, canvas, tents, etc. Flax fibre of fine grade is utilized in the high-quality production of suiting, fabrics shirting, in addition to cloth laces as well as household textiles.

4.3 Hemp

The stem of the hemp plant is used to produce the fibres that are used in clothing. Hemp fibres are being used in the manufacturing industry and the production of

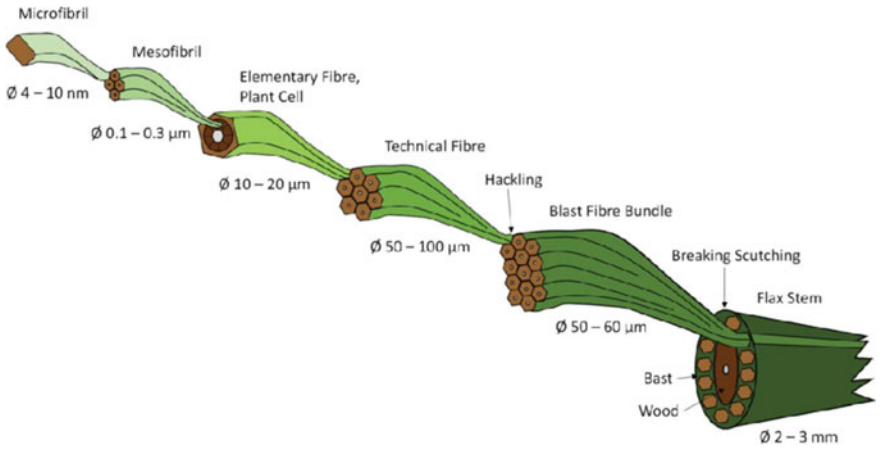

Fig. 3 Structure of flax fibre [18]

insulation composites. Many people are becoming aware of the potential of hemp fibre, and a wide variety of hemp goods made from various parts of the plant are now available on the market. Major producers include Germany, China and France, among others. Certain countries have banned the manufacture of hemp because of the possible extraction of tetrahydrocannabinol, responsible for marijuana effects, from some varieties. In the United States, the cultivation of hemp is restricted to a few selected regions. This is primarily because hemp requires a cool soil temperature at the time of planting. In terms of heat conductivity, hemp fibre performs admirably. As a result, hemp seed has long been harvested for its oil, which is used in various products such as cooking and cosmetics. In addition, hemp has been used as a medicinal plant, and research into its therapeutic and medicinal properties continues [19–21]. Affinity for dyes, mildew resistance, protection from ultraviolet (UV) rays and natural anti-bacterial attributes are some of the characteristics of this plant. Hemp is used to make ropes, paper and canvas, among other things [22]. Pieces of linen-like fabric can be created by weaving the fibres together so that they can be used for home furnishings, textiles, floor coverings and clothing. Moreover, it is also applied in the automobile industry as the reinforcement for moulded thermoplastics.

4.4 Ramie

Ramie is one among the most durable natural fibres available today. The fibre presents silky lustre white colour, high elasticity combined with excellent colouring capabilities. Ramie fibre is produced in large quantities in China, the biggest producer, in the Philippines and Brazil and also more limitedly in India. The cost of ramie fibre is rising as it has become incredibly requested, despite its limited availability on international markets. Yarn made from this fibre could be used to make a wide variety of different clothes. To make it more versatile, it is frequently mixed through many other textile-based fibres. Moreover, it is fit for the production of ropes, nets and twine [23].

4.5 Kenaf

Kenaf is a harvest that has been around for a long time, since 4000 BC, and has been used for many different purposes throughout history. As an “alternative crop”, it was considered simple to grow and produce, and the products made from it were inexpensive to manufacture. Kenaf is considered to be a plant from the Ancient Egypt tradition, therefore it is likely that this fibre was first used in North Africa. Also, kenaf manufacturing has been going on in India for more than 200 years. During the Second World War, researchers in the United States began investigating the use of kenaf, as there was greater competition for cordage material due to the conflict. The manufacturing of kenaf in the United States appeared in the the1940s

as a result of the disruption of jute fibre imports caused by the Second World War [24]. Aside from that, China ranks among the world's largest producers of kenaf [25]. Kenaf fibres are brittle, coarse and also irregular in appearance, making them difficult to process utilizing non-woven fabric or conventional textile equipment [26]. India, China and Bangladesh [27] counties are the three largest kenaf producers at the moment. Recently, it has been discovered that kenaf can be used for a high-end application, namely, that it can be mixed with cotton to create yarn and fabric. In addition to being aesthetically pleasing, the fabrics are made from blended yarn that feels smooth to the touch. This lightweight kenaf blend material has the appearance of linen [28]. Because of the natural absorbency and fire-retardant characteristics, retted kenaf, also in non-woven form has been found to be suitable for applications in a broad range, including outerwear, shoes and furniture [29]. Some countries are experimenting with the use of kenaf as an alternative for wood in pulp and paper production [30]. Also, natural fibre/plastic composites based on kenaf are a viable alternative to glass fibre-reinforced plastics (GFRP) in various applications, including the construction/housing industry, automotive industry and packaging [31].

4.6 Nettle

Stinging nettle is a wild plant that grows mainly in Asia, particularly in Indonesia, and in Europe. Lateng, Jelatang are the Indonesian name for nettle. The fibre of the nettle plant is quite strong, and it contains a lot of fixed carbon. Fine hairs cover nettle plants, especially in the leaves and stems. When it is touched, it releases chemicals, stings and produces inflammation on the skin, resulting in redness, stinging, pimples and irritation. Nettle plants grow wild and are considered a weed in the agricultural business. They are easy to grow and scavenge food from the parent plant.

Some nations have studied stinging nettles in terms of mechanical performance, with research taking place in the Brittany Region in France [32], also Tuscany region in Italy [33], the Netherlands and India. Stinging nettle has a specific weight of 0.72 g/cm^3, therefore, much lower than other bast fibres, and is classified as light fibre [33]. If stinging nettle is utilized as composite reinforcement, it can generate light and robust material with a low average specific weight. The stinging nettle can be used for textile/fibre, medicine, cosmetics, food and animal housing. Also, nettle fibre as a substitute for glass fibre, like reinforcement fibre in polymer matrix composites, is effective. It benefits from being softer and much more flexible than glass, which is a significant advantage. The material has also been discovered to be appropriate for mats in automobile interiors as well as for urea–formaldehyde particleboards. Not only the advancement of nettle research but also the cultivation across several countries is addressed in the literature [34–36].

5 Fibre Extraction Process

Fibre extraction processes can be classified into three main types: mechanical extraction, chemical extraction and retting. All the extracted parts, stem, bast and leaves are rinsed away before drying after fibre extraction using these methods. The moisture concentration in fibre has an impact on fibre quality; thus, proper drying is crucial.

5.1 Mechanical Extraction

The basic goal of mechanical processing of bast fibrous plants is to extract as much fibre as possible of the best possible quality to allow for subsequent processing. The fibre extraction technique creates strands of fibres that are linked to one another. Obtaining technical fibre necessitates the removal of fibre-bearing tissues from the stem or other parts of the plant. The decortication process—the extraction of green fibre without retting—must be included when discussing fibre extraction technologies. Compared to the fibre obtained following retting, the quality of the bast fibre acquired by this technique is poorer. As a result, decorticated fibre is mostly used in non-textile applications. Squeezing and breaking are used in current mechanical processing procedures to separate the fibre from the woody components. The stress (stretching) to which raw materials are subjected during processing, which may be present to a greater or lesser amount, can produce fibre damage or breakage, which can have a direct detrimental influence on its quality. On the one hand, mechanical processing can result in extensive fibre purification, while on the other side, it might result in excessive fibre shortening. A situation in which the ratio of long bast fibres to impurities is inverted may allow for a higher ratio of long bast fibres yet with a higher impurity concentration.

5.2 Decortication

Decorticator is a machine into which plant stalks are fed, to be then subjected to impact, shear and compressive forces. As a result of stalks being broken into many pieces, then bast fibre is separated to variable extents as the core [37]. The major product of decortications is the fibre bundle, which might also consider a by-product in some cases. Various kinds of decorticators, such as crushing rollers, ball mills, hammer mills, drop weight methods and more have been used for fibre processing.

Crushing rollers: There are two or more pairs of crushing rollers whose diameter varies between 0.2 and 0.3 m, also varying the length/diameter ratio [38]. Concerned rollers are situated parallel to each other and will be rotated in opposite directions to each other. Plant stalks are fed in a uniform and constant way between the rollers, which were also subject to force. The rollers for decortication are usually adaptable

[39]. Typically, the surface of the roller could be flat, pinned or fluted to allow compression, crimping or combing of the input materials. Once the relevant rolls are grooved, a force is applied to tear or grind [38]. The rolling gap shows the distance of a couple of rollers. Other than rolling distance, also speed or rotation plays a significant role in evaluating the stalk's primary forces. The crushing roller demands very little energy due to its lower speeds compared to hammer mills. Large fibres with higher market value are also produced by crusting rollers. Crushing rollers work well only for the stalk that is restored. The decorticator fibres are like low pureness for the untouched stalk. A crushing roller decorticator was made, e.g. by Leduc et al. [39] with purity ranges of the fibre between 55 and 60% at a capacity of around 4,500 kg/h. Fibre wrapping can lead to rotating machine parts being overheated and can damage or possibly cause a fire in the decorticator. Baker et al. [40] suggested the ball milling method to resolve this problem and Khan et al. [41] indicated drop-weight methods to be possibly used as fibre decortication alternatives. Fibre stems do not block machine parts when decorticated using these methods, thereby avoiding the possibility of fibre clogging in the system.

Ball mills: Grinding balls are utilized in ball mills in an appropriate container to shred material. The grind balls tumble, while the container rotates. This material is subjected to shear forces created by the tumbling balls. Ball mill outcome is affected by grinding duration and velocity and by the number of grinding balls. The idea is that the fibres are going to be preserved so that the cores will be ground, while the fibres will remain intact. Decortication retting hemp stalks using a lab-scale planetary ball mill was also investigated [40]. The study proved that the grinding period hence the speed of rotation has a major impact on performance. The best ball mill performance, measured in terms of both detaching efficiency and yield of fibre, has been noticed at grinding speeds of 200 and 250 revolutions per minute, depending on the amount of time spent grinding. While ball mills do not even have problems with fibre clogging, fibre damage continues to be significant. Also, the large part of the stalk, after grinding, becomes a powder made of fine particles.

Hammer mills: It is made up of a very fast hammer rotor in a chamber by the rotating hammer. A stalk of the plant is supplied to the chamber and applied forces impact the rotating hammers, which separate the fibres. Hammer mills, especially for unreleased stalks, failed to remove the core of the fibre entirely in many cases. The output of hammer mills is thus frequently poor in fibre, with limited use for applications [42, 43]. A further disadvantage of the hammer mill extraction (decortication) process is mainly supplying short fibres with significant variation in length, e.g. length of decorticated flax fibres, as reported in [44], spans between 17 and 101 mm. Another disadvantage of the hammer mill is its high energy required, increasing the feeding mass [45]. This method has though a significant rate of application over others because of its better extraction–production efficiency, in terms of yield per time [42].

Drop weight method: A new method of mechanical extraction, drop weight, has been suggested by Khan et al. [41]. Solving the fibre wrapping problems around the rotating parts of machines also over fractions of fines in the ball milling's final product. The basic operation of the drop weight technique is the application of plant

stalks being subjected to impact forces by lowering the weight on the trunk so that the fibre is segregated from the core. The impact intensity could be measured by the input stalk's energy, which is evaluated by the weight mass and the drop height. Outcomes by Khan et al. [41] revealed increased inputs of released energy additional fibres from the core and yielded higher produced fibre.

Other extraction methods: hemp decortication was achieved in the field by adopting a forage harvester, as reported by Gratton and Chen [42]. The harvester's cutter-head originally had 12 knives, but it was reduced to 9 scutching bars and 3 knives after modification. The reduction determined how many knives were available to enhance the trimmed hemp stalk length, so the length of the fibres produced. Impact forces are applied to the hemp stalks as a result of the combined presence of scutching bars. When comparing the altered cutter-head setting with the original cutter-head setting, it was found that higher fibre purification was obtained that cores also were removed.

5.3 Retting

Extraction by retting, mechanical and a mixture of both methods for bast fibres from plants are the most common. In biology, the retting process is based on extracting the fibre bundles with the least damage to their surrounding tissue. The quality of the extraction of fibres is significantly related to the retting conditions. (e.g., temperature and duration). Overretting tends to cause connections of weakening areas between cells and even breakage of single fibres [46]. Underretting results in fibre bundles connected with cores, which negatively impacts the purity of the fibre. Mechanical extraction is the process of mechanical stresses to break the bond between a core and its fibre. When it comes to the number of tonnes processed per hour, this technique outperforms retting by a wide margin. Controlling a set of mechanical forces acting on the stalk, on the other hand, is difficult, as well as the bond-breaking does not react very well to mechanical forces. This approach also creates extremely varied length fibre, a further disadvantage in mechanical extraction [46]. In this case, mechanical extraction is not very effective on its own. Plant stalks that have been pre-retted can have better fibre separation during mechanical extraction. Efficacy and efficiency in fibre extraction have thus been improved by combining retting with mechanical extraction.

Chemical retting: In the plant stalk, chemical retting breaks down the hemicellulose, pectin and lignin, causing them to degrade. If there is an excessive amount of retting, the cellulose will degrade as well. Aspects, such as temperature, reaction time and chemical concentration [47], affect the quality and efficacy of chemical retting extraction of fibre. For chemical retting, a variety of substances have been employed, the most common of which are alkalis (e.g., sodium hydroxide), salts (e.g., sodium benzoate), enzymes, mild acids and a mixture of different acids (e.g., oxalic and sulphuric acid) also a detergent [47, 48]. When alkali treatments are coupled with rising temperatures, the adhesive in the stalk plant is effectively degraded without

causing significant damage to the cellulose of the plant. When it comes to the retting of chemicals, removing utilized chemicals and sewage water is the most significant issue to be encountered.

Water retting: Water retting is the most common technique of retting, which involves immersing bundles of stalks in water. Water seeps into the core stalk part, swelling the inner cells and breaking the external layer, boosting moisture and decay-causing bacteria absorption. Over-retting weakens the fibre, while under-retting makes separation harder. The stalks are taken from the water before retting is completed, dried for many months, and then retted again in double retting, a gentle procedure that produces great fibre. Water retting is the most ancient method that has been documented. This process was once well-known for its ability to produce retted bast fibres of superior quality. On the other hand, generating large amounts of sewage water is a significant issue that must not be overlooked [49].

Dew retting: In places with limited water supplies, dew retting is a typical approach. It works best in regions with a lot of dew at night and warm daytime temperatures. The harvested plant stalks are uniformly scattered on grassy fields, where bacteria, sun, air, and dew combine to generate fermentation, dissolving most stem material around the fibre bundles. The fibre can be separated in two to three weeks, depending on environmental circumstances. Dew-retted fibre has a deeper colour and is of lower quality than water-retted fibre.

Enzymatic retting: This process is frequently hampered by the inconsistent and poor quality of fibre and geographic location, which necessitates the use of the optimal moisture and temperature to encourage the growth of microorganisms [50]. Substrate species and their preliminary pH are essential parameters for the enzyme retting process. The types of enzymes used, and the retting parameters set, can be customized to achieve the best fibre characteristic for an exact application. Obendorf and Song [51] found that enzyme-mediated retting has been the most effective technique. Yu and Yu [52] discovered that microbe retted kenaf fibre could remove 85.54% and 91.31% of the pectin and gum, respectively, from the fibre. Yılmaz [53] has confirmed that, this process produces the finest fibre comparisons with NaOH extraction of water retting. A significant decline in breaking force and linear density was noticed in xylanase concentration. Furthermore, higher concentrations of celluloses help improve breakdowns. High crystallinity fibres have reported that the celluloses remove more non-load bearing content [54].

Several enzyme-based processes, like scouring, retting, fictionalization, and bleaching, have been well-established in bast fibre processing over the past few decades. (Table 3) [55, 56].

Steam explosion (STEX) pretreatment before retting: Bast fibre retting processes frequently include pretreatments to improve the efficiency of the retting process. As a result of pretreatment, enzymes can penetrate the fibres more effectively during the retting process, resulting in better fibre quality. The STEX process is the most widely used for the various types of pretreatment techniques because of its high efficiency at a low price. Defibration using STEX is a combination of thermo-mechanical and chemical methods. To increase the efficiency of retting, STEX can

Table 3 In bast fibre processing, enzymes are used in the following ways [56, 57]

Fibre	Composition (%): holocellulose/pectin/lignin	Treatment	Enzyme	Primary function of enzyme	References
Jute	71–77/1–2/ 10–13	Retting	Pectinase	Degradation of pectic substances	[58]
		Scouring	Xylanases	Delignification through polysaccharide degradation	[59]
Flax	82–95/1.4–5/ 2.5–5	Retting	Pectinases	Degradation of pectic substances	[60–65]
		Scouring	Pectinases	Degradation of pectic substances	[66]
		Bleaching	Laccases	Not determined	[67]
Ramie	79–83/4–5/0.8–1	Degumming	Pectinases	Degradation of pectic substances	[68, 69]
Hemp	86.3/3.4/9.3	Retting	Pectinases	Degradation of pecticsubstances	[70, 71]

also be used as a pre-treatment. Fibres are chemically modified as well as mechanically defibrated during the process. The STEX process decomposes lignocellulosic structural components through heating, the formation of organic acids, and applying shear forces to the fibres in a controlled environment. The hydrolysis processes of hemicellulosic components and changes in the chemical properties of lignin and cellulose crystallinity index allow for the opening of the lignocellulosic structure and an increase in retting efficiency. At the end of this period, the instantaneous pressure discharge brings the reaction to a halt, allowing the fibres to be separated from the bundles. The cellulose of chemical grade, or high-quality fibres for textiles and composites, can be purified using the STEX process [72]. A defibrillation and depolymerization process is used in this process, according to one study [73]. When using a beaching agent of sodium percarbonate (SP), Jiang [74] has successfully reduced the gum content on ramie fibre to below 5 and 11.65%. The STEX process has also been approved as a suitable extraction method for bast hemp fibre by the International Hemp Association.

By STEX a significant reduction in the xylan content has also been obtained [75]. Pakarinen [75] has investigated the effects of STEX and alkalization pretreatments before the enzyme retting process on the properties of hemp fibres. The largest increase in enzyme hydrolysis was observed with additional pectinases in anaerobically preserved hemp fibre. Enzymes hydrolyze substrates more readily after a pretreatment in which microfibril swelling is caused because lignin provides fibres structural rigidity, which prevents swelling, delignification has increased the efficiency of the enzyme. In addition, the removal of xylan components also increases the surface area, allowing the fibres to swell, and eventually cleaves certain components. Because enzymes are easily accessible between the substrate and the surface

of the cell wall and the pectin removal process following the procedure for pretreatment, Enzymatic hydrolysis was found to have a significant relationship with the removal of pectin. STEX pretreatment is important to increase the efficiency of the repair process due to a better chemical or enzyme penetration into the internal part of bast fibres. Nonetheless, the STEX process, which occurs after the enzymatic retting process, aids in the preservation of the quality of retted fibres. Retted fibre has had its enzyme activities reduced, preventing an unintentional retting process from occurring, which could have resulted in a decrease in the quality of the finished fibre.

6 Production of Some Important Bast Fibres

Jute, flax, hemp, kenaf, nettle, Sunn hemp, ramie, etc., are among the most popular fibres, which have been commercially marketed around the world, primarily in Europe and in parts of Asia and America. Locally, other bast fibres were explored, and their popularity and demand have grown in both developed and developing countries alike. Since the early 1900s, Jute has been used in sacks and packaging and sacks, carpet backing and geotextiles [76]. Table 4 shows the variety of some important bast fibres according to their retting method.

7 Conclusion: The Future of Bast Fibres

Beyond the major natural fibres of silk, wool and cotton, many less diffuse plant fibres, including banana, ramie, flax and jute, are capable of surviving indefinitely in the future. When it comes to the manufacture of these fibres sustainably, During the duration of the fibre's production, the majority of these plants for bast fibre may not require any additional attention or maintenance (banana, jute, flax, ramie, Sunn hemp, etc.). Consequently, despite significant advances in science and technology, most of those fibres have been produced commercially or in small quantities on a local level for so many centuries. The main natural fibre benefits are that these come from natural sources. The entire process from plant cultivation to the extraction of fibre is environmentally friendly and cost-effective. Although natural fibres have been used in various fibre applications for the past 15 to 20 years, the vaster focus has been placed on the use of synthetic fibres in smaller quantities. As a result of the rising natural fibre production, the specific attention and application of these fibres in several textile fields, automotive fields, construction fields, food packing industries, electronics industries, etc.

Table 4 Variety of some important bast fibres according to their retting methods

Variety	Retting methods	Rationale	Merits	Demerits	Retting time	References
Flax, hemp, kenaf, jute	Water retting	Plant stems plant must be dipped in water and regularly monitored	Produces retted fibre that is exceptionally uniform and of high quality	Extremely serious pollution problem caused by anaerobic bacteria putrid odour, fermentation, environmental issues, and high expenditure It is necessary to treat wastewater extensively	Two weeks	[77, 78]
Flax, Jute	Dew Retting	The plant stems are spaced uniformly across the field to receive adequate sun rays. Fungal colonization is encouraged by atmospheric air, ew, and sunlight, which causes adhesive substances and cellular stem tissues to be broken down, resulting in the release of individual fibres	The pectin materials were easily removed	Inconsistent qualities and reduced strength are some of the consequences of using a product contaminated with soil	Three weeks	[77, 79]

(continued)

Table 4 (continued)

Variety	Retting methods	Rationale	Merits	Demerits	Retting time	References
Flax	Enzymatic retting	Enzymes hydrolyze gum as well as pectin material throughout the stem, releasing the juice. It is permissible to use manageable retting conditions to maximize retting efficiency	It is possible to achieve specific properties for different applications by altering the retting time and enzymes utilized. The procedure is more hygienic also quicker	The strength of the fibre is low	One day	[63, 77]
Kenaf, jute, flax	Chemical retting	Typically carried out with the help of sodium benzoate, sodium hydroxide, hydrogen peroxide	Within a short span of time, a clean and smooth surface could be acquired without consistency	While using a volume of NaOH greater than one percent, fibre strength has deteriorated as a result. Processing costs are high. Also, the colour is unfavourable	Approx less than 2 h	[80, 81]
Kenaf	Mechanical extraction	It is forcing fibre separation on the fed stem, followed by post-cleaning and further filtration of impurities	The short fibre in large quantities should yield results in a small time period	Reduced quality of fibre and increased expenditure	–	[82]

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Chemical Modifications of Natural Fiber Surface and Their Effects

Faris M. AL-Oqla and M. H. Alaaeddin

Abstract Biocomposite materials are a newly established class of composites. They possess desirable properties for numerous applications. This chapter provides an extensive analysis to highlight the recent improvements in bio-composite materials, natural fibers, and their chemical treatments. The importance and novelty of natural fiber composites (NFCs) are specifically examined along with polymers and their classification, characterization, and treatments. The chapter further provides detailed explanations regarding the recent techniques and chemical treatments currently applied to process natural fibers. The various chemical treatments addressed in this chapter include alkaline, silane, benzoylation, and permanganate treatments. Multiple effects of chemical treatments influence various properties of natural fibers, especially; their mechanical properties, which further impact the mechanical performance of composites and matrices. Such effects have been sufficiently explored. The common applications and recent utilization of NFCs have been considered to subsequently highlight current prospects.

Keywords Green composites · Cellulosic fibers · Chemical treatment · Biomaterials · Bioproducts · Mechanical performance

1 Introduction

Since the wide spread of global awareness, the utilization of eco-friendly materials has been increasing. Recycling and sustaining materials are becoming leading research aspects, such as the need to manage waste disposal and limit the use of finite resources [1–3]. Such efforts have contributed to the discovery of various effective

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recycling methods. It has been proven that recycling materials are cost-effective [4, 5]. Recycling is a process that is well emphasized and driven by economic benefits, such as in the metal industry for instance. It is generally more difficult to recycle polymers, partly due to the lack of economic incentives that enable efficient recycling. To ensure effective efforts in protecting the environment, specific legislation and economic incentives must be introduced to motivate, encourage, and promote recycling concepts as well as emphasize the need for recycling such materials [6, 7]. The European Union considers waste management within its top priorities, such as sophisticated policies and procedures, have been introduced to effectively manage waste [4].

Natural fiber materials are alternatives to synthetic fibers. They can gradually become replacements as well. Synthetic materials are usually composed of non-renewable petroleum-based substances [1, 8–11]. Currently, natural fibers are effective alternatives to synthetic and environmentally harmful materials, especially those used in composites to form strong load-carrying materials. This is known as the reinforcement process where strong materials are usually embedded within weaker materials. Certain natural fibers possess beneficial properties, and their advantages (such as low cost, affordability, and abundancy) are of paramount importance [12–16]. Such advantages provide excellent opportunities for new generations of reinforcements in polymer matrices. Naturally, the properties and performance of these fibers are unpredictable depending on their respective treatment and processing. They are unlikely to possess mechanical resilience for most applications. Hence, maintaining an effective combination with polymer matrices is encouraged [17–22]. A biocomposite is a material where at least one natural resource is incorporated in its formation process. Such involvement enhances the value of composites, providing numerous properties such as mechanical, physical, and biological attributes [1, 23–25]. Biocomposites can be manufactured using multiple and varied techniques, e.g., compression molding, injection molding, sheet molding, filament winding, resin molding, extrusion, pultrusion, and hand lay-up. These techniques are implemented for numerous types of fibers (such as woven, unwoven, and differently sized fibers) to structure and place fibers in the required direction. This facilitates the emergence of specified properties in the final product. However, important factors must be considered to achieve the desired outcome and attain the required properties. The factors include the type and length of fiber, the interface quality, corresponding surface energies, chemical compatibility, and the different or scaffolding layers of the composite [26–32]. Since biocomposites consist of different materials, the interfacial bonding between these materials is affected by the hydrophilicity of fiber and hydrophobicity of polymer matrix. Thus, chemical and physical treatments are essential to modify the fiber's surface and improve interaction [33–36]. Composite materials are commonly classified according to the nature of reinforcement, such as ceramic, polymer, and metal matrix composites. Predominantly, resin polymers are categorized into two types. The first type includes thermoplastics such as polyethylene (PE), polypropylene (PP), polyether ether ketone (PEEK), polystyrene (PS), polyvinyl chloride (PVC), and polyolefin. The second type includes thermosets such as epoxy, polyester, and phenol–formaldehyde resin [37–43].

2 Natural Fibers—Classification

Natural fibers are predominantly found in plants, animals, and minerals. The most common fibers from plants are likely cotton, kenaf, hemp, sisal, flax, jute, bamboo, and coconut fibers [44]. Each fiber possesses unique properties, for instance, hemp fibers are likely to be used in the manufacturing of ropes and aerofoils. This is due to their high resistance and suppleness in aggressive environments. They are also used as seals in heating and sanitary works. Overall, natural fibers have been recently seen as significant alternatives to synthetic fibers. Their compatibility and vast properties enable them to replace synthetic and environmentally harmful materials [45–48]. Natural fibers possess excellent advantages, especially in composites. Besides being environmentally friendly, they are abundant, cost-efficient, lower in density, easily processed, and biodegradable [3, 10, 12, 49–55]. Table 1 provides a classification of natural fiber resources according to their origins.

Regularly, composite materials have a bulk phase, referred to as a matrix, which is continuous, and one is dispersed referred to as reinforcement and is noncontinuous. Flakes, particulates, and fibers can be the type of materials which are used in the reinforcement process, the main idea bout composites; is that the bulk phase allows load to take place on the surface area and transfers it to the required reinforcement, as a result, this will increase stiffness and strengthen the composite [12, 56–59]. However, in biocomposites, natural fiber is used as reinforcements, whereas the matrix could be a synthetic or a biopolymer. Reasonably, these fibers are abundant, inexpensive, and renewable. Natural fibers are more affordable to produce in many parts of the developing world. In addition, they exhibit enough strength and stiffness for many applications, due to their low densities and they have enormous potential for producing composites with properties comparable to those of E-glass fibers, for example [2, 60–64]. Figure 1 provides a chart of source-based categorization of natural fibers.

Table 1 A brief classification of natural fibers

Leaf fiber	Plants leave fibers such as fique, sansevieria, sisal, agave, and banana
Seed fiber	Fibers are commonly isolated from seeds or seed cases, such as kapok and cotton
Stalk fiber	Fibers represent the stalks of the plant. For instance, barley, rice, wheat straws, and other crops including grass and bamboo
Fruit fiber	Usually extracted from the fruit of the plants
Bast fiber Skin fiber	Fibers are extracted from the skin or bast over the stem of plants. Such fiber has a higher tensile strength compared to other types. Thus, these fibers are applied in the manufacturing of durable yarn, fabric, packaging, and different types of paper. Common examples are Industrial hemp, kenaf, Jute, flax, rattan, vine fibers, and ramie

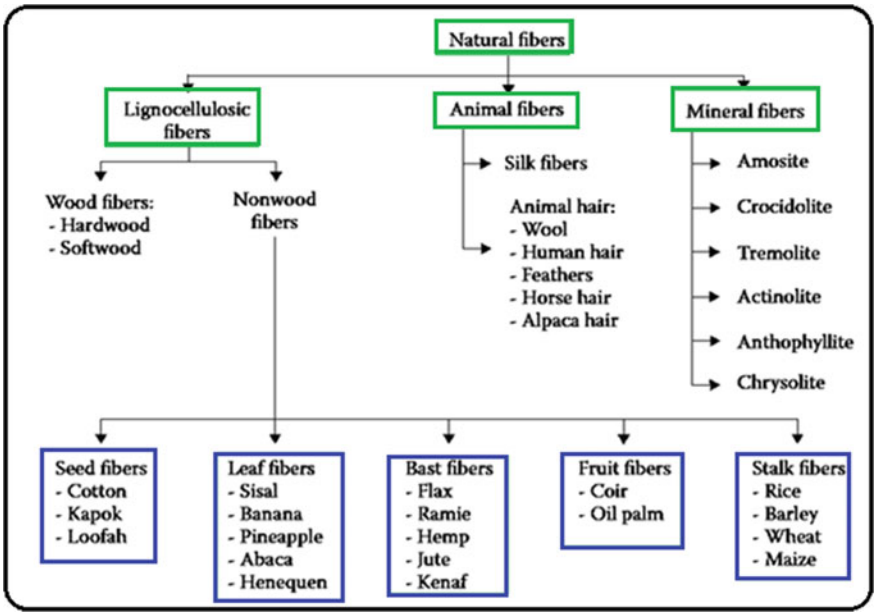

Fig. 1 Source-based categorization of natural fiber

3 Polymers

Polymers, also called resins or plastics, are more known than metal and ceramic composites or matrices. Generally, most inorganic and organic reinforcements can be mixed with polymers to produce different materials known as reinforced plastics and/or polymer composites [65–70]. The mechanical and physical performances of polymers make them attractive in the manufacturing world. They are likely to possess low-density materials and require less complex processes compared to other previous options. The curing temperature and manufacturing of common polymers are normally in a moderate range where an ambient temperature may suffice in some cases. As a result, the manufacturing cost of common polymers is substantially lower, and this is attributed to the low energy input. Polymers also have a wide class of organic materials. Each polymer possesses various features and distinct characteristics. Their wide range of properties and manufacturing cost makes them an outstanding option in developing composites suited for numerous applications and applied throughout various fields, where various selection tools like the Analytical Hierarchy Process (AHP) are utilized to properly determine the proper polymer matrix for certain applications [59, 71–79]. Polymers are mainly classified as either thermoplastics or thermosets. Both have been extensively used to produce different polymers. For instance, polystyrene, polyethylene, polycarbonates, polyamides, polysulfones, and nylon are examples of common thermoplastics. Polyester, polyimide, polybenzimidazole, epoxy, phenolic, bismaleimide, and

Table 2 Some applications of thermoplastics and thermosets

Thermoplastics	Thermosets
Car trims, bearings, phones, toys, handles, electrical products, gears, hinges and catches, cables, ropes, hoses, sheets, and windows	Motor brush holders, electrical equipment, printed circuit boards, circuit breakers, handles and knobs, spectacle lenses, encapsulations, and kitchen utensils

silicone are examples of thermosets [80–83]. Since thermoplastics are commonly employed to manufacture dashboards (as an example), they are used in manufacturing applications likely to withstand higher temperatures and for high-tech applications due to the intrinsic conductivity of some polymer types [84, 85]. Table 2 provides additional examples of the common applications of thermoplastics and thermosets.

4 Natural Fiber Composites

Natural fibers consist mostly of three main elements: cellulose, hemicellulose, and lignin. They can be isolated from different resources such as leaf (e.g., sisal), seed (e.g., cotton), bast (e.g., flax and hemp), fruit (e.g., coir), and from other sources such as birds' feathers [17, 86]. Natural fibers provide numerous merits over synthetic fibers (e.g., carbon and aramid fibers) and environmentally harmful materials. Natural fibers and environmentally friendly materials are carbon positive and absorb more carbon dioxide than they produce; they are also renewable and biodegradable [87, 88]. Their properties vary from one type to another, however, they exhibit advantages over other options. Table 3 states a number of advantages and disadvantages of natural fiber composites.

Natural fibers' surfaces are likely to be uneven, rough, and might require treatment. Uneven and rough surfaces might contribute to a good adhesion in matrices and composite materials. Overall, the mechanical qualities of natural fibers allow them to be utilized in composites. In natural fiber composites, fiber is the reinforcement; specific types of natural fiber provide enough stiffness and good tensile strength. Their mechanical properties have a direct relation with the stiffness and tensile strength of composite products. Hence, it is important to carry out a successful selection process to find out the most appropriate and suitable fiber for the reinforcing process. There are multiple criteria which must be considered when carrying out the selection process, such as fiber-matrix adhesion, elongation at failure, thermal stability, long time behavior, and manufacturing cost [42, 72, 74]. However, most of the biocomposite materials are presently applied in the construction works, automotive industry, packaging, and furniture industries. They are also used in structural applications, often, have low densities, resulting in high stiffness and high strength-to-weight ratios [89, 90]. Moreover, the fatigue damage tolerance and the given fatigue strength to weight ratios provide them with attractive options for various applications. The mechanical qualities of natural fibers are likely to be impacted by

Table 3 Advantages and disadvantages of natural fiber composite

Advantages	Disadvantages
High specific strength, stiffness, and low density	Lower durability is observed compared to synthetic fibers but can be enhanced considerably with the right treatment
For renewable resourcess, less energy is required to produce them, they involve CO2 absorption while providing oxygen to the environment	Higher rate of moisture absorption, the rate can be lowered by specific treatment
Low production cost when compared to synthetic materials	Lower impact strength compared to synthetic fiber composites
	Due to their numerosity, there is greater variability in their properties
Low emissions of toxic fumes when exposed to heat and during incineration process at end of life	Limited matrix options due to lower processing temperatures
Less abrasive damage	Quality is variable, depending on unpredictable conditions
Less equipment is required compared with that to produce synthetic composites	Weather is very influential on their properties

different aspects, such as the distribution of the fibers, the ratio of reinforcement to matrix/composite, and the efficiency of stress transfer between composites and their different components. Composite-based polymers are commonly applied in enormous and major engineering applications. Matrices usually work through bonding the fibrous phase and allocate the stress to a high-modulus fibrous constituent under an applied force. The major properties of the composites rely on different configurations or criteria that are related to the shapes, sizes, and ratios of materials used in the structural arrangement, as well as the distribution of these materials. Figure 2 shows an example of natural fiber composites using hemp fiber.

5 Surface Characterization

There are many techniques, which have been reported to be useful and effective in modifying surfaces and altering their inherited characteristics. Some of these methods are physical treatments such as γ-radiation, vacuum ultraviolet, plasma, corona, and laser treatments. The chemical treatments are also known to provide good results with regard to surface and fiber treatments, such as direct condensation grafting with surface compatibilization over the hydrophobic moieties and copolymerization with the matrices [92–94]. The characterization process of treated surfaces involves different techniques depending on the purpose, type of materials,

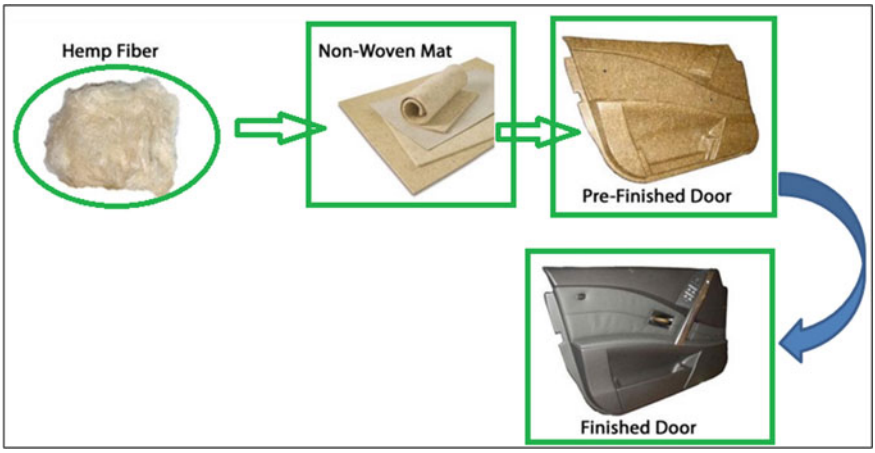

Fig. 2 An example of hemp fiber reinforced materials to produce a door [91]

and applicability, including inverse gas chromatography, contact angle measurements, elemental analysis, water uptake test, FTIR spectroscopy, X-ray photoelectron, etc. [95, 96]. Figure 3 provides an example of structural modification in natural fibers.

6 Chemical Treatments of Natural Fiber Composites

In most of the polymeric matrices, the polar chrematistics of natural fibers might represent a serious problem with regard to bonding compatibility. However, chemical treatments of surfaces might contribute to an enhanced interfacial bonding and adhesion between the different layers and composites, decrease water absorption and improve specific properties. Additional cost must be considered in the processing of natural fibers when treating them and modifying their surfaces [97, 98]. Overall, the chemical treatments are useful and can be considered as effective tools to modify the surfaces of natural fibers. Further elaboration on the types and techniques of chemical treatments is given below.

6.1 Silane Treatment

SiH4 is the chemical formula of silane compound. In various forms of treatments, silanes have been reported to be effective when used as coupling agents; they stabilize composite materials and provide good adherence between glass fibers and polymer matrices [16]. The agents might contribute to a reduction in the cellulose hydroxyl

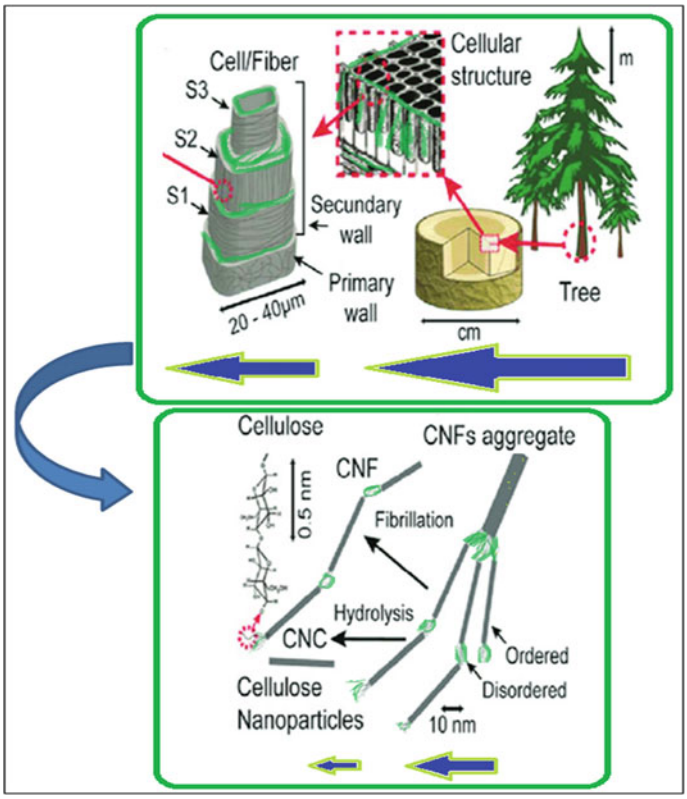

Fig. 3 Structural modification in natural fibers

groups in fiber–matrix interfaces. When water uptake or moisture is present, the hydrolyzable alkoxy group leads to the development of silanol. Hence, the formed silanol reacts with fiber's hydroxyl groups forming stabilized covalent bonds, these bonds are in the cell walls which are chemisorbed onto fibers' surface. The swelling of fiber is likely to be restrained by the hydrocarbon chains, this happens through the forming of a crosslinked network, which also can be developed by the covalent bonding between the composite elements [99, 100]. Figure 4 depicts the impact of silane treatment on the surface of mercerized natural fiber.

There have been numerous studies that applied silane treatment, especially to carry out surface modification in glass fiber matrices or composites. In different research findings, the coupling agents of silane were reported to be beneficial in modifying natural fiber–polymer interfacial strength and treating their surfaces [101]. In the treatment process of flax surfaces, the process was reported to be in a three aminopropyl trimethoxy silane, in an environment of 1% acetone solution (50/50 by volume) for 2 h. In sisal, the fiber was immersed for 5 min at a pH value of 4.5–5.5 the immersion was in 2% aminosilane with 95% alcohol, the process was followed

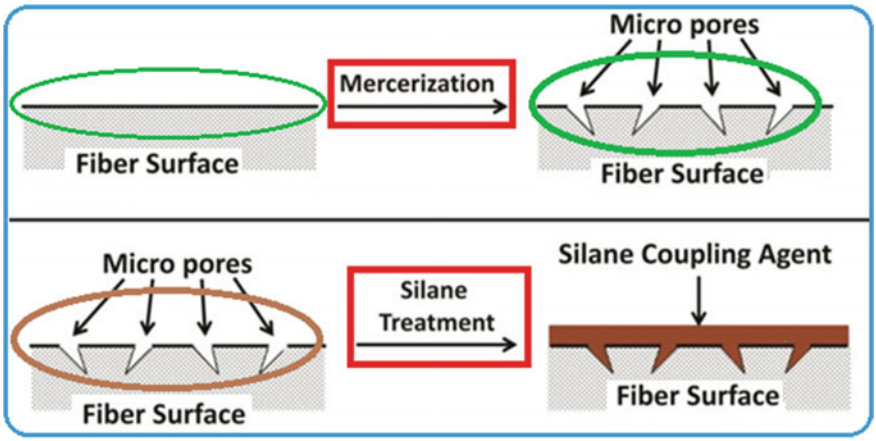

Fig. 4 Effect of silane treatment on the surface of natural fiber

by 30 min of air drying. For silane solutions in ethanol and water mixture with concentration of 0.033 and 1%, it has been found that the bonding and interfacial properties between the matrix and the silane coupling agent were stronger than that of alkaline treatment, the strength was observed in the tensile and thermal stability [11].

6.2 Alkaline Treatment

Alkaline treatment or mercerization is a common chemical treatment, likely applied in the treatment of natural fibers, especially when used to reinforce synthetic polymers. The main part of the treatment causes a disruption in the network structure of the hydrogen bonding and hence increases roughness. To promote the ionization of hydroxyl group in the alkoxide, the aqueous sodium hydroxide (NaOH) is a key factor [11]. Alkaline treatment has a direct influence on the hemicellulosic compounds, extraction of lignin, the cellulosic fibril, and the degree of polymerization. The illustration of the reaction between cell and alkaline is found in Eq. (1).

$$\text{Cell} - \text{OH} + \text{NaOH} \rightarrow \text{Cell} - \text{O} - \text{Na} + \text{H}_2\text{O} + \left[\text{surface impurities}\right] \quad (1)$$

Researchers who carried out alkaline treatment on fibers revealed good grounds to improve their properties in matrices and composites. For example, to treat flax fiber, there has been reported that 2% of alkali concentration for 90 s at 1.5 MPa pressure and temperature of 200 °C would be enough to perform defibrillation and degumming in fibers. Researchers also reported an increase in amorphous cellulose content which

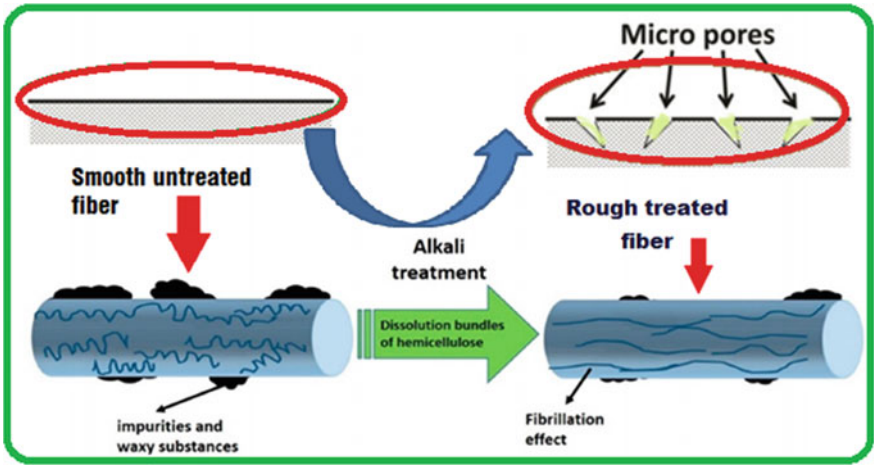

Fig. 5 A schematic illustration of untreated and treated natural fiber using alkaline treatment. Adopted from [103]

likely takes place at the expense of crystalline cellulose content. In addition, two effects were reported due to alkaline treatment, first, an increase in surface roughness which results in enhanced interfacial bonding and mechanical interlocking. Second, the cellulose increases at fiber's surface and an increased potential for reaction also occurs. Hence, alkaline treatment is proven to maintain an enduring effect on the mechanical performance of such fibers, such as stiffness and strength [11, 102, 103]. Figure 5 provides a schematic illustration of untreated and alkaline treated natural fiber.

Moreover, alkaline treatment was reported to offer about 30% enhancement to strength and modulus tensile. The treatment is also reported to increase the dynamic mechanical behavior of composites. Other findings showed that the sisal fiber-treated polyester composite showed a better tensile strength at 5% NaOH than 10% NaOH-treated composites. This can be justified by the higher NaOH concentration, leads to excess delignification which eventually weakens or damages the fiber. Hence, upon certain or high NaOH concentrations, a decrease in the tensile strength might occur [100, 103].

6.3 Benzoylation Treatment

Benzoyl chloride is commonly used in the treatment of many types of fibers. Benzoylation is an essential treatment mechanism in organic materials. The treatment enhances the adhesion between fibers and matrices or composites. Hence, significantly decreasing water absorption, increasing thermal stability, and improving the strength of composites and/or matrices. NaOH and benzoyl chloride (C6H5COCl)

solutions have been reported to be used in the treatment of sisal fibers' surfaces at different concentrations [92, 100, 103]. The chemical reaction can be found in Eqs. (2) and (3).

$$\text{Fiber} - \text{OH} + \text{NaOH} \rightarrow \text{Fiber} - \text{O} - \text{Na}+ \tag{2}$$

$$\begin{aligned}\text{Fiber} - \text{O} - \text{Na} + + \text{Cl} - (\text{C} = \text{O}) - (\text{C}_6\text{H}_5) \rightarrow \\ \text{Fiber} - \text{O} - (\text{C} = \text{O}) - (\text{C6H}_5) + \text{NaCl}\end{aligned} \tag{3}$$

The thermal stability of preprocessed or treated fiber composites was reported higher than that of untreated ones, this is attributed to various factors. Similarly, it has been reported that the treatment improves the interfacial bonding of flax fiber and polyethylene (PE) matrix. This works by stimulating the hydroxyl groups of the cellulose and lignin of fibers. In this particular treatment, the alkaline pretreatment was essential, and the fiber was therefore suspended in 10% NaOH and benzoyl chloride solution for 15 min [100, 104].

6.4 Acetylation of Natural Fibers

This method introduces an acetyl functional group (CH3COO–) into an organic compound. It is a common esterification mechanism in natural fibers; the mechanism causes plasticization in cellulosic fibers. When the reaction occurs, a generation of acetic acid (CH3COOH) takes place as a by-product, which must be removed from the lignocellulosic materials ahead of fibers' utilization process. Acetic anhydride (CH3-C (=O)-O-C (=O)-CH3) chemical modifications alternate the cell wall polymer hydroxyl groups with acetyl groups, modifying their existing properties to become hydrophobic [16, 100, 104]. The acetylation method increases composites' dimensional stability and decreases the hygroscopic behavior of natural fibers. The fiber reaction with the acetic anhydride can be demonstrated in Eq. (4).

$$\begin{aligned}\text{Fiber} - \text{OH} + \text{CH}_3 - \text{C}(= \text{O}) - \text{O} - \text{C}(= \text{O}) - \text{CH}_3 \rightarrow \\ \text{Fiber} - \text{OCOCH}_3 + \text{CH}_3\text{COOH}\end{aligned} \tag{4}$$

The method is common for surface treatments, particularly, in fibers reinforcing polymers or composites. In sisal fiber, various studies reported improved adhesion between the fiber and matrices or composites after acetylation treatment is applied [16, 100, 105]. Initially, the procedure starts with alkaline treatment and then followed by the acetylation treatment. In a reported case, the dewaxed sisal fiber was immersed in NaOH concentrations of 5 and 10% at 30 °C for an hour. The alkaline-treated fiber was immersed for an hour at 30 °C in glacial acetic acid, decanted and immersed in acetic anhydride. A concentrated drop of H_2SO4 was added for 5 min. However, the raw sisal fiber was treated with NaOH concentration of 18%. The process was

followed by glacial acetic and then with acetic anhydride and two drops of concentrated H2SO4 for an hour [100]. As a result, the treated fiber exhibited roughness on surface with notable voids,this can be advantageous to achieve better mechanical interlocking with the matrix. In addition, higher thermal stability was reported in treated fiber, enhanced fiber–matrix interaction was also observed in the treated fibers. Furthermore, in cases of fiber-reinforced polyester composites, the acetylation method exhibited less tensile strength loss and higher bioresistance compared to composites with silane-treated fibers [100].

6.5 Acrylonitrile Grafting

The reaction of acrylation is initiated by cellulose molecule free radicals; high energy radiation is shed on the cellulose to generate radicals with chain scission. To modify glass fibers, acrylic acid (CH2 = CHCOOH) should be graft polymerized. It is also used to modify surfaces in natural fibers. Graft copolymerization on the fiber surface is illustrated in Eq. (5)

$$\text{Fiber} - \text{OH} + \text{CH}_2 = \text{CHCN} \rightarrow \text{Fiber} - \text{OCH}_2\text{CH}_2\text{CN} \tag{5}$$

In one case, oil palm fibers were blended for 30 min with 10% of NaOH and then the fibers were treated for an hour in acrylic solution at 50 °C and within different concentrations. Then, the fibers are washed with an aqueous alcohol solution and dried accordingly. As a result, oil palm fiber's tensile strength did not increase. However, various chemical treatments have been investigated on similar fibers including peroxide, acetylation, permanganate, alkaline, silane, and acrylation. The last is proven to form strong covalent bond formation and, hence, tensile strength and Young's modulus of targeted fibers were slightly enhanced [100]. It was also reported that the acrylic acid-treated flax fiber-HDPE enhanced the tensile strength. A decrease in water absorption was also observed and acrylonitrile is employed to modify fibers [100]. Sisal fibers' graft copolymerization through AN method was studied by Mishra et al. [106] by preparing a mixed CuSO4 and NaIO4 initiator at a temperature between 50 and 70 °C and in an aqueous medium. The AN concentration, initiator, fiber loading, treatment time, and medium affected the graft effect. It has been shown that the untreated fibers uptake more water and the treated fibers absorb the least amount of water [106]. Hence, the study suggested that the changes caused by the chemical reaction to the surface of fibers decreased their affinity to moisture. In addition, the chemically modified fibers' grafting with 5% AN exhibited an improved Young's modulus and tensile strength compared to the ones with 10 and 25% AN. This is attributed to the fact that low AN concentration might provide an orderly arrangement of polyacrylonitrile units. The study concluded that treatment of 3 h offered an optimum graft yield [106].

6.6 Maleated Coupling Gents

The maleated coupling agents are commonly known for their ability to strengthen composites, especially the ones containing fillers. In addition to its ability to modify fibers' surface and carry out fiber treatment, unlike some coupling agents, maleic anhydride is also capable of modifying the polymer such as PP matrix. This makes it attractive when better interfacial bonding and enhanced mechanical properties are required [94, 100, 107]. To obtain a covalent bond across the interface and it is recommended to treat the cellulose fibers with heated MAPP copolymers. The activation mechanism happens through the reaction of maleic anhydride with PP and fibers. By exposing fibers to 170 °C ahead of treatment and later performing the esterification of cellulose fiber, upon the completion of this process, the surface energy of cellulose fibers increased close to the surface energy of matrix. As a result, a higher interfacial bonding of fiber and enhanced wettability was observed. However, when MAPP was utilized as a coupling agent to change the surface of jute fiber, it has been observed that a 30% loading of fiber with a concentration of 0.5% MAPP in toluene and 5 min of impregnation with 6 mm average fiber lengths, optimum results were obtained, the flexural strength increased to 72.3% in the treated composites. The PP matrix showed that the maleic anhydride treatment decreased water absorption in hemp, sisal, and banana fibers reinforced novolac composites. In addition, it has been reported that, upon the implementation of maleic anhydride treatment, enhancement observed in specific mechanical properties of natural fiber-reinforced composites such as impact strength, hardness, flexural modulus, and Young's modulus [16, 100, 107].

6.7 Permanganate Treatment

The permanganate compound has the permanganate group MnO4–. The treatment of permanganate and MnO3—ion formation offers the formation of cellulose radical. However, initiating graft copolymerization can be achieved through the highly reactive Mn3 + ions [107, 108]. Permanganate treatments are highly likely to be carried out by applying potassium permanganate (KMnO4) solution in acetone within varied categories of concentrations. The reaction is illustrated in Eqs. (6) and (7).

$$\begin{aligned} &\text{Fiber} - \text{H} + \text{KMnO}_4 \rightarrow \\ &\text{Fiber} - \text{H} - \text{O} - (\text{O} = \text{Mn} = \text{O}) - \text{OK} + \end{aligned} \tag{6}$$

$$\begin{aligned} &\text{Fiber} - \text{H} - \text{O} - \text{Mn} - \text{OK} \rightarrow \\ &\text{Fiber} + \text{H} - \text{O} - (\text{O} = \text{Mn} = \text{O}) - \text{OK} + \end{aligned} \tag{7}$$

The immersion period can be carried out from 1 to 3 min [109]. Upon the completion of alkaline pretreatment, sisal fibers were dipped in permanganate solutions

at 0.033, 0.0625 concentrations, and in acetone for 1 min. The results showed a reduction in the hydrophilic behavior of used fibers,hence, the water absorption of composite was reduced. This reduction took place when the KMnO4 concentrations were considered at higher levels. Hence, at higher than 1% KMnO4 concentrations, cellulosic fiber degradation occurs, resulting in polar groups between fibers and matrices. In another aspect, peroxide treatment is also offered in organic chemistry. There is a decomposition tendency in organic peroxides, this allows them to free radicals followed by reacting with the groups of hydrogen in the cellulose fibers and matrices [16, 109, 110]. For instance, the peroxide was able to initiate free radical reactions between the cellulose fibers and PE matrices. Dicumyl peroxide (DCP, (C6H5C(CH3)2O)2) and benzoyl peroxide (BP, ((C6H5CO)2O2) are chemicals within the family of organic peroxides,they are used in the treatment of natural fiber surfaces. Before the peroxide treatment is carried out, alkali pre-treatment takes place and then fibers are typically coated with DCP or BP in the solution of acetone for 30 min. In the reported study, the concentration of peroxide solution was 6%, in addition to saturated peroxide solutions in acetone. To achieve decomposition of peroxide, it is recommended to maintain a high temperature throughout the process. It has been reported that peroxide-treated oil palm fibers incorporated with PF composites are likely to withstand tensile stress maintaining improved strain levels [110]. The study examined DCP and BP treatments on short sisal fiber-reinforced PE composites [110]. The study reported enhanced tensile strength of composite when an increase of peroxide concentration is considered up to 6% for BP and 4% for DCP, then remained constant. The peroxide treatment decreased the fiber's hydrophilicity and increased its tensile characteristics.

6.8 Isocyanate Treatment

The compound isocyanate has the functional group of –N = C = O; this makes it vastly vulnerable to react with the hydroxyl groups in fibers. The reaction with the cellulosic fiber is illustrated in Eq. (8):

$$-\mathrm{N} = \mathrm{C} = \mathrm{O} + \mathrm{HO} - \mathrm{Fiber} \rightarrow -\mathrm{N}\,(-\mathrm{H}) - \mathrm{C}(=\mathrm{O}) - \mathrm{O} - \mathrm{Fiber} \quad (8)$$

The mechanism of treatment is to function as a coupling agent applied in the treatment of fibers and fiber-reinforced composites, and the reaction takes place between the coupling agent and fibers [16].

7 Applications of Natural Fiber Composites

The applications of natural fiber composites are widely spreading; they are used in enormous products and engineering fields. Natural fibers are also abundant and

available in different sizes, shapes, and types, e.g., kenaf, jute, hemp, bamboo, oil palm fibers, etc. They are employed in the preparation of composites and matrices. Lately, they received attention in important manufacturing industries such as automotive applications, constructions, building materials, structural components, and packaging [1, 8, 27, 60, 111, 112]. They are also used in aerospace, sports, recreational equipment, boats, machinery, and electrical and electronic industries. Table 4 provides examples of the types of fibers and their current industrial applications.

8 Chemical and Physical Treatment Impact on the Mechanical Properties

The concept of chemical treatment refers to the utilization of chemical agents, they are meant to modify the surface of fibers or the whole fiber according to the type of treatment. Chemical treatment of fiber is frequently performed to improve the adhesion and bonding of the fiber surface to the polymer matrix. Such treatment is expected to increase the strength of fiber, modify surfaces, enhance moisture resistance, and boost the mechanical properties of the composite itself. The extraction and separation processes of fibers are essential and can influence the overall quality of the anticipated outcome. Generally, the separation process of fibers from crops is known as the retting technique or method. This technique works by removing non-fibrous tissues joined with fibers through the degradation and decomposition of hemicellulose and pectin, the outcome result will be releasing separated fibers. However, due to its high specificity, low environmental impact, short duration, and better controllability; enzymatic retting is an encouraged method in many treatment cases. The cost of the process is considered high compared to other options; this makes it infeasible on an industrial scale. Once the retting process is accomplished, the non-fibrous materials have to be entirely removed. Hence, extracted fibers have to be cleaned, refined, and processed according to the required application. In order to maintain a suitable interfacial bonding in matrices or/and composites, it is encouraged to modify the surfaces of natural fibers. Due to their moisture absorption and wettability [16, 114], natural fibers are entitled to additional chemical and surface treatments. Applying the suitable treatment is likely to enhance their properties as reinforcement agents. Expectedly, results may lead to a remarkable improvement in surface roughness and hence, provides enhanced adhesion between the matrices or composites.

Overall, there are various chemical treatment methods, which can be utilized to treat fibers and their surfaces. Those chemical methods are likely to work on the reduction of hydroxyl groups aiming to improve the adhesion properties and compatibility between the elements. The sodium chlorite, acrylonitrile grafting, zirconate, triazine titanate, fungal, and enzyme treatments are also known treatments. Chemical treatments are wide and considered among the most important methods to improve the adhesion and compatibility between constituents. The treatments modify the

Table 4 Examples of natural fibers–polymers applications [26, 27, 29, 111–113]

Application	Fiber type	Example
Automotive industry	Wood, Cellulose-Polymer, Kenaf, Flax/Sisal, Wood Fiber, Jute, and Coconut	Bumpers, covers, door covering (racks), side and back doors panels, electronic device boot-liners, engine shields, floor trays, insulators, rear parcel shelves, seat backs, and hat rack seats, spare tire-lining, upholstery, and wheel shield
Buildings, constructions, furniture, and others	Sisal, Coir, Cotton, Ramie, Hemp, Oil Palm, Wood, Stalk, Rice Husk, Jute, Coir and Bagasse	Bank notes, panels, papers, packaging, bricks, construction panels, constructing drains and pipelines construction products, cordage, decking, door shutters, electrical, fencing, Furniture industry, furniture panels, furniture, geotextiles, pipes, railing systems, textile and yarn, textiles and frames of windows
Marine and Mechanical applications	Glass fiber reinforced plastic	Antenna dishes, both synclastic and anticlastic shells, bridge decks, chemical and water tanks, cooling towers, doors, folded plates of various forms, ladders, skeletal structures, staircases, walls, panels, and windows
Aerospace industry	Basalt fiber reinforced polymer, Carbon fiber-reinforced plastic	Access doors, antenna dishes, arrays, cockpit and fuselage of helicopters, control surface fin & rudder, engine cowlings, fairings, floorboards of helicopters, floor boards, front fuselage, fuel tanks, fuselage structures, interior decorative panels, main torsion box, motor casings, nose cones, other pressurized systems, partitions, pressure bottles, propellant tanks, ribs and longerons, rotor blades, solar booms and solar horizontal and vertical tail, stiffening spars, undercarriage doors, wing root, wing skin, cabin baggage racks, and several similar applications

(continued)

Table 4 (continued)

Application	Fiber type	Example
Sports	Glass fiber-reinforced plastic, Carbon fiber reinforced plastic	Bicycle frames, fishing rods, golf clubs, hockey sticks, poles (pole vault), ski poles, skis, tennis and badminton rackets, etc.

microstructures of fiber which results in enhanced tensile strength, morphology, and wettability, and offer chemical groups.

Physical treatments are common methods and are reported in various studies. The physical treatments are effective and can improve surfaces' adhesion and compatibility. They are likely to work by functionalizing fibers' surfaces using different mechanisms such as plasma, UV light, and ultrasounds. However, the plasma method is the most common method, especially in the modification of surfaces [115, 116]. For example, cold plasma is applied when impurities are targeted. The method influences different surface properties such as flame resistance, printability, and wettability. In addition, the method increases surface roughness resulting in greater mechanical properties and interfacial bonding [115]. In addition, with the incorporation of free radicals, surface hydrophilicity can be changed, and the free radicals have to react with oxygen or other suitable gases [115, 116]. In plasma treatment, ionized gas reacts with fiber's surface, this requires an electrical field between electrodes to transmit energy and accelerate gas electrons. Then, the gas electrons will collide with atoms or neutral gas molecules in a vacuum and under atmospheric pressure. In plasma vacuum, gas is allowed in a vacuum chamber at a low pressure. However, in plasma vacuum, the gas is supplied at a low-pressure triggering ionization by means of bond rupture or atoms removal, hence, causing a rise in crosslinking and free radicals. Plasma method is a batch process which requires a closed system with special arrangements [116]. On the other hand, the atmospheric plasma treatment is attractive in various manufacturing platforms. The method allows the specimens to be tested and modified in situ and is not limited to a vacuum chamber, it is a reproducible, reliable, and suitable treatment. The method is divided into multiple discharge methods, e.g., dielectric barrier discharge, atmospheric pressure plasma jet, glow discharge, and corona discharge. The last one is a method that applies a low-frequency discharge through the grounded metal roll and two opposing electrodes. The ionization of atmosphere generating plasma is induced by these discharges. Technically, the fiber is positioned in the void between the electrodes, then exposed to high-speed electrons, increasing the number of free radicals and stimulating surface oxidation [116, 117]. Compared to other plasma treatments, the process requires less energy and lower cost. The dielectric barrier discharge (DBD) is relatively similar to the corona treatment method. However, there are one or more insulators or dielectric barriers in the void between the electrodes. The transported charge is accumulated and then distributed around the area of electrodes. The gas available between the electrodes will not be ionized at this point and will function as a reservoir to absorb dissipated energy.

Some disadvantages of the dielectric barrier discharge are having a short duration and not being completely uniform [116, 117]. Another method is the atmospheric pressure glow discharge (APGD), the method is known for its stability, uniform and homogeneous surface treatment compared to other methods like the dielectric barrier discharge (DBD). The method functions in an argon or helium environment, at high frequencies, a low voltage passes within the conductive electrodes. The glow of the discharge is caused by the excitation collisions followed by de-excitation. In another approach, the atmospheric pressure plasma jet (APPJ), a quartz cylindrical tube is inserted where gas flows, this takes place between two tubular metal electrodes. The plasma method provides a very accurate treatment, it is applied in the surrounding air and occurs in the form of a plume in the sample [117]. Each process is preferred based on specific conditions and the availability of the process, for example, the APPJ is likely to be used in industrial and research applications, such as several biomedical devices, biological material sterilizations, and treatment of heat-sensitive materials. Another physical treatment is the ultrasound treatment [118], the method is different and effective, especially in modifying surfaces. Through ultrasonic irradiation, the method applies an effect known as the "cavitation effect", forming small collapsing bubbles that produce immense shock waves. The surface of fibers will start peeling andshow particle breakdown and erosion,this is due to the impact of waves shock. Ultrasound treatment has low and high frequencies. At low frequency, violent cavitation is generated,its effect is highly likely to be localized, whereas the high frequency provides less violent cavitation considering the shorter lifetime of generated bubbles [118, 119]. The treatment uses UV light representing electromagnetic radiation with energy potential. It should be capable of promoting photochemical reactions in fibers' surface by targeting its molecular structure [118]. The UV treatment is clean, cost-effective, and reliable for industrial applications [119]. Besides, there are additional types of physical treatments such as the ozone treatment, gamma-ray irradiation treatment, ion beam treatment, and laser treatment.

Reinforcements and plasticizers have a vital role in influencing various biocomposite properties, e.g., density, rheology, degradability, water sensitivity, gas permeability, shelf life, and antimicrobial behavior. Biocomposites' mechanical properties are influenced by several processing factors such as applied force, cooling and heating rates, temperatures and deformation rates. Those factors are associated with the type of polymer used in the process. Polymers' factors are also influencing and vital, such as chemical composition, morphology, molecular orientation, molecular weight, degree of crystallinity, copolymerization, cross-linking, concentration, plasticization, and the type of reinforcement. The processing environment, type of polymer, orientation of fiber, and type of reinforcement are factors influencing the tensile strength in biocomposites. There are various mechanical characteristics which have to be considered in composite materials, e.g., impact strength, compressive strength, hardness, tensile modulus, flexural modulus, flexural strength, tensile strength, yield modulus, yield strength, loss modulus, storage modulus, micromechanical performance, rate of elongation, and creep and poison's ratio. Various studies observed an enhancement in composites' tensile strength following chemical treatments on natural fibers utilized in these composites. Such progress is attributed to the reduction of fibers'

hydrophilicity which increases compatibility and enhances fiber–matrix interaction. Similarly, an enhanced behavior was observed in Bio-Polyethylene-based composites with alkali and palmitoyl chloride-treated coffee silverskin [120, 121]. Furthermore, improvements in flexural and tensile strength were also reported in alkali-treated fiber-added composites. Loading application on the composite determines the orientation of the. Several findings revealed that incorporating natural fibers as reinforcements in different types of polymer matrices would increase impact strength, flexural strength, hardness, and interlaminar shear strength.

9 Conclusions and Future Perspectives

The use of biocomposites and biomolecules with attractive systematic processing (such as the selection of suitable fibers and polymers, surface treatments, and chemical modifications) is becoming widespread. This work explored the different classifications of natural fibers, common polymers, composites, surface characterization, chemical treatments, and recent applications. It has been found that the compatibility of selected materials and the interactions formed are predominant factors that determine the rate of success of biocomposites. Maintaining the appropriate pre-treatment of natural fibers and the accurate modifications to their surfaces will essentially provide optimized results. Natural fibers possess a wide range of different and modifiable properties. However, the predominant challenge is to accurately determine their specific properties. Each fiber possesses different characteristics that must be individually assessed, pre- and post-treatments. This work further concludes that the features of the final biocomposite product are greatly influenced by several factors, such as the selection process of the fiber and polymer, the production stage, techniques, treatments, as well as modifications conducted during composite production and/or assembly. Nevertheless, natural fiber composites possess specific and commonly known characteristics that can be adjusted and modified to meet certain properties when possible. Hence, precise manufacturing is crucial to ensure successful optimization, one that conforms to the required arrangement of the reinforcement phase. This will contribute to their compatibility, further strengthening the bonds between all components. Additional research efforts are encouraged to enhance the mechanical, thermal, adhesive, and physical properties reported in biocomposites. This would ultimately improve their reliability and compatibility, especially when successfully used in medical or sensitive applications such as implants for example. Finally, biocomposites and biomolecules exhibit integrated features that can be potentially applied in biomedical applications. Nanomaterials, particularly nanofibers, are believed to govern their mechanical properties. However, it is extremely essential to develop and introduce compatible biocomposites. Chemical modifications are also required to alter certain properties and enhance others as a result. Further research efforts are required to obtain accurate conclusions regarding chemical treatments and modifications.

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Physical Modification of Bast Fibre Surface and Their Effects

M. Ramesh, J. Maniraj, S. Ganesh Kumar, and A. Felix Sahayaraj

Abstract Among the naturally available biodegradable materials, bast fibres (BF) play a significant role in engineering applications. The key attributes of BFs are dielectric, hygroscopic and surface properties, insulation properties and mechanical strength. The properties are variable based on chemical composition and environmental conditions. This study exhibits the extraction of BFs and the effect of physical modification of BFs and their effects. BFs can be extracted from herbs such as flax, hemp, ramie and other wild plants. The common extraction processes are microbial retting, mechanical extraction and alkali extraction techniques. The outer bark or phloem of jute, kenaf flax and hemp plants are called cellulose fibres and it is annually renewable crops growing in 9–100 days. The modifications of these BFs through biodegradable and eco-friendly techniques such as plasma treatment, fungi, microbial and biochemical reactions of enzymes. The treatment of BFs using these techniques varies the properties of the material. In the textile industries, natural fibres extracted from the plants play a vital role in the production of cloths, fabrics and mats that are ecologically sound. Cultivators involved in the growth of the herbs, as well as the extraction and treatment of the fibres, may benefit from the BFs. Hence, researchers show interest in discovering novel fibre and focusing on the various techniques to modify the surface of the already-discovered fibre for industrial requirements. These modifications can be accomplished by various eco-friendly methods such as physical, chemical and physicochemical treatments. In this paper, we have discussed and reviewed the various physical, chemical and surface behaviours of BFs and various surface treatments to modify the surface behaviour of the extracted BFs.

Keywords Bast fibres · Cellulose · Biodegradable · Surface treatment · Bark layer · Vegetable fibre

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1 Introduction

Natural products are now increasingly popular in recent years. These natural products are made from sustainable and environmentally friendly materials. The importance of biodegradable materials in the manufacturing of engineering components leads to show attention by biodegradable material researchers [1, 2]. On a global scale, 58% of clothing is made of synthetic fibres; from the report, it was clear that synthetic fibres are used more frequently than natural fibres [3]. Synthetic fibres are typically manufactured from polymers that are synthesized from chemical compounds which leads to environmental pollution. Therefore, synthetic fibres are more harmful to human health and the environment since they do not degrade easily after usage [4]. All countries are increasingly becoming more interested to use natural products to reduce both health problems as well as environmental issues. As a result of growing environmental concerns over synthetic products, manufacturers are looking for environmentally friendly, biodegradable and recyclable materials. Natural fibres are one of the most effective and popular fibres for replacing synthetic fibres. Jute, hemp, sisal and cotton are just a few of the natural fibres that are utilized commercially and are considered traditional fibres. Apart from these fibres, bio-fibres (BFs, also known as vegetable fibres) [5, 6]. Vegetable fibres have been used for textile processing even before recorded history. BFs have been abundantly available in the world since the dawn of history. Cotton is the most popular natural fibre in the world, with 25 million tons produced each year. Cotton production is predicted to be on average. Vegetable fibres are divided into numerous sub-categories based on morphological classification, including leaf fibres, seed fibres and BFs [7].

At the global level, the usage of synthetic fibres and natural fibres is increased for manufacturing of clothes, packaging sectors, manufacturing of nets, etc. The synthetic fibres are made artificially by synthesizing the chemicals which creates pollution to the environment and the synthesized polymers are not biodegradable which destruct the environment. To overcome health-related problems, people are attracted to the usage of eco-friendly materials in developed and developing countries. Biodegradable materials grab the attention of researchers and manufacturing industries in the manufacturing of engineering components [8]. The use of natural polymers and biocomposites have increased drastically due to their degradable property. Non-degradable plastics like polyethylene, polystyrene, polypropylene, etc., are threats to the planet due to the increasing accumulation. The environment-centred engineering products are based on biodegradable components [9]. In the manufacturing of exterior panels for automotive and agricultural components, thermal insulation components, sports, and furniture products, BFs are widely used due to their availability and mechanical properties. Based on the applications, BFs play a dominant role in textiles and cordage industries. BFs are very strong and relatively long; for these reasons, these fibres are widely used in manufacturing applications. There are several shreds of evidence found in the usage of BFs and biodegradable materials by ancient people for protecting their bodies, shelters, mats and utensils. The soft

woody fibre is extracted from the dicotyledonous plants. BF characteristics are specified based on the surface roughness and flexibility. It is categorized based on soft fibre and hard fibre. Jute is a BF category that is cultivated widely to extract fibres that are composed primarily of cellulose. Cellulose is the major component of the plants secondarily composed of lignin which is the major component of the wood fibre. BFs made of cellulose are often called cellulosic BFs, classified based on their anatomical origins such as seed fibre, BF, leaf fibre, wood fibre, grass and reed. In the BF category, the classification is based on applications and objectives of cultivation. The plant is cultivated for fibre extraction on the one hand and, on the other hand, plant is cultivated for fruits, vegetables and fibre is extracted as a by-product [10].

Flax is a type of BF cultivated majorly in France, China, Egypt and Belgium. The fibres are extracted from the phloem surrounding the stem of the plants. Flax has 36–42% of phloem content in dry straw. After extraction of the phloem content of BFs, there are various treatment methods such as physical methods and chemical methods to increase the mechanical properties. These techniques are used to enhance the bonding of layers and to reduce the deficiency of the BF layers [11]. The physical methods such as surface fibrillation, electric discharge, cold plasma methods, etc., increase the thermoplasticity and dimension stability of the BFs. The cold plasma method is often used technique and a very effective method to increase the surface properties of the BFs. This technique is used to increase the adhesion and compatibility between two natural polymers. The physical treatment of BFs leads to enhancing the mechanical bonding with the matrix, which leads to increasing the structural and surface properties [12].

2 Bast Fibres

BFs are generally long and have better tensile strength. Therefore, the BF is regarded as the most vital part of the plant. BFs are extracted from stems. The bark layer is covered on the outside of the stem, and the cuticle contains the BF which covers the stem. Between the bark layer and the stem core, the bast layer lies, which is a fibrous plant layer. Woody tissue and pith are the two elements of the stem core [13]. In a single fibre unit, BF molecular structure consists of chains of cellulose, hemicellulose, lignin, pectin and wax. As a result, BF is completely cellulosic in origin. The BF category has a large number of plants as compared to the number of plants farmed and processed commercially. The structure of bast natural fibre is exhibited in Fig. 1.

3 Background Information

BF doesn't have a well-defined history. However, various evidence suggests that prehistoric humans employed BF. Plant materials were used to cover and protect the

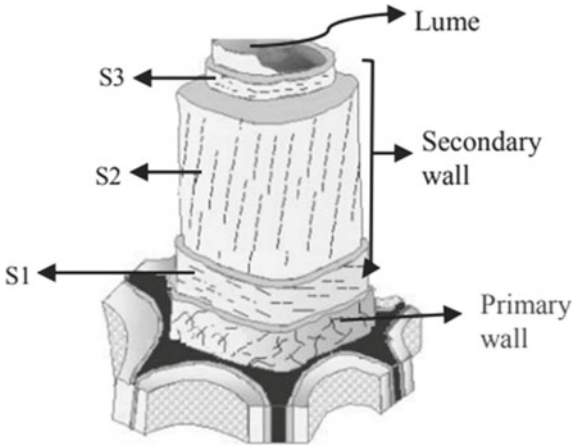

Fig. 1 Structure of BF

ancient people's bodies and thatched leaves were used for manufacturing the mats and fabrics and also used for domestic and other day-to-day life product applications. As a result, individuals developed BF specifically to meet their demands [14]. *Excavations in Switz*erland's ancient lake regions, dated back to roughly 10,000 B.C., have uncovered linen fabric fragments. Before 4000 B.C., actual weaved linen specimens were unearthed in Egypt. Kenaf fibre was first cultivated in West Africa from 4000 B.C. Ramie has been grown in south Asia and to some extent Africa and Egypt for hundreds of years. Hemp is one of the large volumes producing plants, with roots in the Southern part of India and an outspread to China. In Egypt around 3400 B.C., The technology of handloom linen cloth had advanced significantly, indicating that flax was produced earlier. Jute, hemp and flax are the most well-known BF, whereas kenaf, urena and nettle are lesser-known BF [15].

4 Physical and Chemical Properties of BF

Length and finesses of fibres are the two significant parameters to consider when evaluating the quality of natural fibres. The fibre's width might range from a few millimetres to several metres. The fineness of natural fibres varies based on the location of the single fibre [16]. Fibre strength is required for the spinning and weaving processes, as well as for the final cloth's strength. Because of its particular properties, flexibility lends a drape to a cloth. Many experts believe that a standard single-mode fibre strength of 6 g/D is required for the majority of fabric uses, while some fabrics have indeed been identified with properties as minimal as 1.1 g/D to be appropriate for a limited number of applications. Because individual threads in fabrics are commonly exposed to unexpected pressures, elasticity is critical and the fabric ought to be capable of yielding and rebounding without causing significant overall distortion [17]. A fibre's density is governed by its molecular functional

group and the layout of polymer molecules within it. The fibre volume of fraction has a significant impact on its aesthetic attractiveness as well as its utility in specific utility. Fibre contribution can be used as a tool for identifying fibres [18]. Chemical composition assessment is required for a better knowledge of fibre nature and the production of various equipment. The most important component of the plant fibre structure is composed of lignocellulosic materials. Other compounds found in vegetable fibres include lignocellulosic, pectin, cellulose, latex, inorganic materials, triglycerides and hydrocarbons, in addition to cellulose [19]. BF was traditionally used to make rugs, bags, dustpans and packing materials, styrofoam or canvas. In domains such as construction, automobile and packaging, representatives of many companies and research institutes are now manufacturing a variety of textile and non-textile goods. BF can be used to make geotextiles, fibreboard materials, as well as insulators, resistance elements, bonding agents, rigid pavement and tiles. in the construction industry. BF is used to create in the automotive industry, slightly elevated polypropylene and polymerizing lightweight structures such as trunk lids, consoles, rear headrests, box compartments, toppers and shoe liners are used. Because of its internal walls, the vehicle and aviation sectors have been working to develop various types of plant fibres, primarily cannabis, jute and agave, as well as bio-resin methods [20]. Natural fibre composites are appealing for a variety of applications due to their high specific characteristics and affordable pricing [21]. Non-woven fibres made from BF are utilized in a variety of applications and goods, including tissues and hygiene items, sorbents in diapers and disposables, insulating mats, mattress filler material and geo-textiles [22]. BF also makes biodegradable plant pots, mulching materials and agricultural packing cloth [23]. As a result, it may experience a revival, not only in the manufacturing of traditional industrial items but also in the development of new goods in industries such as papermaking and construction. Both the consumer and the manufacturer have benefited from the use of BF. The producer can improve the properties of BF with dyes and finishes, resulting in increased demand and sales of their end goods [24]. BFs are also important for industries in terms of a diverse selection of polypropylene synthetic fibre with an additional benefit. Apart from that, BF can help poor farmers better their livelihoods by assisting them in plant cultivation, fibre extraction and fibre processing, all of which are critical to their economic survival [25].

5 Surface Properties of Bast Fibres

As it pertains to the bonding strength, fibre surface behaviour determines the interaction between the matrix and reinforcement [26]. Fibre surface characteristics are the most important parameters influencing interfacial adhesion on fibre surfaces of BF-reinforced polymer composites [27]. It is essential to increase the adhesion property of BF for use as reinforcement since the fibres and polymer matrix are chemically different, according to the researchers [28]. Biodegradability of BF in alkaline treatment method [29], resulting in brittle composites with poor impact strength. Inverse gas chromatography (IGC) was used to examine the surface characteristics of raw and processed lignocellulose fibres, and it was observed that BF had greater surface dispersive energy than leaf fibre [30]. Plasma-induced modification affected the surface structure of coir fibres, resulting in considerable water absorption, according to researchers [31]. According to various researches, surface modification leads to an increase in shear strength. Fibre surface qualities affected by the following factors are fibre surface modification, chemical properties of fibre and fibre processing techniques [32].

6 Fibre Surface Modification Techniques

Fibre surface properties can be improvised by enhancing matrix–fibre adhesion, fibre fineness, and hydrophilicity, as well as minimizing water absorption behaviour. Fibre surface treatment can improve the tensile properties of BF in polymer composites. Impurities and wax compounds on the BF surface, on the other hand, cause poor surface wetting and impair fibre matrix bonding [33]. Fibre modification techniques are exhibited in Fig. 2.

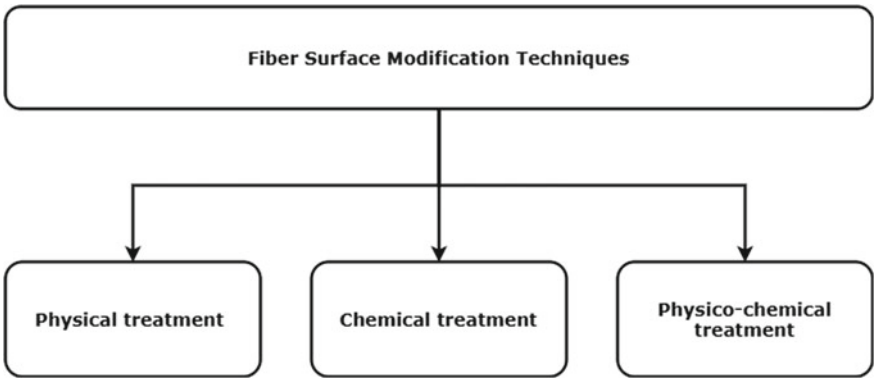

Fig. 2 Fibre surface modification techniques

7 Physical Treatment

Mechanical treatments enough to alter BF can have a significant impact on rheological and mechanical characteristics, boosting thermal properties and their influence on compressive and flexural bonding without affecting the BF's chemical makeup [34]. These treatments are split into three categories: mechanical treatment, solvent extraction treatment and electric discharge method, and they are used on BF to separate fibre clusters divided into discrete strands and enhance fibre surfaces for composite applications. Each physical treatment resulted in a different increase in mechanical properties and contact area, as well as increased crystalline size and resilience of the treated BF, as shown in Fig. 3. The effect of plasma and cationizing physical surface treatment processes are presented in Fig. 4 [35].

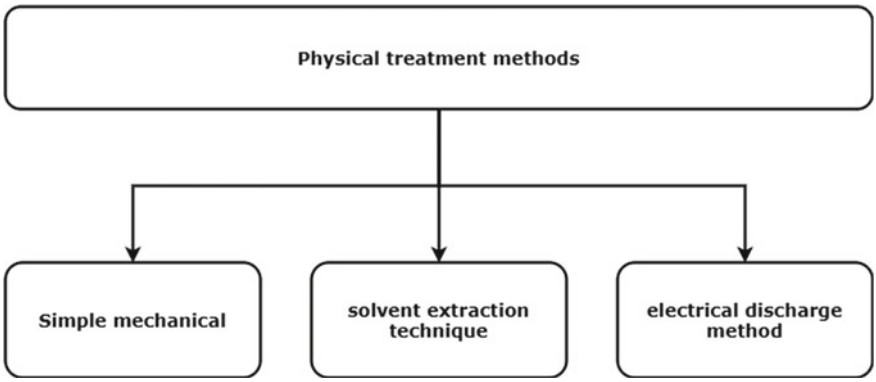

Fig. 3 Fibre physical treatment methods

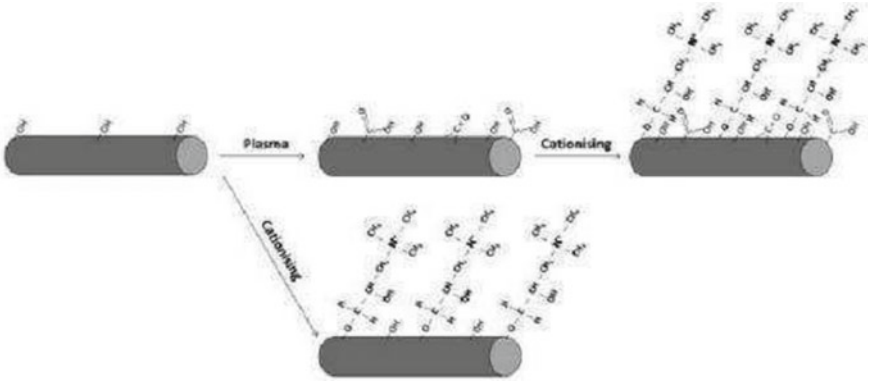

Fig. 4 Effects of plasma and cationizing surface treatment processes [35]

7.1 Simple Mechanical Treatment

The use of a variety of physical treatments to treat BF is increasing all the time, and they may be divided into three categories: simple mechanical treatment, chemical solvent extraction and electrical discharge. Stretching, calendering, rolling and shaping are examples of common mechanical treatments for long BF that impact fibre bonding with the polymer matrix [35, 36]. Stretching increases the ductile nature of the fibre, but it can also cause extension by allowing the BF to glide over each other during stretching, causing elongation and additional extension [37]. Calendering turns BF into a sheet that will fit into appropriate moulds by applying pressure from calendar rollers. According to some researchers [38], the calendering technique achieved the goal of improving the surface smoothness and density of BF. As a result, the pore size has decreased and bigger particles have been repelled from the BF sheet. BF bundle separation is achieved by rolling and swaging, with the rolling effect improving dispersibility and adhesion with the polymeric matrix. Traditional treatments can reduce fibre loss and improve the surface area available for fibre–matrix interaction in composites [39, 40]. In contrast, Varshney and Naithani discovered that fibres tend to tangle and consume a lot of energy during treatment. The BF chemical structure, on the other hand, will remain constant throughout the process, improving the composite's reinforcement performance [41]. This discovery was later confirmed by Ragoubi et al. [42] who found that applying BF significantly increased the fracture energy of composites while having no effect on strength or stiffness. Different treatment processes are listed in Table 1.

7.2 Solvent Extraction Treatment

Solvent extraction is the simplest way to employ mechanical fractionation to enhance surface area and eliminate soluble contaminants for short BF and fillers [43]. Solvent extraction was used to separate lignocellulosic fibres from BF sources, resulting in cellulose-rich fibres. This approach is a good way for separating a chemical on the basis of water solvent blending behaviour [44]. However, because BF will degrade as the fibre aspect ratio decreases, this therapy was not widely adopted. Furthermore, dangerous steam is created during this process which is damaging to the environment [45, 46]. Many novel bioderived solvents have recently been identified, however, not all of them can be employed for this purpose.

7.3 Electric Discharge Treatment

The electrical discharge utilized in this procedure is intended to separate cell walls, raise rheological characteristics and improve BF physical behaviour. By hardening

Table 1 Surface treatment processes of BFs

Process	Remarks
Stretching	Heat treatment will increase the tensile modulus of the fibres and improve their strength
Calendering	The density and permeability of the fibres were controlled and the filtration efficiency was improved
Rolling	Properties of the surface and structure of The fibres of lignocelluloses were partially modified
Solvent extraction	New bioderived solvents that are both renewable and biodegradable are attracting a lot of attention since they can aid with ecologically friendly solvent extraction
Corona treatment	It is used as a surface modification technique to remove unwanted material from the surface. Also used primary process for chemical treatment methods
Plasma treatment	In composites, PF had a better mechanical interlocking of the fibre matrix and had a stronger connection with the matrix
Ionized air treatments	Increases fibre wettability, however, the benefits in interfacial properties are less than chemical treatments
Thermal treatment	The use of a mixture of chemical and heat treatments on fibres can improve their initial strength and durability
Steam explosion	Temperatures range from 120 to 220 °C, with periods ranging from 30 min to 2 h
Ultraviolet	Surface oxidation increased mechanical properties and fibre–matrix adhesion when compared to normal electrochemical treatments
Electron radiation	Composites developed by this process having better mechanical characteristics and surface behaviour with lower resilience
Fibre beating	There is an increase in durability but a loss in mechanical strength as a result of defibrillation
Dielectric barrier	Due to the modification of fibre surface, roughness rises, resulting in improved wettability but a decrease in tensile strength

the interfacial bonding and morphology, electric discharge is an effective therapeutic technique for enhancing the interaction of hydrophobic resin and hydrophilic reinforcement. In contrast to solvent extraction, the electric discharge method has a minimal ecological effect [47, 48]. The most common electrical discharge approach for changing the heat stability preserves the personal appearance of the substance while retaining the molecular structure of the BF. Dry-up causes the lignocellulose fibre bundles to split into single filaments when BF is heated to temperatures between 100 and 200 °C for varying periods of time [49]. Fibre bundles will release or depolymerize emerged as an alternative/organic compound having lower glass transition temperatures or lignin-like glass transition temperatures [50, 51]. It is also observed that when BF is subjected to lower temperature ranges, the crystallinity of the material rises owing to fibre stiffness and improved physical adhesion between the fibre and the matrix. It was determined that the precise temperature at which BF

crystallizes is 150 °C, and it was deduced that heat therapy increased mechanical properties and modulus more than chemical modification [52]. Plasma treatment is another electrical discharge approach for fibre modification, and it is particularly successful in BF interfacial stimulation of the base. Due to growing concerns about environmental contamination, plasma treatment has been adopted as a strategy to decrease the usage of chemicals for surface treatments [53]. This technique may be utilized to enhance the fibre–resin interaction in biodegradable polymers and was discovered to be an efficient and sustainable method of changing the interface of BF [54] without the use of a chemical agent. This traditional approach first alters the outermost layers on the outside of the BF surface, resulting in considerable changes in BF surface morphology [55] and increased wettability. However, plasma-treated BF was shown to have a reduced strength value due to BF disintegration after treatment and is not recommended for use as reinforcement in composites [56]. Corona treatment, in combination with a dielectric barrier, is a sort of atmospheric plasma technology. Corona surface treatment [57] communicates modifications in fibre content and influences surface morphology of BF by using a low-temperature corona discharge plasma. The surface energy of the cellulose fibres is changed by an electric current used to provide an electrical discharge to a BF surface charge at or near atmospheric pressure [58]. Corona treatments aren't frequently employed because of the challenges of using them on three-dimensional fibrous materials [59], their intrinsic complexities, as well as the scarcity of studies devoted to comprehending their behaviour on BF [60]. These physical approaches, on the other hand, offer various advantages, including the lack of a requirement for exact alteration criteria, reduced, minimal energy, price technique and the capacity to handle a big volume of material on a large scale, all of which can improve industrial production. The dielectric barrier forms a non-thermal, non-equilibrium plasma at atmospheric pressure, which modifies the surface characteristics of fibres via the plasma process [61], which is similar to the corona therapy method. And though the ions and electrons in a plasma discharge can be elevated to temperatures ranging from 10,000 to 100,000 K, the gases themselves can be retained at lower temperatures as low as 100 °C. During discharge, collisions generate high-energy electrons, which can efficiently form activists and electrically enthusiastic elements. Even though the treatment process can help activate the BF's interface, the discharge is not consistent and only seems to last a short time [62]. Even though the therapy will minimize fibre strand agglomeration by centralized air procedure using electric discharge, which is analogous to corona discharge [63]. Furthermore, dissociation of fibre bundles and ionized air penetration through the BF improves BF during the therapy, fibre and resin interactions occur due to the presence [64]. As well as revealing significant changes in after-treatment surface finish, which might improve BF wettability [65]. In addition to alkaline extraction, one of the most effective methods is hydrothermal therapy. for removing hemicellulose fibres with minimal energy is required for hydrothermal technologies quick process [66]. In comparison to the alkaline treatment, the steam explosion methods require less energy, dangerous chemicals, human toxicity and environmental effect to achieve better fibre yields [67]. This process includes processing BF with superheated steam at different conditions and

response periods, which is triggered by shear strength caused by humidity extension and acetone, and created by acetyl group hydrolysis in BF-derived hemicellulose [68]. The fibres are physically shattered from within, releasing contaminants and forming fibrils without affecting the chemical composition. Some cellulose fibres may degrade depending on treatment gradation, time and temperature, but the BF is not considerably affected because of their superior tensile properties, crystallinity and less moisture content of BF. The electron energy approach [69] is a surface modification procedure for BF that develops contacts between BF and the polymeric matrix to improve properties, such as fibre/matrix, additives, temperature, pressure, morphology, crystallinity and surface–volume ratio, all influence the reaction [70]. The free radicals generated on the exterior of the BF by the charged particle radiotherapy procedure can activate carbonyl groups cross-linking polymerization, and the customized BF can be used in nanocomposite to enhance reinforcement–resin bonding or as an external consulting material for toxic metal ions adsorbent and sewage treatment purification. As a result of the irradiation, cellulose's architecture, responsiveness, physical qualities and physicochemical properties will be dramatically altered [71]. The BF polymer composite's performance was improved by fibre beating, a well-known physical surface modification method in the paper industry. Beating is a mechanical treatment used to prepare pulp for the paper-making process. It creates a regulated quantity of smaller fibrils, increases fibre bonding and provides optimal strength. It's not only widely utilized in wood-related materials but also been employed in BF. BF fibre angle acquired from the beating technique may be used to modify the microstructure and mechanical characteristics of BF [72]. The use of ultraviolet light is the least prevalent physical method (UV). When compared to standard electrochemical treatment, UV treatment has a higher degree of surface oxidation [73]. Lignin, wax and hemicellulose which function as UV radiation absorbers, give good UV protection in BF including jute, hemp and cotton fibre. One of the clean options is UV treatment, which is popular since UV sources are very affordable, versatile and simple to install [74, 75].

8 Future Scope

BF plays the dominant role in replacing synthetic fibres which pay the way for an eco-friendly environment. The cultivation of BF crops strongly influences the production of biodegradable products [76, 77]. The hybrid techniques in agriculture increase the production rate proportionally and the researchers have been carried out on the extraction of fibres using mechanical methods, chemical methods, etc. [78]. The scope of economical degradable materials is increasing and prevention of environment from recyclable materials can be achieved by cultivating the fibres. Kenaf fibres play a significant role and strongly influence the textile industry [79, 80]. It can be inferred that these environmentally acceptable ways for altering the surface of BF are green approaches. The drawbacks of BF can be rectified, and the demand for BF application at a marketable level can be enhanced, using these

ways. As a result, we can conclude that it is a green concept-based solution to natural resource sustainability in the form of surface-modified BF. At different stages, investigations such as the manufacture and removal of cells, microbial viscose, fungus and catalysts from environmental assets, as well as the deployment of these materials as a result of BF and other related fibres and the defining of their final products, can be conducted. The development of bio nanomaterials with enhanced bioactive components, such as those utilized in proactive and intelligent flexible packaging, are being developed and have opened up new possibilities for materials technology [81, 82]. Such biodegradable nanocomposite materials are likely to be the subject of extensive research to substitute or limit the usage of existing petrochemical-based goods Another unique approach is genetic engineering of natural BF to increase productivity, quality and application areas [83, 84].

9 Conclusion

BF plays the dominant role in replacing synthetic fibres which pay the way for an eco-friendly environment. The cultivation of BF crops strongly influences the production of biodegradable products. The hybrid techniques in agriculture increase the production rate proportionally and the researche have been carried out on the extraction of fibres using mechanical methods, chemical methods, etc., The scope of economical degradable materials is increasing and prevention of environment from recyclable materials can be achieved by cultivating the fibres. BFs play a significant role and strongly influence the textile industry. It can be inferred that these environmentally acceptable ways for altering the surface of BF are green approaches. The drawbacks of BF can be rectified, and the demand for BF application at a marketable level can be enhanced, using these ways. As a result, we can conclude that it is a green concept-based solution to natural resource sustainability in the form of surface-modified BF.

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Green Methods for Surface Modification of Bast Fibers

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Abstract The lack of fundamental knowledge in providing an eco-friendly surface modification of bast fibers for enhancing the composite's properties is still a major challenge in creating next generation composite products. The remarkable characteristics of bast fiber composites, which are ideal for manufacturing innovative green materials, have piqued the interest of researchers. Unfortunately, the fibers' chemically inert and smooth surface restricts their applicability. There has been much effort taken by the researchers to provide organic modification on fiber surface for enhancing the fiber-matrix interaction adhesiveness. In order to improve the chemical activity of the fiber's surface, reduce its hydrophilic character, and improvement in surface roughness, it is necessary to modify the fiber's surface. The major focus of this chapter is to briefly summarize the recent progress in surface modification of bast fiber in an eco-friendly approach and how far it affects the composite properties are being highlighted.

Keywords Bast fibers · Bio-surface modification · SEM · Fiber- interlocking

1 Introduction

The high specific strength, specific modulus, and low weight fiber reinforced polymers have seen widespread in use recently [13, 42]. Thus, these novel high performance composites are vital in the development of materials for the automotive,

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military, aerospace, and sports industries. Bast fibers from plant stems are being used by many researchers in manufacturing sustainable composites. High specific strength, high specific modulus, equivalent temperature resistance, and low density have made BFRPs a popular choice for polymer composite reinforcement[1]. Bast fibers reinforced polymer composite (BFRPs) are made up of strong fibers as reinforcement and the matrix is polymer-based. Fibers provide strong mechanical qualities, while the matrix aligns and protects the fibers [38]. Adhesion between the fiber and the polymer matrix affects the fiber reinforcement's ability to transfer stress. The strength of the bond between matrix/fiber has a direct impact on the durability and safety of the composite material. Owing to the increased crystallinity, chemical inertness, as well as smooth surface, bast fibers require extensive surface treatment to promote chemical and mechanical interlocking [10, 35, 41]. Moreover, the use of eco-friendly surface modification is also an indispensable key factor in developing sustainable engineering materials.

Thus, it is vital to outline the many eco-friendly surface modification approaches in bast fiber to guide future research and expedite wide applicability for polymer reinforcement. Table 1 highlights the various surface treatment and their advantages and disadvantages. In this chapter, we highlight some of the key breakthroughs in bio-modification technologies on the bast fiber surface and discuss their potential impact on composites. The following sections elaborate on this issue.

2 Bast Fiber Surface Modification for Polymer Reinforcement

It is necessary to ensure better bonding of BFRPs for reducing the risk of failures like fiber pull-out, de-bonding, and slide while increasing the composite's safety and durability of use. The surface treatments reduce the -OH functional groups present on the fiber surface that leads to an increase in surface roughness, hydrophobicity, chemical activity, thermal property, enhanced tensile strength/modulus, elevated cellulose content/crystallinity, and reduced moisture intake [27, 28, 37].

Figure 1 showed several green surface modification approaches. Surfaces can be modified biologically, physically, or chemically. Previously, many researchers used the chemical approach to modify surface properties [3, 28, 29]. However, when the used chemical is discarded, it harms the environment. Environmentally friendly treatments include new eco-friendly chemical processes, biological treatments, nanocellulose coatings, and plasma fabric coatings were being employed.

In the following sections, we shall describe the various green surface modification techniques of BF and its reinforcing impact on polymers.

Table 1 Bast fiber surface modification procedure's, benefits and drawbacks

Treatments	Method	Outcomes	Advantages	Disadvantages	References
Fungi	Fungi from the zygomycetes, ascomycetes, and basidomycetes groups	Elimination of non-cellulosic and amorphous substances	Eco-friendly, low-cost, and efficient	Care must be used while deciding on the treatment duration, since a lengthy treatment might weaken the fibers	[15, 19, 33]
Chemical treatment	Alkaline, Silane coupling agent, Acetylation, Benzoylation, Peroxide, Maleated coupling agents, Sodium chlorite, Isocyanate, Stearic acid, Oleoyl chloride, Permanganate, Triazine, Isocyanates, Graft copolymerization, Bleaching	Cleaning surface of fiber, chemical modification, reduced moisture intake, roughness enhancement leading to increased fiber strength	Easy to process, effectiveness and appropriate in a wide range of applications, industrial scale implementation, and the most widely used ways	Hazardous chemical used, skilled handling required, waste management need, and higher final product costs	[17, 18, 20, 21]

(continued)

Table 1 (continued)

Treatments	Method	Outcomes	Advantages	Disadvantages	References
Physical treatment include plasma, UV, corona, treatment	Treatments such as ultraviolet, heat treatment, plasma, corona, and fiber beating	Eliminating weakly adhered layers, synthesis of novel functional groups, better mechanical characteristics, increased hydrophobicity, and improved interfacial adhesion all result in a favourable influence on mechanical properties	There are no toxic chemicals required, resulting in a shorter treatment time, a lower operating cost, a lower environmental impact, and more flexibility	The only way to produce low-pressure plasma is through batch procedure, which requires a well-designed plasma reactor equipment and costly vacuum	[4, 11, 19, 26]
Nanocellulose Coating	Microbial or Bacterial cellulose	Improvement in interfacial adhesion since a new material has been deposited on the fibers surface	Simple procedure, low-cost, and environmentally friendly,	Because of the hydrophilic nature of bacterial cellulose, certain fibers lose their strength	[16, 19, 24]
Enzymes	Pectinase, amylase, xylanase, laccase, hemicellulase, cellulose etc.	Fiber separation from non-fiber components leads to interfacial adhesion enhancement, the fiber surface is cleansed	Little fermentation waste, well-controlled environmental treatment, and Eco-friendly, high-quality fibers	Expensive and only available on a trial scale	[6, 12, 19, 25]

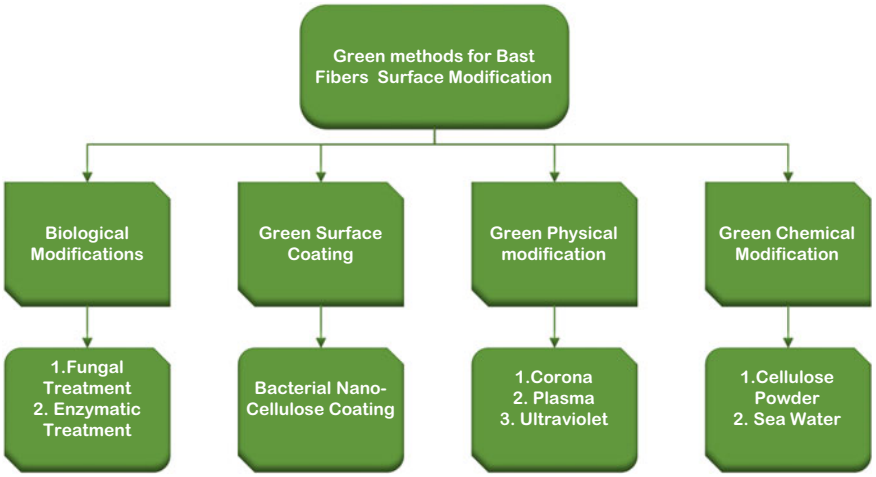

Fig. 1 Diverse methods used for Bio-fiber surface modification

2.1 Biological Modifications

Chemical or physical surface modifications are among the most frequently used types of surface treatments today. Nonetheless, they have a number of drawbacks, including the excessive use of solvents and high energy consumption, toxic chemicals, hazardous waste disposal, pollution, and the expensive cost of some chemicals and equipment. Over chemical and physical approaches, biological modifications have significant advantages. Microorganisms such as fungus, bacteria, and enzymes may be employed to circumvent this limitation; they may change the surface of natural fibers with a minimum energy input, after each usage. Furthermore, after each usage, enzymatic systems may be recycled [32].

Biological agents such as bacteria and fungi can alter the fiber surface. The fiber surface is modified by enzymes that remove wax and other non-cellulosic components. Lignin is removed by extracellular oxidase enzymes (such as those found in white rot fungi) and the solubility of hemicellulose is dramatically enhanced. It reduces hydrophobicity. For example, microorganisms may degrade surface materials to produce cellulose nanofibrils [34]. Increased interfacial adhesion between fibers and polymeric matrices was discovered by depositing 5–6% bacterial cellulose nanofibrils on the fiber surface. Aside from physical and chemical approaches, biological treatment has a potential future in fiber-based composite production [7].

2.1.1 Fungal Treatment on Bast Fibers

Fungi treatment of fiber is an effective, eco-friendly and an alternative to chemical treatment. Fungi produce lignin degrading enzymes that help to remove lignin from

fiber surfaces. Thus, fungi improve the hydrophobicity of fiber surfaces by eliminating hydrophilic lignin components. They create tiny holes or pits on the fiber surface, resulting in increased surface roughness and interfacial adhesion [7]. The fibers must be sterilized for 15 min at 120 °C before treatment. After drying, the fibers are cultured with fungi for 2 weeks at 27 °C [18]. The fungal hyphae on the surface of fiber decompose the lignin component of the fiber by secreting the enzyme xylanase, resulting in a doubled surface area of the fiber relative to untreated fiber [30]. The treated fiber also had a greater crystallinity index than untreated fiber, owing to the fungal exclusion of non-cellulosic amorphous components from the fiber surface.

Alkali pretreated hemp fibers were fungal treated to improve their bonding in polypropylene composites. Two weeks of incubation at 27 °C were used. The fungi used were *phanerochaete sordida, pycnoporus* species*, Schizophyllum commune*, absidia, *Ophiostoma floccosum.* Integrated fungus and alkali-treated fiber composites had a 32% increase in tensile strength over untreated fiber composites. Basidiomycetes (white rot fungus) is the only fungus capable of degrading non-cellulosic substances from fibers, and therefore increasing their mechanical qualities [33].

For the biosoftening of jute fiber, white rot fungus, specifically, *P. chrysosporium* and *C. subvermispora* FP, were selected [15]. There was no noticeable loss of cellulose in their biosoftening procedure lasting 30 days, which is considered the ideal incubation time for lignin degradation. Use of biosofteners like these fungi improves the fiber's spinnability by increasing the strength and elongation % and decreasing its stiffness.

Trametes hirsuta laccase was employed to biocatalyze the attachment of functional phenolic compounds to the fibers. A 5% insertion of functional molecules might improve polymeric matrix compatibility [2]. In an investigation, hemp fibers were treated with the fungus *Ophiostomaulmi* [12], and found that treated fibers are quite clean. This might be due to the elimination of water-soluble compounds by fungal activity on hemp fibers. Moreover, these fiber reinforced composite exhibited higher moisture resistance and increased composite strength. Fungal treatment is a low-cost, green treatment that is set to take the composite industry away from standard synthetic materials.

2.1.2 Enzymatic Treatment on Bast Fibers

The enzymatic treatment is recognized as an efficient method for fiber modification nowadays. Most preferably, enzymatic treatment is employed in textile industries for the alteration of fiber properties to prepare good quality fabrics. The use of enzymes for fiber treatment is on the upswing due to the fact that it is beneficial to the environment and precise action when compared to microbial modification. Enzymes such as hydrolases and oxidoreductases are suitable for polymer modifications. In hydrolases, glycosidases, proteases and lipases are studied for their effects on polymer modification. The enzymes such as tyrosinase, laccase and peroxidase are coming under the oxidoreductase class and were effective for surface treatments.

Fig. 2 Abaca fiber surface morphology micrographs: **a** Raw, **b** and **c** fungamix modified [6]

Enzymatic treatments are widely considered as an effective fiber modification approach today. Enzymatic treatment is preferred in textile industries to modify fiber characteristics and produce high-quality textiles. Enzymes are increasingly used to treat fibers due to their environmental friendliness and precision in modification. Enzymes like hydrolases oxidoreductase, tyrosinase, laccase, and peroxidase were used widely due to their excellent surface treatment properties.

Fiberboards were prepared by *myceliophtera thermophila* laccase treated beech (Fagus sylvatica) fiber. In enzyme bonded boards, lignin crosslinking occurs owing to covalent connections between fibers, which acts as an adhesive and improves adhesion. Laccase-bonded fiber boards are said to be stronger than urea formaldehyde-bonded fiber boards [36]. Flax fiber surfaces were modified by eliminating pectin and calcium using enzymes with chelators. To make the surface hydrophobic, they go hand with wall stripping and microfibril production, which helps to improve the interfacial bonding between fibers and their matrix [19]. Surface treatment of hump fibers with pectate lyase (EC 4.2.2.2) enzymes at various concentrations and treatment times resulted in the elimination of pectin components and increased the thermal stability of the fiber. Furthermore, the mechanical characteristics of the fibers were found to be inferior in the absence of the pectic component, which acted as a binding agent in the cellulose fiber bundles [31].

Surface roughness, waxy and projecting sections of untreated Abaca fiber (Fig. 2a) were removed by the Abaca fiber treated with Enzyme (fungamix) (Fig. 2b and c). Fibrillation occurs as the binding materials are removed from the treated fiber surfaces. Due to the modification, the outer surface of the fiber is removed and the diameter gets decreased [6].

2.2 Coating with Bacterial Nanocellulose

The treatment of natural fibers in the preceding sections entailed eliminating chemicals from the natural fibers. But here presents a novel modification of bast fibers, one that involves not removing but rather adding new material to the surface. Nanosized cellulose components are placed on the fiber surface to increase its adherence to the

matrix in this process. This allows for the creation of a hierarchical structure [22]. A. xylinum, a cellulose-producing bacterium, would deposit nanocellulose on the surface of plant fibers in the presence of a suitable growth medium.

By integrating bacterial cellulose into the fiber, interfacial contact between fiber and matrix is improved. However, for improved bonding, a proper distribution of bacterial cellulose on the fiber is required. The cellulose coating increases the mechanical interlocking of the interface, resulting in the introduction of nanocelluloses at the interface, which also improves the stiffness surrounding the plant fibers [34]. By establishing hydrogen bonds with the hydroxyl groups on the surface of the plant fibers, the deposition of bacterial nanocellulose improves the hydrophobicity of the fiber surface.

The bast fibers non-cohesive structure causes a severe decrease in fiber strength and Young's modulus when the hemp and sisal fibers are exposed to the fermentation media containing bacteria (Fig. 3a–e). This is due to a further splitting of the technical fibers into smaller fibers. The modified fibers were mixed with poly-L-lactic acid (PLLA) and acetate butyrate CAB, and the composite's characteristics were studied. When comparing the composite tensile characteristics to the unmodified sisal and hemp fiber reinforced composite, the Interfacial Shear Strength (IFSS) increased slightly. The attaching method significantly improves the interfacial adherence of both polymers which is seen from the SEM image (Fig. 3f, g) taken after fiber pull-out test [16, 34].

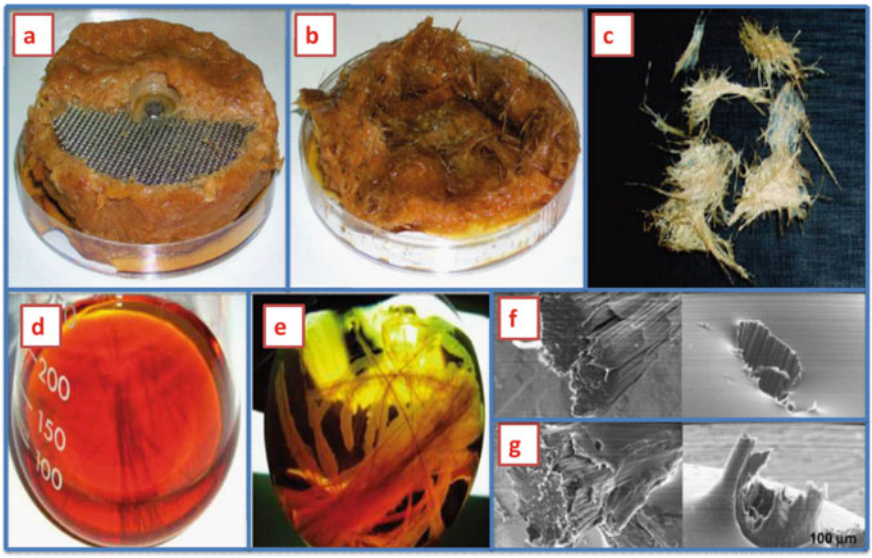

Fig. 3 Photograph **a** Bacterial cellulose pellicle upon fermentation; **b** Fibers inside cassette; **c** Bacterial cellulose-coated sisal; **d** Prior to bacterial culturing; **e** Bacterial culture after 2 Days; Single fiber pull-out SEM of **f** bacterial cellulose-modified sisal and the associated matrix voids; **g** acetone-treated and bacterial cellulose-modified sisal fibers and the associated matrix voids [34]

2.3 Physical Modifications

Chemical treatments attempt to diminish the fibers' hydrophilic character; however, surface treatments can improve the adhesion processes between the fiber surface and the polymer matrix by not only modifying the fiber surface but also increasing fiber strength. Surface fibrillation and electrical discharges are two major physical modification methods that modify the cellulose structure and consequently enhance the surface characteristics of the fibers. These treatments not only improve the surface qualities of the fiber, but also improve its mechanical strength and surface properties. Despite the necessity of surface treatment, there are just a few ways that have been tried to address this problem owing to a shortage of surface treatment equipment. Corona, plasma, ultraviolet (UV), fiber pounding, and heat therapy are examples of physical treatment procedures. In the plasma stream, ions, free radicals, and electrons are formed, which affect the surface properties of fibers. The substrate is subjected to plasma treatment, which involves blasting it with extremely energetic particles travelling in the plasma stream. Consequently, the surface attributes of the substrate, such as surface roughness, surface chemistry, and wettability may be changed without the use of toxic chemicals. The removal of loosely adherent surface layers is a common plasma treatment method for altering the surface of natural fibers (i.e., abrasion and cleaning) and establishing strong covalent connections with the matrix, which aid in excellent bonding [8]. The type of the plasma input gas determines the functional groups generated on the surface of natural fibers. As a result, plasma treatment minimizes the environmental effect caused by toxic chemical surface treatments. Jute fibers were treated for 5, 10, and 15 min with argon cold plasma before being composited with unsaturated polyester resin [39]. Plasma treatment resulted in the development of hydrophobicity, roughening the surface morphology of the fiber, and the generation of pits. The fiber pull-out length of plasma-treated composites was shorter than that of untreated fiber composites. Flexural strength composites made with 5 and 15 min plasma-treated fibers have lower strength values, which might be related to fiber breakdown caused by plasma treatment. However, after 10 min of plasma treatment, some crosslinking may have happened as a result of the production of new intermonomer linkages in fiber basic ingredients, which may account for the increased flexural strength attribute (14%) compared to raw fiber composite.

Except for hemp fiber, which is naturally hydrophilic, untreated abaca, flax, and sisal fibers are intrinsically hydrophilic. However, continuous exposure to air pressure plasma increases the hydrophilic behaviour of fibers to rise [4]. This could be due to the breakdown of hydrophobic chemicals in the fiber, re-deposition of an apparently denser hydrophobic protective layer or hemp fibers surface cross linking during high-pressure plasma treatment which limits swelling.

The helium plasma employed in this process creates radicals on the flax fiber surface that react with unsaturated polyester chains and eliminate surface contaminants [26]. Plasma treatment reduces the permeability coefficient of these fibers. The morphological modifications in untreated and plasma-treated flax fibers were studied. Figure 4 a shows the SEM micrograph of untreated flax fiber, and Fig. 4b shows

the plasma-treated fibers. The treated fibers appear cleaner. The surface topography shows the reduced contaminants in the treated fiber surface. The fiber pull-out fracture is visible. Figure 4c shows the composite reinforced by untreated flax fibers has poor interfacial bonding as seen by large holes and Fig. 4d shows a clean surface owing to greater bonding during fiber pull-out. Thus, the plasma-treated fibers enhance the interfacial bonding between the matrix and the fibers in composites. This is in line with the idea that helium plasma therapy should promote fiber/matrix attachment.

In plasma, the concentration, pressure flow, and gas type are controllable but in corona treatment, this is not possible. The experimental setup comprised two flat electrodes (aluminium) and a dielectric spacer (quartz plate). 1 g cellulose fiber in a 5 cm^3 corona cell was treated for 1 min at 15 kV, 60 Hz, 25 °C and 50% relative humidity. The corona treatment of the jute fiber leads to increasing the content of hydroxyl and carboxyl groups [11]. Optimum treatment conditions raise fiber modulus of elasticity by 15%. UV treatment is another physical modification process, in which the fibers were housed in a stainless-steel cylinder of 140 mm long and 35 mm in diameter. Irradiation uses high intensity radiation below 200 nm. The trials are done at room temperature with a 2.5 Torr pressure. The Xe KsR-$2A^8$ source lamp with MgF_2 window was employed at a 30 mm distance from a sample holder. Exposed with 147 nm radiation and the photodiode measures 3×10^{15} photons/(s cm^2) [38]. Fiber surface polarity is improved by UV treatment, which results in greater wettability of the fibers and increased composite strength. The polarity of the jute yarn was raised by up to 200% after being exposed to UV light for a longer period of time at a consistent distance from the bulb to the substrate [11]. It was

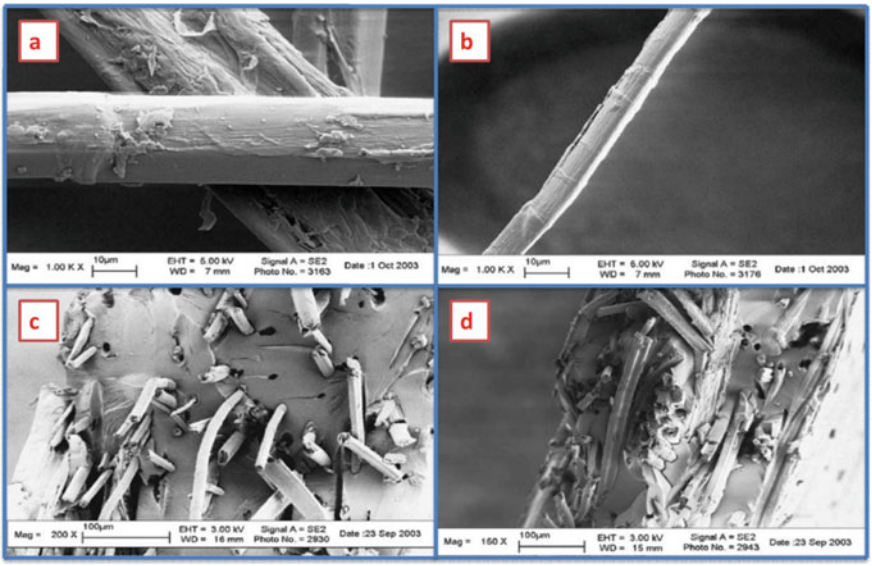

Fig. 4 SEM image of flax fibers **a** untreated; **b** plasma-treated; Tensile fracture of composites **c** reinforced with untreated fibers and **d** reinforced with plasma-treated fiber [26]

shown that the treatment distance had a significant impact on the yarn's polarity and tenacity. Jute epoxy composites' mechanical characteristics can be improved by UV-oxidation treatment of fiber; however, this requires finding an appropriate ratio among increasing surface polarity and decreasing fiber strength. A 30% improvement in composite flexural strength was attained under ideal treatment circumstances. UV surface treatment done in banana fibers had an impact on mechanical properties and chemical structure [9]. The fiber's tensile strength was dramatically increased when UV light was administered at acceptable levels. This material has better E values (238.94 MPa) and greater strength after seven days of being exposed to irradiation.

2.4 Chemical Modifications

The hydrophilic nature of natural fibers is one of the key issues in employing them as reinforcement in polymer composites. However, chemically-treated natural fibers reduce their hydrophilic properties [5, 28]. Chemical treatment or mercerization can also increase the thermal stability and surface morphology of fibers. The most significant chemical treatments include sodium chlorite, malleated coupling agents, benzoylation, peroxide, acetylation, silane coupling agent, alkaline, isocyanate, stearic acid, oleoyl chloride, permanganate, triazine, isocyanates, graft copolymerization, bleaching, etc. However, inappropriate disposal of chemicals after treatment causes environmental damage. Alternatives that do not harm the environment such as sea water and cellulose powder chemical treatment can also be used by researchers. After testing the pH and salinity of the saltwater, the fibers can be submerged in it for up to 30 days. The fibers were then washed and dried at room temperature [40]. Time-dependent surface morphologies of sugar palm fibers treated with saltwater or freshwater [23]. According to tensile testing, the fiber treated with saltwater for 30 days had the greatest stress value, 23,042.48 kPa. Preparing natural fibers with cellulose powder requires soaking the fibers individually in steel vessels with 2–10% cellulose pulp. It is produced in 30 min in hot distilled water. Then they were dried for 3 h at 70 °C [14]. It was found that cellulose treated fiber shows increased mechanical strength and interfacial bonding effect.

3 Conclusion

In the process of surface modification of bast fibers using chemical procedures, a significant amount of toxic chemicals were used. Proper chemical waste treatment and disposal can further raise the manufacturing cost of BFRP. Novel methods for bast fiber's surface modification with ecologically friendly technologies are the viable answer to this problem. Treatments which are more environmentally sustainable for modifying the surface of bast fibers such as plasma treatments, enzymatic, fungal, and coating with bacterial nanocellulose can be employed. Because bacterial cellulose

creates hydrogen bonds with the hydroxyl groups on the surface of plant fibers, coating them with bacterial nanocellulose boosts their hydrophobicity. When the non-cellulosic constituents are removed from plant fibers by fungal and enzyme treatment, cellulose and hemicellulose constituents are exposed more. Bacterial cellulose may hydrolyze cellulose in plant fibers. Plasma treatment causes a cleansing of bast fibers as well as surface etching. All of these approaches can alter the crystallinity, thermal behaviour, mechanical characteristics, and surface morphology of bast fibers without the use of toxic chemicals. Modified blast fibers can be utilized to create green composites, which suit in replacing the conventional materials for the automobile, aeronautical, structural/semi-structural, antibacterial and textile sectors.

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Surface Modification and Its Effect on Interfacial Properties

S. Govender, T. P. Mohan, and K. Kanny

Abstract Bast fibres are natural fibres that are sourced from the stem region around plants. Bast fibres show great potential as fibre reinforcement in composites. The inherent characteristics of unmodified bast fibres such as being hydrophilic in nature resulting in low fibre-matrix interfacial adhesion, natural wax and impurities on the fibre surface cause poor surface wetting, hydroxyl groups and free water reduce the fibres adhesive characteristics with resins, swelling and plasticising due to high moisture absorption causing dimensional instability and poor mechanical properties and high crystalline regions preventing resins from penetrating the fibre. Various methods, modifying the structure and surface of fibres, improve their inherent characteristics. The surface modifications of bast fibres achieve varying improvement of fibre-matrix adhesion in fibre-reinforced composites depending on the method of treatment. Treated fibres offer increased strength, improved hydrophilic nature, reduce water absorption, and remove impurities from the fibre surface. Surface modification methods are characterized as physical methods, chemical methods and biological methods.

Keywords Bast fibres · Chemical treatments · Surface modification · Interface properties · Characterization · Mechanical properties · Thermal properties

1 Introduction

Bast fibres show great potential in the field of composites as fibre reinforcement. Lack of adoption in bast fibre being used in composite materials is due to the inherent characteristics of untreated bast fibres, such as being hydrophilic in nature, there is a decrease in the compatibility with hydrophobic polymer matrices leading to poor fibre-matrix adhesion at the composite interface. Surface modifications are used to

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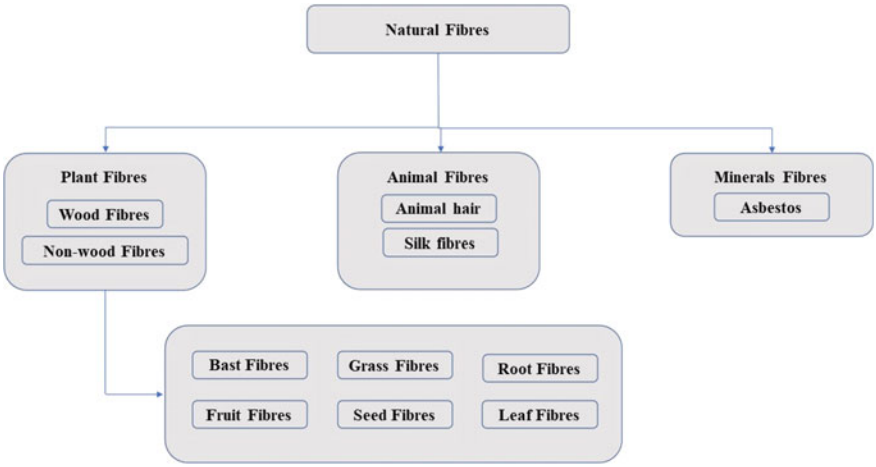


Fig. 1 Classification of natural fibres

treat the fibres in various methods to improve such undesirable characteristics. These modifications can be in the form of various mechanical, chemical, and biological treatments to improve the fibre's interfacial properties.

2 What Are Bast Fibres?

Bast fibres are cellulosic fibres sourced from the phloem or bast surrounding the stems of dicotyledonous plants. Bast fibres have found commercial application in cordage and the textile industry. Flax, Hemp, Ramie, Jute, Sisal, Pineapple, Kenaf, and Urena are common types of bast fibres. Bast fibres are characterized into "soft" or "hard" fibre groups, distinguished by fibre fineness and flexibility.

Extracting the valuable fibre, located in the phloem often requires separating from the xylem and sometimes the epidermis. This process is called retting. (Fig. 1) outlines the classification of bast fibres among common natural fibres.

3 Extraction Methods/Retting

looseness-1The first step in fibre processing is fibre extraction, where the fibre bundle's outer layer must be removed from the plant. Retting releases, the fibres from attachments and removes glueing agents due to breaking off the fibres from the core of the stem. The extracted fibre properties depend on the retting type, process,

and parameters. After retting, cellulose-rich fibres remain with high-strength properties [1]. Under-retting results in inadequate fibre separation and over-retting causes fibre weakening [2].

4 Surface Modification

High mechanical property composites require critical strong fibre-matrix interface bonds. An optimal level for fibre-matrix adhesion appears in polymer matrix composites, providing the best mechanical properties for the composite. A major constraint of natural fibres is their hydrophilic nature, this decreases compatibility with hydrophobic polymer matrices. Biocomposites have high water absorption characteristics due to the hydrophilic nature of bio fibres, reducing their applications. Insufficient fibre-polymer matrix bonding and poor surface wetting are caused by the presence of natural wax on natural fibre surfaces. Hydroxyl groups and free water, mainly in amorphous regions, reduce the natural fibre's adhesive characteristics with binder resins. The natural fibre's high water and moisture absorption nature results in swelling and plasticising causing dimensional instability and poor mechanical properties. Bio-fibres having a high content of cellulose, also have high crystalline contents. Crystalline regions are assemblages of cellulose, held together by strong intramolecular hydrogen bonds. In the fabrication of composite process, resins can only penetrate the fibre if the cell wall is swollen. Therefore, fibres must undergo various treatments. The complex structure of lignin and the crystallinity of cellulose make natural fibre undesirable materials of choice.

4.1 Physical Methods

Physical treatments roughen and clean surface impurities on the fibre, improving interlocking between the fibre-matrix. Physical treatments are eco-friendly as they do not involve the use of environmentally harmful methods. These treatments only affect the external cell walls and do not affect the chemical composition. Hence, they can be considered as pre-treatment for chemical treatments.

4.2 Plasma

Plasma treatment modifies the surface of natural fibres in a process that is carried out using high-frequency microwave energy to apply ionized gas molecules (plasma) to the surface of the fibres, as seen in Fig. 2.

Plasma treatment has been found to significantly improve mechanical properties [3]. Further, plasma treatment leads to the introduction of functional groups on the

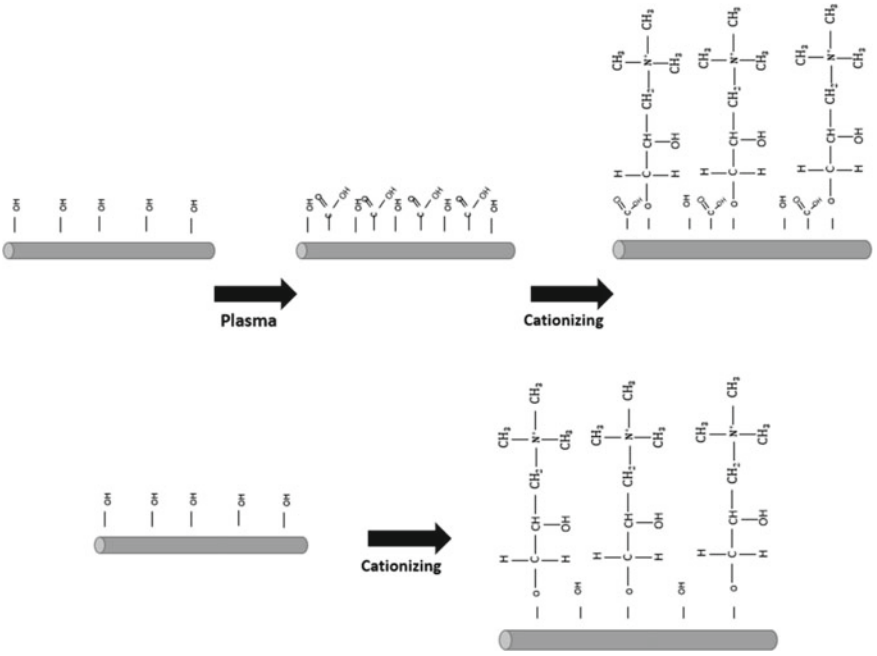


Fig. 2 Plasma treatment process

surface of the fibre. The functional group's bonds (covalent bonds) to the matrix provide a strong fibre-matrix interface. Plasma treatment also causes surface etching, which improves surface roughness resulting in mechanical locking leading to a better interface with the matrices as displayed in Fig. 3.

Plasma treatment increases mechanical properties, shear rigidity and bending, due to changed surface properties and surface roughness [4]. Plasma treated fibre composites have been reported to show an approximately 14% increase in flexural strength over raw fibre composites [5]. Plasma treated ramie fibre has shown up to a 50%

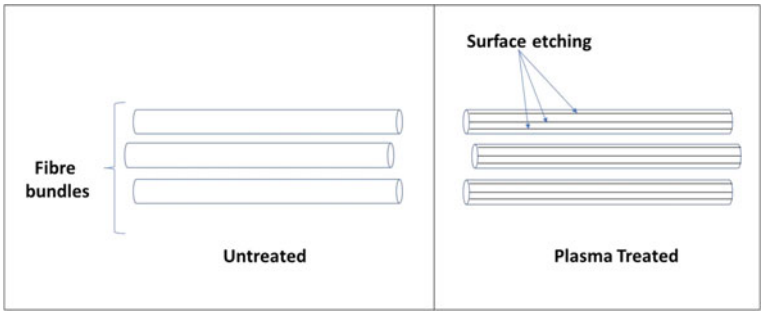


Fig. 3 Illustration of untreated and plasma treated sisal fibre

increase in the interfacial shear stress to polypropylene (PP) compared to untreated due to plasma etching increasing the treated fibre surface roughness [6]. Plasma treated jute/PP composites have been reported to show a 114% increase in tensile strength compared to untreated jute/PP composites [7]. Plasma treated banana fibre has shown increased tensile strength and increased elastic modulus, showing a stiffer fibre. The treated banana fibre decreased elongation at breakage [8]. The observed increase in performance of the fibre after being subjected to plasma treatment can be explained by surface etching resulting in improved interfacial mechanical locking at the fibre-matrix interface. The effect has an impact on improved mechanical properties. At a greater duration of treatment, the fibre may decompose due to softened interfacial adhesion as a result of the surface grains being subjected to continuous impact [9, 10].

4.3 Electric Discharge

Corona discharge treatment (CDT) is a treatment process where a large electric field is applied to the fibres through a sharp electrode. The CDT process can be seen in Fig. 4. The number of functional groups and the polarity increase, leading to improved fibre-matrix interface adhesion. CDT is applied in a continuous process, a common practice in the textile or paper industry [11]. This treatment can also be applied as a preparation stage for grafting the surface of the cellulosic fibre to hydrophobic, nonpolar polymers [12].

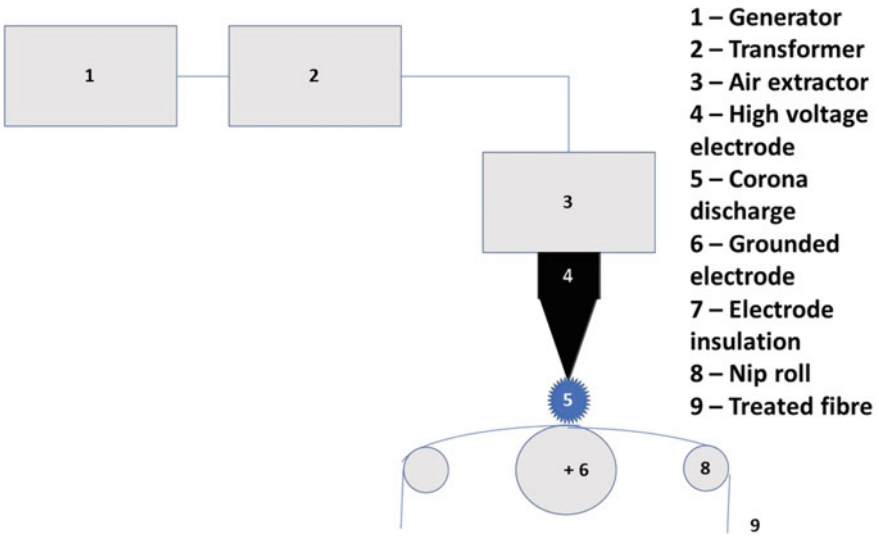


Fig. 4 The CDT processes

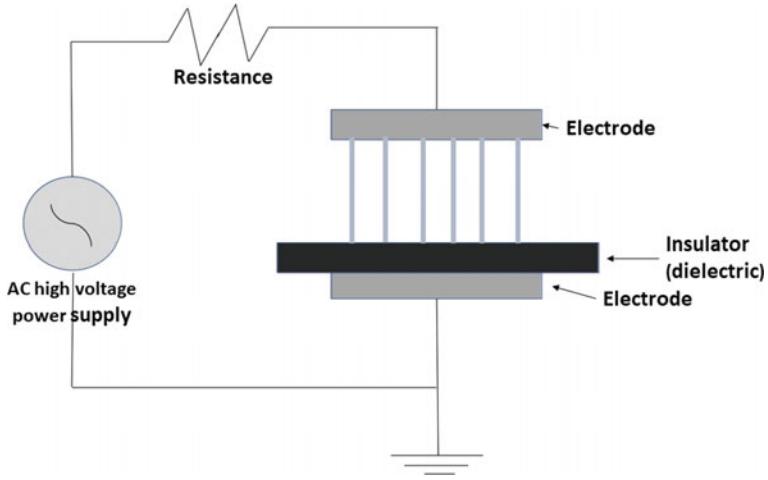


Fig. 5 DBD apparatus

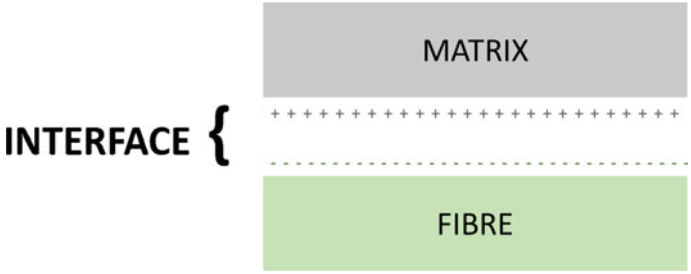


Fig. 6 Illustration depicting interfacial electrostatic adhesion

Dielectric Barrier Discharge (DBD) is a treatment process where a glass (silica or ceramic), is set between electrodes coupled to an AC high voltage power source. Figure 5 shows the DBD apparatus. The high voltage current ionizes gaseous molecules, producing plasma. The degree of plasma produced is dependent on the gas pressure in the apparatus and therefore the chance of collision between molecules.

Electric discharge treatment on fibres incorporates electrostatic adhesion to its interface and offers improved properties to their composites [13]. Figure 6 illustrates interfacial adhesion between the fibre-matrix.

4.4 Ultrasound

The fibres are submerged in a watery medium while exposed to an ultrasonic field and fibre and subsequently washed. This procedure ensures the fibres are cleaned

and freed of attendants like dust, soluble organic matter, microorganisms, colour and scents. The degree of cleaning can be determined by adjusting the process parameters [14].

4.5 Ultraviolet

Ultraviolet (UV) radiation is commonly used as a natural fibre surface modification. UV light causes the fibre surface to be more hydrophilic [15]. It was found that UV radiation can affect hydrogen bonds and reduce cellulose crystallinity [16].

It has been well described that UV significantly degrades the material strength of natural fibre composites. The duration of UV exposure was found to decrease the fibre tensile strength. The degradation rate of bast fibre composites depends on UV wavelength, exposure time, and UV intensity [17]. Compared to plasma and corona, UV treatment achieved larger polarity and can be controlled by extending the treatment duration or varying the distance of the sample. The UV treatment attributes increased fibre polarity at the surface and an improved fibre structure that is reduced once passed an optimal number of UV treatments. The strength of the fibre begins to decrease with each treatment passed the number of optimal treatments. The resulting surface polarity improves interfacial physical adhesion at the treated fibre-matrix interface due to improved wettability. Good wetting leads to the development of permanent adhesion due to molecular attractions like Van de Waals, covalent and electrostatic. Better adhesion results in greater composite mechanical properties.

5 Chemical Methods

This method of fibre surface modification uses varying chemical processes to modify the surface and structure of the fibre. Depending on the treatment method, chemical treatments commonly remove impurities, clean the fibre surface, release fibre bundles, breakdown the cellulose chain and in some instances, change the fibre's chemical structure, leading to improved interfacial interactions in composites between fibres and the matrix. Chemical treatments on fibres can be individually carried out or also as a combination of multiple methods to improve the characteristics of the fibre.

5.1 Alkali

Alkaline treatment or mercerization is a commonly used chemical treatment of natural fibres. During the alkaline treatment, hydrogen bonds are broken down in the fibres, increasing surface roughness, as visible in Fig. 7. It removes wax, lignin,

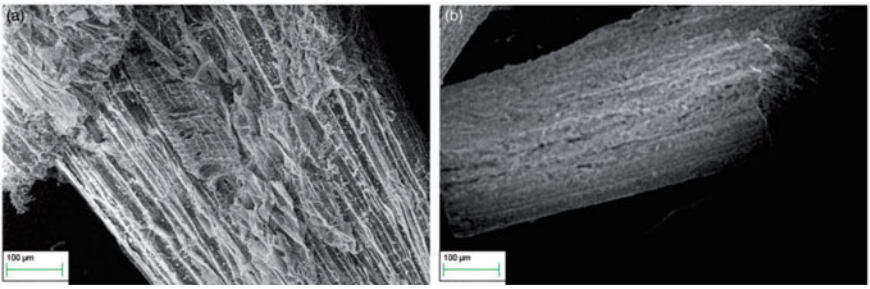


Fig. 7 The longitudinal surface of **a** untreated, **b** NaOH treated banana fibre [20]

and oils from the fibre surface, breaks down cellulose polymer chains and reveals shorter length crystallites [18]. Alkaline treatments have two effects on fibres: (1) increased surface roughness causing improved mechanical locking; and (2) increased cellulose exposure on the fibre surface, increasing the number of reaction sites [19].

Adding aqueous NaOH to fibres promotes alkoxide ionization in the hydroxyl group [21]. This reaction can be seen below:

$$Fibre - OH + NaOH \rightarrow Fibre - O - Na + H_2O$$

Alkaline treatments on bast fibres have shown to have a permanent effect on their composite's mechanical behaviour, specifically on fibre stiffness and strength [22]. Both the tensile strength and modulus for flax fibre-epoxy composites increased up to 30% after the fibres were subjected to alkaline treatments [23]. Fibre reinforced composites impact fatigue and dynamic properties are significantly improved due to alkaline treatments [24–26]. The optimal concentration of NaOH, for maximum tensile strength, is 4% for treating sisal fibre reinforced composites [26]. 5% NaOH treated sisal fibre reinforced polyester composites showed better tensile strength than 10% NaOH treated composites [27]. Excessive delignification of natural fibres occurs at higher alkali concentrations resulting in damaged or weaker fibres. A drastic decrease in the composite's tensile strength occurs after the optimum NaOH concentration. Fibre composites subjected to alkali treatment have shown increased desirable properties and interfacial adhesion due to the treatment roughing the fibre surface, having a positive effect on mechanical interlocking at the fibre-matrix interface. A 173% increase in interfacial adhesion shear strength has been reported [28].

NaOH treated sisal fibre shows better improvement in tensile properties than untreated sisal fibres (12%, 12% and 9.5% increase in tensile strength, modulus, and elongation respectively), however lower than that of NaOH–clay treated sisal fibre. A 17% increase in fibre pull-out was observed in NaOH treated sisal fibre over untreated sisal fibre in polypropylene. The fibre pull-out was examined by viewing the fractured fibre–matrix interface region by using SEM. Figure 8 shows the micrographs of the fractured surface of fibre–matrix interface region of untreated and NaOH treated

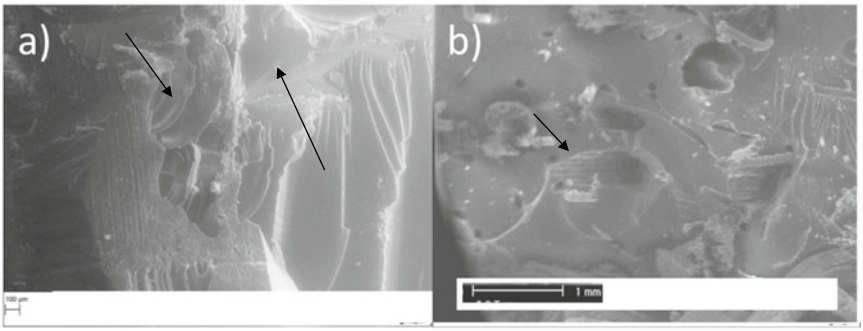


Fig. 8 The fracture surface of matrix–fibre interface surface of **a** untreated sisal fibre composites and **b** NaOH treated sisal fibre composites

sisal fibres–PP composites. The fracture surface of fibre–matrix interface is smooth in untreated and NaOH treated sisal fibre composite [29].

Alkali treatment is commonly considered a standard to improve the effectiveness of additional treatment methods [30].

5.2 Silane

Silanes stabilize composite materials as coupling agents allowing the fibre to adhere to the polymer matrix. Silane coupling agents reduce cellulose hydroxyl groups present at the fibre–matrix interface. Hydrocarbon chains form a crosslinked network caused by covalent bonds between the fibre and matrix, restraining swelling. Silane coupling agents were found to effectively modify the interface of natural fibre–polymer matrixes, increasing their interfacial strength. It has been reported that silane-modified fibre composites displayed improved fibre–matrix interaction and much higher tensile strength compared to alkaline treated fibre composites [19]. Silane treatment also improved the thermal stability of the composites [21]. The reaction can be seen below:

$$CH_2CHSi(OC_2H_5)_3 \xrightarrow{H_2O} CH_2CHSi(OH)_3 + 3C_2H_2OH$$

$$CH_2CHSi(OH)_3 + Fibre - OH \rightarrow CH_2CHSi(OH)_2O - Fibre + H_2$$

A 32% increase in fibre pull-out was observed in NaOH–clay treated sisal fibre respectively over untreated sisal fibre. The improvement in pull-out strength is indicative that the NaOH–clay treated fibre increases interface properties and fibre–matrix adhesion. The fibre pull-out was examined by viewing the fractured fibre–matrix interface region by using SEM. Figure 9 shows the micrographs of the fractured

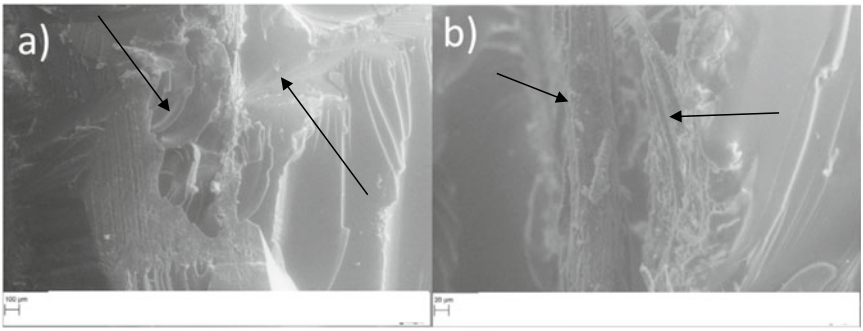


Fig. 9 The fracture surface of matrix–fibre interface surface of **a** untreated sisal fibre composites and **b** NaOH–clay treated sisal fibre composites

surface of fibre–matrix interface region of untreated, NaOH treated and NaOH–clay treated sisal fibres–PP composites. The fracture surface is rough in NaOH–clay treated sisal fibre composite (fibre–matrix interface fractured surface shown by arrows).

5.3 Maleated Coupling

Composites containing fibre reinforcements and fillers are commonly strengthened using maleated coupling agents. Unlike other chemical techniques, the maleic anhydride modifies the fibre structure and the matrix, improving interfacial bonding and mechanical properties. This treatment increases the surface energy of fibres to a surface energy level closer to that of the matrix [31]. Resulting, in better wettability and increased interfacial adhesion of the fibre. Mechanical properties of plant fibre reinforced composites are improved after maleic anhydride treatment. It has been reported that reduced water absorption in banana, hemp and sisal fibre reinforced PP composites due to MAPP treatment [32]. Aside from alkalisation maleic anhydride treatment is a widely popular method of treatment [30]. Treatment improves interfacial adhesion of hydrophilic bast fibres and hydrophobic matrices resulting, in composites with improved mechanical properties compared to untreated composites [33]. The notable effect maleated coupling has on the surface wettability of fibres and matrix improves interfacial adhesion and leads to improved composite performance. The relative cost and effectiveness of the treatment warrant the popularity this method of surface modifications has gained.

5.4 Acetylation

Acetylation is a common, esterification method for cellulosic fibres causing plasticization. The reaction introduces an acetyl functional group ($CH_3COO–$) into an organic compound. Acetic acid (CH_3COOH) is generated and needs to be removed before using the fibre.

Acetic anhydride modification, replaces the polymer hydroxyl groups with acetyl groups, resulting in hydrophobic polymers [34]. The fibre reaction with acetic anhydride is as follows:

$$Fibre - OH + CH_3 - C(=O) - O - C(=O)CH_3 \rightarrow Fibre - OCOCH_3 + CH_3COOH$$

The surface of natural fibres undergoing acetylation became very rough and contained numerous voids that provided improved mechanical locking between the fibre–matrix, this can be observed in Fig. 10. Treated fibre composites were found to have higher thermal stability than untreated fibres composite due to the treated fibres improved thermal stability and fibre–matrix interaction [35]. It has been reported that acetylated fibre reinforced composites compared to silane treated fibre composites exhibit lower tensile strength loss and higher bio-resistance [36]. Flax fibres having undergone different concentrations of acetylation treatment reported 50% higher thermal properties. An increase of around 25% in flexural and tensile properties was observed in 18% acetylated flax fibre–polypropylene composites compared to untreated fibre composites [37]. Acetylated fibres have been seen to significantly affect fibre–matrix interfacial adhesion. A 435% increase in interfacial adhesion shear strength has been observed with enhanced cellulose exposure facilitating improved fibre–polymer matrix interlocking at the interface due to the acetylation treatment process in sisal fibre and polypropylene composites [28]. Figure 11 illustrates the process of mechanical interlocking between fibre–matrix.

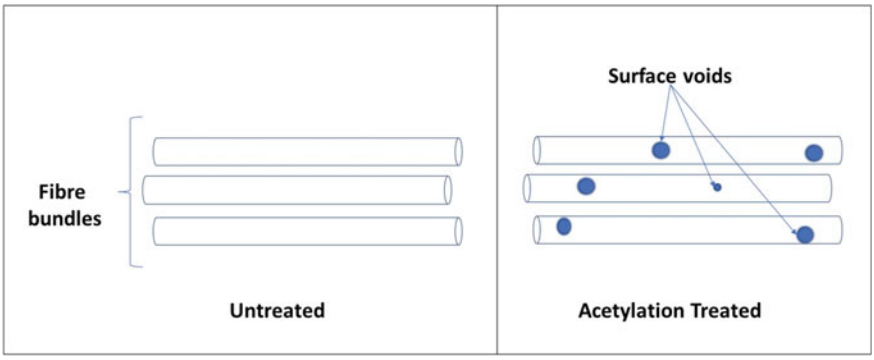


Fig. 10 Illustration of acetylation effect on the fibre surface

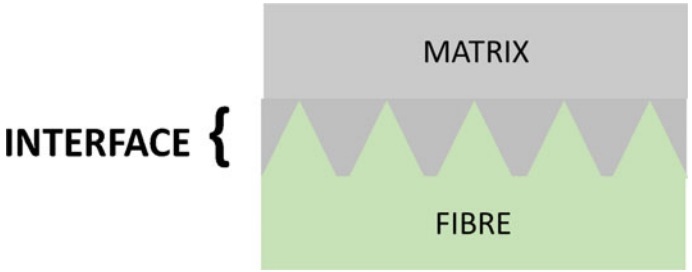
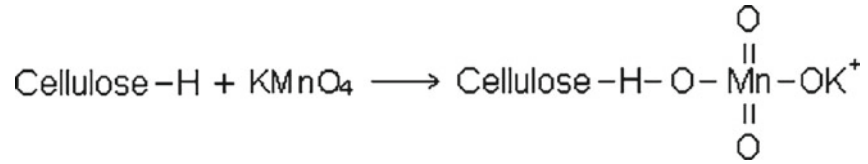
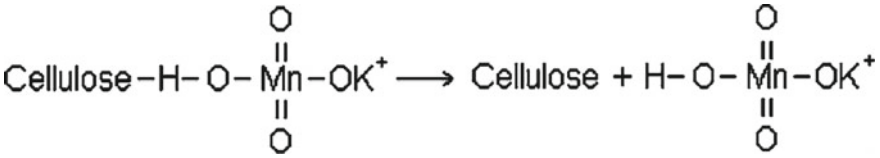


Fig. 11 Illustration displaying interfacial mechanical locking in fibre–polymer composites

5.5 Permanganate

Permanganate treatment forms cellulose radicals through fibre and MnO_3^- ion forming. Followed by highly reactive Mn^{3+} ions initiating graft copolymerization. This reaction is seen below [38]:





Potassium permanganate ($KMnO_4$) is the most used solution in permanganate treatment. Permanganate treatment reduces the hydrophilic tendency of fibres, decreasing water absorption of fibre reinforced composites. Permanganate treatments improve chemical interlinking resulting in improved interfacial adhesion between the fibre–matrix. The formation of cellulose-manganate improves thermal stability. Due to an oxidation process, the fibre surface becomes rough due to the potassium permanganate engraving the surface of the fibre, improving mechanical interlocking at the fibre–matrix interface [39]. Flexural properties in modified banana fibre–polypropylene composites have shown an improvement of 10% [40]. Potassium permanganate-treated flax fibre-high density polyethylene and linear low-density polyethylene composites offer higher tensile properties than untreated fibre composite counterparts [41]. Figure 12 illustrates the chemical interlinking.

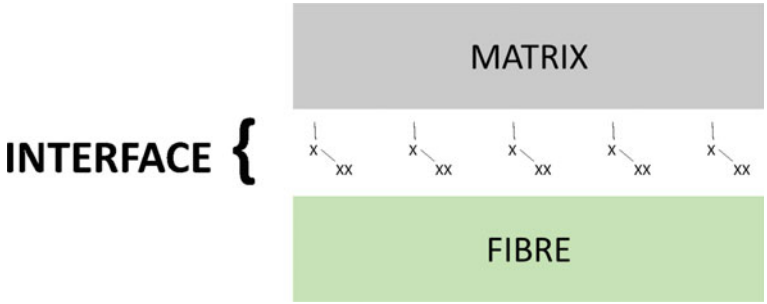
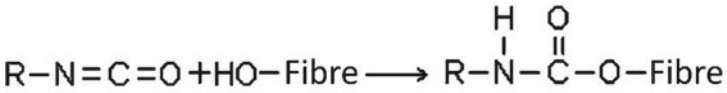


Fig. 12 Illustration of chemical interlinking at the fibre–matrix interface

5.6 Isocyanate

The isocyanate functional group (N=C=O) is prone to react with the hydroxyl groups in fibres. Isocyanate works as a coupling agent in fibre reinforced composites [24, 42, 43]. The isocyanate reaction [38]:

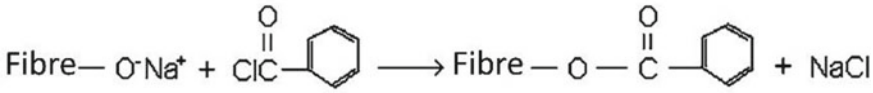


Where R could be various chemical groups. It has been shown that isocyanate treatments were more effective than silane treatments in improving the mechanical properties of cellulose fibre–PS composites [44]. Toluene diisocyanate-treated sisal fibre–LDPE composites reduced the hydrophilic nature of the sisal fibre, enhancing the composite's tensile properties [24]. Toluene diisocyanate-treated oil palm fibre composites when compared to alkaline-, peroxide- and permanganate-treated fibre–PF composites have reported lower Youngs modulus and tensile strength but reported similar young modulus and higher tensile strength with acrylic acid-and silane-treated fibre–PF composites [45]. On the flax surface, the free –OH reacts with isocyanate, leading to reduced fibre surface polarity, and strengthening interfacial adhesion between fibre and resin [46].

5.7 Benzoylation

Benzoyl chloride, used in fibre treatment, results in decreased hydrophilic nature of the treated fibre and increases reaction with the hydrophobic matrix. The fibre reaction with benzyl chloride can be seen below:

$$\text{Fibre} - \text{OH} + \text{NaOH} \rightarrow \text{Fibre} - \text{O}^{-}\text{Na}^{+} + \text{H}_2\text{O}$$



The benzoylation treatment of fibres improves fibre-matrix adhesion, leading to a considerable increase in the composite's strength, a decrease in water absorption and increased thermal stability. NaOH benzoyl chloride treated fibre–PS composite interface has been hypothetically modelled [35]. The thermal stability of treated composites shows improvement over untreated composites. A similar process has been reported for interfacial adhesion of flax fibre and polyethylene [47].

5.8 Peroxide

Organic peroxides decompose easily to free radicals which react with hydrogen groups of the fibre–matrix. This reaction can be seen below:

$$RO - OR \rightarrow 2RO$$

$$RO + PE - H \rightarrow ROH + PE$$

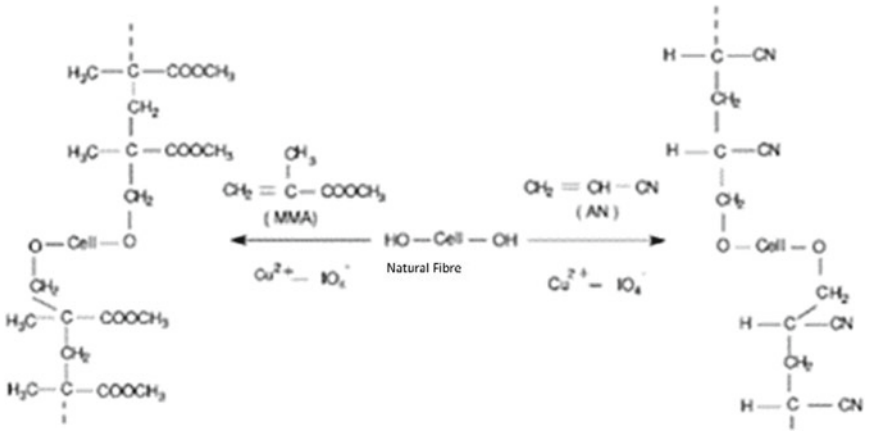
$$RO + Cellulose - H \rightarrow ROH + Cellulose$$

$$PE + Cellulose \rightarrow PE - Cellulose$$

Benzoyl peroxide (BP) and dicumyl peroxide (DCP) treated short sisal fibre–PE composites were observed to increase tensile strength with an increase of peroxide to a level and then reached equilibrium (4% DCP & 6% BP) [24]. The Peroxide treatment resulted in a decrease in the hydrophilicity of the fibres and an increase in the tensile properties [42]. Kenaf fibre bleached with hydrogen peroxide after alkalisation reported increased crystallinity and surface roughness, leading to improved mechanical interlocking between the kenaf fibre–PLA matrix. Resulting, in increased mechanical properties of treated kenaf fibre–PLA composites [48]. Among treatment methods, hydrogen peroxide treated kenaf fibres in polypropylene matrix composites contributed the highest mechanical and physical properties, this is described by good fibre–matrix adhesion observed by SEM [49].

5.9 *Graft Copolymerization (Acrylation/Acrylonitrile Grafting)*

The procedure involves chemical copolymerization where biopolymers are used as the backbone of the polymer. Free radicals of cellulose initiate this reaction. An aqueous solution with ions is utilized to treat cellulose while exposed to radiation. The cellulose molecules crack forming radicals. These radical sites are then treated with a compatible solution. Cellulose fibres treated with hot polypropylene-malice anhydride (MAH-PP) copolymers yield covalent bonds across the interface. The fibre treatment increases the surface energy near the matrix's surface energy, providing higher interfacial adhesion and better wettability. The graft copolymerization reaction can be seen in detail below:



Graft copolymerization forms a chemical bond between fibre and matrix, improving interfacial adhesion. After treatment, the fibre and matrix surface energy are closer resulting in improved interfacial adhesion and better wettability [50]. The following equations are used for percentage graft yield and efficiency [51].

$$\%Grafting(P_g) = \left(\frac{W_g}{W} - 1\right) \times 100$$

W_g = weight of grafted fibre, W = weight of ungrafted fibre

$$\%Percentage(P_c) = \left(W_g - \frac{W}{W_m}\right) \times 100$$

W_m = weight of monomers

5.10 Effect of Multiple Chemical Treatments on Interfacial Properties

Surface modified composites have higher storage modulus than unmodified fibre composites. Alkali treatment and silane treatment of ramie fibre improved adhesion with a PLA matrix compared to untreated ramie fibres. The alkali-treated composite had the highest storage modulus, suggesting alkali treatment of fibres is an optimal method to improve interfacial adhesion between ramie and PLA matrix. A significant decrease in the storage modulus was observed in the range between 50 °C and 70 °C. The treated composites were observed to have a decreased damping peak compared to neat PLA, indicating improved compatibility between PLA and the treated fibres [52]. The impact properties of surface-modified ramie fibres are higher than unmodified ramie composites. The major factor that influences impact strength is toughness. Fibre reinforced polymer composite's toughness depends on the polymer matrix, the fibre, and the strength of their interfacial bonding. Composites with alkali-treated fibres get the highest impact strength. Alkali treatment under impact testing appears to provide effective resistance to crack propagation because of the improved interfacial adhesion. The treated composites improved mechanical properties due to the alkali treatment improving the fibre surface adhesion characteristics due to removing artificial and natural impurities, producing a rough surface. Improved thermal properties were observed using DSC due to improved adhesion between both faces [52].

Akali, permanganate pre-treatments, benzoyl chloride, malic anhydride and silane have been shown to improve jute/epoxy-polyester composite mechanical damping properties when compared to untreated fibre composite caused by the improvement of interfacial bonding. DMA testing has shown that silanized and benzoyl chloride treated jute fibre reinforced polymer composites improved thermal stability and storage modulus compared to composites reinforced with untreated fibre. This appears to be due to silanization improving interfacial bonding resulting in reduced frictional damping. Silanized and benzoyl chloride treated jute fibre reinforced composites displayed improved strength compared to untreated jute fibre composites at the same concentration [53].

Polylactic acid (PLA) reinforced with alkali-treated short jute fibres (5%, 10%, 15%) with H_2O_2 composites demonstrated a considerable increase in tensile strength compared to untreated fibre composites. The tensile modulus of 10% NaOH/H_2O_2 treated fibre composites at higher fibre loading was 40% and 125% than untreated fibre/PLA composites and neat PLA respectively [54].

Various chemical modified jute fibre reinforced LDPE polycaprolactone composites showed better mechanical properties compared to untreated fibre reinforced composites. Jute fibres that were subjected to silane treatment displayed increased roughness of the fibre surface and diameter. Further, these composites showed increased mechanical properties such as tensile strength and modulus and a decrease in elongation at break, with an increase in the weight fraction of fibres [55].

Untreated, as well as NaOH and NaOH/clay treated, fibre reinforced epoxy composites were subjected to fibre pull-out testing to study the fibre–matrix interfacial properties. The maximum debonding load of untreated fibre was 240 N and increased for NaOH and NaOH/clay treated fibres to 255 N and 330 N, respectively [20]. The NaOH/clay treated fibre showed the maximum increase possibly due to the presence of nanoclay and the high-strength crystalline phase of treated fibres possibly caused the higher load reduction force and debonding. The treated fibre's higher deboning load suggests the fibre–matrix bonding is stronger and a higher load can be applied to the fibre before failing. The fibre–matrix interface of the debonded fibre is examined by SEM images in Fig. 13.

Untreated fibres displayed a smooth interface at pull-out with relatively less friction and strength. The interface of the chemically treated fibre displayed fibres sticking to the matrix due to increased binding, increasing the pull-out strength. The presence of nanoclay on the fibre surface resisted fibre pull-out in the opposite direction of loading, possibly causing the increase in pull-out strength [20].

Nanoclay particles infused sisal, kenaf and banana fibre composites increased surface area, fibrillation, and crystallization. The nanoclay is attached to the fibre surface with hydroxyl bonding and acts as a barrier for water uptake and moisture. Adsorption results display a reduction in swelling and water uptake in nanoclay infused samples providing dimensional stability to the fibres. Nanoclay infused fibres

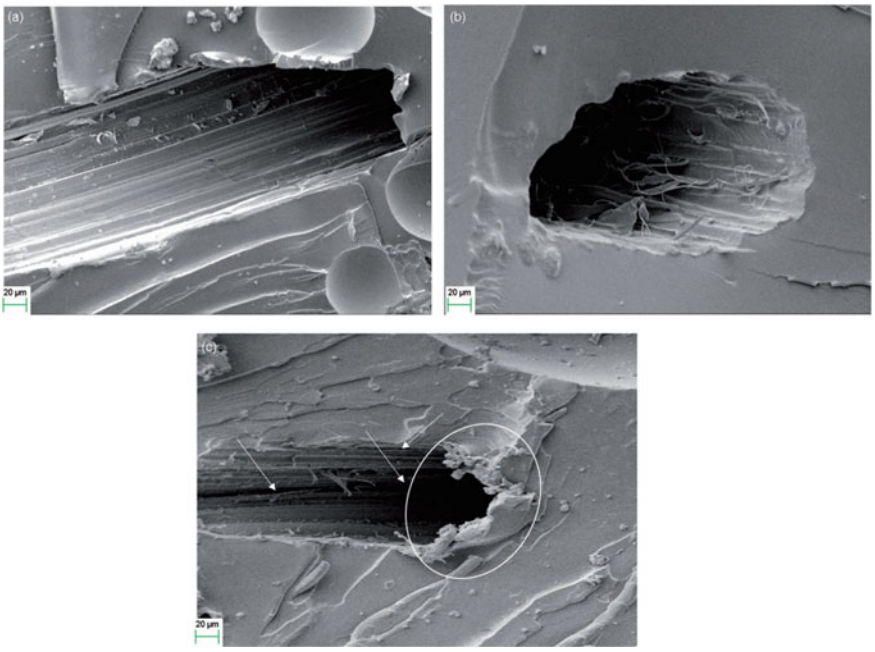


Fig. 13 Fibre–matrix interface fracture of **a** untreated, **b** NaOH, and **c** NaOH/clay during fibre pull-out test [20]

displayed less moisture uptake compared to untreated fibre equilibrium moisture content (EMC) values. The equilibrium moisture content values reduced up to 3% for nanoclay infused fibres.

Table 1 displays the degree of hysteresis (dH) of the adsorption–desorption curves of values for untreated and nanoclay infused fibres. Untreated fibre results show higher dH values compared to nanoclay treated fibre. Nanoclay reduced the forming of the polylayer moisture absorption at the fibre surface for intermediate vapour pressure resulting in reduced dH.

The nanoclay effect on absorption and swelling characteristics are displayed in Table 2. Nanoclay infused fibre swelling is substantially reduced compared to untreated fibres (Table 2). The equilibrium molar water uptake (Q) values of nanoclay infused banana, kenaf and sisal fibre are 42%, 36% and 18% lower than that of their corresponding untreated samples [56].

Table 1 Degree of hysteresis (dH) of untreated and nanoclay treated fibres

Relative vapour pressure (P/P_0), %	Degree of hysteresis(dH)					
	Sisal		Banana		Kenaf	
	Untreated	Treated	Untreated	Treated	Untreated	Treated
0	0	0	0	0	0	0
10	16	11	19	13	16	12
20	17	12	19	13	17	12
30	16	10	20	12	18	11
40	15	10	20	12	18	11
50	16	10	17	11	17	10
60	13	9	19	10	14	9
70	11	8	16	9	12	8
80	13	9	13	8	12	8
90	5	3	16	7	6	7
100	0	0	0	0	0	0

Table 2 Swelling properties of untreated and nanoclay infused fibres [56]

Fibre	Swelling % increase
Untreated	
Banana	24
Kenaf	16
Sisal	11
Nanoclay infused	
Banana	9
Kenaf	8
Sisal	9

6 Biological Methods

These methods of fibre surface modification utilize biological bodies to improve fibre properties. They include cultivating fungi, bacteria or enzymes on the surface of fibres, modifying their characteristics and improving their composite's interfacial interaction between the fibre and matrix.

6.1 Fungi (White Rot Fungi Treatment)

Fungal treatment increases interfacial adhesion due to the formation of pits, providing a rough texture to the fibre surface. White rot fungi are selectively able to breakdown lignin at a higher rate compared to cellulose. It also produces extracellular oxidases that mimic lignin molecules. Fungi naturally produce extensive hyphae networks possibly roughening up the surface. White rot fungi are aerobic microorganisms that require oxygen to develop.

Further, the use of Ophiostoma ulmi as fungi has been shown to free treated fibres of impurities possibly due to the removal of water-soluble compounds by the fungi as seen in Fig. 14 [57]. It has been found to have a marginally positive effect on moisture absorption and improved durability of the composites. Additionally, it was found to increase basic and acidic components after fibre treatment, exposing the hydroxyls and phenolic hydroxyls groups, and improving adhesion with both basic and acidic resins. Fungal treatments are environmentally friendly and low-cost and show promise in shifting the composite industry towards greener pastures.

A combination of alkali and fungal modified hemp fibre–polypropylene composites reported tensile strength of 48.3 MPa, a 32% increase over untreated fibre composites. The results have suggested due to improved mechanical locking at the fibre–matrix interface, fungal treated fibre composites strength has improved by up to 22% [58].

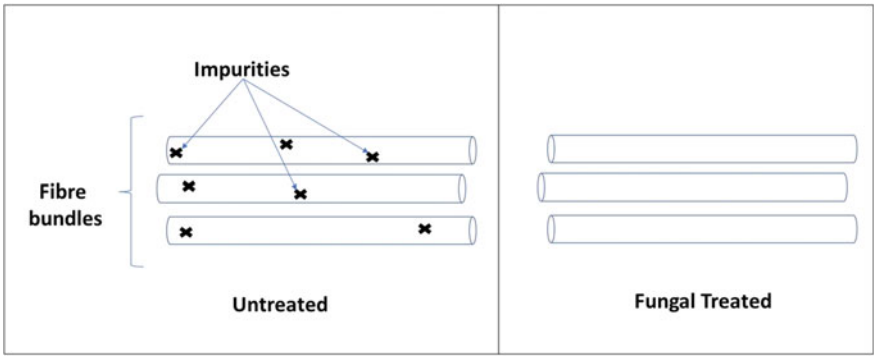


Fig. 14 Illustration of the effect fungal treatment has on fibres

6.2 Bacteria

Bacteria belonging to genera Acetobacter, Agrobacterium, Alcaligenes, Pseudomonas, Rhizobium or Sarcina, produce bacterial or microbial cellulose. Acetobacter xylinum was found to be the most efficient producer of bacterial cellulose [59]. Producing extracellular cellulose microfibrils that form a firm matrix allowing bacteria to stay in contact with the atmosphere. The cellulose produced protects competitors and promotes colonisation on the substrate. It has been found that a single bacterial cellulose fibril has a greater elastic modulus (78 GPa) than that of natural fibres (30 GPa) and is much close to that of glass fibres (70 GPa) [60]. Growing cellulose-producing bacteria on natural fibre shows bacterial cellulose significantly covers the fibre surface due to their hydrophilic and rough surface. It has been shown to improve interfacial adhesion to bio-based polymers, resulting in, enhanced properties and better durability. The hydroxyl groups present at the substrate surface and of the bacterial cellulose help promote hydrogen bonding between them. The interfacial adhesion of the deposited cellulose with the surface of the natural fibre can be improved by subjecting the fibres to a pre-treatment by solvent extraction, removing the hydrophobic compounds from the surface. This results in significant increase in interfacial adhesion to polymers and consequently leads to the improved mechanical performance of composites. Modified hemp and sisal fibres in polylactic acid and cellulose acetate butyrate matrices composites were fabricated and interfacial shear stress were characterized. The interfacial shear stress significantly increased in bacterial cellulose treated fibre composites compared to untreated. An optimized treatment process of sisal fibre leads to cohesive fibre failure under single fibre pull-out testing, implying interfacial adhesion exceeds the sisal fibre sub-fibres [61]. The strengthened interface can be described due to the present nanoscale cellulose on the fibre surface, increasing roughness and molecular entanglement of bacterial cellulose fibrils and the polymer. Figure 15 illustrates molecular entanglement at the fibre–matrix interface.

Fig. 15 Illustration of molecular entanglement at fibre–matrix interface

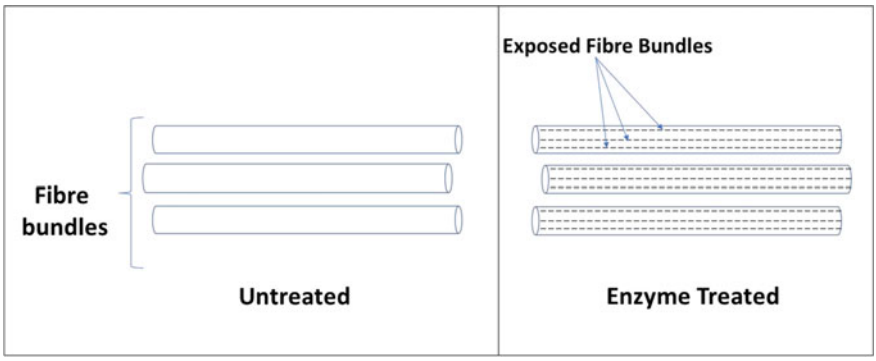


Fig. 16 Illustration of the effect enzymes treatments has on fibres

6.3 Enzymes

There are various enzyme mixtures developed for the treatment of natural fibres, generally rich in pectinase. Enzymes separate the fibres into individual bundles. The treatment exposure of fibre bundles can be seen in Fig. 16. It has been found that enzymatic treatments can improve crystallinity and thermal properties [62]. Enzymes effectively remove hygroscopic pectin and hemicellulose, resulting in a more homogeneous fibre surface with improved thermal properties. The removal of hygroscopic pectin and hemicellulose increases individual bundle exposure and improves surface roughness.

Fibre pull-out results report enzyme-treated fibre composites to have better fibre–matrix adhesion due to mechanical interlocking suggested by SEM displaying sharp-edged fracture regions [63].

7 Future Perspective

As world-leading industries realize gains by investing in greener processing, the use of natural fibres as an alternative to non-renewable sourced fibre has gained traction. Bast fibres offer several notable advancements to synthetic alternatives documented in Table 3.

Bast fibre however has a fair share of constraints, limiting its performance in applications as fibre reinforcement in composite materials. These drawbacks are due to the inherent nature of the natural fibres. The use of surface modification techniques on bast fibres has successfully shown improvement in fibre performance in composite material systems. Depending on the surface modification technique utilized, various effects have been noted to improve the fibre surface interfacial adhesion with polymer matrices. This allows for natural fibre reinforced composites to possess comparable properties to non-environmentally friendly materials, currently utilized in global

Table 3 Comparison between natural bast fibres and synthetic fibres

Property	Bast fibre	Synthetic fibre
Mechanical properties	Moderate	High
Thermal sensitivity	High	Low
Moisture sensitivity	High	Low
Renewability	Infinite	Limited
Manufacturing	Low	High
Recyclability	Good	Moderate

Table 4 Application of bast fibres and their composites in major industries

Industry	Current and possible application
Automotive	Interior components, decorative accents, upholstery, packaging and exterior panels
Aerospace	Interior components, partitioning, storage compartments and low stress structures
Building and construction	Partitioning, decking, fibre reinforced cement, insulation, furniture and decorative accents
Healthy and beauty	Absorbents, wound dressing, clothing, medical cast and implants
Packaging	Chemical storage, logistics boxes and bags and insulation

industries. This has not gone unnoticed and has gained extensive interest from major stakeholders in several industries around the globe.

Current trends show promising signs for the mass adoption of bast fibre, with their composites already being currently utilized in industries ranging from beauty and health to aerospace and automotive as seen in Table 4. While there is much to still learn about surface modification techniques and their effects on interfacial adhesion, there are numerous companies and industries that have readily begun using natural fibres in their products. Figure 17 shows the relationship between knowledge and product value for respective industries.

The surface modification of bast fibre composites has varying effects on its interfacial properties depending on several factors such as the type of fibre, modification method, duration of treatment, etc. Figure 18 shows the governing range of composite performance.

8 Conclusion

Extensive research has been carried out over the years into surface modifications and their effect on the interfacial properties of bast fibres. Most of the research found shows positive effects on bast fibres and their composites properties due to changes in the interfacial properties.

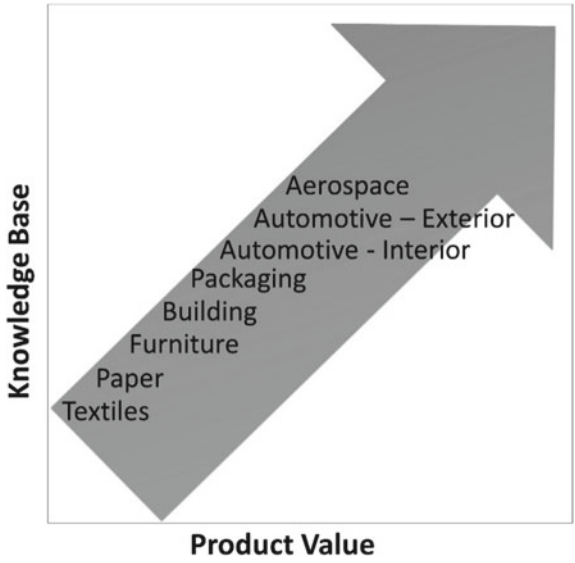


Fig. 17 Application of bast fibres

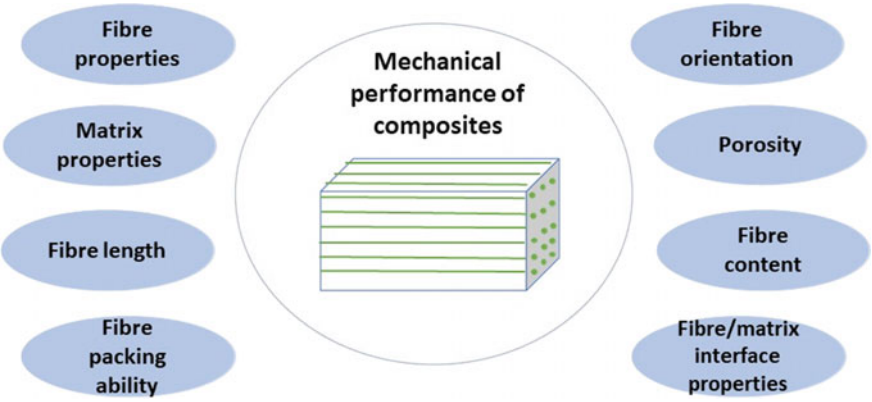


Fig. 18 Parameters governing composite performance

Physical treatments roughen and clean surface impurities on the fibre, improving interlocking between the fibre–matrix. These treatments only affect the external cell walls and do not affect the chemical composition. Hence, they can be considered as pre-treatment for chemical treatments. Plasma introduces functional groups to the fibre surface as well as causes surface etching, both significantly improving interfacial adhesion with matrices, leading to improved mechanical properties. Electric discharge uses a voltage to increase the number of functional groups and polarity, it can be used as a preparation stage for grafting. Ultrasound cleans and frees impurities on the fibre surface, improving interfacial properties. Ultraviolet cause the fibre

surface to be more hydrophilic. It has been found that ultraviolet degrades the strength of the fibres.

Chemical treatments commonly remove impurities, clean the fibre surface, release fibre bundles, breakdown the cellulose chain and in some instances, change the fibre's chemical structure, leading to improved interfacial interactions in composites between fibres and the matrix. Chemical treatments on fibres can be individually carried out or also as a combination of multiple methods to improve the characteristics of the fibre. Alkali treatments increase fibre surface roughness, improve mechanical interlocking and increase the exposure of reaction sites on the fibre surface. Silane acts as a coupling agent allowing the fibre to adhere to polymer matrices. Silane-treated fibres displayed improved fibre–matrix interaction and have a positive influence on mechanical properties. Maleic anhydride modifies the fibre structure and the matrix, improving interfacial bonding and mechanical properties. Acetic anhydride modification, replaces the polymer hydroxyl groups with acetyl groups, resulting in hydrophobic polymers. Permanganate treatment reduces the hydrophilic tendency of fibres, decreasing water absorption of fibre reinforced composites. Benzoylation results in decreased hydrophilic nature of treated fibre and increases reaction with the hydrophobic matrix. Graft copolymerization increases the surface energy near the matrix's surface energy, providing higher interfacial adhesion and better wettability.

Biological surface modification utilizes biological bodies to improve fibre properties. They include cultivating fungi, bacteria, or enzymes on the surface of fibres, modifying their characteristics and improving their composite's interfacial interaction between the fibre and matrix.

The surface modification of bast fibres has been extensively studied for decades, providing key knowledge and possibilities to utilize bast fibres in a variety of current products and industries.

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Manufacturing Aspects of Bast Fiber-Based Composites

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Abstract Each day, an increasing number of objects for several industries, including automobile, aerospace, biomedical, construction, and even electronics industry, are manufactured using bast fiber-based composites. Composite manufacturing depends on the matrix and reinforcement configuration. Many techniques can be used, and the selection depends on the final use or purpose of the composites. For example, it is possible to obtain simple laminates by casting or using a most expensive technique like compression molding. In this chapter, the main aspects of the processing techniques are discussed. Comments are provided on alternatives to improve the final quality of composites, and in some cases, aspects of how nanostructures can be included during composite manufacturing are introduced. The analyzed techniques are for polymer and cementitious matrices.

Keywords Bast fiber · Processing composites · Polymer matrix · Self-reinforced composites · All-cellulose composites · Cementitious composites

1 Introduction

For several centuries, humanity has been looking for material alternatives to improve the behavior of their tools, machines, and constructions. Notably, the mixing of several materials to obtain advantages is always a constant. Since ancient times, natural fibers have been used to develop composite materials. The behavior of bricks fabricated by ancient Egyptians using mud and straw is a known process [1], and this situation is mentioned in the Bible (Exodus 5:6–14). With the industrialization

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process and the introduction of assembly lines, the material supply requirements represent new opportunities to use natural fibers for materials required in new industries, such as automobiles. One of the most iconic events corresponds to the development of the Ford industry and its car panels fabricated using natural reinforced composites, some of which are based on soy-protein plastic [2]. During World War II, fuselages of fighter aircraft were made with flax fiber composites [3]. Currently, these composites are of increasing interest because of their potential to improve and implement solutions around the circular economy, which is focused on reducing fossil resources, increasing the use of waste streams, and increasing biological recycling [4]. Some industrial applications where natural fiber composites are considered include automotive [5] and aerospace components [6], materials for construction and building [7], electronic devices including sensors [8], sports [9], and household and materials handling and storage [9].

Composite fabrication can be divided into two processes: primary processes that produce objects or samples near the desired shape at the required size and second processes that use joining or machining processes to adjust the object or sample to the final required conditions. The machining processes include drilling, milling, or turning activities, and adhesives or mechanical elements are used for joining [1].

Regarding primary processes, independent of the composite, some aspects that can be considered concern the matrix, bast fiber configuration, and fiber–matrix adhesion. In this chapter, general considerations of these key factors and their influence on composite processes are commented on. A large variety of matrices are used to fabricate bast fiber composites. The most common type corresponds to polymer matrices, followed by cementitious matrices. Currently, the use of green matrices is a prime concern. Some of these matrices are biodegradable polymers, such as biodegradable polyesters [10], polymers based on starch [9], and bio-based polymers, such as a bio-based epoxy resin [11]. Additionally, interest in self-reinforced composites is relevant due to the non-inclusion of additional matrices. Some details of these green composites are also mentioned in this chapter.

Alternatively, the techniques mentioned in the chapter can be useful to include nanostructures that improve bast fiber-based composite behavior and the use of non-bast fibers or synthetic fibers to develop hybrid composites. For this reason, in this chapter, the manufacturing technique is discussed as a function of the matrix, and it is divided into the following parts: basic aspects of manufacturing bast fiber composite materials; processing polymer matrices (PMCs), including some comments on the secondary process; and finally general aspects for processing of cementitious composites.

2 Basic Aspects of Manufacturing Bast Fiber Composite Materials

As Fig. 1 shows, aspects linked to the matrix and fiber configurations are essential to fabricate composites. The continuous lines indicate parameters conditioned by the matrix or reinforcement characteristics. The dotted lines indicate interactions between different parameters, as in the case of the influence of the fabrication process on the fiber length of discontinuous composites. According to Fig. 1, the matrix can affect fiber–matrix adhesion and limit the fabrication process. These parameters indicate the necessity to know the chemical and physical changes of the matrix during manufacturing. For example, when the matrix is a thermoset polymer, it is imperative to know how matrix crosslinking occurs. Additionally, when matrix solidification occurs during crystallization, the conditions essential to obtain the required final crystallization grade, crystal size, and distribution need to be known. In the case of thermoplastic matrices, requirements such as the melting temperature or solidification temperature must be considered.

Concerning fiber–matrix adhesion, the most common alternatives are fiber modification before composite fabrication, matrix modification, and the addition of coupling agents during fabrication. In this book, this topic is deeply considered in other chapters. Independent of the selected alternative, there is a nonsignificant impact on composite manufacturing.

Depending on the process, see Fig. 1, the reinforcement content and void content vary. For example, composites manufactured using a thermoset matrix such as an unsaturated polyester resin with discontinuous bast fibers by hand lay-up have a maximal reinforcement content that oscillates between 20 and 30 wt%. However, when preimpregnated reinforcements such as bulk molding compounds (BMCs) are used and transformed using compression molding, the fiber content reaches 90 wt%, and the void content is less than 3%.

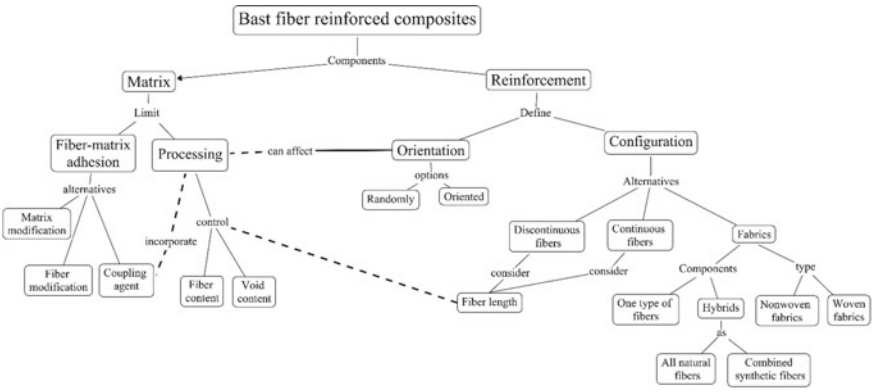

Fig. 1 Basic aspects of manufacturing bast fiber composites

Figure 1 shows that the parameters directly linked to the reinforcements are the orientation and configuration. Some processes can control the orientation. For example, during the extrusion process, the flow of the melted matrix orients the discontinuous reinforcements. When composites are made by stacking reinforcement layers, the orientation can be adjusted as a function of the final properties.

Using different textile techniques, bast fibers can adopt several configurations that include discontinuous fibers, rows, nonwoven fabrics, woven fabrics, or even 3D textiles [12]. Additionally, it is possible to combine bast fibers with other natural and synthetic fibers, such as carbon fibers or glass fibers, to obtain hybrid systems [6]. The isolation of cellulose nanostructures from the bast fiber configuration is another area of interest to produce composites, including nanocomposites.

Liu et al. [13] reported promising results for increasing the mechanical behavior of poly(lactic acid) (PLA) reinforced with nanofibrils isolated from flax yarns.

Figure 1 indicates that when reinforcements are chosen as discontinuous fibers, the fiber length is a principal parameter. The reduction in fiber size during processing is linked to fiber fractures or fibrillation [14].

Figure 2 shows that during the process, chemical and physical variations in the matrix and reinforcement can affect physical or mechanical behavior. For example, Zafeiropoulos et al. [15] analyzed the effect of the varying crystallinity of isotactic polypropylene matrices at the interface with green flax and how modification of the crystallinity of the matrix alters the strength and cracking behavior of this region. In the case of thermoset matrices, Gañán and Mondragon [14] commented on the importance of analyzing the alteration of the curing process of an unsaturated polyester matrix due to the presence of untreated and treated fibers.

On the other hand, bast fibers exhibit the physical changes listed below: (a) size reduction due to a process observed by Bos et al. [16] when analyzing the effect of different processes for mixing short flax fiber with PP; (b) disintegration of the bundle and increased defibrillation; (c) alteration of the fiber lumen that supposedly collapses due to pressure applied during compression molding: as observed by Pupure et al. [17], this change on fiber morphology contributes to reducing the effect of

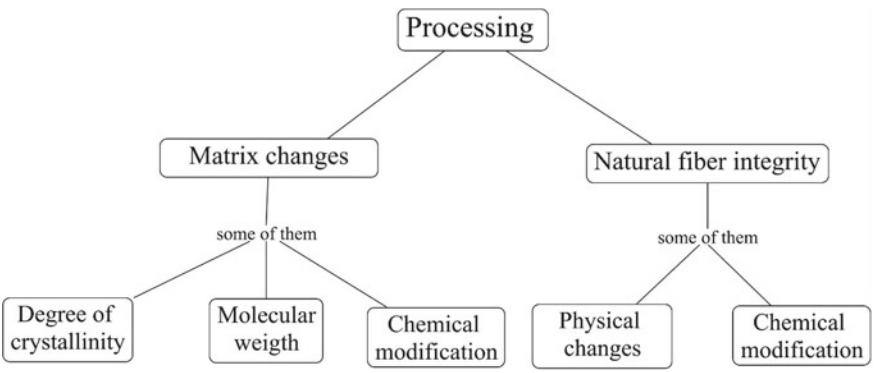

Fig. 2 Alterations introduced by processing

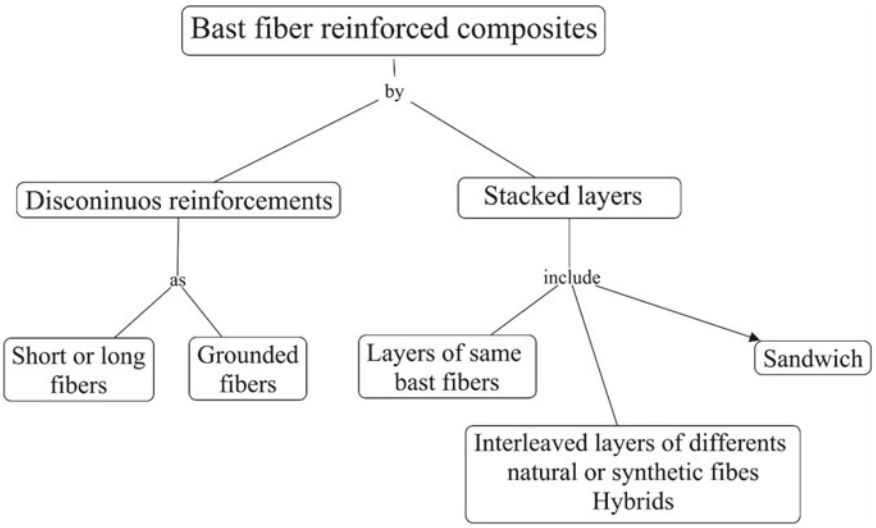

Fig. 3 Bast fiber-reinforced composites as a function of the reinforcement configuration

the lumen on the final porosity of composites; (d) potential alteration of cellulose fiber crystallization; (e) chemical modifications that include variations in the surface composition or alterations of the polar fiber surface; and (f) reduction of noncellulosic components.

Composites or natural composites can be classified as a function of the reinforcement used configuration or matrix nature. As a function of the reinforcement configuration, as shown in Fig. 3, it is possible to use discontinuous reinforcements such as short, long, and grounded fibers or stacked layers. Bast fiber-reinforced composites can also be fabricated using combinations with other natural or synthetic fibers, forming hybrid composites. Additional alternatives include the production of sandwich materials.

One of the most common classifications of composites corresponds to the matrix. Accordingly, composites are polymer/plastic matrix composites called reinforced plastics or reinforced polymers (PMCs), ceramic matrix composites or reinforced ceramics (CMCs), metal matrix composites (MMCs) or reinforced metals, carbon/carbon composites, and cementitious composites. In the case of bast fiber composites, this classification can be expanded, as shown in Fig. 4, where the inclusion of binder-less or self-reinforced composites is included. In these composites, noncellulosic components or cellulose can act as a matrix, excluding the addition of an external matrix. The advantages of these composites are their biodegradability, making them a type of green composite. Samples of fabrication alternatives are discussed above.

In some opportunities after fabrication, the piece requires a secondary process to adjust the object to the final shape or surface specifications [1]. These secondary processes can produce defects or alterations in composite materials. The most

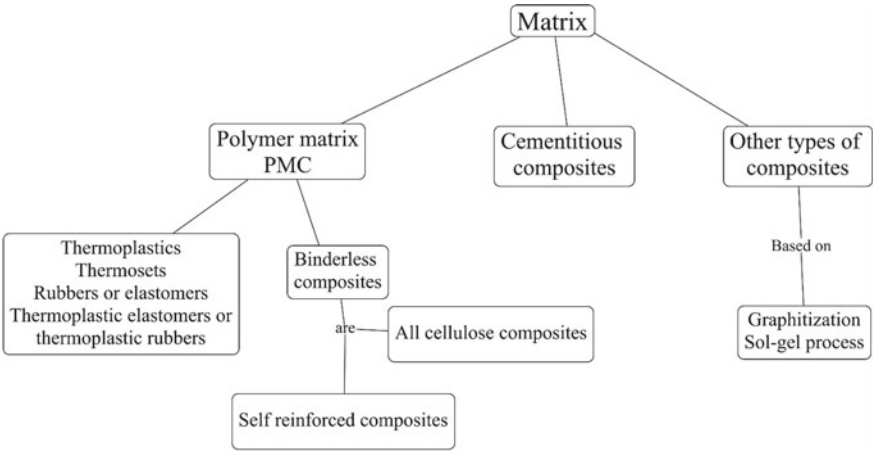

Fig. 4 Bast fiber composites as a function of the matrix

common alterations are linked to heating generated during machining actions. This extra heat can alter the matrix, which is linked to partial melting or even the generation of burning zones. Additionally, this extra heat affects the reinforcements. On some occasions, fibers peel out or delaminate. To control these problems, it is important to reduce the heat generated using the machining process while avoiding the use of water as a refrigerant due to the tendency of the bast fibers to capture water or moisture [1].

3 Polymer/Plastic Matrix Composites or Reinforced Plastics (PMCs)

Bast fibers are used as reinforcements in many polymers to develop composites. As an example of this tendency, Fig. 5 shows the published papers reported in the *Scopus* database for some common polymers used as bast fiber matrices. The common thermoplastics are polypropylene (PP), different types of polyethylene (PE), and polyamides (PA); while the common biodegradable polymers are polycaprolactone (PCL) or different types of polylactic acid (PLA). Figure 5 also shows information on the publications of two of the most common thermoset matrices, unsaturated polyester (UP) and epoxy resins. In the last case, epoxy resin is the matrix with more published papers in 2020, and some of them include bio-based epoxy resin [11].

As mentioned before (Fig. 1), the selected matrix affects or defines the manufacturing process. For example, when using a crosslinking polymer such as an epoxy resin, the curing cycle and postforming process (postcuring) control the mechanical and physical properties of the matrix and its composites. The definition of thermal cycles is vital because, as Campana et al. [18] observed, the conditions

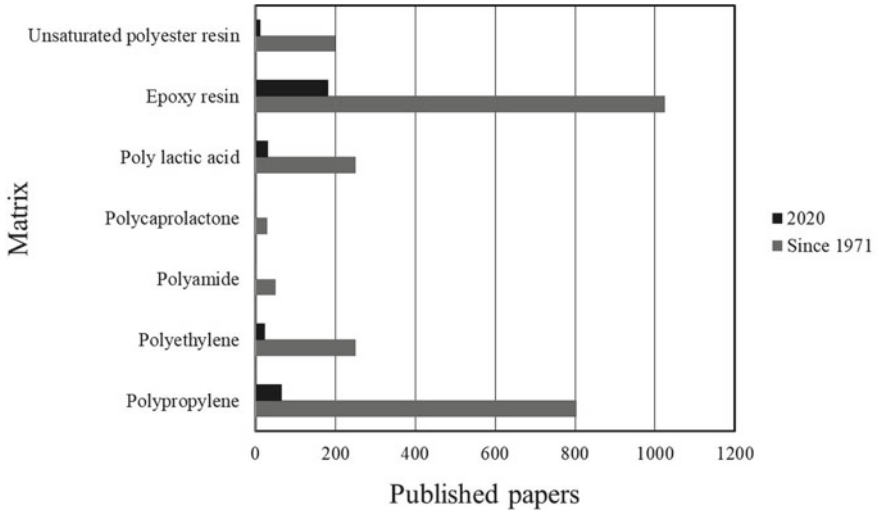

Fig. 5 Published papers about natural polymer composites in the *Scopus* database

of the postcuring temperature cycle reduce the ultimate stress and strain of the unidirectional flax-reinforced epoxy composites, whereas the modulus increases. For a rubber matrix, the vulcanization process affects the final behavior of the material; for instance, Wang et al. [19] observed that the elongation of composites based on styrene–butadiene rubber reinforced with hemp fibers was controlled by modifications during matrix crosslinking.

Regarding composite manufacturing, the general aspects to consider are as follows:

- State of the polymer matrix at room temperature: When the matrix has a low viscosity, as in the case of an unsaturated polyester, casting is an option. In contrast, if the matrix is solid (e.g., pellets), such as PP or PCL, a melting process is required to produce composites.
- Temperature requirements: During the process, polymers undergo physical or chemical modifications. When the matrix must be melted and after the required cooling process for solidification, properties such as the melting temperature; cooling temperature, including the crystallization temperature; and degradation temperature are important. Additionally, rheological conditions in the molten state must be considered. For a matrix that undergoes chemical modifications, such as a thermoset polymer, the required parameters include the curing and postcuring cycles. In the case of rubbers, the vulcanization conditions are also key factors to control during processing.
- Reinforcement content: As shown in Fig. 1 and commented above, depending on the process, the final weight percent can vary.
- Void content: The mechanical or physical properties are limited by the void content in the final composites. Additionally, regarding the fiber lumens, the voids can

link to the air entrapped during processing and the formed gases during fabrication. These gases can be associated with the moisture content of the bast fibers or formed gases due to the thermal decomposition of noncellulosic components that have a lower thermal resistance than cellulose. Thus, it is important to verify the thermal resistance of the polymer or other used additives to avoid thermal depolymerization or decomposition due to the heat required or produced by friction actions during processing.

- Processability conditions: Depending on the final objective of the composite, aspects that can be considered for selecting a manufacturing process are as follows: easiness to produce samples or pieces, manufacturing cost, accessible technology, reproducibility of samples or pieces, sample size, shape complexity, production rate, energy consumption, and waste generation.
- Monitoring of the manufacturing process: As mentioned in the other part of this chapter, matrices may undergo physical and chemical changes. Some of them can be monitored, for instance, the evolution of crosslinking in thermoset matrices or the vulcanization of rubbers using strategies that monitor increasing hardness.
- Alterations of reinforcements: Reinforcements can be affected by process changes, leading to alterations in the fiber length, size distribution, lumen dimensions, aspect ratio, and chemical structures. These alterations must be monitored to establish the potential effect on composite behavior.
- Secondary process: Again, as mentioned before, depending on the final form or shape of the composite sample, machining or joining may be required. These operations can affect the integrity of the composite due to the potential heat required or produced that can introduce new defects and affect the final behavior of the material.

Before manufacturing, the bast fiber reinforcements (untreated or treated fibers) must be dried or conditioned at low specific relative humidity (RH) to reduce the void content (porosity) of the composite due to the evaporation of moisture during processing. The effect introduced by moisture evaporation can introduce alterations to the fiber–matrix interface and potential alterations to matrix consolidation or delamination. Moudood et al. [20] reported that when flax fabrics were conditioned at 70% RH and 95% RH, the mechanical behavior of epoxy composites fabricated by vacuum infusion was lower than that of composites fabricated at a lower RH. This reduction was due to the increase in porosity and alterations in the fiber structure and fiber–matrix interface.

In some cases, it is necessary to initially dry the matrix, as is the case for thermoplastic matrices with relatively high moisture content, such as PCL or PLA. The matrix can be dried following matrix production using a vacuum oven [10].

Based on the composite process, early mixing steps among the matrix, additives, and reinforcements can be required. However, when nanostructures are added to improve the specific properties of bast fiber composites, it is necessary to consider their inclusion by using a mixing process.

Regarding early mixing processes, the plastic industry uses several alternatives. All of them can be useful for composite fabrication even with the inclusion of nanostructures. Some of these processes are listed below:

- Mix dried fibers and matrix pellets. All the materials are solids, and the mixing process occurs using mechanical mixers, including rotary drum mixers, static tubular mixers, conical screw blenders, planetary mixers, and equipment that combine mixing and milling actions, such as ball milling or planetary ball milling machines.
- Mix dried fibers with a dissolved matrix. In these situations, it is possible to use mechanical mixers and ultrasonic or high-speed dispersers. For example, Correia et al. [21] used an Ultraturra disperser to mix natural rubber latex with an aqueous suspension of treated jute fibers, a process that provides a homogeneous distribution of fibers into the latex.

  On some occasions, during the mixing process, it is possible to include degasification steps to reduce the content of trapped air [22].
- Mix dried fibers with a melted matrix. This process is frequently performed using a plastic extruder with one screw or a twin screw. The twin screw is more useful to obtain a better distribution of reinforcements into the matrix. Traditional machines used in the rubber industry can also be useful, as in the case of the two-roll mixing mill or the Brabender batch mixer.
- In situ polymerization onto the reinforcement surface. Some polymer reactions can be catalyzed by the OH groups of bast fibers. Using this idea, it is possible to graft polymer chains onto the fiber surface. Ring open polymerization of PCL has been investigated as an alternative for fiber modification [23]. Notably, this alternative can be useful for fabricating cellulose nanocomposites based on polymers with fabrication limitations, such as polyaniline (PANI). Nepomuceno et al. [8] reported the successful formation of nanocellulose/PANI nanostructured nanocomposites in which the cellulose works as a good backbone for the conductive polymer; their study shows the possibility of producing a new type of sensor.

3.1 Processing Techniques

As mentioned above, there are many processing alternatives to produce bast fiber composites [2, 3]. Tables 1 and 2 summarize key factors and examples of the usual manufacturing techniques used to produce bast fiber composites.

Some of the techniques in Tables 1 and 2 require a mold to final consolidation of the composites. The mold material is chosen considering its chemical and thermal resistance to support the processing steps, including the heating conditions or use of solvents. Before processing, all molds must be dried and cleaned. Eventually, the demolding product can be used to facilitate the removal of composites from the mold. The demolded product cannot be altered or suffer changes under the thermal

Table 1 Basic aspects of bast fiber composite techniques using a low-viscosity matrix

Processing technique	Matrix state before composite fabrication	Bast fiber configurations	Mainly aspects	Type of matrix	Processing parameter control	Examples
Cast molding	Liquid/dissolved polymer	Discontinuous fibers or grounded fibers	Reinforcements and low-viscosity matrix mix before casting into a mold	Low-viscosity resins at room temperature	Gel point and demolding time	Bamboo fibers/castor oil-based polyurethane resin [24]
				Dissolved thermoplastics	Solvent evaporation and demolding time	Kenaf fibers/aqueous PVA solutions [25]
Hand lay-up molding	Liquid/dissolved polymer	Layers or sheets of discontinuous or continuous fibers	Each layer or sheet is impregnated by low-viscosity matrix	Low-viscosity thermosets at room temperature	Gel point and demolding time	Kenaf fiber/epoxy resin [26]
Spray up molding	Liquid/dissolved polymer	Short randomly fibers	Simultaneously projection of low-viscosity matrix and short fibers	Low-viscosity thermosets at room temperature	Gel point and demolding time	Jute fiber/unsaturated polyester [27]
		Short randomly fibers and layers or sheets of discontinuous or continuous fibers	Ejected matrix with or without short fibers impregnate layers or sheets of reinforcement resting on a mold			
Vacuum bag molding	Liquid/dissolved polymer	Layers or sheets of the discontinuous or continuous fibers	Impregnated layers are covered by vacuum films before applying vacuum	Low-viscosity thermosets at room temperature	Gel point that conditioned time to applied vacuum. Other aspects: demolding time	Carbon/flax hybrid composites [28]

(continued)

Table 1 (continued)

Processing technique	Matrix state before composite fabrication	Bast fiber configurations	Mainly aspects	Type of matrix	Processing parameter control	Examples
Vacuum infusion	Liquid/dissolved polymer	Layers or sheets of the discontinuous or continuous fibers	Injection of a low-viscosity matrix in a mold after vacuum. Inside the mold is located dry reinforcement	Low-viscosity thermosets at room temperature	Gel point that conditioned time to applied vacuum and demolding time	Flax fiber reinforced vinyl ester, flax fiber-hybridized basalt reinforced vinyl ester [29]
Resin transfer molding (RTM)	Liquid/dissolved polymer	Layers or sheets of the discontinuous or continuous fibers	Injection of resin in a closed mold after vacuum application. Inside the mold is located dry reinforcement	Low-viscosity thermosets at room temperature	Gel point that conditioned time to applied vacuum and demolding time	Hemp fabrics/bio-based epoxy [30] or kenaf fiber/epoxy composites [31]
Filament winding	Liquid/dissolved polymer	Continuous reinforcement	Continuous reinforcement is impregned with matrix before wound onto a mandrel	Low-viscosity thermosets at room temperature	Gel point and demolding time	Flax fiber yarn/epoxy resins [32]
Pultrusion	Liquid/dissolved polymer	Continuous reinforcement	Impregnated continuous reinforcements are introduced into a heated mold, after passing through the die	Low-viscosity thermosets at room temperature	Gel point, curing temperature, speed of pulling	Flax fiber/epoxy resins [33]

(continued)

Table 1 (continued)

Processing technique	Matrix state before composite fabrication	Bast fiber configurations	Mainly aspects	Type of matrix	Processing parameter control	Examples
	Pellet or powder matrix. The matrix must be melted or dissolved previously to reinforcement impregnation	Continuous reinforcement	Impregnated continuous reinforcements are introduced into a heated mold, after passing through the die, composite is cooled	Melted or dissolved matrix with low-viscosity as possible	Temperatures of die, matrix viscosity, pull forces	Flax fiber/different thermoplastic matrices [34]
Compression molding	Liquid/dissolved polymer	All type of the reinforcements	Previously impregnated reinforcement located in the mold after closing the plates	Dissolved matrix with low-viscosity as possible	Temperature cycle adapted in function of the polymer. Pressure cycle	Jute fiber/unsaturated polyester [35]
	Pellets, powders, or sheets	All type of the reinforcements	Stacked reinforcement layer between matrix layer	Thermoplastics matrices	Temperature cycle. Pressure cycle	Layers of kenaf fabric in polypropylene matrix and aluminum [36]
	Bulk molding compound (BMC)	Discontinuous fibers or grounded fibers	Prepreg that contains all components	Bulk is placed on two parts mold and cured using temperature. Thermoset matrices	Curing cycle, compression cycle	Kenaf fibers/unsaturated polyester resin [37]

(continued)

Table 1 (continued)

Processing technique	Matrix state before composite fabrication	Bast fiber configurations	Mainly aspects	Type of matrix	Processing parameter control	Examples
	Sheet molding compound (SMC)	Discontinuous fibers or grounded fibers and continuous reinforcement	Prepreg that contains all components	Stiff sheet is placed on two parts mold and cured using temperature. Thermoset matrices	Curing cycle, compression cycle	Hemp/unsaturated polyester resin [38]

Table 2 Basic aspects of bast fiber composite techniques using a thermoplastic matrix

Processing technique	Matrix state during conformation	Bast fiber configurations	Mainly aspects	Type of matrix	Processing parameter control	Examples
Extrusion molding	Pellet melted during process	Discontinuous fibers or grounded fibers	Matrix melted during the process; a homogenous blend emerges from die	Thermoplastic	Barrel temperature, speed of screw, pressure in die	Jute fibers/PP [39]
Injection molding	Pellet melted during process	Discontinuous fibers or grounded fibers	Melted composite is injected into a close mold	Thermoplastic	Barrel temperature, speed of screw, injection pressure, mold temperature	Jute fibers/PP [39]
Rotational molding	Polymer powder melted during process	Discontinuous fibers or grounded fibers	Rotate mold is heating and them cooling to solidification of material	Thermoplastic, specific for polyethylene's	Melting temperature, cooling temperature, mold rotational speed, rotational axis	Torres and Aguirre [40]

conditions of fabrication because this undesirable situation introduces defects, such as increases in the void content.

Cast molding is one of the simplest and easier techniques to manufacture fiber composites due to its low requirements. As indicated in Table 1, a combination of reinforcements and a low-viscosity matrix is cast into a mold. After the complete consolidation of the composite, it is possible to remove the material from the mold. Typically, molds have one part (open mold), but it is possible to use closed molds. Polymers with low viscosities at room temperature are usually selected as matrices for example unsaturated polyesters; other polymers may include polyurethane, in which polymerization occurs at the same time as composite consolidation. This is the case of castor oil-based polyurethane resin composites reinforced with bamboo, as developed by Dias Machado Lopes et al. [24]. It is also possible to use dissolved thermoplastics. In these cases, a heated composite blend is cast into a mold, and the consolidation of the material is linked to solvent evaporation. This is similar to the flax fiber reinforcement polyvinyl alcohol (PVA) produced by Ali et al. [25]. This technique can also be useful for foam production or the addition of nanostructures to bast fiber composites.

A usual drawback of cast molding is the low velocity of production, especially when composite consolidation occurs at room temperature. Increasing composite consolidation by heating molds can be an option because altering the crosslinking of thermoset matrices during the curing process or increasing solvent evaporation modifies matrix solidification. Objects produced by cast molding have a higher void content than other manufacturing techniques, such as closed molding. A reason is that air is trapped during mixing and casting into a mold. Alternative solutions include (a) the use of degassing strategies, such as the addition of degassing chemical agents to composite blends before casting, (b) the use of degassing or vibrational systems to remove or induce the liberation of trapped air in the material before casting, and (c) the use of vacuum equipment, such as a vacuum oven during the casting process.

When continuous reinforcement is used or the final composite corresponds to a laminate, the easier and cheaper technique to produce composites corresponds to the hand lay-up process. In this process, the arrangement sequence of reinforcement layers or sheets is organized on the surface of a clean and polished mold until the required composite thickness is reached. Each layer or sheet is impregnated by a low-viscosity matrix that contains all additional additives required for the final composite. Regarding the impregnation of layers, a common, inexpensive brush is used. To reduce the content of trapped air bubbles, after the impregnation of a layer, it is gently pressed to the rest of the laminate using a simple tool such as a metal roller. However, one of the main drawbacks of this technique is the void content of the final composites. To reduce the void content, alternatives similar to those for cast molding can be used.

Despite the low production rate of this technique, it is useful for all types of bast fiber combinations even those using synthetic reinforcement [6], thereby leading to the creation of innumerable hybrid composites [6]. Additionally, it is possible to use layers of discontinuous fibers, instead of only continuous reinforcement, to modify the disposition of a layer, thereby producing anisotropic laminates.

Hand lay-up is essentially defined as an open mold technique; for improving the surface quality of composites, a closed mold can be used. Additional options are available after finishing the arrangement of impregnated layers. For instance, it is possible to put the composite in a compression machine, as in the case of Devadas et al. [26], or into an oven to achieve better consolidation of the polymeric matrix, thereby improving the surface quality and reducing the void content.

The spray-up technique is the second most common open-mold process for composite manufacturing. In this case, a low-viscosity polymer at room temperature is projected onto a mold surface. Usually, the matrix is projected along with short fibers that are cut by the same projection machine. Its main advantage with respect to hand lay-up is the decrease in impregnation time and the possibility of creating homogeneous layer of short randomly dispersed fibers. In addition, the resin was projected onto a dry reinforcement located on the mold. This technique can be combined with hand lay-up. The skill of the person who operates the spray-up machine was analyzed by the works of Kikuchi et al. [27] during the fabrication of a panel based on jute fiber reinforced-unsaturated polyester.

As mentioned in Table 1, bast fiber composites can be manufactured by techniques that include the use of vacuum, as in the case of vacuum bag molding, vacuum infusion, and resin transfer molding (RTM). These techniques are considered an improvement over open-mold techniques such as hand lay-up. In the case of vacuum techniques, impregnated layers of reinforcement located on a mold are covered by vacuum flexible films before applying vacuum. The vacuum films are pressed to a sealing tape located outside the mold area before vacuum steps. This step occurs before the gel time of the matrix. The main advantage of the technique is the decrease in void content. However, the vacuum flexible films conformed by perforated release films developed most of the time in polypropylene, a bleed fabric that absorbs excessive matrix, a vacuum bag film commonly developed in polypropylene, and the sealing tacky tape are useful just once because they are impregnated by the matrix; thus, they are discarded and thrown away after consolidation of the composite. Decreasing the content of residues is a challenge of this technique.

One of the key aspects to evaluate for the above technique includes the effect of fiber–matrix adhesion and fiber orientation on the resin flow to obtain a homogeneous distribution of the matrix, thereby avoiding defects such as resin-rich areas or resin-poor areas. Using this technique, it is possible to achieve a fiber volume fraction between 30 and 50%. Combining this technique and the hand lay-up process makes it possible to create a large number of laminate composites, even hybrid composites, for example, the carbon and flax-reinforced composites manufactured by Fiore et al. [28].

As commented in Table 1, a difference that occurs in vacuum bag molding is that during vacuum infusion, dried reinforcements are placed inside the mold, and the low-viscosity matrix is injected into the mold after vacuum. This technique is extensively used for thermoset matrices, and again, the gel time is the key aspect to control the process. Using this method, Almansour et al. [29] obtained composites reinforced with flax fibers and hybrid systems with flax and basalt fibers with void contents of less than 3% and volume fractions between 30 and 40%.

Regarding resin transformer molding (RTM), on some occasions, the low-viscosity matrix is degassed using a vacuum system prior to injection in a closed mold, where the dried reinforcements are laid down. Most of the time, all processes occur at room temperature, and after matrix injection, the mold can be placed under vacuum. Heating the mold can be performed after matrix injection. For instance, the bio-based epoxy reinforced with hemp fabrics fabricated by Di Landro and Janszen [30], utilized a curing process at 120 °C for 2 h followed by a postcuring step at 180 °C for 2 h.

According to Table 1, in the RTM technique, it is also relevant to control the gel time of the matrix that conditioned the matrix injection time and degassed steps, and the reinforcement impregnation that, additionally, is limited by the fiber–matrix adhesion.

The RTM process has advantages over the open-mold process, such as a lower void content due to the vacuum steps during matrix injection, a higher production rate, and a volume fraction that is usually approximately 40 wt%. The mold cost can be higher than other processes but can be used for hundreds of pieces.

RTM techniques have variations, such as vacuum-assisted resin transfer molding (VARTM), especially for pieces with strict control of the reinforcement volume fraction. This is essentially similar to traditional RTM with stricter control over the vacuum process.

Techniques specialized to produce hollow profiles using low-viscosity matrices are filament winding and pultrusion. In both techniques, the reinforcements are impregnated by a matrix before being put in a die. Impregnated reinforcements require alignment before molding. In the case of filament winding, the impregnated continuous reinforcements are wound onto a mandrel, whereas in pultrusion, they are pulled to enter a die by a force applied outside the mold. In filament winding, the curing process basically occurs at room temperature, whereas in the pultrusion process, the molds must be heated because a cured composite must emerge from them. The pultrusion is often considered a continuous process.

Regarding both techniques, it is possible to use thermoplastic matrices, especially in the case of pultrusion. These matrices must be melted or dissolved to achieve impregnation of the reinforcements as the case of the Van de Velde and Kiekens work [34].

In the case of composite steps requiring a postcuring process, an oven system can be used.

The compression process can be carried out under hot or cold molding conditions, making it a versatile technique because it is possible to develop composites in which matrix solidification occurs under the temperature conditions of the compressing machines. Some alternatives are commented on in Table 1.

Regarding closed molding, the compression process has advantages over open-mold techniques, such as a higher production rate, higher quality of the surface finish, lower void content, and higher reinforcement content (between 35 and 90 wt%). Aspects to consider are (a) the matrix viscosity, (b) the impregnation process, and (c) avoiding fiber cracking or size alterations due to the compression cycle, warpage, and residual stress. As a function of the reinforcement configuration, the stacking of layers defines the final anisotropy of the composite.

By compression molding, it is possible to transform prepregs or compounds. Typical prepregs are bulk molding compounds (BMCs) as the work of Sreenivasan et al. [37] and sheet molding compounds (SMCs), for example, technique used by Patel et al. [38]; some variations include thick molding compounds (TMCs) or dough molding compounds (DMCs). Regarding prepregs or compound development, different reinforcement configurations are placed over a slurry matrix and passed through rollers to make them thinner and more stable. Then, they are wrapped and stored under appropriate conditions before being unpacked for shape and compression molding.

When prepregs are made using thermoset resin, it is necessary for them to be stored under refrigeration before compression molding. This step helps prevent potential precuring that can affect the properties of the final composites [14]. After the storage time is finished, the prepregs are cut to the required size and put into a mold. After closing the plates, pressure and temperature cycles are applied [3].

Compression molding can produce hybrid composites that include fiber metal laminates (FMLs) formed by layers of bast fabric composites and metal sheets that act as the skin [36]. These hybrids are lightweight with good mechanical properties. Essentially, the most common metal that is used is aluminum. Before consolidation of the FML, the metal sheets are roughened to improve adhesion to the bast sheet composites. The final consolidation of the FML takes place using compression machines. A complete study of this process is reported by Ishak et al. [36].

Conventional techniques for thermoplastic polymers used for manufacturing composites are listed in Table 2. The most common process corresponds to injection molding. This technique is useful to achieve complex shapes, even when the sample has inserts or cores. This technique offers high repeatability, a high productivity rate, a high surface finish, reduced warpage and shrinkage, and low material loss. On the other hand, the extrusion process is used to mix discontinuous reinforcements with the matrix to obtain a blend or compound that can be processed using other techniques, including injection molding or compression molding. Additionally, extrusion is used to produce profiles, rods, plates, films, or sheets with a specific cross section. When continuous reinforcements are selected, pultrusion, extrusion, and compression molding can be used. Regarding all processing, the wettability of the reinforcement is the key aspect to consider [3].

In both the extrusion and injection techniques, the material is heated, compounded, matrix melted, and transported toward the mold using a screw. During the process, the fiber size can be reduced or altered due to a variety of friction sources, including fiber–fiber, fiber–metal, fiber–matrix, and other friction forces when passing through the die orifice or entrance into an injection mold. In these techniques, fiber agglomeration is a factor to control. The reinforcement fraction is approximately between 30 and 50 wt%.

In the extrusion process, granulate polymers, discontinuous reinforcements, and other additives, including coupling agents, improve fiber–matrix adhesion or nanostructures at the solid-state entrance by a hopper into a heated barrel. After mixing actions, a homogeneous compound emerges outside the extruder by a die. Conventional extruders have one screw, but a better mixing process is achieved when the extruder uses a twin screw.

Some authors, for example, Oksman et al. [41], produce homogeneous compounds, such as their flax/PLA composites, by the extrusion process, while the last forms are achieved by compression molding.

Producing composite profiles with specific cross sections requires defining a specific extruder die and components in the postextrusion line.

Injection molding has a high production rate of complex and net-shaped pieces. In this process, a solid compound is introduced into an injection machine, at which point it is melted and pushed into a cold mold. During the solidification of the material inside the cold mold, it is necessary to maintain pressure. This technique is one of the usual options for making automobile pieces with thermoplastic bast-reinforced composites [5].

Large hollow objects are produced by rotational molding. This technique is based on loading powder polymer and its additive into a mold. After closing the mold, it

rotates at low speeds while being heated for polymer melting, followed by a cooling process. The process takes between 0.5 and 1 h. This technique is less commonly used than injection but was used by Torres and Aguirre [40] to manufacture jute/HDPE composites with good fiber dispersion.

Additive manufacturing techniques such as 3D printing are useful to rapidly produce geometrically complex forms of prototypes or pieces with high precision. In these processes, the piece is formed through layer-by-layer deposition, and several thermoplastic polymers can be used. Currently, it is one of the most promising alternatives to produce bast thermoplastic composites with a low production rate or even to produce unique forms with an insignificant generation of waste [42].

As commented above, for specific purposes, composite materials require a secondary process. They are useful to achieve the final appearance of the piece. Some of them include machining activities such as milling, turning, grinding, drilling, and joining using adhesive or mechanical systems. When selecting these activities, it is necessary to consider the potential defects that are introduced due to new thermal, mechanical, or chemical actions that are performed. Typical defects that are introduced into composites are fiber pull-out, peel-up, delamination, debonding, reinforcement cracking, and residual stress.

3.2 Self-Reinforced Composites

In these cases, composites are manufactured without an additional matrix because they are formed by bast fiber components. The matrix can be generated using two strategies: (1) by the chemical modification of non-cellulose, which is usually a strategy used to produce binder-less boards [43] and (2) by altering amorphous cellulose, such as in the case of all-cellulose composites [44]. The hot compression technique is one of the most used techniques to achieve the final consolidation of these green composites. These materials are biodegradable, are useful because they use a higher number of raw materials, and have limited delamination problems.

Regarding binder-less boards, the bast fibers are previously treated by enzymes such as laccase [45] or by a chemical process that uses steam treatment as a steam explosion [43, 45]. During these treatments, the formation of reactive sites and the generation of degradation molecules from hemicellulose or lignin molecules can occur, such as in the case of furfural [45]. Both aspects are vital for the potential chemical reactions that occur during hot processing steps, which determine the final behavior of the composite [45].

On the other hand, one of the pioneers of the development of all-cellulose composites is Nishino and Arimoto [46]. In their processes, natural fibers are soaked using a solvent to promote the partial dissolution of amorphous cellulose; after this step, the materials can be molded by casting [44], compression molding, or even using an electrospinning technique to form fibers that are close to the nanoscale range [47].

Solvents such as DMAc/LiCl, NMMO, NaOH/urea, and ionic liquid are used in the above process. DMAc/LiCl is widely used because it can be used at room

temperature, thereby reducing energy consumption [44]. Additionally, some authors report that using this process leads to higher mechanical properties compared with the use of other systems, such as a NaOH/urea system [47].

4 Cementitious Composites

Bast fibers have been extensively used to produce cementitious composites because they improve mechanical properties such as flexural toughness and ductility while also contributing to controlling crack formation [48].

The main concerns to manufacturing these composites are the level of porosity in the matrix, which affects the mechanical properties of the final materials; the optimal volume content of reinforcements, the homogeneous reinforcement dispersion inside the matrix, the reinforcement orientation, and the fiber–matrix interaction through the interface to avoid fiber pull-out [7].

As commented by Ferrara et al. [7], several fabrication methods for cementitious composites reinforced with natural fibers are based on the inclusion of pulp patented by L. Hatschek in 1900. The semi-continuous process of the Hatschek method has three steps: sheet formation, next to the board conformation, and finally matrix setting and curing [7]. Inspired by this method, several modifications have been considered [7]. Currently, cementitious composite fabrication includes the following steps: preparation of blends by mixing natural fibers with water or the addition of substances, such as plasticizers [49]. Then, cement is incorporated along with other ingredients required by the formulation, such as pozzolanic material or sand. The mixing process is performed until obtaining a homogenous, consistent, or well-dispersed mortar. In several works, additional components are added to the blends to modify or improve the final behavior of the composites.

During the mixing process, aspects such as the mixing speed, time, and ingredient addition sequence are important to control. Additionally, when short fibers are used, it is important to monitor the potential alteration of the size ratio, size distribution, and inclusion of air, which can be controlled or reduced by the use of vibrational actions. When the mixing process is finished, the mortar is cast in a mold, and the curing process occurs.

To reduce the void content after the mortar is cast in the mold, these alternatives can be considered: the application of vibrational actions or vacuum [7].

Cementitious composite laminates can be produced by casting the mortar in a mold and then placing a layer of continuous reinforcement. The sequence of the layer of continuous laminates and mortar is determined depending on the composite design [7].

When short fibers are used, one of the parameters to consider during blend conformation is altering the rheology. Some authors indicate that alterations are related to the water capture of natural fibers and the specific surface area [7].

The degradation of natural fibers due to long-term exposure to an alkaline cement matrix during use is one of the concerns that needs research for this type of composite.

As indicated by author Page et al. [48], natural fiber degradation is associated with a partial dissolution of noncellulosic and even cellulosic parts due to the presence of alkaline media for alkaline hydrolysis; furthermore, the possible precipitation of carbonates on the surface or in pores affects the mechanical behavior of fibers. Some alternatives to control these problems include fiber treatment using organic coatings [48], reducing the pH of the mortar by matrix modification [48], or the inclusion of nanostructures as cellulose nanofibers, which also contribute to improving some of the mechanical properties of the composites [49].

5 Conclusion

Bast fiber composites are progressively being considered for a large number of industrial applications. Part of this tendency is related to the number of techniques available for composite manufacturing. They can be manufactured with simple and inexpensive techniques, such as casting, which is recommended for low-viscosity matrices; or more expensive techniques, such as compression and injection molding.

This chapter summarizes a compendium of the usual techniques when polymer or cementitious matrices are used, emphasizing the main aspects related to reinforcements and matrix characteristics and how they can be altered during processing.

This general information can be useful to address the inclusion of nanostructures and the fabrication of hybrids, which are promising scenarios for the increasing use of bast fibers.

Acknowledgements The authors acknowledge the Universidad Pontificia Bolivariana and NANOCELIA network of the Iberoamerican Program of Science and Technology for Development (CYTED), for their support.

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Thermal Stability of Polymer Composites Reinforced with Bast Fibers

Pragya Bharti, Sanya Verma, Manoj Kumar, and G. L. Devnani

Abstract Bast fiber application as a reinforcing material in natural fiber composites is increasing day by day, which is currently a trending topic in the composites industry. Bast fibers are derived from the fibrovascular group located between the epidermis or bark surface and the inner woody core of dicotyledonous plants, known as bast (or phloem). It has a variety of advantages, including low cost, low density, excellent durability, acceptable explicit strength qualities, improved energy recovery, carbon dioxide sequestration, and biodegradability. However, in this study, the main focus is on the concerns surrounding bast fibers such as their low thermal stability. The major issue reviewed in this paper is the thermal stability of bast fibers reinforced composites. Compilation of recent literature is done along with state of art thermal characterization techniques. Improvement of thermal stability of bast fibers and their reinforced composites are also compiled.

Keywords Composites · Thermal · Biodegradability · Energy recovery

1 Introduction

Due to the production of extensive amounts of waste plastics and wood or other waste materials, various ecological issues and concerns got together with the issues like quickly topping off landfills have prompted the advancement of new cycles and materials to deal with these arising issues. For this purpose, increasingly more exploration is being completed to utilize squandered materials through reducing or potentially reusing. Reducing the use of synthetic fibers and shifting towards

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natural fibers is the ultimate solution to achieve this. Substantial work and progress have been achieved in the exploration, extraction, fabrication and characterization of traditional and newer natural fibers and their reinforced polymer composites [4, 5, 7, 9–11, 15, 28, 30]. Various surface treatment and filler addition methodologies have also been suggested to improve the adhesion between fiber and matrix in order to have composites of improved quality [6, 8]. Bast fiber is a special category of natural fibers in which fiber is extracted from the stem of the plant. Flax, hemp, ramie, kenaf and jute are the main bast fibers (Fig. 1) produced all over the world.

Fibrous plants can be classed based on their function. According to another classification scheme, the majority of technically essential bast fibers come not just from agricultural plants like flax, hemp, or ramie, but also from wild plants like nettle. Other countries, like Brazil and Chile, cultivate small amounts of the crop. Hemp is mostly grown for fiber in Europe, Canada, and China. Cannabis sativa indica plants are cultivated for medical medications, and a small amount of low-quality hemp fibers are harvested from them. Kenaf is grown for its fiber in India, South Africa, Vietnam, Thailand, and other regions of Africa, as well as to a lesser extent in Southeast Europe. India is the world's largest producer of jute. Bangladesh, China, Nepal, Myanmar, and Thailand are among the countries that grow jute. Local jute is mostly purchased by India, Pakistan, and China, while raw jute is imported from Bangladesh by the United Kingdom, Spain, Ivory Coast, Germany, and Brazil. China

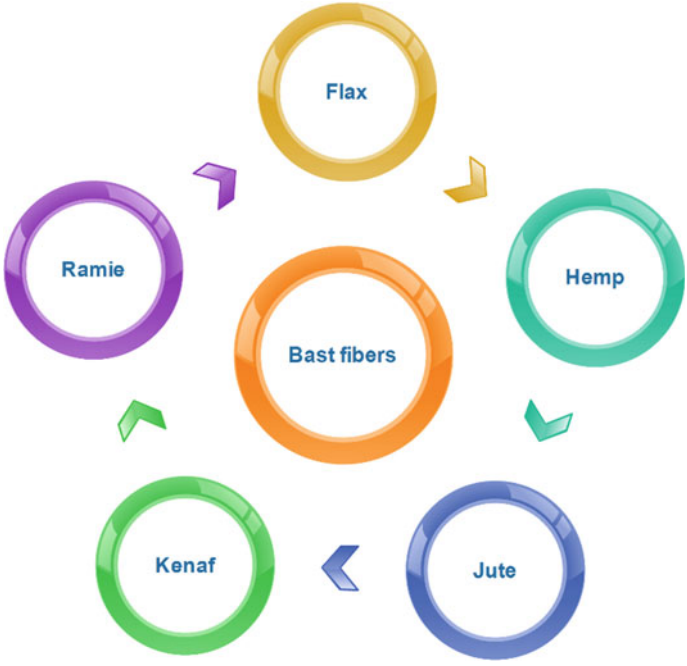

Fig. 1 Types of bast fibers

is the world's leading producer of ramie, which it mostly exports to Japan and Europe. The Chinese cultivated plant is the real ramie or China Grass, often known as the Chinese plant or white ramie. Japan, Taiwan, the Philippines, and Brazil are among the other producers. On the foreign market, just a small percentage of the ramie produced is available. Green ramie, also known as rhea, is a second form of ramie that is thought to have originated in the Malay Peninsula. This variety has smaller, green-on-the-bottom leaves and looks to be better adapted to tropical environments [34]. Urtica dioica is a European native that can also be found in Asia, northern Africa, and North America. Isora can be found all throughout India, from the Jamuna eastwards to Bihar and Bengal, and southwards in central, western, and southern India, as well as the Andaman Islands. Being lignocellulosic these bast fibers have hydrophilic in nature and thermally they are less stable. Physical, chemical and biological treatments and filler addition is a method by which we can enhance the properties of these fibers and ultimately their reinforced composites. A lot of work has been done on the thermal characterization of these fibers and their reinforced composites. Neto et al. [24] did a comprehensive review on the thermal characterization of natural fiber based composites. He explained the importance of thermal stability of these materials because processing temperature is an important parameter in the fabrication of composites and properties of lignocellulosic fiber change at higher temperatures. So thermal characterization is important to understand the suitability of these bast fiber composites for different applications. Asim et al. [2] compiled the literature on thermal behavior of plant fiber based composites, hybrid composites along with nano composites.

2 Methodology of Thermal Analysis

Thermal analysis is an important area of materials science engineering where the properties of samples are examined as their variation with the temperature. Lot many methodologies are normally used, different from each another by the parameter which is analyzed. Different methods for thermal analysis of bast fiber is based on a different principle and fundamental. In some analysis weight loss is measured with temperature while in some methods heat flow is the measuring parameter. Dimension is also measured for understanding the effect of temperature. Diagrammatic representation is shown in Fig. 2. Characterization techniques based on these different measuring parameters are shown in Fig. 3.

2.1 Thermogravimetric Analysis

Thermogravimetric analysis (TGA) is a characterization technique of thermal examination in which the weight of the specimen is analyzed with varying time and temperature. This analysis gives information about physical processes, such as phase change,

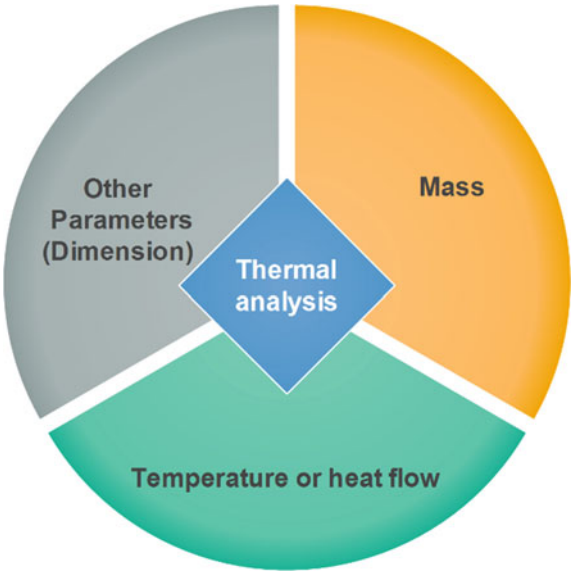

Fig. 2 Mechanism of thermal analysis

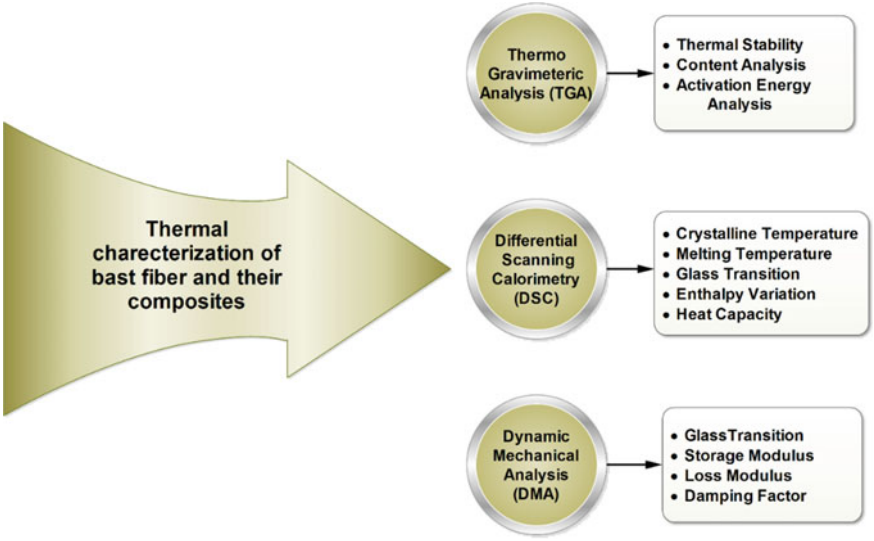

Fig. 3 Different characterization techniques for evaluating thermal stability of bast fiber composites

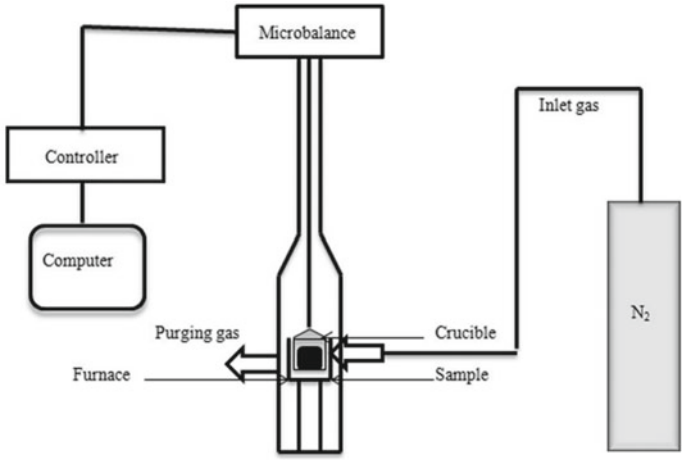

Fig. 4 Representation of TGA (Thermogravimetric analysis) [16]

adsorption, absorption, etc. as well as chemical processes like thermal decomposition and reactions of solid gas. Thermogravimetric analyzer is used as an instrument for performing the thermogravimetric analysis. This instrument continuously measures the weight of the sample with changing temperature or time. Weight of the sample, temperature range, and time are taken as measuring parameters in thermogravimetric analysis while more results and analysis may be extracted from this base analysis. A typical instrument consists of a very high precision mass balance with a sample pan situated inside of the furnace. A programmable computer is there to control the temperature values. Normally the temperature increases at a constant rate or sometimes the constant mass is kept during a thermal reaction. The atmosphere is kept different like ambient, nitrogen, or other gas depending on the need and type of analysis. Pressure can also be kept high low or moderate depending on the process. This data is collected for the whole thermal reaction and the graph is plotted between mass of the sample (%) and temperature known as the TGA curve. The differential of this data (mass) is also plotted with temperature known as DTG (Differential thermogravimetry). Analysis is very useful for understanding the thermal degradation pattern of bast fibers and their composites (Fig. 4).

2.2 Differential Scanning Calorimetry

Differential scanning calorimetry (DSC) is another important thermal analysis technique in which the increase of temperature of sample and reference sample is done and the difference in the amount of heat required is measured and this measurement is done as a function of temperature. Both the specimen and reference material are kept approximately at the same temperature during the whole experiment. It is

programmed in such a manner that the temperature increases linearly and reference material heat capacity should be accordingly over the temperature range. Heat flux DSC and power differential DSC are two main types.

2.3 Dynamic Mechanical Analysis

Dynamic Mechanical Analysis is another major characterization technique which examines the mechanical and viscoelastic properties of bast fiber composites. In this analysis, the material is kept under a periodic stress. Different modes of deformations are applied such as bending, compression, shear and tension. Modulus of the materials as a function of temperature or time is examined.

3 Thermal Characterization and Thermal Stability Analysis of Bast Fiber Composites

Compilation of recent advancements in thermal characterization techniques and thermal stability analysis of important bast fiber composites is discussed as follows.

3.1 Thermal Characterization and Thermal Stability Analysis of Jute Fiber Composites

Epoxy composites reinforced with jute fiber were fabricated by a simple hand layup method [27]. 18.5 volume percentage of jute fiber was used. The thermogravimetric analysis showed that the decomposition temperature for jute fiber reinforced composite is less as compared to neat epoxy resin. Reinforcement of jute fiber caused the shift towards a higher temperature. Chatterjee et al. [3] developed jute fiber polypropylene composites by a compression moulding technique (Fig. 5).

Three different factors fiber length (3, 6, 9 cm), fiber loading (0, 5 and 10%) and fiber ply (1, 2 and 4) were varied. Thermal characterization and Differential scanning calorimetry (DSC) is performed in the temperature zone (20–180 °C) at a heating rate of 10 °C/min. Dynamic mechanical analysis (DMA) is done in 3-point bending mode in the temperature range from 25 to 125 °C at a heating rate of 3 °C/min at a frequency of 1 Hz. Optimum values of the performance parameters like tensile strength storage moduli were obtained for 2-ply composites in all cases of fiber loading. Thermal stability of reinforced composites also improved due to the reinforcement of jute fibers. The findings of DMA analysis suggested that PP-Jute composites have shown high values of storage modulus which further enhance due to increasing the number of plies and fiber percentage at 6-cm length of jute fiber (Fig. 6).

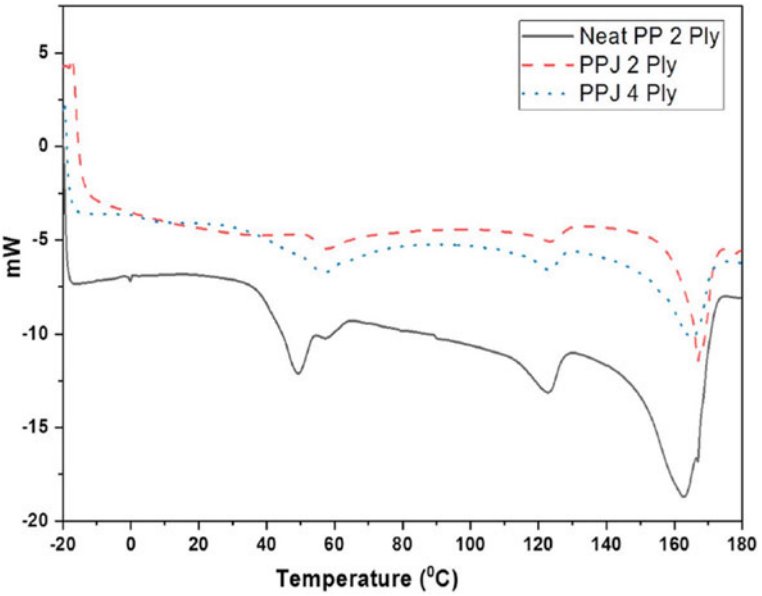

Fig. 5 DSC curves of neat PP and jute PP composites [3]

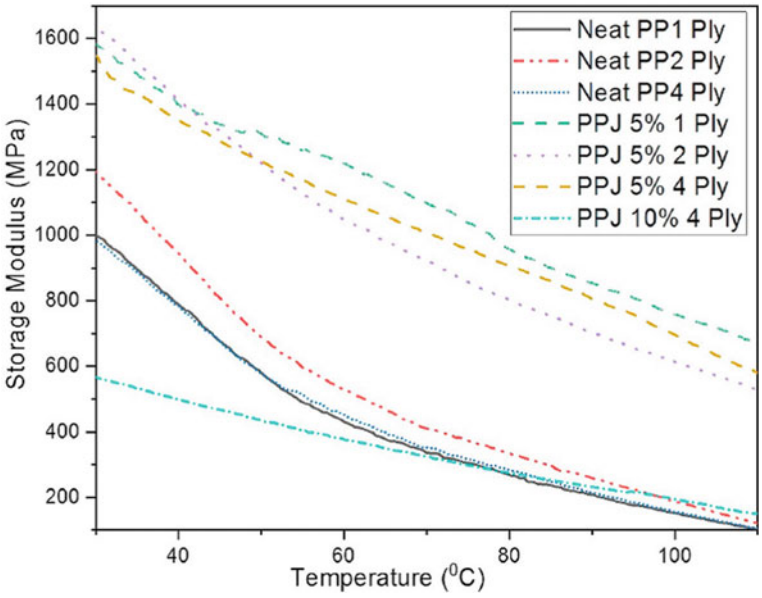

Fig. 6 Storage modulus curve for different combination of jute pp composites [3]

Flammability and thermal characterization of jute fiber reinforced epoxy composites were done after treating fiber with 0.06 M sodium periodate solution [22] and it was observed that composites developed from chemically treated fiber have high thermal stability as compared to the untreated one. Chemically treated jute fiber [29] reinforced polymer composites were fabricated by a compression moulding technique. Jute being lignocellulosic material degraded in the temperature zone of 220–330 °C. and degradation of polyester was observed in the range of 340–450 C. Table 1 is showing the summarized results of the work.

3.2 Thermal Characterization and Thermal Stability Analysis of Flax Fiber Composites

Flax reinforced phenolic composites were prepared by diffusion bonding techniques. Different fiber loading was kept that is 2, 4, 6 and 8 for the fabrication. 6% fiber loading was recommended for the best results. van de Velde and Baetens [32] did thermal and mechanical characterization of Flax Fiber and evaluated their suitability for possible reinforcement in composite manufacture. Shukla et al. [31] adopted hand layup technique for the production of hybrid epoxy composite materials reinforced by bamboo and flax mat. The thermal properties were evaluated by standard characterization techniques DSC, TGA. El-Sabbagh et al. [13] evaluated the possibility of flax reinforcement in engineering plastics such as PA6 and PB6. Injection moulding was used for the fabrication of composites. Molaba et al. [23] fabricated composites of phenolic resins reinforced by flax fabric. 70% fiber loading was kept for the composites and flax fabrics were treated with a 5% alkali solution. Silane and flame retardant treatments were also done on the fabric. It was observed that flame retardancy of the composites improved but at the same time degradation temperature shifted to lower values. Table 2 is giving a brief summary of the key points of thermal analysis of flax fiber reinforced composites.

3.3 Thermal Characterization and Thermal Stability Analysis of Hemp Fiber Composites

Lu and Oza [21] developed hemp fiber reinforced HDPE composites. Their work investigated the effect of Chemical modifications (silane and NaOH treatments) of hemp fiber on the thermal and thermo-mechanical characteristics of their HDPE composites. The observations suggested that the thermal stability of composites reduced as we keep on increasing the fiber and treated composites had improved thermal stability as compared to untreated composite samples. DMA observation revealed an enhancement in the value of storage modulus values of the Alkali and silane treated composites as compared to untreated samples. The improvement in

Table 1 Thermal stability analysis of Jute Fiber reinforced composites

S.No	Matrix	Fiber	Fabrication method/Fiber loading/Filler or treatment applied	Key findings	References
1	Epoxy LY 556 (bisphenol-A-diglycidyl-ether) is	Jute and glass	Hand layup/16.6% Glass and 18.5% Jute/-	In case of neat epoxy material, the first step of weight loss is over by 120 °C due to the removal of moisture. Neat epoxy is stable upto 335 °C. In case of jute reinforced epoxy Thermal stability of the material improved because of addition of jute fiber, which is lignocellulosic in nature	Raghavendra et al. [27]
2	Polypropylene	Jute	Compression moulding (0, 5, 10)/-	Thermal degradation stability of reinforced composites is improved by the reinforcement of jute fibers. Outcome of DMA analysis suggested that polypropylene-jute composites have improved values of storage moduli which improved by increasing the number of plies	Chatterjee et al. [3]

(continued)

Table 1 (continued)

S.No	Matrix	Fiber	Fabrication method/Fiber loading/Filler or treatment applied	Key findings	References
3	Epoxy	Jute	Hand layup/20, 25, 30, 35 percentage/0.06 M sodium periodate solution	TGA results analysis revealed that untreated jute/epoxy composite started lose weight earlier as compared to treated jute fiber reinforced composite	Mathubala and Nandhini [22]
4	Polyester	Jute	Compression moulding/40% alkali treatment	Weight loss was observed from 220 C to 330 mainly because of degradation of lignocellulosic reinforcement	Sajin et al. [29]

Table 2 Thermal stability analysis of flax fiber reinforced composites

S.No	Matrix	Fiber	Fabrication method/Fiber loading/Filler or treatment applied	Key findings	References
1	Phenolic resin	Flax	Diffusion bonding/2, 4, 6, 8%/	DSC curves revealed the endothermic curve which are having variations in shape and area	Ashok Kumar et al. [1]
2	–	Flax fibers	–	Exposure to different temperatures on the flax fibers results in a reduction in mass and alter the mechanical characteristics of the flax fiber. At 60 °C, moisture was removed from the flax fiber. Exposure to 120 C leads to degradation of waxes and removal of water	van de Velde and Baetens [32]

(continued)

Table 2 (continued)

S.No	Matrix	Fiber	Fabrication method/Fiber loading/Filler or treatment applied	Key findings	References
3	Epoxy	Bamboo and flax	Hand layup/28% fiber loading/10% NaOH treatment for 30 min	(a) TGA thermograms showed two-step decomposition process. The first stage, decomposition occurred in the 215.C.-390C temp. range. The second step decomposition in the temperature range of 431C.-860C. Residual amount of char at 860C-6.815% (b) In DSC analysis observed glass transition temperature is 195C. (Tg) value of HYBRID composite increased as compared to neat epoxy	Shukla et al. [31]
4	Flax	Engineering plastics (PA6 and PB6)	Injection moulding/0–50%/Alkali treatment	Potential of producing PBT and PA6 composites reinforced by flax	El-Sabbagh et al. [13]

(continued)

Table 2 (continued)

S.No	Matrix	Fiber	Fabrication method/Fiber loading/Filler or treatment applied	Key findings	References
5	Flax fabric	Phenolic resin	Compression moulding/70% fiber loading/5% alkali solution, silane and flame retardant treatment	Flame retardant treatment of flax fabric was observed as an effective technique for improving flame retardancy of the composites TGA analysis revealed that degradation temperatures shifted to lower values	Molaba et al. [23]

storage moduli was experienced till 40% loading of fiber but at 50%, it came down significantly. Composites in which fibers were treated by silane showed high storage modulus as compared to alkali treated ones as a result of improved fiber matrix adhesion. Table 3 gives the compiled information of work done on thermal characterization and stability analysis of hemp fiber based composites. Activation energy analysis was used to determine the thermal stability of hemp-PLA composites [26]. Different chemical treatments were applied on hemp fibers and it was observed that acetic anhydride treated composites had higher activation energy values as compared to others. Dhakal et al. [12] fabricated polycaprolactone composites reinforced by hemp fibers by press moulding technique and found a thermal degradation temperature of 432 C for the composites. Khoathane et al. [18] analyzed the thermal behavior of I pentene/polypropylene composites reinforced with hemp fibers and observed improved thermal properties as compared to neat matrix. Khoathane et al. [18] produced polyamide composites reinforced by hemp fibers and it was concluded that composite performance is strongly affected by the addition of fibers. Hemp fiber produces a positive effect on thermal and mechanical characteristics of composites.

Table 3 Thermal stability analysis of Hemp Fiber reinforced composites

S.No	Matrix	Fiber	Fabrication method/Fiber loading/Filler or treatment applied	Key findings	References
1	High density Polyethylene (HDPE)	Hemp	Compression moulding/20–50%/Alkali and silane treatment	Increased fiber loading has an adverse effect on thermal stability. DMA observation showed an enhancement storage moduli after Alkali and silane treatment as compared to untreated composites	Lu and Oza [21]
2	PolyLactic acid	Hemp	Compression moulding/20–40%/Acetic anhydride, alkali and silane	Acetic anhydride treated composites had higher activation energy values as compared to others	Oza et al. [26]

(continued)

Table 3 (continued)

S.No	Matrix	Fiber	Fabrication method/Fiber loading/Filler or treatment applied	Key findings	References
3	Polycaprolactone	Hemp	Press moulding/20%	Highest thermal decomposition temperature of 432 °C is obtained	Dhakal et al. [12]
4	1-Pentene/Polypropylene	Hemp	Injection moulding/30% fiber loading	Composites exhibited excellent thermal properties as compared to fiber and matrix individually	Khoathane et al. [18]
5	Polyamide	Hemp	Compression or Injection moulding	It was observed that composite performance is strongly influenced by the addition of fibers. Hemp fiber created a positive effect on thermal and mechanical properties of composites	Nishitani et al. [25]

3.4 Thermal Characterization and Thermal Stability Analysis of Kenaf Fiber Composites

Jaafar et al. [14] fabricated completely biodegradable composites by PLA and hemp fibers. Chemical treatment of fiber was done by three different concentrations of alkali that are 1.5 M, 1 M and 0.5 M. 1 M alkali treatment for the fibers was recommended for improved thermal performance of composites. Lee et al. [20] did their research work on Kenaf fiber reinforced polypropylene composites with the addition of magnesium hydroxide as a flame retardant and found it had a positive effect on thermal properties as well. Khan et al. [17] studied the effect of stacking order of kenaf and jute on epoxy composites. 30% fiber loading and hand layup method was used for the development of composites. Fiber addition induced a positive effect on the thermal stability of composites (Table 4).

Table 4 Thermal stability analysis of Kenaf fiber reinforced composites

S.No	Matrix	Fiber	Fabrication method/Fiber loading/Filler or treatment applied	Key findings	References
1	Polylactic acid	Kenaf	Compression moulding/-/Alkali and acid treatment	1 Molar NaOH treated Kenaf fiber composites had shown better thermal properties as compared to other treated fiber composites	Jaafar et al. [14]
2	Polypropylene	Kenaf	Compression moulding/0–25%/Magnesium hydroxide as flame retardant	In TGA analysis improved thermal stability is observed due to MH addition in spite of the decrease in tensile strength	Lee et al. [20]
3	Epoxy	Kenaf/Jute	Hand layup/30% fiber loading	Stacking order of kenaf and jute improved the properties of neat epoxy because fiber addition improved the Tg values of the composites. Similar improvements were also observed in the case of storage modulus	Khan et al. [17]

Table 5 Thermal stability analysis of Ramie fiber reinforced composites

S.No	Matrix	Fiber	Fabrication method/Fiber loading/Filler or treatment applied	Key findings	References
1	PLA	Ramie	Hot pressing/30%/alkali and silane	Incorporation of ramie fibers had a positive effect on thermal properties of PLA. Similar improvements were observed in storage modulus	Yu et al. [33]
2	Soy Resin	Ramie	Hand layup	Modified soy flour composites had better thermal stability	Kim and Netravali [19]

3.5 Thermal Characterization and Thermal Stability Analysis of Ramie Fiber Composites

Yu et al. [33] used the hot pressing method for making PLA composites reinforced by ramie fiber. Silane and alkali were used for the surface treatment of fibers. The incorporation of fibers had improved the performance of composite materials. Kim and Netravali [19] developed 100% biodegradable green composites by ramie fiber and MSF(modified Soy Flour). Significant improvements in tensile properties were observed so on the thermal characteristics which were evident in TGA and DTG curves (Table 5).

4 Conclusion

Thermal characterization techniques like thermogravimetric analysis (TGA), Differential scanning calorimetry (DSC) and dynamic mechanical analysis (DMA) have been used extensively for thermal characterization and thermal stability analysis of bast fiber composites. Bast fiber was successfully reinforced in the majority of polymer matrices and in most of the cases, improvement in thermal stability was observed due to reinforcement. Composites developed with chemically treated bast fibers had high storage modulus, glass transition temperature and increased decomposition temperature. High values of activation energy of thermal decompositionwere also observed in the case of composites developed by surface modified fibers as a result of improved thermal stability.

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Water Absorption Behavior of Bast Fibers Incorporated Polymer Composites

Aswathy Jayakumar, Sabarish Radoor, Jun Tae Kim, Jong Whan Rhim, Jyotishkumar Parameswaranpillai, and Suchart Siengchin

Abstract The demand for biodegradable materials are increasing due to their environmentally friendly properties and for the replacement of petroleum-based products. Bast fibers are the most important renewable materials for many engineering applications. Bast fiber-based composites are receiving great attention due to their good mechanical properties, biodegradability, and non-toxic nature. These fibers consist of cellulose, hemicellulose, and lignin. The major drawbacks associated with bast fibers are their low thermal stability, low water barrier, and low hygroscopicity. To improve the functional properties of bast fibers, it is generally modified with several physical and chemical agents. The current chapter is designed to give an overview of some of the important bast and leaf fibers, their modifications, and water barrier properties.

Keywords Bast fibers · Composites · Polymer · Water barrier properties · Biodegradable

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1 Introduction

The increasing demand for environment-friendly materials paved the way to explore the application of natural fibers. Nature fibers are renewable, non-toxic, and biodegradable materials having great promises in day to day life [1–3]. Among the natural fibers, bast fibers received great attention due to their good strength, low-density, and low price [4, 5]. The commonly used bast fibers are obtained from hemp, flax, jute, ramie, and kenaf [6–8]. The stem of a plant consists of the outer skin (epidermis), followed by phloem, xylem, and hollow space. Bast fibers are extracted from the phloem of plant stems. The fiber exists in bundles in the phloem. Here, the fibers are connected or bound by a natural adhesive called pectin. Single fiber consists of primary and secondary walls composed of cellulose, hemicellulose, lignin, and pectin [9]. A cross-section of the hemp stem is shown in Fig. 1 [10].

The individual fibers are separated through the retting process [11]. The isolation of fibers through retting includes chemical retting, enzymatic retting, water retting, dew retting, and mechanical retting. The mechanical retting includes decortication and post-decortication cleaning [12]. The retting process successively cleaves the chemical bonding that holds the stem.

Bast fibers typically have high cellulose content (hemp (68), flax (71), jute (61–71)) and low lignin content (hemp (10) and flax (2.2), jute 12–13)) [13]. The high cellulose content in bast fibers makes them more crystalline. The functional properties of bast fibers depend on the growing conditions of plants and the retting process. The higher water absorption of fibers could reduce the mechanical qualities of composites because of interfacial cracking [14]. The presence of hydrophilic groups is the main reason for the absorption of moisture by the natural fibers [15]. Note, the higher the moisture absorption, the higher is the microbial attack and lowers the overall performance of the bast fibers. The moisture absorbance of some of the natural fibers is given in Table 1 [16]. Generally, surface modifications and pretreatment

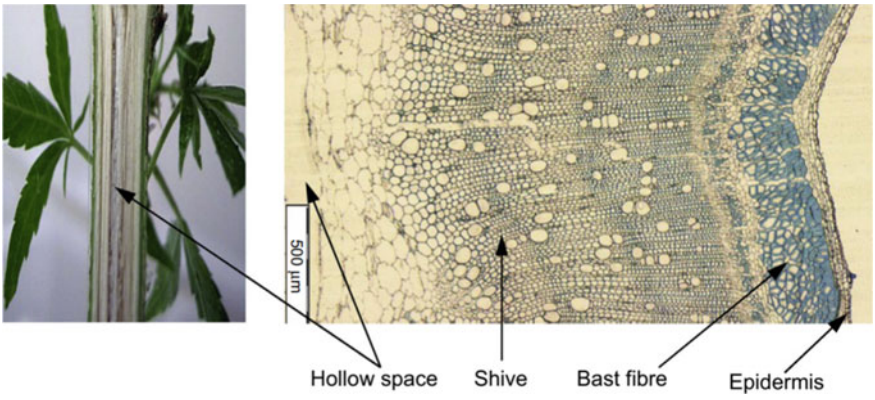

Fig. 1 Cross-section of hemp stem. [Reproduced with permission from Elsevier License Number: 5214570391858] [10]

Table 1 Percentage of moisture content in bast fibers measured at 65% relative humidity and 21 °C. [Reproduced with permission from Elsevier, License Number 5184681348075] [16]

Natural fiber	Moisture content (%)
Hemp	9.0
Jute	12
Flax	7
Ramie	9

are required for enhancing the interfacial interaction between the fiber and polymer matrix. The current chapter will discuss the various modification and the different kinds of bast fiber-based composites.

2 Modifications of Bast Fibers

Generally, bast fibers have low thermal stability, low hygroscopicity, high moisture absorption, and high impurities. Physical, chemical, and biological treatments can overcome these drawbacks associated with bast fibers. The physical methods include plasma and ultrasound. The chemical methods are treatment with alkali, silane, liquid ammonia, esterification, acetylation, isocyanate, and grafting [17]. Biological methods include treatment with nanomodification, bacteria, fungi, and enzymes. The chemical methods consume and waste many solvents and chemicals and need large energy input. The green methods (physical and biological methods) can overcome these issues [18]. Plasma treatment is a dry technique that introduces reactive free radicals and other functional groups on the surface of bast fibers [19]. Bacteria (Clostridia species) treatment is used to modify the structure of natural fibers. The mechanism involves the release of specific enzymes for the degradation of hemicellulose, pectin, and wax. The action of bacteria can improve the crystallinity, mechanical properties, chemical stability, and water holding capacity of bast fibers [18, 20]. The treatment with fungal species, including *Absidia*, *Schizophyllum commune*, *Phanerochaete sordida*, *Ophiostoma floccosum*, and *Pycnoporus* can improve the crystallinity and the interfacial interaction between the bast fibers and the polymer matrix. During the treatment with fungal species, surface holes are introduced in the bast fibers that could provide high roughness [21]. Enzymes such as lipases, laccases, and peroxidases were also used for the enzymatic treatment of bast fibers. The modified bast fibers have improved mechanical, hydrophobicity, and antimicrobial properties [22]. Nano-modification is the modification of natural fibers with nanoparticles such as nanocellulose, inorganic nanoparticles, etc., that could enhance the water barrier, mechanical, antimicrobial, and thermal properties of bast fibers [23].

3 Water Absorption Behavior of Bast Fiber Reinforced Composites

3.1 Bast Fiber-Based Composites

Bast fiber incorporated composites have found applications in various fields such as automotive, aircraft, insulation materials, food packaging, temporary outdoor applications, construction, sports, and soundproofing [17, 24]. The bast fibers tend to absorb moisture from the surrounding atmosphere. The water uptake is due to the existence of CH_2OH groups in bast fibers [17]. The absorbed water in bast fiber-based composites can be bound or free or both. The polar groups found in the fiber tend to bound with the dispersed water, whereas the free water will move through the cracks or interfacial space. The main problem is that when these composites are exposed to moisture, water molecules infiltrate into the composites and remain attached to the hydrophilic groups of natural fibers [25]. This will affect the overall performance of bast fiber-based composites.

3.2 Hemp Fiber-Based Composites

Hemp fibers are one of the widely used strong fibers of the bast fiber family obtained from *Cannabis* species [26]. Hemp fiber's inherent mechanical and thermal properties have significant importance in reinforcing polymer composites. The biodegradable nature and its low-density over artificial fibers make them widely acceptable [27, 28].

The diameter of fibers also has a significant role in imparting the mechanical and water barrier properties of bast fibers incorporated composites. Alkali-modified hemp fibers were used to reinforce polybenzoxazine, and their mechanical and water barrier properties were studied [29]. The different volumes of hemp fiber percentage (10, 15, 20, and 30 vol%) and mesh size [20 (311.5 $\pm$ 42.9 μm), 40 (251.8 $\pm$ 45.3 μm) and 80 (198.3 $\pm$ 40.7 μm)] were used, and a maximum in tensile strength and impact strength were observed for 30% loading (80 mesh). However, the water barrier properties of the composites were reported to increase with increased hemp fiber loading and mesh size. The high surface area and the increased availability of free OH groups have contributed to the uptake of a higher amount of water. Han et al. [30] studied the effect of water-washing and alkali treatment on the ultraviolet aging properties of hemp fiber incorporated polypropylene (PP) composites. The water absorption property of the composites at different UV aging times was studied. It was observed that the water absorption capacity of PP composites was decreased for the treated composites and the least water absorption was observed for alkali-treated composites. Wu et al. [31] studied the water barrier properties of hemp fiber reinforced composite protected polyethylene (PE-NFRC) films. The incorporation of polyethylene (PE) has led to the enhancement in the water contact angle of the composite (Fig. 2). Due to this, the surface hydrophobicity of the composites has been reported to get enhanced. In addition, the water barrier properties of hemp fiber

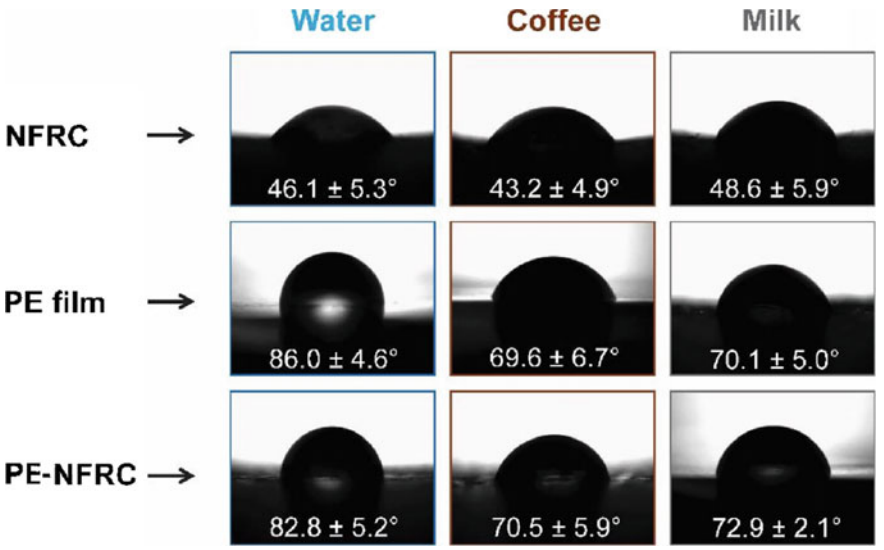

Fig. 2 Contact angle measurement of NFRC (natural fiber reinforced composite), PE (polyethylene), PE-NFRC (polyethylene protected natural fiber reinforced composite) using water, coffee and water droplets [Reproduced with permission from Elsevier License Number: 5265790875991] [31]

reinforced composite protected PE films were enhanced than its pure hemp fiber reinforced composite (Fig. 3).

Dixit et al. [32] utilized alkali-treated hemp fiber to develop PE/PP-based packaging film. Due to the hydrophilic nature of native hemp fiber, the water vapor transmission rate and water vapor permeability of PE/PP-untreated composite is higher than PE/PP-modified hemp composites, real polythene packaging, and PE/PP packaging films. Both WVTR and WVP values of PE/PP-untreated composite were considerably reduced for PE/PP—modified hemp composites from 120.2 g m^{-2} day^{-1} to 51 g m^{-2} day^{-1} and from 6.34 × 10^{-11} g m^{-1} Pa^{-1} s^{-1} and 2.69 × 10^{-11} g m^{-1} Pa^{-1} s^{-1}, respectively. The water contact angle of the alkali-treated fiber-based films was reported to be 119°, whereas 121° for polymeric films, 120° for real packaging, and 91° for native fiber-based packaging. The researchers recommend alkali-treated hemp fiber-based films for green applications.

3.3 Kenaf Fiber-Based Composites

Kenaf fiber is known for its high strength and is one of the widely used natural fibers for the reinforcement of polymer composites obtained from the herbaceous annual plant [33, 34]. Moustafa et al. [35] treated the kenaf fiber with the resorcinol-hexamethylenetetramine mixture (R-HMT) for enhancing the interfacial adhesion

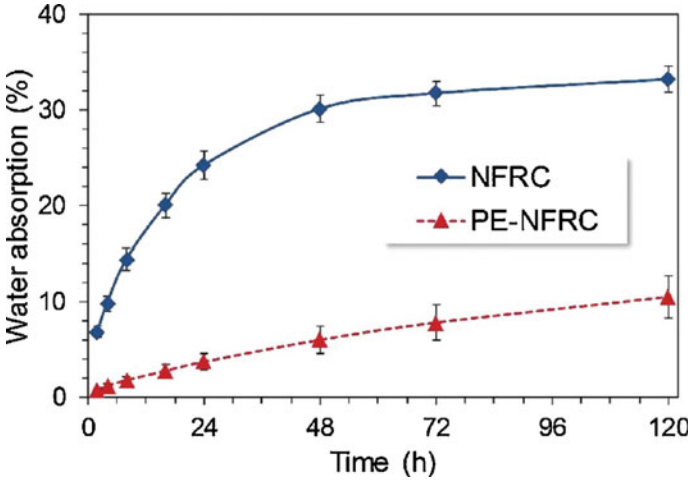

Fig. 3 Water absorption behavior of NFRC (natural fiber reinforced composite) and PE-NFRC (polyethylene protected natural fiber reinforced composite) [Reproduced with permission from Elsevier License Number: 5265790875991] [31]

between the kenaf fiber and the polystyrene (PS) matrix. Their studies suggested that the hydrophobicity of the composites depends on the reinforcement efficiency, polarity, and compatibility between fiber and polymer. The treatment resulted in the loss of OH groups in the fiber. Therefore, the treated fiber reinforced composites showed higher hydrophobicity and barrier resistance due to the increased compatibility and interfacial interaction between the fiber and polymer.

Ramesh and his coworkers developed polylactic acid (PLA) based composites based on kenaf fibers and montmorillonite clay filler [36]. The researchers utilized 30% of NaOH treated kenaf fiber with different concentrations of montmorillonite (0, 1, 2, and 3) for the composite preparation. The water absorption studies were carried out for the composites by immersing the samples in water at room temperature. The results showed that the developed composites have higher water absorption than the neat PLA, and the percentage of the water absorption of the composites increased with submersion time. The addition of treated kenaf fiber has resulted in maximum water absorption. But incorporating montmorillonite reduced the water barrier properties of PLA/kenaf composites. This is because the montmorillonite clay restricted the water movement, thereby improving the water barrier properties of PLA/kenaf-based composites. Later, Ramesh et al. [37] utilized kenaf fiber and aloe vera fiber treated with 6% sodium hydroxide solution to develop bionanocomposites based on PLA incorporated montmorillonite. The hybrid composites with 15 wt% treated kenaf fiber, 15 wt% treated aloe vera fiber, and 3% MMT showed the best water resistance. The enhanced water barrier properties were due to the addition of montmorillonite clay.

Oyekanmi and his coworkers [38] utilized cellulose nanofibrils obtained from kenaf fibers and macroalgae from seaweed to develop biofilms. (Fig. 4). The biofilms

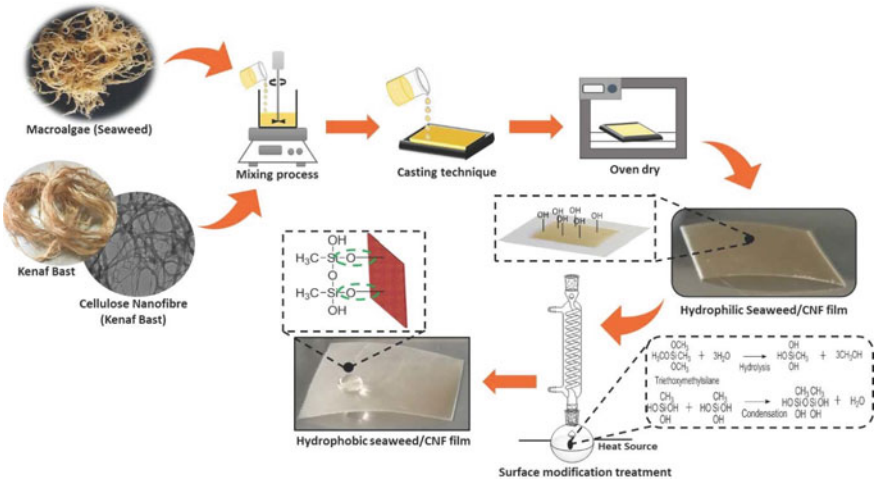

Fig. 4 Representation of macroalgae-based films by incorporating nanofibrils obtained from kenaf fibers [38]

treated with silane reported improved water barrier properties. The strong intermolecular interaction between the microalgal hydroxyl groups with the silane could be the reason for the improved water barrier properties. This interaction resulted in the reduction of free hydroxyl groups.

3.4 Flax Fiber-Based Composites

Flax fibers have high cellulose content and are known for their high strength, an excellent substitute for synthetic fiber for composite applications. It is extracted from the bast or skin of the flax plant *Linum usitatissimum* L [39–41]. Fathi and his coworkers [42] developed bioepoxy composites using modified flax fibers. The flax fibers were modified using 2,2,6,6-tetramethylpiperidine-1-oxy radical (TEMPO)-mediated oxidation followed by amino silane. The silane-modified composites have shown better water barrier properties than the non-treated ones. The researchers concluded that the reduction in water absorption properties could be due to (i) a decrease in free hydroxyl groups in the silane-modified flax composites, (ii) strong interaction between silane-treated flax fibers, and epoxy resins that might cause the reduction in interfacial gaps. Similarly, Alix and coworkers [14] studied the effect of chemical treatment on water barrier properties of flax fiber-based polyester composites. The composites with fibers treated with silane and styrene have shown better moisture resistance than the non-treated fiber composites. Dhakal and his coworkers utilized the compression molding method to develop flax and carbon-flax

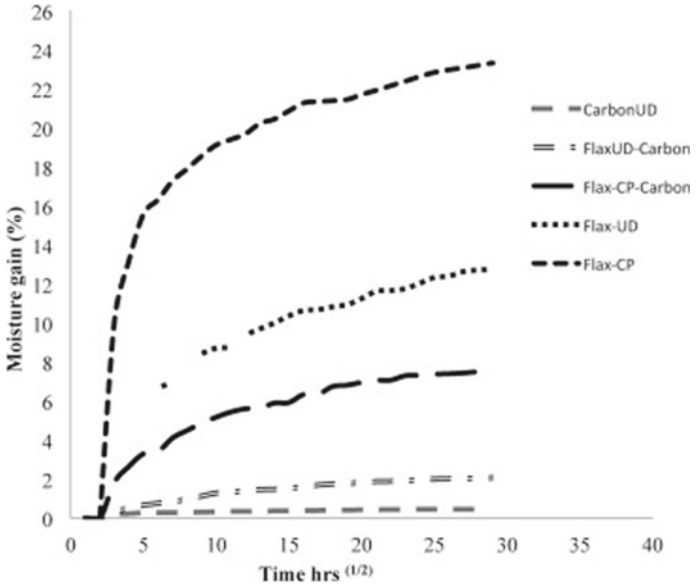

Fig. 5 Water absorption properties of flax and flax/carbon hybridized materials [Reproduced with permission from Elsevier License Number: 5265830646456] [43]

hybridized epoxy composites [43]. The researchers observed a reduction in water absorption by incorporating carbon fiber in flax epoxy composites (Fig. 5).

3.5 Sisal Fiber-Based Composites

Sisal fibers are considered as one of the important fibers in reinforcing polymer composites. It is generally obtained from the leaves of the sisal plant (*Agave sisalana*) [44, 45]. They are ecofriendly in nature and have good strength, Young's modulus, cheaper, and easily available. Generally, these fibers are surface modified to enhance the interfacial interaction between the sisal fiber (hydrophilic) and polymer matrix (hydrophobic) [45]. The major surface modification methods of sisal fibers include silane treatment and alkali treatment. Mohan et al. [46] incorporated different weight percentages of nanoclay (1, 2, and 3 wt%) into the epoxy polymer, which is reinforced with sisal fibers. Their studies revealed that the increased nanoclay concentration has resulted in a decrease in the water absorption properties and water transmission rate of composites (Figs. 6 and 7). The loading of 5 wt% nanoclay has shown better properties than the 1 and 3 wt% nanoclay concentrations. The decrease in water uptake of nanoclay filled polymer composite might be due to the formation of the tortuous pathway by the nanoclay. This could result in the narrowing of pores and restrict the movement of water molecules.

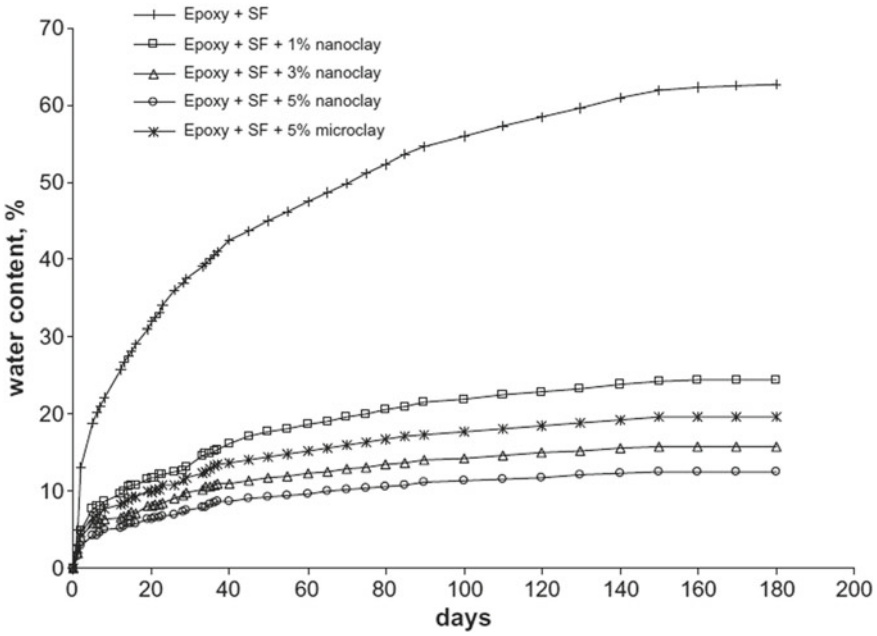

Fig. 6 Effect of different concentrations of nano clay on water uptake properties of sisal fiber reinforced epoxy-based composites [Reproduced with permission from Elsevier License Number: 5265840550820] [46]

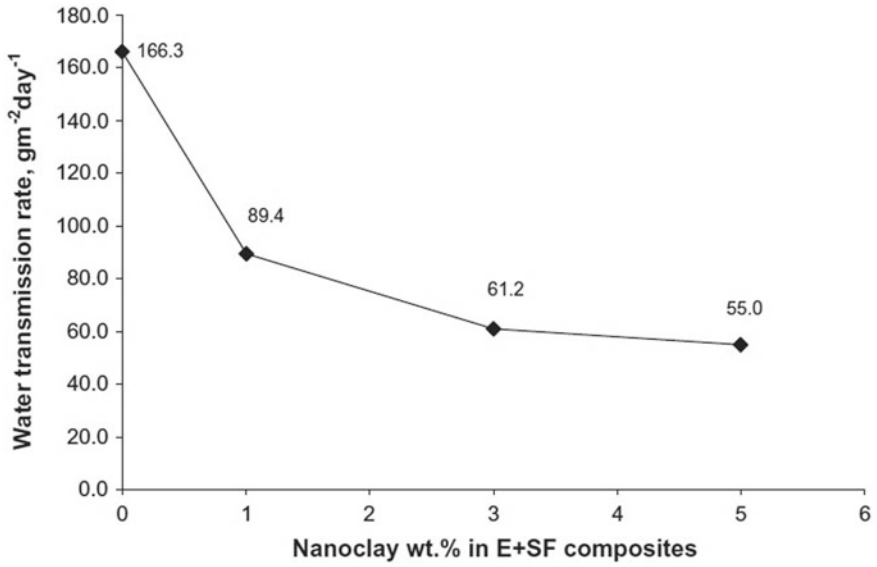

Fig. 7 Effect of different concentrations of nano clay on the transmission rate of sisalfiber reinforced epoxy-based composites [Reproduced with permission from Elsevier License Number: 5265840550820] [46]

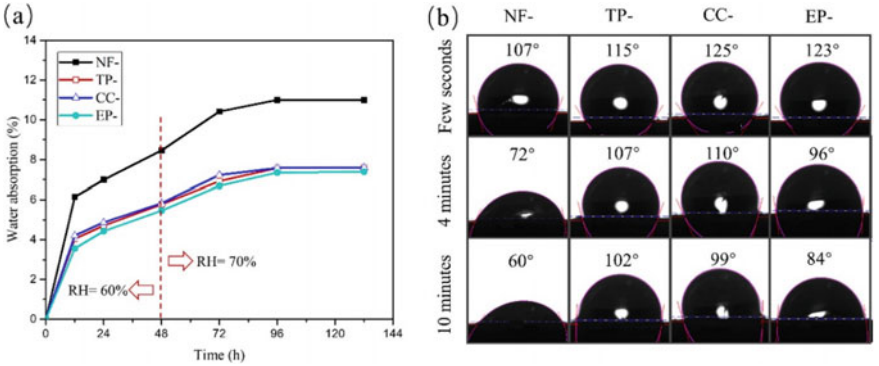

Fig. 8 **a** Water resistance of starch-sisal composites incorporated with TP, CC, eggshell powder (EP) and without filler (NP) after 132 h. **b** water contact angles of TP, CC, eggshell powder (EP), and without filler (NP) at initial, 4, and 10 min

Similarly, Ji and coworkers [47] used different organic [eggshell powder (EP)] and inorganic fillers [talcum powder (TP) and calcium tri carbonate (CC)] to reinforce starch-sisal fiber-based composites. Their studies have shown that for the developed composites' relative percentage water absorption capacity reaches 11.0, 7.6, 7.6 and 7.4 for non-filler (NF), TP, CC, and EP, respectively. The non-filler composite has a large pore size compared to the other filler incorporated composites. Hence, the large pore size may contribute to the greater water absorption in non-filler composites. The relative increase in water resistance properties by the EP and TP composites could be due to the strong interaction of hydroxyl groups of eggshell powder and talcum powder with the starch matrix. The water contact angle of TP, CC, EP, and NF were 102, 99, 89, and 60°, respectively (Fig. 8).

3.6 Pineapple Fiber-Based Composites

Pineapple fibers are easily available natural fibers with a good impact on polymer-based composites as reinforcing agents. It is composed of 70–82% cellulose, 5–12% lignin, and 1.1% ash [48]. It has a glossy silk nature with a smooth texture and white color. The higher cellulose in pineapple fiber makes them hydrophilic. The base fibers are extracted by the mechanical or retting process [49]. Rahman and his coworkers [50] studied the effect of gamma radiation on the water absorption properties of low-density polyethylene (LDPE) incorporated with pineapple leaf fiber (PALP). They developed the composites by loading 10 to 60% pineapple leaf fiber. The radiated (7.5 kGy) composites have less water absorption than the non-radiated PALP/LDPE (50/50) composites. Interestingly, the water absorption increases when using 10.00 kGy radiation. The researchers concluded that the increase in gamma radiation (10.00 kGy) could decrease the cross-linking efficiency, increasing the

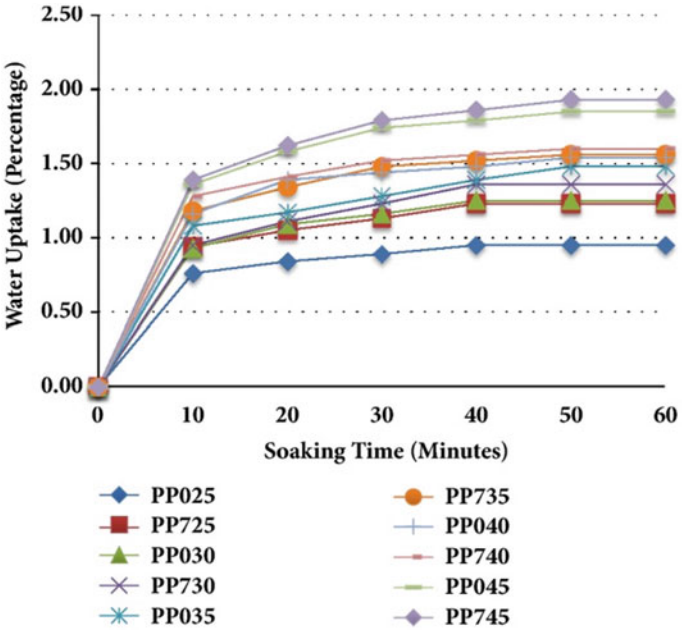

Fig. 9 Water absorption properties of pineapple leaf fiber (PALF) reinforced PP composites [48]

amorphous region in composites; therefore, water absorption increases. In the post irradiated composite (7.5 kGy), the gamma radiation reduced the hydroxyl groups and further increased the crystalline regions of PALP and LDPE. The improved interfacial interaction between the PALP and LDPE might be the reason behind the enhancement in the water barrier properties of the composites. Motaleb and his coworkers utilized the compression molding technique to develop pineapple leaf fiber reinforced PP composites [48]. They analyzed the water barrier properties of composites by incorporating different weight (25, 30, 35, 40, and 45) percentages of pineapple fiber. The percentage of water absorption capacity of the films was increased with respect to the soaking time. The maximum absorption of water was found at 50 min of soaking time (Fig. 9). The alkali-treated fibers have shown increased water absorption than non-treated. The researchers concluded that the alkali treatment resulted in the removal of non-cellulosic impurities that could increase the micro-gaps.

4 Effect of Various Treatments on the Water Absorption Behavior

The major drawbacks associated with natural fiber composites can be overcome by various chemical treatments. Usually, treatment with alkali, silane, benzoyl groups, acetyl groups, permanganate, peroxides, and isocyanate are used. The most efficient treatment for improving the hydrophobic nature and water barrier properties is alkaline treatment. Alkali-sensitive hydroxyl groups are eliminated during the reaction with alkali and thereby contributing to the improvement in properties of fibers. The first step in the treatment of silane, with fiber, involves hydrolysis which will result in the formation of silanol. The formed silanol has the ability to provide a continuous molecular network by reacting with hydroxyl groups of cellulose and the functional group of polymer matrices. Peroxide treatment can induce the production of free radicals that can react with the hydroxyl group of polymer matrix and the fiber which favors the enhancement of water barrier properties. Treatment like benzoylation can reduce the hydrophilic nature of fibers as it replaces the hydroxyl groups within the fibers. Isocyanate is commonly used as a coupling agent during the modification of fibers. During the treatment, strong covalent interaction is evolved between the functional groups of isocyanate and hydroxyl groups of cellulose and lignin. Stearic acid treatments enhance the water resistance properties by reacting with the hydroxyl groups of fiber through carboxyl groups [51].

The effect of treatments on water barrier properties of bast fiber-based composites is summarized in Table 2.

5 Conclusions

Bast fibers received great attention as reinforcing agents in polymer-based composites. Due to its biodegradability, lighter weight, strength, and non-toxic nature, it found applications in automotive, packaging, aerospace, and other industrial sectors. The major drawbacks associated with bast fibers could be eliminated by several physical and chemical methods. Several studies have shown that the reinforcement of bast fibers in polymer-based composites resulted in improved mechanical and thermal properties of composites. The modification of bast fibers has a significant impact on improving the water barrier properties of bast fiber-based composites as it reduces the free hydroxyl groups. Hence, the bast fiber-based composites with better properties can be used as an alternative for petroleum-based products.

Table 2 Bast fiber-based composites and their water barrier properties with respect to various treatments

Bast fiber-based composites	Modifications	Water barrier properties	Reference
Hemp fiber/PP composites	Water and alkali treatment	Water absorption decreased	[30]
Hemp fiber-based composite	Protected PE films	Reduced water absorption	[31]
PE/PP /native hemp fiber	Alkali treatment	Higher water vapor transmission rate and water vapor permeability	[32]
Kenaf fiber/PS	Resorcinol-hexamethylenetetramine	Showed variation in hydrophobicity	[35]
PLA/kenaf fiber/montmorillonite clay	NaOH	Enhanced water barrier properties	[36]
Kenaf fiber/aloe vera fiber/PLA/montmorillonite clay	NaOH	Improved water barrier properties for nanocomposites	[37]
cellulose nanofibrils obtained from kenaf fibers/macroalgae-based films	Silane coupling agent	Reduced pore size and restricted the movement of water molecules	[38]
Flax/bioepoxy-based composites	2,2,6,6-Tetramethylpiperidine-1-oxy radical (TEMPO)	Modified composites showed improved water barrier	[42]

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Rheological Properties of Bast Fibre Composites

Ishwi Varshney and G. L. Devnani

Abstract Bast fibre composites offer tremendous properties to material science such as lightweight, sustainability and cost effectiveness. Ramie, Jute, Hemp, and Flax are the major bast fibres which are extracted from the stem of the plant. Rheology is the science which deals with the flow behaviour of the material and plays a major role for the fabrication of bast fibre composite materials. It deals with changing viscosity and non-Newtonian behaviour of the molten polymer sample. Complex viscosity, storage modulus, and loss modulus are the key parameters to be studied in this field. Proper understanding and analysis of rheological properties are very important to develop improved quality bast fibre composites having diverse applications.

Keywords Complex viscosity · Storage modulus · Loss modulus · Rheology

1 Introduction

How deformation of material (mainly liquid and liquid like) takes place under the action of a force is known as rheology. The area of discussion is basically the rheological behaviour of polymer composites reinforced by bast fibres. Due to their physical behaviour, the visco-elastic nature of these composites is a very important domain to be studied. The study of the rheology of bast fibre based composites facilitates the quality aspects of constituent materials, fabrication processes/finished product and analyzing composite performance. We can say that rheologyanalysis is important in order to have better knowledge of the role that fibres/fillers on the rheology of reinforced composites. Before going to a detailed discussion on the rheological behaviour of bast fibre composites we should understand the natural fibres, bast fibres, their types, properties, reinforced composites and various rheological parameters to be examined. Present study is classified into three parts, first section deals with the introduction of natural fibres with emphasis on bast fibres, second section deals

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with different rheometers and rheological properties which are important to analyse in order to have improved quality composites and the last section summarizes the research work done by various academicians and researchers on this important field.

2 Importance of Natural Fibres with Emphasis on Bast Fibres, Their Types and Properties

Due to a growing understanding of the interconnection of global environmental issues, the importance of the concept of sustainability, ecology of industries, eco-efficiency, and green engineering as well as chemistry are being merged into the production of the new era of materials, goods, and methods. The decline of petroleum supplies, along with stricter environmental regulations, is driving the development of novel materials and products that are both ecologically benign and fossil-fuel free [1–9]. Scientists and engineers have a tremendous task ahead of them in inventing the technologies that will enable the biobased materials revolution to become a reality. Natural fibres, such as bast fibres, also provide vital, renewable resources to the business. Bast fibre is made from the phloem that surrounds the stems of fibrous plants, mostly dicotyledonous. The plant's bark or skin protects it from evaporation and unexpected temperature changes, as well as provides some mechanical support to the stem. Fibres are found in the phloem and are normally found in bundles beneath the skin; they support the conducting cells of the phloem and give the stem strength. The xylem material, the woody core of the plant is found in the centre of the plant. Flax and curaua are good reinforcing materials for high-mechanical-strength composites when coupled with carbon fibres. Different bast fibres like jute etc. are used for rope, canvas, pulp, and paper, as well as to reinforce moulded thermoplastics. The short core fibres can be applied in insulation moreover fibre boards can also be made along with mats which can control erosion. Shives are also utilized as a source of particle board raw materials and bedding for edible fungi. Kenaf, jute, and hemp shives can be used as bedding in horse stables and chicken farms. Natural fibres have attracted a lot of attention as a reinforcing material for polymer-based matrices due to environmental concerns, as well as their low cost and several intrinsically fascinating features (density, shape ratio, mechanical behaviour) [10]. [11] like good performance profile, environmental sustainability, biodegradability, lightweight, and low cost. Functionalization has piqued interest as a means of overcoming the challenges associated with natural fibres and, as a result, achieving good performance in natural fibre–reinforced polymer composites [1, 2]. Bast fibres have been used in a variety of applications, including geotextiles, insulation, and composite materials. Bast fibre biocomposites are reliable, economical, having low density. Flax, Kenaf, ramie, and Jute are the most important bast fibre plants. Hemp and flax are grown in nearly every country and harvested for grain, seed, and fibre. Hemp (Cannabis sativa) is a high-fibre, high-biomass plant. Hemp plants grow to be 1.5–2.5 m tall, having a diameter of the stem in the range of 7 to 16 mm. Hemp is a plant that originated

in Central Asia and has since been grown on every continent, in every region, and in every country. Hemp was reintroduced as an industrial crop in Canada in 1998, after a 60 year ban. Clothing, ropes, and insulating materials have all been made from hemp fibres for a long time. Hemp fibre has recently become more popular as a reinforcement in composite materials. Flax (Linumusitatissimum L.) is a member of the Linaceae family. The flax stem's height and diameter are around 1–1.3 m and 4–5 mm, respectively. The plant is native to the Mediterranean and Southwest Asian regions, and it is today one of Canada's most significant crops. Flax fibre was formerly utilized as a textile material, but it has recently been found useful in other uses, such as the manufacture of cigarette papers in North America. The Malvaceae family includes kenaf (Hibiscus cannabinus L.), which can be grown in a variety of climates. Under ideal temperature (21–29 °C) and soil pH (5.9–7.0) conditions, kenaf plants can reach a height of 2.6–3.9 m. The kenaf stem's diameter ranges from 10 to 20 mm. C. capsularis L and Colitorius L are two commercial types of jute, which belong to the Tiliaceae family. It reaches a height of 2.6–3.9 m and has a diameter of 20 mm. Raw jute and its associated fibre are mostly produced in Bangladesh, India, China, and Thailand. Jute is the cheapest and strongest of the natural fibres. Ramie (Boehmeria nivea) is a China grass that belongs to the Urticaceae family. China gathered almost 94% of the World's ramie in 2014. The plant grows to be between 1.0 and 2.5 m tall, with leaves measuring 6–16 cm long and 7–13 cm wide. Ramie stalks range in size from 4.4 to 9 mm in diameter. Ramie is grown largely for its fibres. The best conditions for ramie production have been found to be in warm, humid climates with an average annual rainfall of 1 m [12] (Table 1).

3 Rheology, Rheometers and and Rheological Parameters

The science of matter flow and deformation is referred to as rheology. The measurement of viscosity is used in the study of rheology for low-molecular-weight fluids. The viscosity of such fluids is mostly determined by temperature and hydrostatic pressure. However, because polymeric fluids exhibit nonideal behaviour, the rheology of high-molecular-weight liquids, whether plain or filled, is significantly more complicated. Polymeric liquids have elastic features, such as uneven normal stresses in shear and a noticeable tensile viscosity in extension, in addition to complicated shear viscosity behaviour. The deformation rate along with the molecular weight and structure and morphology of the polymer, the quantity and quality of different additive materials and fillers, as well as the temperature, all influence these rheological parameters. Furthermore, even at a constant rate of deformation, stress is observed to be time dependent. For polymers and polymeric composites alike, rheology is a critical topic. A quantitative description of polymer and composite rheology is clearly necessary for building models of various polymer processing procedures, which can then be utilised to optimise operations and predict the development of flow instabilities. When it comes to polymers, though, the mechanical properties are usually what count. Rheological behaviour, on the other hand, has an impact on mechanical

Table 1 Various bast fibres and their properties

FLAX [13, 14]	
Chemical properties	Flax fibres are mostly made up of cellulose, with minor amounts of hemicellulose, lignin, pectin, oils, and waxes. The cell walls contain cellulose, hemicellulose, and pectins
Physical properties	The fast absorption and desorption of moisture are the main physical qualities of flax that set it apart from other fibres. High crystallinity of the cellulosic component of the fibre, which results in: • linen fabrics having high creasability • flax yarns of low extensibility • fibres and yarns with improved tenacity
HEMP [15]	
Chemical properties	Hemp fibre is mostly made up of cellulose, which accounts for roughly 77% of the overall weight. Pectins, lignin, vegetable waxes and lipids, other water-soluble compounds, and around 10% hygroscopic water make up the rest. Hemp has a lower cellulose content than flax because it is more lignified, yet it is less sensitive to chemicals. It is resistant to bases and can only be damaged by powerful acids. Hemp resists decay better than flax

(continued)

Table 1 (continued)

Physical properties	The unit cells range in size from 15 to 50 microns in diameter. The cells' average length is 35–40 mm, however, it can range from 5–100 mm. Fibre bundles range in length from 1500–2500 mm. Hemp fibre has a somewhat better breaking strength than flax fibre, however, its elongation is poor (23%). The fineness of the bundle determines its flexibility. The longer the bundles, the less twisting is required while spinning. Although bundles have a low elongation, they have high flexibility, which might present complications when spinning. Blending flax with hemp enhances the elongation and flexibility of the yarns, which are both lacking in hemp yarns made entirely of hemp. These mixtures, on the other hand, reduce the yarn's strength
JUTE [16]	
Chemical properties	Jute fibre is acidic and contains a significant amount of hemicellulose (21–25%) with an acidic group and lignin (12–14%). Alkali has a negative effect on the fibre. Even a modest alkali treatment lowers the fibre's wet strength. The carboxylic and phenolic hydroxyl groups in the fibre contribute to its acidity. Because of the carboxylic acid, the fibre holds ash particles cationic in nature and has an affinity for dyes like methylene blue. The chemical characteristics of jute fibre are determined by alpha cellulose, hemicelluloses, and lignin, which account for more than 96% of the total jute constituents
Physical properties	Jute is a tough fibre with a low extension due to its specific structure with closely aligned long chain molecules. Jute fibre has low extensibility values ranging from 1 to 1.7% breaking elongation. White jute has been found to be less durable than Tossa jute. In terms of tensile strength, jute is comparable to steel

(continued)

Table 1 (continued)

RAMIE [17]		
Chemical properties	Constituent	Content (%)
	Cellulose	68–76
	Hemicellulose	12.98–17.2
	Lignin	0.61–0.72
	Pectin	12
	Fats and wax	0.25
Physical properties	The typical length of a raw ramie fibre strand is 0.61–1 m. The longest fibres can reach 1.5–2 m in length. Ramie is having long final fibre cells, which are around 150 mm and have the highest length-to-breadth ratio. It's lustrous, has a high tensile strength, improves in strength when wet, and is microbial, mildew, insect, and rot resistant. Ramie absorbs and releases moisture quickly, without shrinking or straining. Ramie has a variety of problems, including a lack of elasticity, abrasion resistance, robustness, stiffness and brittleness, the requirement for degumming, and a high cost	

behaviour, which is why polymer rheology is being studied. The mechanical properties of moulded items, fibres, and films, for example, are influenced by molecular orientation. For short fibre composites in unfilled systems, fibre orientation serves as molecule orientation. The kind and degree of molecule or fibre orientation is determined by the polymer's rheological behaviour and the nature of the flow in the manufacturing process. Rheology is a component of many other aspects of polymer science. Many polymers, for example, are made from emulsions of monomers in stirring reaction containers. The resulting latices pass through pipes and may end up as a paint that is applied to a surface using a method that necessitates exact control of the latex's chemical properties. Rotational moulding is one method of converting plastisols, which are polymer suspensions in a liquid, into useful things. Powdered polymers or grains must be able to flow freely out of bins and perform well in fabrication processes such as rotational moulding and powder coating. The rheology of polymer powders is crucial in the early portions of extruders and injection moulding machines before the polymer softens to a liquid. At a more fundamental level, rheological measurements, both transient and steady-state, can be employed for product characterization and quality control. These measurements are commonly used to examine and comprehend the interaction of the numerous parts of a multicomponent or multiphase mixture, as well as their impact on the flow and other properties of such materials.

3.1 Rheometers

There are two categories of instruments when we talk about measuring the viscosity of polymers and their composites (a) Viscometers (b) Rheometers. Both the instruments measure the viscosity of specific nature. A viscometer is used for measuring fluid viscosity and the corresponding flow behaviour of liquids. Limitation of viscometers is that it only examine the viscosity and flow behaviour under one flow condition. Viscometers either works when the fluid is static and an object moves through it or in other case the object is static and the fluid goes past it. Viscometers work in the Laminar regime of flow where the value of Reynolds number is less. In other categories a Rheometer is a very important instrument for analysing the rheology of polymer melts and composites in response to an application. It is also used for those fluids which does not have a single value of viscosity and for characterizing their flow behaviour more parameters are required (Table 2).

Table 2 Comparison between a rheometer and viscometer

S. no	Point of discussion	Rheometer	Viscometer
1	Basic functionality	For determination of rheological properties of polymer melts and their reinforced composites	For determination of viscosity of liquids under flow conditions
2	Classification/types	Capillary, extensional, rheometer and torque rheometer interface rheometer	Rotational type, vibrational or oscillation type, Saybolt and redwood, etc
3	Characteristic	It is used for those fluids/polymer melts which does not have a single value of viscosity and for characterizing their flow behaviour more parameters are required	Only for viscosity of liquids
4	Range of shear rate	Very wide range	For short range over limited values
5	Multitasking	It can serve as a viscometer	Not possible to work as rheometer

3.2 Types of Rheometers

Rheometers can be classified into these three categories.

(1) **Capillary Rheometers**: It can be applied in extrusion as well as injection moulding. These types of rheometers are useful in obtaining the correct measurements. These rheometers analyse the flow through a narrow space to examine the viscosity changes with respect to changing rate of shear.

(2) **Torque Rheometers**: These types of rheometers are for relatively small extruders or mixers. These rheometers evaluate the values of torque on mixing screws or rotors to analyse the difficulty level in mixing of different materials It does not measure the true or absolute viscosity of the sample as the measurement is based on comparison.

(3) **Dynamic Rotational Rheometers**: Dynamic Rotational Rheometers examines the visco-elastic behaviour along with the molecular structure of the polymers which are kept between the two parts of the instrument, a static component and other one which rotates. The rotation speed can be changed. The processing effect on resin can also be analysed. These rheometers are useful in analysing the behaviour of thermoplastic polymer materials. Figure 1 is showing the different shear planes for evaluating rheological properties.

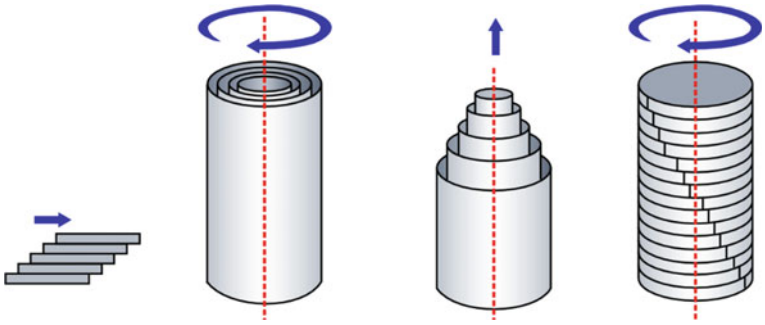

Fig. 1 Different shearing planes that can be employed to measure rheological properties. From the left—Couette drag plate flow; cylindrical flow; Poiseuille flow in a tube and plate-plate flow. (*Source* Wikipedia)

3.3 Important Properties in Rheological Examination of Bast fibre Composites

(a) **Complex viscosity**

[18] developed agrofibre/HDPE composites and in Fig. 2 variation of viscosity of composites with frequency is shown. Rise in viscosity is because of the addition of fillers in polymer.

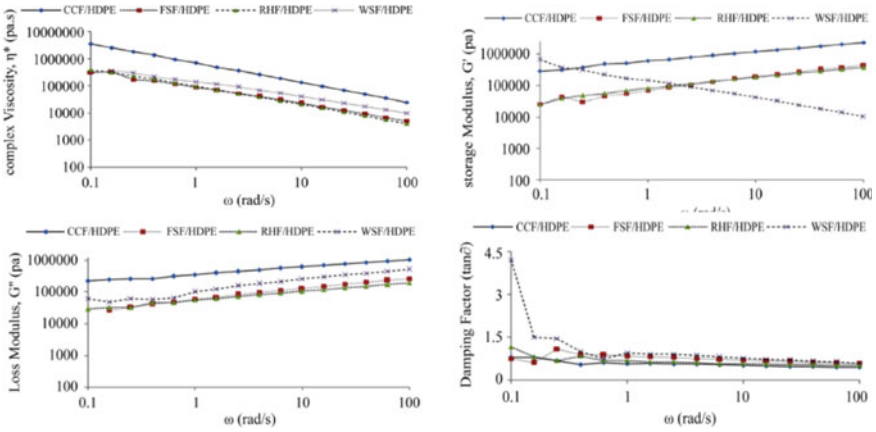

Fig. 2 Variation of complex viscosity, storage modulus, loss modulus and damping factor of agrofibre filled HDPE composites [18]

(b) **Storage modulus**

Figure 2 is showing the change of storage modulus with respect to frequency. Because of highfibre percentage (65%) [18] causes agglomeration and intrinsic rigidity due to which the storage modulus of composites is high.

(c) **Loss modulus**

Figure 2 is showing the change in the loss modulus (G'') with frequency (ω), for fibre filled HDPE (High density polyethylene composites) composites. The value of the loss modulus also increased with the increased values of frequency.

(d) **Damping factor**

Damping factor is a tan value of G''/G' where G'' is loss modulus and G' is storage modulus. In the case of perfect elastic behaviour there is no viscous part that's why G'' is zero leads to $\tan \delta = G''/G' = 0$.

4 Rheological Analysis of Bast Fibre Composites

[19] Studied melt viscosities of jute and kenaf reinforced polypropylene composites at 30% loading of fibre. Capillary rheometer was used for the analysis. The effect of the coupling agent on melt viscosities was also analysed. Agrofibre including flax shives were used in HDPE for developing composites [18]. They have determined the meltrheological by hybrid rotational rheometer under strain-controlled conditions. [20] Prepared flax and hemp fibre composites and rheological behaviour were studied with the help of a dynamic mechanical analyser. [21] summarized a review of the various visco-elastic and rheological properties of composite materials. [22] examined the rheological behaviour of jute composites reinforced PP and It was observed that with the reinforcement of fibres and MAPP, the melt viscosity of the composites increased due to better interlocking between fibre and matrix. [23] analysed the effect of hemp fibre on the elongation rheology and shear of polypropylene composites. The fibre loading range was 0–30% and MAPP addition 0–0.6%. ARES rheometer with parallel plate geometry was used for rheological characterization (Table 3).

5 Conclusion

Composite materials with a polymer matrix and natural fibre reinforcement are gaining popularity in research and industry. The rheological properties of a natural fibre polymer composite are heavily influenced by the effectiveness of bonding

Table 3 Compilation of available work on rheological properties of natural fibre reinforced composites

S. No	Title	Fibre used	Observations	References
1	Rheological properties of natural fibre reinforced PP composites	• Cellulose • Sawdust • Wheat straw	The addition of fibres to PP enhanced the composites' stabilising torque. At 40 wt% cellulose loading, the rise was roughly 11%. Because of increased interactions between fibre and matrix, silane treatments raised stabilising torque values, regardless of the fibre used. Because of MAPP's plasticizing effect, it reduced stabilisation torque	[24]
2	Rheological properties of natural fibre polymer composites	• Rice hull • Flax shive • Corncob • Walnut shell	The rheological properties of the final composites are affected by the type of fibre, content materials, particle's shape and size	[25]
3	Melt rheological properties of natural fibre reinforced polypropylene	• Coir • Jute • Kenaf	The viscosity of jute was the highest, followed by kenaf and coir. When the power-law indices (n) are compared, it is clear that jute had the most shear thinning. Jute had a power-law index of 0.32, while polypropylene had a power-law index of 0.37. The application of (MA-g-PP) coupling agent had little influence on viscosity. The benefits of the compatibilizer can be seen in the jute composites' improved tensile characteristics	[19]

(continued)

Table 3 (continued)

S. No	Title	Fibre used	Observations	References
4	Mechanical and rheological behaviour of composites reinforced with natural fibres	• Flax • Hemp • Wood particle	The disposure of fibre, size of fibre and types of matrix affected both tensile and rheological behaviour	[20]
5	Rheological properties of molten flax- and Tencel VR-polypropylene composites: influence of fibre morphology and concentration	• Flax • Tencel	The activation energy increases as the fibre content increases. Greater viscosity, viscous and elastic moduli, and the apparent yield stress resulted from the increase in fibre concentration. A larger number of fibre and higher flexibility resulted in more fibre–fibre interactions for the same fibre concentration. As a result, apparent yield stress, composite viscosity, and activation energy all increase	[26]
6	Characterization and comparison of rheological properties of agro-fibre filled high density polyethylene bio-composites	• Rice hull • Flax shive • Corncob • Walnut shell	The complex viscosities were quite high at low frequencies, but they decreased as frequency increased, showing that the agro fibre composites were shear thinning. Complicated viscosity is excessive with very good filler due to aggregation of filler particles. However, complex viscosity will grow at a very large particle size due to the difficulties of aligning fillers along the drift path	[18]

(continued)

Table 3 (continued)

S. No	Title	Fibre used	Observations	References
7	The evaluation of rheological properties of composites reinforced with hemp, subjected to photo and thermal degradation	• Hemp	Surface degradation due to matrix chemical changes reduces visco-elastic properties and, as a result, the carrier life of composite constructions reinforced with hemp mat and polyurethane resin	[20]
8	Comparative rheological studies on jute fibre and glass fibre filled polypropylene composite melts	• Jute fibre • Glass fibre	With increasing fibre content, pseudoplasticity reduces at a given temperature and increases with temperature in both circumstances. The melt viscosity of each filled system increases with increasing fibre content material and decreases with the rising shear rate at a given temperature, becoming virtually similar at extreme shear cost	[27]
9	Rheological characterization of PP/jute composite melts	• Jute	With the addition of fibres, the composites' steady-state viscosity improved.	[22]

(continued)

Table 3 (continued)

S. No	Title	Fibre used	Observations	References
10	Rheological properties of polypropylene/hemp fibre composites	• Hemp	Beneath shear strain, growing hemp attention expanded the crossover modulus (Gc) and frequency (xc) of the composites. The yield strain and the zero shear viscosity improved with hemp concentration. Increased hemp content material leads to better values of the consistency index and the maximum strain	[23]
11	Dynamic and capillary shear rheology of natural fibre reinforced composites	• Flax • Sisal	There is a decrease in composites viscosity due to the presence of MAPP which also counterbalances the growth in viscosity because of fibres. Elastic behaviour and yield stress increase due to higher fibre concentrations	[28]

(continued)

Table 3 (continued)

S. No	Title	Fibre used	Observations	References
12	Rheological properties of sisal fibre/poly (butylene succinate) composites	• Sisal	Because of the shift in fibre aspect ratio, the power-law index n decreased as fibre content rose. When the angular velocity was held constant, the force given to the fibres by the melt and other fibres increased as the fibre content rose, reducing the fibre length and, thus, the aspect ratio. The shorter the fibres became at constant fibre content as the angular velocity increased: the shorter the fibres, the easier for fibres to orientate along the flow direction and the lower the composites' resistance to flow. Composites with higher fibre content reduced their aspect ratio faster than composites with lower fibre content	[29]

at the fibre-matrix interfacial boundary. The interface's main job is to make it easier to transfer stress from one fibre to the next throughout the matrix. The weak bonding properties of cellulose fibre with a polymeric matrix are well recognised. The hydrophilic character of cellulose, as well as the presence of organic and inorganic compounds on the fibre surface, inhibit efficient adhesion between the fibre surface and the polymer matrix. Fibre surface modifications with treatments such as heat treatment, acetylation, silanisation, and plasma have successfully enhanced bond strength. The topic of compiling existing material has been considered.

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Dielectric Properties of Bast Fiber Composites

Astha Dixit, Kriti Sharma, and G. L. Devnani

Abstract With increasing use and research in the field of natural fibers and their reinforced composites, it is important to explore the potential of these novel materials in diverse fields. Bast fiber is an important category of natural fibers consisting of mainly jute, ramie, hemp, kenaf, etc., extracted from the stem of the plant. Extensive work has been done on the mechanical properties of these fibers and their reinforced composites, but the dielectric property aspects of these materials have been less explored. Dielectric properties like dielectric constant, resistivity, conductivity, etc., decide whether the material is useful in insulation or conducting products. Bast fibers with low resistivity have a high tendency to allow the flow of electric current. Lignocellulosic with these bast fibers have (OH) hydroxyl groups that affect the dielectric constant. Moisture content of the material is also responsible for the dielectric properties. This study compiles the work done on dielectric aspects of natural fiber, especially bast fiber-reinforced composites in order to improve the understanding of commercialization aspects of these materials in the production of electrical and electronic goods.

Keywords Resistivity · Conductivity · Dielectric constant · Electric current

1 Introduction

A dielectric substance is considered as a non-metallic substance having a high value of specific resistance and large insulation resistance too. In other words, we can say that dielectric material is basically a non-conducting material which has the capacity to store electrical charges. When a dielectric substance is placed in an external electric field, the electric charges do not flow through the substance. Dielectric polarization is responsible for + ve charges to flow in the direction of the applied external electric field and -ve charges to move in the opposite direction of the applied electric field. An internal electric field is generated, and due to this reduction in the external electric

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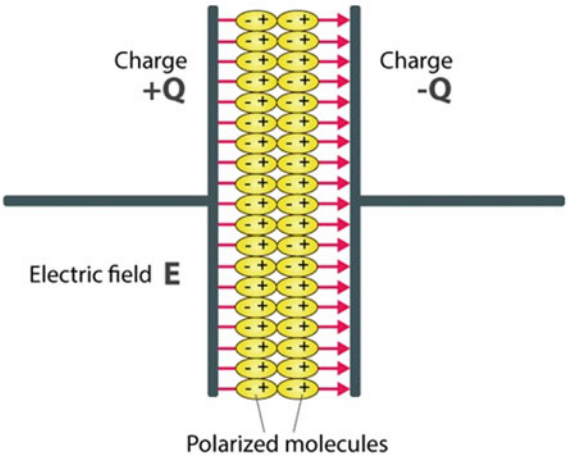

Fig. 1 Polarization of dielectric molecules

Table 1 Dielectric versus insulators

S.No	Dielectrics	Insulators
1	A substance can develop electric field without much loss of energy	Having low conductivity and obstructs the current flow
2	Bonding is weak as compared to insulators	Covalent bonds are there
3	Storage of charge	Obstruct charge
4	Applied in capacitors, power cables	Used in conducting wires and high-voltage system

field, it opposes the original electric field. Electric susceptibility accounts for the ease of dielectric material can be polarized when situated in an electric field. Figure 1 is showing the polarization of dielectric molecules when an external electric field is applied. Dielectric substances are solids mostly. Use of dielectric materials is for the storage of energy. Dielectric materials are often confused with insulators. A comparison between dielectric substances and insulators is given in Table 1.

The increasing use of electrical and electronic goods in this century has created an issue of E-waste also which is basically non-biodegradable in nature. This has also encouraged the academic and scientific world to look for alternative sustainable and biodegradable materials for developing electrical and electronic goods in the near future. Extensive work has been done in the last decade on exploration and utilization of natural fibers [7, 10, 11, 36] and their reinforced composites [20, 31], and a lot of emphases is given on mechanical and thermal characterization of these novel materials. Various treatment methodologies and filler addition is also tried [8, 9] to improve the properties of these composites. With the increasing use of these natural fiber-reinforced composite materials, diverse application is another area of exploration which makes this research field more attractive and useful. Bast

fibers are derived from the phloem, a substance inside the stem of stringy plants and it is an important category of natural fibers. Substantial work also has been done on bast fiber characterization and development of various composite materials [17, 25, 35] Jute, kenaf, hemp. Ramie and flax are the main bast fibers [24, 33, 34]). The presence of different functional groups on bast fibers which are friendly to chemical modification and their flexibility to fabricate into different sizes and shapes is the desirable characteristic of bast fibers for electronic and electrical utilization. Variation in properties of bast fibers is a challenge to control the property of fabricated products. A critical review of type of bast fibers that are utilized in the development of dielectric materials and their electrical characterization can be very useful for the researchers and industries that are looking to explore these sustainable materials in dielectric application [3]. This study elaborates on the important dielectric properties of bast fibers and their reinforced composites. The effect of chemical modification on dielectric characteristics of these materials is also reviewed.

2 Dielectric Properties of the Materials

The dielectric properties of bast fiber composites decide whether can be utilized in electrical applications or not. The major dielectric properties to be evaluated are as follows.

2.1 Dielectric Constant of Composite

The dielectric constant or relative permittivity of composite or any other material is also called the relative permittivity of the material. It is the ratio of the permittivity of a given substance to the permittivity of vacuum. In terms of mathematics, the dielectric constant $Kor\varepsilon_r$ or relative permittivity can be expressed as.

$$Kor\varepsilon_r = \varepsilon/\varepsilon_o$$

where ε is the permittivity of material, ε_o refers to the permittivity of the free space. The value of dielectric is essential to describe a capacitor. A capacitor is an important electronic device which is fabricated by putting a dielectric material inside the conducting metal plates. The type of dielectric material and its dielectric constant is responsible for the storage of charge inside the capacitor. The value of dielectric constant is always greater or equal to 1.

2.2 Dielectric Loss and (ε'') Dielectric Loss Factor ($\tan\delta$)

Dielectric loss ε'' is proportional to the value of conductance and if we represent permittivity by ε' the loss factor $\tan\delta = \varepsilon''/\varepsilon'$. Other parameters are $\varepsilon' = C/C_0$ and

$$Co = \frac{\varepsilon o.A}{d}.$$

2.3 Dielectric Strength

The dielectric strength of polymeric materials is determined utilizing the accompanying estimation of dielectric power or strength as

$$DS = \left[\frac{dv}{dx}\right] = \frac{V_b}{d},$$

where DS is the dielectric strength in kV/mm, Vb is the maximum voltage, and d is the thickness of material.

2.4 Resistivity

Electrical resistivity is basically the property of material due to which it creates resistance in the movement of current from one end to the other. It is the reciprocal of conductivity given as

$$Resistivity\ \rho = \frac{RA}{L}.$$

3 Dielectric Properties of Bast Fiber Composites

The advancements and developments in electronic and electrical equipment (EEE) are responsible to make it a leading industry. It is a fast-growing business worldwide. The innovations and technological upliftment are giving a boost to the replacement of outdated and old equipment and instruments responsible for a substantial increase in Electronic or E-waste The disposal of this E-waste is a big issue which has been faced by industry persons. If some of the components can be replaced by biodegradable material like bast fiber or natural fiber composites, it can reduce the burden on environment. The detail of work done on dielectric properties of bast fiber composites is as follows.

3.1 Dielectric Properties of Jute Fiber-Reinforced Composites

Jute fiber is a class of the Tiliaceae family. It develops to a stature of around 2.5–3.5 m and a width of around 20 mm. Asian countries like India, Bangladesh, and China along with Thailand are the main makers of crude jute and related strands. Among regular filaments, jute is considered the least expensive and most grounded. Low-density polyethylene composites were fabricated by the reinforcement of jute fiber. Heat press molding methodology was used for the development of composites. Chemical treatment with HEMA was used to improve interlocking between fiber and matrix. Dielectric parameters mainly dielectric constant and loss tangent of jute, polymer, and composites were analyzed. Dielectric properties like dielectric constant and loss tangent (tan d) of jute and low-density polyethylene were examined. Transition from Ferro to paraelectric phases had been observed in untreated and chemically jute-reinforced composites having fiber loading of more than 20% [4] studied dielectric properties of jute fiber-reinforced polypropylene composites. The value of the dielectric constant had an increasing trend up to 30% fiber loading after that a sudden increase was noticed [18] studied the dielectric properties of jute woven fabrics. The evaluation of AC-specific electrical conductivity and its variation enables to analyze the characteristics of the jute fabrics for real applications [26] examined the dielectric properties PLA and PLA/PBS blends and their reinforced composites by jute fibers. The temperature range was kept from 20 to 140 °C and the frequency range was 10^{-1} Hz–1 MHz. In another study [21], the dielectric properties mainly dielectric constant, dielectric loss factor, and dissipation factor were evaluated for polypropylene and polyester composites reinforced by jute and bamboo fibers. The value of dielectric parameters increased with increased fiber percentage over the entire range of frequencies. Figure 1 is showing the variation of dielectric constants of jute/bamboo-based polypropylene and polyester composites.

3.2 Dielectric Properties of Flax-Reinforced Composites

Flax fiber was once utilized as a material texture, however, more as of late, it has tracked down the use in different applications, for example, making paper cigarettes in North America. Dielectric behavior of flax fiber-reinforced ethylene–propylene–diene monomer (commonly known as EPDM) composites having different fiber percentages. The EPDM/flax composites had more value of dielectric parameters in comparison to the neat EPDM sample [1]. A dielectric study was done to analyze the effect of addition of strontium titanate on polypropylene composites reinforced by flax fiber. The increase in dielectric properties is due to accumulation of charge carriers at fiber–polymer–filler interface. These materials were recommended for application in layered printed circuit boards. Modeling studies were also performed

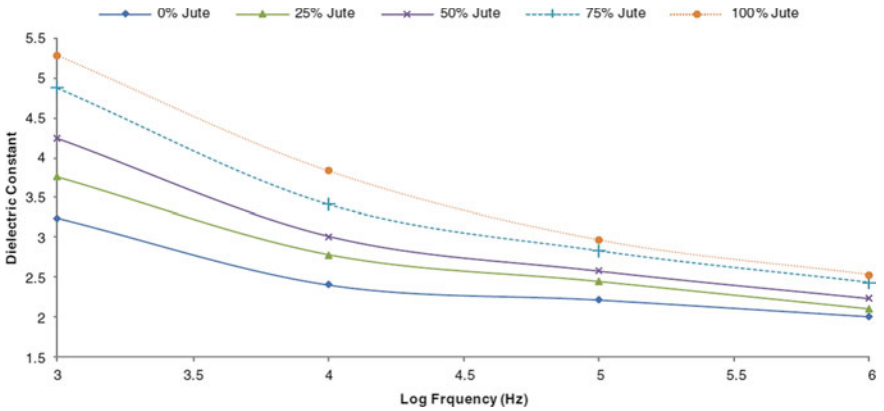

Fig. 2 Effect of fiber ratio on dielectric constant of jute and bamboo hybrid composites [21]

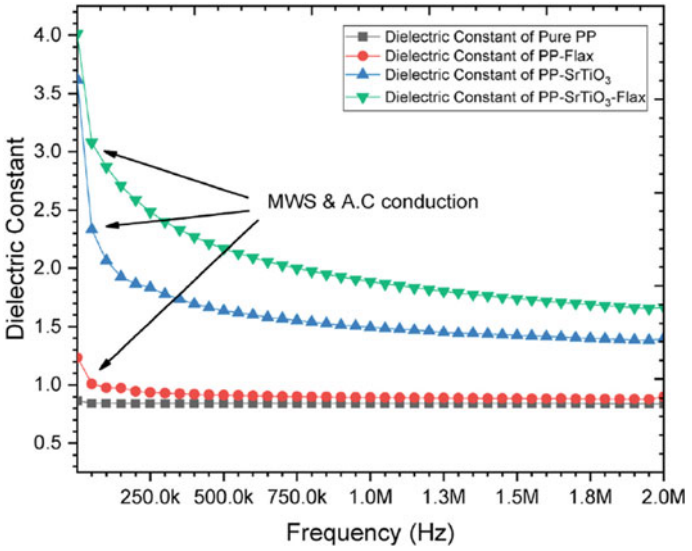

Fig. 3 Dielectric property variation with frequency of various samples

in that work. Figure 2 shows dielectric constant variation of composite as well as pure pp samples with and without addition of filler material [15] (Fig. 3).

3.3 Dielectric Properties of Hemp Fiber-Reinforced Composites [15]

Hemp (Cannabis sativa) is a decent wellspring of fiber and biomass. Hemp plants range from 1.5 to 2.5 m tall and have a stem measurement somewhere in the range of 7–16 mm. Hemp began in Central Asia and has since been developed in all main lands, locales, and nations. Composites were fabricated with isotactic polypropylene (iPP) polymer and hemp fibers [23]. The dielectric measurements were performed using SEM Mirror Effect method coupled with the calculation of induced current (ICM) value. The mechanical and dielectric characterization of epoxy composites was done at room temperature and different fiber loading values [5]. Increased value of dielectric constant and loss factor were observed with increased fiber loading values. Increased value of conductivity was also observed. It was concluded that there is a direct relation between structure and mechanical and dielectric properties of bast fiber composite material [28] recommended hemp fiber-reinforced polypropylene composites for packaging electronics because of their excellent dielectric properties.

3.4 Dielectric Properties of Kenaf Fiber-Reinforced Composites

Kenaf (Hibiscus cannabinus L.), which can be developed under various environments, is an individual from the Malvaceae family. Kenaf plants can grow up to 2.5–4.0 m underneath typical temperatures (22–30 C) and soil conditions pH (6.0–6.8). Stem width of kenaf goes from 10 to 20 mm [27] developed polyurethane foam filled with kenaf fiber and studied the effect of fiber loading on the dielectric characteristics of the material. The foam was prepared by the free rising method. The fiber loading varied from 0 to 15% and the temperature range was kept between 30 and 200 °C. The range of electric field frequency was set between 20 Hz and 2 MHz. The increment of dielectric parameters was observed by increasing the fiber loading. The values were very high in the frequency range from 10 to 100 Hz. Higher temperature facilitated high dielectric constant but over 120 °C the value of loss tangent decreased dramatically. Table 2 summarizes the recent developments in the dielectric characterization and application of bast fiber-reinforced composites.

4 Dielectric Properties of Other Natural Fiber Composites

A significant amount of work has been done on other natural fibers (other than bast) based composites for dielectric applications in the last decade [32] studied dielectric properties of epoxy composites reinforced by Prosopis juliflora, a plant-based fiber.

Table 2 Recent developments in the dielectric characterization of bast fiber composites

S. No	Matrix	Fiber	Fabrication method/Fiber loading/Filler or treatment applied	Key findings	Reference
1	Unsaturated polyester along with Polypropylene	Jute and bamboo	Hot pressing method was used/10 to 30%fiber loading was kept/alkaline treatment was done	(a) At high-frequency dielectric constants were decreased (b) Fiber loading has a proportional behavior with dielectric parameters (c) Alkaline-treated composites showed less value of dielectric constant	Jayamani et al. [21]
2	High-density polyethylene	Banana, **Hemp,** and Agave	Compression molding/ 55:45, 50:50, 45:55 wt ratio of fiber to resin /2% maleic anhydride in xylene was used for treatment	(a) Decrease of surface resistivity on increasing fiber content (b) Increase in volume resistivity on increasing fiber content	Naik and Mishra [30]
3	Unsaturated polyester	Chicken feather fiber, **kenaf fiber**	Compression molding/ 10–45% fiber loading was kept/unsaturated composites were cured by MEKP	(a) The dielectric parameter values were high at low frequencies (b) values were high for the fiber content of 40%	Siong Kiew et al. [39]
4	None	Hemp and flax fiber yarns	Fabrics were covered withNi, NiO, Cu, CuO, and Al coatings	(a) Metal conductivity of bast fiber fabrics slightly increased due to metal oxide coatings. but the distribution is strongly influenced by moisture due to the high hygroscopic properties of the bast fibers (b) Metal oxide coating on both sides is recommended in order to have low-density and high-conductivity fabrics	Sidorovica et al. [37]
5	**Poly propylene**	**Jute yarn**	Compression molding/ 37.1 to 55.9% fiber loading was kept/ $KMnO_4$, MAPP, Stearic acid, and (TDI) toluene diisocyanate	(a) The dielectric performance of polypropylene and jute yarn commingled composites was analyzed by varying the fiber loading, various chemical treatment methodologies, and amount of moisture present in the (b) All the dielectric parameters including dielectric constant, dissipation factor, loss factor, and conductivity improves by enhancing fiber loading. This increase is because of increased polarization effect (c) Decrease in the values of the volume resistivity was observed because bast fibers attract moisture and the moisture presence supports the current flow in the composite	George et al. [13]

(continued)

Table 2 (continued)

S. No	Matrix	Fiber	Fabrication method/Fiber loading/Filler or treatment applied	Key findings	Reference
6	Bisphenol-C-Formaldehyde resin	jute mat along with rice, wheat, sugar cane and jamun fibers were used	Simple hand layup method had been used and silicon spray was applied for mold releasing agent	(a) The solid strength of composite had been decreased by 53–72% because of the randomness in the structure of the composite fibers. (b) The volatility at the same time has increased by 53–153% (c)	Mehta and Parsania [29]
7	**Polyester**	jute fiber	-	(a) Water sorption measurements are made of composites of polyester matrix consisting of glass, jute, and washed jute fibers. Glass fiber-based composites have less tendency of water absorption as compared to jute and jute blend compounds, because of its low hydrophilic properties (b) The highest water absorption rate was higher in the washed jute mixture after removing the measuring agent with hot water (c) Mathematical model was used to all evaluate to diffusion coefficient. The high diffusivity was found in the natural composite fibers in comparison with pure resin and glass fiber composite (d) Dielectric constant of composites reinforced with jute is higher as compared to composites reinforced with glass fiber (e) Variation in the values dielectric permittivity had been observed at different frequency values. Time was also a variable parameter for this water absorption process. An ambient temperature condition was used. A nonlinear trend was observed in the variations of values of permittivity values with respect to water absorption. frequency range was set (200 Hz–1 MHz.)	Fraga et al. [12]
8	Polyethylene/Poly propylene	Jute fabrics	Compression molding /50% fiber loading/Gamma radiation was applied on polypropylene, polyethylene, and jute	The dielectric constant parameter along with and loss tangent of the jute fabrics composites was enhanced but the conductivity values of the composites were decreased as a result of gamma irradiation. It can be inferred that gamma radiation is one of the useful techniques to enhance the mechanical as well as dielectric parameters of jute fabrics based polymer matrix composites	Zamam et al. [40]

(continued)

Table 2 (continued)

S. No	Matrix	Fiber	Fabrication method/Fiber loading/Filler or treatment applied	Key findings	Reference
9	**Epoxy**	Sunn Hemp	Hand lay up/5,10,15,20,25 percentage of fiber/ pre-sonication was done on epoxy resin and degasification was done at 80° under vacuum	Dielectric parameters including dielectric constant and loss factor are enhanced with increasing loading	Dash and Bisoyi [5]
10	**Epoxy**	Hemp	Hand lay-up/20% of fiber/KOH alkali treatment was done with varying alkali concentrations from 5 to 20% for various soaking time	The dielectric behavior of composites reinforced with sun hemp fiber was analyzed with respect to varying KOH alkali concentrations The highest values of parameters like ε0 and tan δ are repoerted in the case of composites with 20% KOH treatment for 10 h and lowest in case of 10% KOH treated composites for 6 h. The complex modulus and impedance graph sugget a non-Debye type of electrical relaxation behavior	Dash and Bisoyi [6]

Alkali treatment was applied to enhance the performance of composites. Significant improvements in dielectric behavior of the chemically treated composites were observed if we compare them with untreated fiber-based composites. The reduction of hemicellulose percentage and lignin percentage in the fiber also facilitated the improvements [19] did the hybridization of Kevlar 29 fiber with bamboo to develop epoxy composites and performed the conductive and dielectric characterizations. Kevlar's incorporation in bamboo substantially improved the dielectric properties of composites [14] used a new fiber Phaseolus Vulgaris for the development of polymer composites and studied the dielectric performance. Central composite design was used for the optimization of parameters, that is, extraction time and alkali concentration [2] utilized short palm tree fibers and developed polyester composites. Maleic anhydride was used for the surface treatment of these fibers. A frequency range of 1–1000 Hz was used for the analysis of dielectric properties [22] analyzed the electrical behavior of phenol formaldehyde composites reinforced with banana fibers. A reduction in dielectric constant was observed with increased fiber loading and frequency values. A reduction was also observed due to different chemical treatments such as alkali, silane, etc., higher value of dielectric constant was observed in case of banana–phenol formaldehyde composites if we compare it with glass–phenol formaldehyde composites [16] examined the dielectric performance of rubber composites reinforced by a hybrid mixture of sisal and coir. Different parameters were varied like fiber percentage, chemical treatment, fiber ratio, presence of bonding agent, and frequency. Filled composites have a high dielectric constant as compared to neat rubber. Incorporation of fiber in the matrix is responsible for this improvement as it created polarization [38] fabricated composites reinforced with a novel natural fiber Grewia optiva. Raw and surface-treated fibers were used for the development of unsaturated polyester-based composites. Similar improvements in dielectric properties were observed due to the incorporation of this lignocellulosic fiber.

5 Conclusion

The importance of natural fibers and their reinforced composites is evident due to their increased use and diverse applications. A lot of research has been done on the application of these composites in semi-structural applications. Extensive work has also been done on the mechanical and thermal characterizations of these novel materials. New diverse applications are also explored with the growth of these materials. Electronic and electric application is one significant area where these sustainable materials can be used and can solve a lot of waste disposal and environmental issues. Proper evaluation of dielectric property is very important for the application of these materials in electrical and electronic goods. It was experienced that the incorporation of fiber increases the dielectric performance due to polarization. Chemical treatment also affects the dielectric parameters. Natural and bast fiber-reinforced composites has tremendous potential to be utilized in this field.

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Biodegradable Bast Fiber-Based Composites

Seyda Eyupoglu

Abstract Recently, the using of plant-based fibers especially bast fibers has been investigated as substitutes for the synthetic fibers such as aramid, carbon, and glass in the polymers composites as reinforcement because of their lightness, biodegradability, low cost, and high mechanical behaviors. Biodegradable composites are defined that a biodegradable polymeric matrix is reinforced with biodegradable fibers. Biodegradable polymers are known as the polymers degraded with enzymes. In order to produce a biodegradable composite, plant-based fibers are frequently used. The behaviors of these composites are fundamentally dependent on the fiber kind, aspect ratio, orientation of fibers in the matrix, volume fraction, and adhesion of fibers and matrix. Especially, fiber properties affect the biodegradable composite behaviors. The plant-based fibers are mainly categorized into grass bast, leaf, seed, and straw fibers. In plant-based fibers, bast fibers are obtained in the bast of plant and it is known that these fibers have been utilized by earlier civilization. Common examples of bast fibers are jute, ramie, kenaf, flax, hemp, and bagasse. Because these plants are annual crops, they are getting rising interest in a variety of biodegradable composite production processes. This chapter, it is reported an overview of the current investigation of biodegradable materials. These consist of some developments substantiated in the field of biodegradability, biodegradable polymers biodegradable composites, bast fibers, and biodegradability testing methods. This chapter could help in the investigation of this area and using of composites in industries.

Keywords Biodegradability · Biodegradable polymers · Biodegradable composites · Bast fibers

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1 Introduction

Biodegradation is commonly described as the degradation of organic substances by enzymes produced by living microbial organisms to small components. Organic substances are transformed through metabolic or enzymatic processes. Although the biological degradation process varies generally, the last product of biological degradation is commonly biomass, methane, and carbon dioxide. Organic sustenance can biodegrade in oxygen (aerobic) or anaerobic environments without oxygen. Aerobic biodegradation is defined as organic substance degradation with microorganism in the presence of oxygen in the environment. Aerobic biodegradation is characterized by oxidative conditions. A variety of organic pollutants are decomposed rapidly under aerobic conditions by aerobic bacteria (aerobes). Aerobes have an oxygen-based metabolism and utilize oxygen to oxidize substrates (sugars and fats) to obtain energy in a process called as cellular respiration. Before cellular respiration, glucose molecules are degraded into two small molecules. This occurs in the cytoplasm of aerobes and then these small molecules enter the mitochondria. In the degradation of small molecules into H_2O and CO_2, oxygen is used, and energy is also released due to the reaction. Aerobic degradation does not release pungent-smelling gases unlike anaerobic degradation. The aerobic process also improves the environment of workers and animals and helps keep pathogens under control. Anaerobic biological degradation (without oxygen) occurs when the number of anaerobic microorganisms is higher than aerobic microorganism. Biodegradable wastes are degraded without oxygen. Compared with aerobic and anaerobic degradation, it is a widely used method for the treatment of wastewater and biodegradable wastes, as it allows us to reduce the volume and mass of input materials. Anaerobic biodegradation is a renewable energy source. In this process, biogas is produced including methane and carbon dioxide for suitable energy production. In addition, nutrient-rich solids left behind after decomposition can also be used as fertilizer [1].

Biodegradation is a commonly microbial natural process that regenerates biological fundamental elements within the World's biogeochemical cycles. Biological degradation is generally catalyzed by enzymes which are converted chemicals into the last product. A chemical substrate is converted to entirely oxidized products by enzymes, and this process is called as mineralization. Biotransformation consists of the conversion of a chemical substance into another without whole mineralization and is shown commonly during microbial metabolism of synthetic substance [2].

In the words, a variety of wastewaters including organic substances are drained. Bio-treatment is accepted to be the well-liked treatment; however, all the wastewaters cannot be applied by bio-treatment for economic or technical reasons. Therefore, the biodegradability of wastewater or substance is needed to evaluate before biological application. In order to determine of biodegradability of a substance, many methods have been provided. When organic substances are degraded by microorganism, basic inorganic substance occurs which are H_2O, CO_2, and energy delivery. Microorganisms utilize energy to transform ADP into ATP. It then uses the energy from the

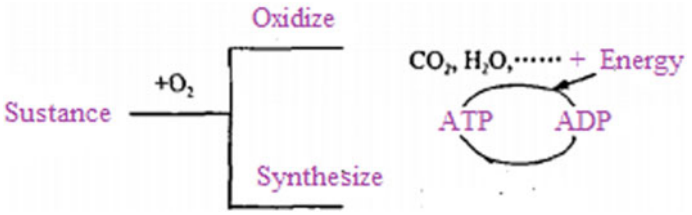

Fig. 1 The degradation of organic substance

reaction of hydrolysing ATP to synthesize the new cytoplasm on the other side. The degradation of organic substance is given in Fig. 1 [3].

Plastics are used in many fields due to their advantages. However, the demand for using and development of biodegradable materials have increased in approximately all industries in recent years. Biodegradable polymers can degrade with the enzymatic activities of bacteria, fungus, and algae in a bioactive environment. As a result of the biodegradation of biodegradable materials polymers turn into CO_2, CH_4, biomass, water, humus, and other natural substances and do not cause environmental problems in any way.

Biodegradable polymers are classified as primary, seconder, and tertiary generation polymers. Primary generation polymers involve the mixture of low-density polyethylene, 5–10% starchers, and pro-oxidative and auto oxidative of additives. During the production of primary polymers, starch granules are blended homogeneously into low-density polyethylene. During the mixing process, microbial degradation of starch results in losses in the properties of low-density polyethylene films. At the same time, low-density polyethylene undergoes chemical degradation with oxygen. First-generation biodegradable polymers are not considered as biodegradable since it takes 3–5 years to degrade in the soil. Second-generation polymers comprise of ethylene acrylic acid, vinyl acetate, and hydrophilic copolymer additions such as polyvinyl alcohol, low-density polyethylene, and gelatinized starch. These polymers degrade for 2–3 years. Tertiary biodegradable polymers consist of fully biobased materials and are synthesized from biomass monomers by classical chemical synthesis. Furthermore, polymers extracted directly from biomass are also used in the tertiary biodegradable polymers. In addition, polymers produced by natural or genetically modified microorganisms are also used in the production of biodegradable plastics [4].

Polymers have been used in almost all industries such as the textile industry for the production of synthetic fibers. Synthetic fibers have been utilized in the textile industry for many years. In literature, it is reported that synthetic fibers lead to skin irritation for the users, environmental pollution, and abrasion to the devices. Especially, because of environmental pollution, researchers, and engineers have focused on the development of alternative sources to synthetic fibers and many natural fiber sources have been investigated. Natural fibers have many advantages which are highly specific stiffness and strength, non-abrasiveness, low cost, sustainability, easy manufacturing, biodegradability, and abundant availability. The disadvantages of natural

fibers are reported as brittleness, moisture absorption, low processing temperature, and incompatibility with polymers [5].

This chapter presents an overview of current literature compared of biodegradable materials. The aim of this chapter is to throw light on the biodegradability, biodegradable polymers, biodegradable composites, and bast fibers. This could help in the investigation in this area and using of composites in industries.

2 Biodegradable Polymers

In the polymer market, most of the polymers are originated from non-renewable and non-sustainable petroleum. Moreover, it is reported that the production of petroleum from biomass takes 106 years. Because of biodegradable and renewable composite production, polymeric matrix and reinforcement must be reproduced from the regenerated resource, commonly produced from plants that are cultivated in a period of less than a year. Commonly, these materials biodegrade in a year and time to degrade of some biodegradable materials are given in Table 1 [6].

The usage of the topic of biodegradable fiber and polymer started approximately three decades ago. Most of the research has investigated biodegradable metrics,

Table 1 Time to degrade in the environment for some biodegradable materials

Material	Time to degrade
Cotton	1–5 months
Poly(caprolactone)-g-maleic anhydride/starch	2 months
Poly(caprolactone)-starch	2 months
Waste gelatine/poly(vinyl alcohol)	1 month
Waste gelatine/sugar cane bagasse	1 month
Conventional copy paper	1 month
Waste gelatin	1 month
Waste gelatine/poly(vinyl alcohol)	1 month
Waste gelatine/sugar cane bagasse	1 month
Waste gelatine/sugar cane bagasse film	1 month
Wool stocking	1 year
Bamboo stick	1–3 years
Chewing gum	5 years
Painted wood	13 years
Plastic	450 years
Glasses and tyres	Uncertain time

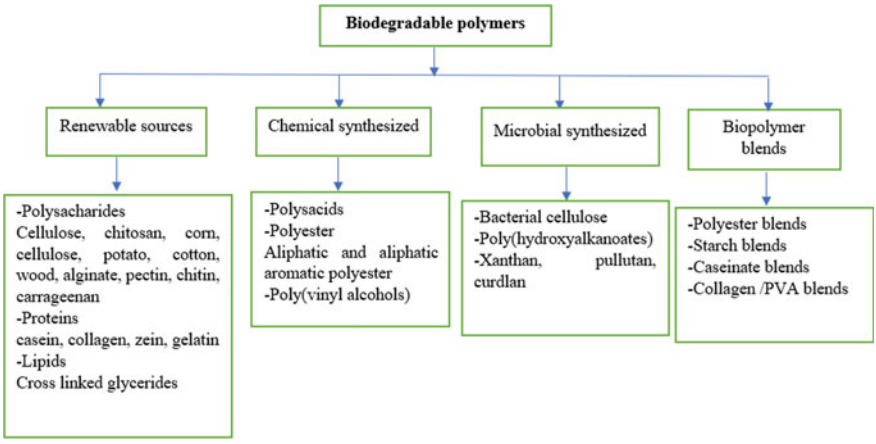

Fig. 2 The basic classification of biodegradable polymers

lignocellulosic fibers, fiber and matrix compositions, fiber extraction, biodegradable composite production, cost, mechanical and psychical properties, chemical compositions, challenges, and trends [6].

The basic classification of biodegradable polymers was given in Fig. 2.

3 Biodegradable Composites Reinforced with Bast Fibers

In the present times, there has been an increasing demand for raw materials achieved from regenerated, biodegradable, and sustainable resources, fundamentally due to the growing concern about the protection of recycling and natural sources. The manufacturing and utilization of plastic substances causes many problems regarding waste disposal owing to their environmental determination. Furthermore, the production of traditional composite structures obtained from non-biodegradable plastics reinforced with inorganic fibers is evaluated critically due to the rising environmental consciousness. Researchers have focused on the development of polymers that can degrade easily. In literature, biodegradable substances which are poly(ester amide)s, poly(vinyl alcohol), poly(lactic acid), polyester, starch, starch derivatives, polyhydroxyalkanoates, and cellulose have been researched for the manufacturing of composites reinforced with natural fibers. Many advantages are reported with the utilizing of natural fibers which are low density, high mechanical stability, low cost, lack of hazardous, and relative abundance. Bast fibers, such as flax, jute, ramie, kenaf, hemp, and sisal, are the most frequently utilized as reinforcement in polymer composites and for industrial treatments [7]. A variety of researchers have investigated the characterization of natural fiber reinforced composites. The mechanical behaviors, cultivation conditions, fiber dimension and morphology, fiber aspect ratio,

the disruption and orientation of fibers in the matrix, moisture absorption, and matrix-fiber adhesion effect on the composite properties [8]. There are many different fibers that can be utilized in the development of biodegradable composites especially bast fibers. The main ingredient of these fibers is cellulose, and the fundamental unit of a cellulose macromolecule is anhydro-D-glucose which includes hydroxyl groups. These hydroxyl groups in the macromolecule lead to making all the natural fibers hydrophilic [9].

Bast fibers comprised of cellulose, hemicellulose, and lignin are obtained from the outer bark of the plant. These fibers are formed from cellulose fibrils held together with hemicellulose and lignin. Every fibril includes a complicated structure and a thin major wall surrounding a secondary wall, which affects the mechanical behaviors of the fibers. The mechanical behaviors of these fibers are related with polymerization degree of cellulose, cellulose content in the fiber, and microfibril angle [9]. In order to produce a biodegradable composite, many matrix polymers and bast fibers are used and some of these polymers are introduced below.

3.1 Bast Fiber

At the present time, sustainable and renewable sources attract attentions in the world, and many comprehensive research have been carried out through worldwide. For this reason, characterization, and preparation of bast fibers are a crucial development and research area. In light of this information, many novel bast fibers have been comprehensively investigated [10].

Bast fibers may be described as those extracted from the outer cell layers of the stems of different plants. Among the plants utilized for cultivation of bast fibers are jute, ramie, flax, and kenaf. Because these plants are annual crops, they are getting rising interest in a variety of nonwood composite production processes. Bast fibers involve a bundle of tube-like cell walls. Every cell wall consists of primary and secondary S1, S2, and S3 layers [11]. A variety of bast fibers are reported in the literature, and some bast fibers and properties are abstracted below.

***Thespesia populnea* tree fiber:** Thespesia populnea (T.P.) tree belongs to the Malvaceae family. This tree is cultivated in Tamilnadu, India. Thespesia populnea tree fiber has 52.2 wt% cellulose, 20.5 wt% hemicellulose, and 20 wt% lignin. The crystallinity index of the fiber is reported as 41%. The TGA analysis indicates that these fibers are stable at 210 °C [5].

Jute fiber: Jute fiber is extracted from the Corchorus plant and belongs to the Malvaceae family. Jute fiber is identified as a lignocellulosic fiber that is partially wood and textile fiber. The fibers are obtained from the skin or bast of the plant. The chemical composition of jute is reported as 64.4% cellulose, 12% hemicellulose, 11.8% lignin, 0.2% pectin, 10% water, 0.5% wax, and 1.1% water-soluble. Jute fiber has a variety of cells which are the microfibrils based on cellulose. These cells are connected with amorphous hemicellulose and lignin. Jute fibers are completely recyclable and biodegradable, and eco-friendly materials. These fibers have high

thermal and acoustic insulation capacity. It is reported that the annual worldwide manufacture of jute fiber is about 3.2 million tons and these fibers have been utilized in many fields. The bag cloth industry has a high demand for jute fibers. In the world, jute bags have increased in popularity compared with nonbiodegradable poly bags that are produced from petroleum-based polymers. However, a variety of jute fibers is wasted either in the form of jut cloth or bag every year. For this reason, researchers have investigated of the recycling of jute production [12].

3.2 Okra Fiber

Okra bahmia (*Abelmoschus esculentus*) is an herbaceous plant belonging to the Malvaceae family, cultivated mainly in the tropical countries. Okra bahmia (*Abelmoschus esculentus*) bast fiber includes 60–70% cellulose, 5–10% lignin, and 15–20% hemicellulose gain from the stem of the okra plant. In the world, vegetable of plants is collected and another part of the plant is an agricultural waste product. After the collecting of vegetables, there are many waste products. For this reason, okra fiber could be a significant source of natural fiber for various applications. Furthermore, okra fiber has high mechanical properties, sustainability, and biodegradability. It has been used as reinforcement material for composites as an alternative to synthetic fibers [13].

3.3 Flax Fiber

The flax plant has approximately 80 cm height fibers all along the stem. Flax fiber is obtained from the stem or bast of the flax plant. These fibers are founded in the form of filaments which are arranged circularly around a central wooden cylinder. Flax fiber is a cellulosic, natural, and multi-cellular bast fiber that has a 40–80 μm diameter. Flax is an attractive fiber because of having high mechanical properties, low density, biodegradability, and availability. In the present day, flax is a luxury fiber that has limited manufacturing and high cost. These fibers are commonly used to produce warm weather, professional wear, and high fashion aspects [14, 15].

Kenaf fiber: Kenaf fiber is extracted from the kenaf plant and belongs to the Hibiscus cannabinus L. family. Kenaf filaments commonly include 2–6 mm individual fibers. Characteristic properties of fibers can vary from the environmental condition, age, separating technique, source, and fiber history. Kenaf fibers consist of 60–80% cellulose, 20% moisture, and 5–20% lignin, and the fibers have high mechanical properties [16].

Hemp fibers: Hemp originated in central Asia and has been known for 12,000 years. The plant is cultivated commonly in China, Central Asia, Europe, and the Philippines. The fibers occur in the stem tissue which keeps the plant erect. For this reason, the fibers are high strength and stiffness. Hemp is the most used

plant-based natural fiber for reinforcement after sisal fiber due to its high mechanical strength, sustainability, biodegradability, and ecologic. In 1941, hemp fibers were utilized as reinforcement for the body of the Henry Ford car; however, the car did not produced commercially because of economic limitations. During the Second World War the production of synthetic fiber reinforced composites increased, but ecological concerns eventuated in interest in natural fiber reinforced composites again [17].

Kusha fibers: Kusha grass is cultivated in Asian countries, and kusha fiber is obtained from kusha grass. It is reported that kusha fiber comprises of 70.58% cellulose, 14.35% lignin, and 1.52% wax. The density of fiber was measured as 1.1025 g cc^{-1}. X-ray diffraction analysis reveals that the crystallinity index of fiber is 55.4%. Thermogravimetric analysis shows that the fibers are stable up to 357 °C [18].

***Pennisetum Purpureum* fibers:** *Pennisetum Purpurem* belongs to the Poaceae family called as Napier grass. This plant is cultivated in tropical and subtropical regions worldwide. *Pennisetum Purpureum* fibers were extracted from this plant and the fibers have 54.26% cellulose, 18.85% hemicellulose, 25.33% lignin, and 5.93% water. Thermal stability occurs in the ranges of 120–330 °C. The fibers have high mechanical behaviors [19].

4 Biodegradable Matrix Polymers

Polymeric materials can be produced from regenerated sources such as plant cellulosic fibers, vegetable oil, bacteria as well non-regenerated petroleum (polyester). Biodegradable polymers can be classified as origins that are natural or synthetic. Furthermore, these polymers can be categorized based on their origins such as agro polymers (cellulose or starch), microbial (poly(hydroxyalkanoate)), polymers synthesized from agro-based sources (poly(lactic acid)) and synthesized from conventionally synthesized monomers. Some of the most used matrix polymers were introduced below.

Poly(butylene succinate): Poly(butylene succinate) is one of the biodegradable polymer which is synthesised with butanediol and succinic acid. These two polymers originate from bio-based regenerable sources. PBS, which is a thermoplastic polymer, has melting point at about 90–120 °C and glass transition temperature of about −10 °C to −45 °C. Moreover, PBS is known as commercially available, biodegradable, high thermal and chemical resistance. However, softness, high price, and gas barrier properties limit the application of PBS. Due to decreasing of the PBS limited usage, PBS is reinforced with natural fibers which are low density, high mechanical properties, low cost, sustainable, renewable, and biodegradable [20].

Polylactide: Polylactide (PLA) is a biodegradable polymer generally synthesized cyclic lactide dimer. PLA can be manufactured with polycondensation of lactic acid. In the second way of PLA production, PLA is achieved with low molecular weight. The raw material of PLA is a renewable natural product which is obtained from corn starch. PLA polymer is commonly used in commercial packing material due to

regenerated and having good mechanical properties. However, some disadvantages which are high prices and brittle character in thicker material limit the usage of PLA compared with other thermoplastic polymers [9].

Starches: Starches produced with conventional methods are natural hydrophilic polymers. Starches have low melt processability and are high hydrophile, for these reasons, a plasticizer needs them to make them suitable for using engineering fields. In this context, glycerol and water can be used as plasticizers to make starches suitable for the thermoplastic process. In the thermoplastic process, the melting point and glass transition temperature of starches diminish owing to the heat and mechanical energy. With using fillers which are cellulose-based natural fibers, the durability of starches can be increased [9].

5 Biodegradability Testing

In industry, laboratory testing methods are utilized to determine biodegradability, a crucial factor for the evaluation of the eco-friendly behavior of substances. Biodegradability is an important parameter because a biodegradable substance will lead to no longer risk in the environment.

Biodegradation test of plastics: In order to determine of biodegradation of plastics, ISO 14852 method is utilized. The ISO 14852 method is a plastics-specific method used to determine the aerobic biological grade in an aqueous matrix by measuring CO_2, ISO 14852 analysis typically takes between 6 months and 28 days and compares the biodegradability of the test sample against the control samples.

Soil Burial Test: The soil burial test is used to determine the biodegradation of textile fibers. According to the burial method AATCC 30–1993, natural soil is used to fill the pot to a depth of 11 cm. The prepared samples are first weighed, buried 3 cm from the bottom, and allowed to reduce for 28 days after the weight of the samples was measured.

6 Bast Fiber-Based Bio-Composites

Composite material, which has recently introduced itself to the world in a short time, has become indispensable in many daily areas such as space-air systems, automotive, and sports. Generally, composite materials, although they can be designed in various forms, are mostly formed by the reinforcement of glass, carbon, aramid, or ultra-high molecular weight polyethylene (UHMWPE) fibers to a polymer matrix such as epoxy, polypropylene, polyethylene, etc. In addition to the advantage of increasing the use of composite materials, the wastes consumed for the material to be formed create a problem. Furthermore, recycling composite materials is hard because of forming two materials which are fiber and matrix. Disposal and usage of these composites occur environmental problems. Furthermore, sustainability and cost of traditional

composite raw materials may be a problem in the future. In order to reduce environmental problems, a variety of attempts have been carried out to use biodegradable polymers in the composites as reinforcement. These disadvantages cause to production of bio-composites. Environmental studies that promote themselves as green composites also focus on the composite material being made from renewable resources. The common usage areas of bio-composites are the construction, automotive, furniture, and packing industries. In the manufacturing of bio-composites, bio-fibers (generally cellulose-based fibers) and bio-matrix are used. Bio-composite materials can be produced with different techniques which are hand lay-up, spray-up, filament winding, resin injection, and pultrusion [21].

Hand Lay-Up Method: The matrix is impregnated on the fiber layers by manually laying them on a mold with reinforcing fabrics prepared in woven or broken form. Before the fiber is put into the mold, the mold surface is cleaned. The matrix is applied to the material at the final stage, where it is important that the matrix penetrates into the fibers optimally [21].

Spray-Up Method: This method is the manual laying method with mechanical parts. The fibers are mixed with resin by a hardener. Due to the roughness that may occur on the surface after spraying, the surface is prepared by smoothing it with the help of a roller [21].

Fiber Winding Method: The fiber winding method is used for mass production of products with a special shape. In this method, fibers are continuously heated with resin and pulled out of the reel. With the increase of the wrapped fiber layers, the product hardens and the rotating mold is separated from the product. Materials such as tanks and pipes with circular geometry are generally produced with this method [21].

Resin Injection Method: In this method, a two-piece mold is used. Felt, fabric, and both of these are used as reinforcement elements. The reinforcement material is placed in the mold in such a way that it fills the mold and is closed. It is coated with late-dissolving resins in the matrix to prevent the fibers from being dragged into the mold. Matrix injection can be applied in cold containers, warm or up to 80°. Due to the closed mold, harmful gases are reduced. This method is used in the manufacture of complex parts [21].

Pultrusion Method: Pultrusion process continuous constant section composite profile. It is a low-cost mass production method in which the products are produced. The reinforcement material is introduced into the resin bath before the forming mold is heated up to 120–150°. In this method, the orientation of the fibers affects the strength of the material [21].

The increasing number of studies have been performed on the growing significance of the new bio-composites. Some of these studies were summarized in Table 2.

Table 2 Properties of some bast fiber-based bio-composites are investigated in the literature

Fiber	Bio-based matrix	Properties investigated	Reference
Pineapple leaf	Polycarbonate	– Mechanical behaviors – Thermal stability	[22]
Ramie	Poly lactic acid/poly caprolactone	– Tensile and impact strength	[23]
Coir	Natural rubber	– Tensile strength	[24]
Bamboo	Natural rubber	– Tensile strength	[25]
Flax	Polylactic acid	– The interfacial characterization	[26]
Kenaf	Poly lactic acid	– Tensile strength – Storage modules	[27]
Kenaf	Poly-L-lactide	– Storage module – Thermal stability – Crystallinity	[28]
Hemp	Poly lactic acid	– Glass transition temperature – Melting point – Mechanical behaviors – Thermal stability	[29]
Short flax fiber	Poly(ε-caprolactone)	– Mechanical behaviors – Crystallinity	[30]
Flax	Poly(ε-caprolactone)	– Crystallinity – Tensile strength – Stroge module	[31]
Jute	Polybutylene succinate	– Weight loss – Biodegradability	[32]
Hemp and kenaf	Cashew nut shell	– Tensile properties – Porosity – Fracture surface	[33]

7 Biodegradability

Biodegradability is described as the decomposition ability of material after interactivity with biological micro-organisms. Biodegradable material refers to a substance that can be converted into basic substances such as CO_2 and water after being biodegraded with the help of micro-organisms in nature and participate in the cycle in nature. Due to biodegradation, time limit and compostability are not required. Biodegradation occurs after the biological interaction in the biological environment and can be classified as enzymatic and hydrolytic biodegradation. In enzymatic degradation, an enzyme is required actively for degradation. Enzyme includes a specific functional group that can interact with a biodegradable polymer chain. Most of the biodegradable polymers contain unstable, easily hydrolysable functional groups such as amin and ester. When the biodegradable substrates are placed in a physiological environment, water diffuses into this polymer matrix and hydrolyses polymer chains to break them down. Biodegradation of products is removed from the matrix surface

with diffusion. Some properties such as water permeability and unstable hydrolytic bonds fundamentally determine the rate of hydrolytic degradation and surface erosion occurs. When the rate of water penetration into the polymer is greater than the rate of water diffusion, the polymer is deteriorated by surface erosion. The biodegradation of polymers by hydrolysis can be catalyzed using acids, bases, and enzymes [34].

A variety of factors affect biodegradation, and these factors can be organized below;

Hydrophilicity: Hydrophilicity can be identified as the attraction to water, while repellent of water is assigned as hydrophobic. Hydrophilic molecules are known as polar molecules [35].

Chemical structure: Chemical structure is defined as the arrangement of atoms in a molecule (in a radical or ion with atoms) and the chemical bonds. The structure theory was developed in about 1860s because of the determination of the chemical structure of the benzene derivatives. In order to determine the chemical structure of any substituent, the constitution and empirical chemical composition of the molecule can be known.

Hydrolysis mechanism: Hydrolysis of a substance commonly occurs in the environment. Hydrolysis is a significant reaction in the ground, surface, fog, and porewaters. Frequently, the hydrolysis mechanism occurs in two types which are nucleophilic substitution and addition–elimination. In nucleophilic substitution commonly takes place in epoxides and phosphate esters. Addition–Elimination frequently takes place when the leaving group is connected to sp^2 hybridized acyl carbon group, such as with carboxylic acid derivatives consisting of anhydrides, esters, amides, urease, and carbamates.

Glass transition temperature: Glass transition temperature is defined as the temperature that the carbon chains start to move. At the glass transition temperature, the amorphous regions experience the transition from a rigid state to a more flexible state making the temperature at the border of the solid-state to rubbery state. It is believed that the gap between the molecular chains rises by 2.5 times at this temperature [36].

Molecular weight: The molecular weight is defined as the mass of a molecule. Distinctive molecules of the same substance may have different molecular weight since they consist of different isotopes of an element. Molecular weight is calculated from the atomic weight of each molecule.

Environmental condition: Environmental conditions affect the biodegradability of substances. Most substances are affected by the moisture and temperature of the environment. Especially, the substance having hydrophilic groups tends to absorb moisture compared with the substance without hydrophilic groups. The biodegradation of these substances enhances with the increase in moisture of the environment.

Porosity: Porosity is defined as the ratio of the total volume of the material to the total pore volume in the material. The porosity of a material can be calculated with this equation;

$$h = \frac{V_p}{V}$$

where h is the porosity, V_p is the total volume, and V is the total pore volume of material. The increase in the amount of porosity represents the increase in the air of material [37].

8 Hydrolytic and Enzymatic Biodegradation

Biodegradation mechanism occurs via abiotic (such as photodegradation, simple hydrolysis) or biological (such as enzymes) sources. Hydrolytic biodegradation is defined as the forming of oligomers and monomers resulting in the chemical bonds in the polymer main chain combining with water. Hydrolysis reaction materializes with acids, alkalis, and enzymes. After the biomaterial is placed in the degradation environment, it absorbs water and swells, and biodegradation proceeds from the outer surface of the material to the inner surface. Hydrophilic or hydrophobic character of polymer affects the degradation. The susceptibility of the polymer to hydrolysis follows; (i) hydrophilicity of hydrolysable bonds, (ii) hydrophobicity of hydrolysable bonds, (iii) hydrophilicity of non-hydrolysable bonds, and (iv) hydrophobicity of non-hydrolysable bonds. Biodegradable polymers which are esters, glycosides, orthoesters, anhydrides, amines, urethanes, and urea involve hydrolysable bonds. Polymers with strong covalent bonds in the main chain (such as C–C bonds) and without hydrolysable groups require a very long time to degrade. Polyesters, polycarbonates, polyanhydrides, polyurethanes, polyorthoesters are some of the polymers subject to hydrolytic degradation. Different types of hydrolysis mechanisms have been investigated and it was obtained that it is hydrolysed in the main chain as well as in the stalactite groups. With enzymes, the degradation occurs from the substance surface to the inside. Enzymes are biological catalysts that accelerate the rate of reaction in living organisms without undergoing any permanent changes. Without enzymes, a variety of reactions in cellular metabolism does not become. Hydrolysis reactions can be catalysed with many enzymes which are protease, esterase, glycosidase, and phosphatase. In this context, some of these enzymes are expected to play an important role in the biodegradation of biomaterials by catalysing hydrolysis reactions. For example, the degree of biodegradation of polyurethane was observed to be approximately 10 times faster in the presence of cholesterol esterase enzyme than in phosphate buffer solution [38].

9 Conclusion

The current generation has focused on environmental preservation because of the increasing environmental concern. Furthermore, a variety of investigations have been carried out with the use of natural fibers in enhancing thermoplastics and thermoset composites in order to develop their mechanical behaviors. Recently, natural fibers especially bast fibers drawn attention as an alternative to synthetic fibers owing to having many properties which are low density, high specific strength, high stiffness, low cost, biodegradable, sustainable, and ecological.

Composite materials are defined as the combination of two or more different materials to manufacture a new engineered material. The mechanical behaviors of composites depend on the ratio of reinforcement, the interfacial adhesion between reinforcement and matrix, the mechanical behaviors of matrix and reinforcement, the surface modification of fibers, the fiber orientation and dimension, test condition, and volume fraction. Crucial progress has been achieved in the advance of biodegradable composites which are like conventional products. Availability, low cost, and biodegradability gains many advantages to these composites. Biodegradable composites are in a world of depleting resources. With the increasing climate crisis, the demand for recyclable materials will increase even more and it is deemed that biodegradable composites will be here to stay. In the present day, bast fibers are frequently used in the production of biodegradable composites. Because of climate change, researchers and industry may focus on discovering novel plant-based fibers to reinforce polymer composites.

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Free Vibration Characteristics of Bast Fiber-Based Polymeric Composites

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Abstract Fiber-reinforced composites have been widely used in industries for their enhanced stiffness and damping characteristics. Recent advancements in the use of biodegradable or natural fiber reinforcements in composites have gained acceptance worldwide because of their biodegradability and as well as favorable mechanical and vibration properties. This article reviews the free vibration characteristics of various bast fibers (jute, flax, hemp, kenaf, ramie, roselle and banana) incorporated composites. The natural frequencies of composites reinforced with these fibers have been studied under various weight percentages, fiber treatments, fiber layering patterns, fiber orientation, and hybridization with secondary reinforcements and so on. Notable improvements in both natural frequency and damping have been observed by many authors on varying the mentioned parameters. This chapter gives an insight into the selection of composite constituents for better vibration characteristics.

Keywords Free vibration · Natural frequency · Damping · Natural fiber · Bast fibers

1 Introduction

High usage of synthetic materials and the new sustainability progress have made researchers and industries move for natural fiber instead of synthetic fibers due to their environmentally friendly and degradable nature [1]. Natural fibers have low density, considerable tensile strength, high moisture absorption rate and lower processing temperatures [2, 3]. Fiber reinforced composites replace conventional polymers because the fiber acts as the load-bearing element in the material [4]. These fiber-reinforced composites attract various industries like aircraft, automobile, food, environmental, civil, and marine [5–7]. This shows that natural fiber-reinforced composites have a great future ahead of conventional synthetic polymers. The chemical treatment of the fiber increases the mechanical properties and surface

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roughness of the fiber which contributes to enhanced properties of resulting composites [8]. These fiber-reinforced polymers are manufactured using many methods such as injection moulding, resin moulding, 3D printing, and hand-layup method [3, 9, 10]. The recycling of fiber-reinforced composites is also possible, and this will also lead to a circular economy and a sustainable environment [11, 12].

Vibrations are very important in certain industries like aviation, automotive, marine, civil, etc. [13, 14]. The usage of fiber-reinforced polymers is increasing in every industry, so the study of vibrations in the fiber-reinforced composite is very important. In particular, the understanding of the properties like damping, frequency, and resonance will be important. The natural frequency of the fiber should be high to avoid resonance at the same time the stiffness of the fiber should be maintained. The angle of the fibers is one of the important factors that influence the natural frequency of composites [15, 16]. The damping value of fiber-reinforced polymer is mostly higher than the polymer matrix because of the viscoelasticity of the polymer [17]. The stacking sequence of the composite affects the damping of the composite [18], and temperature also makes some considerable differences in the damping [19].

2 Classifications of Natural Fiber

Natural fibers are derived from three different sources, and they are classified accordingly as plant/vegetable fiber, animal fiber and mineral fiber. Plant fibers are mainly classified into wood and non-wood fibers. Further non-wood fibers are classified based on their derivations from the plant such as bast/stem, leaf, seed/fruit, stalk and grass/reed [20]. Bast and leaf fibers are used commonly compared to other fibers because of their high mechanical property. Generally, animal fibers comprise animal hair and silk fibers. Animal fibers are classified into Keratin and Non-Keratin-based fibers. Keratin-based fibers include sheep wool while Non-Keratin based fibers include fur-type fibers and even human hair. Mineral fibers include asbestos [21] (Fig. 1).

3 Experimental Modal Analysis

Modal analysis is performed to determine the natural frequency and damping ratio of the natural fiber incorporated polymer composites. The free vibrations of the composite beams are studied in cantilever conditions where one end is fixed and the other end is free [23]. The system is assigned with one degree of freedom for simplicity reasons. An impulse hammer is used to induce the vibrations in the setup. The response of the specimen is detected by an accelerometer, which measures the displacements/vibrations. The signals that are recovered from the transducers are

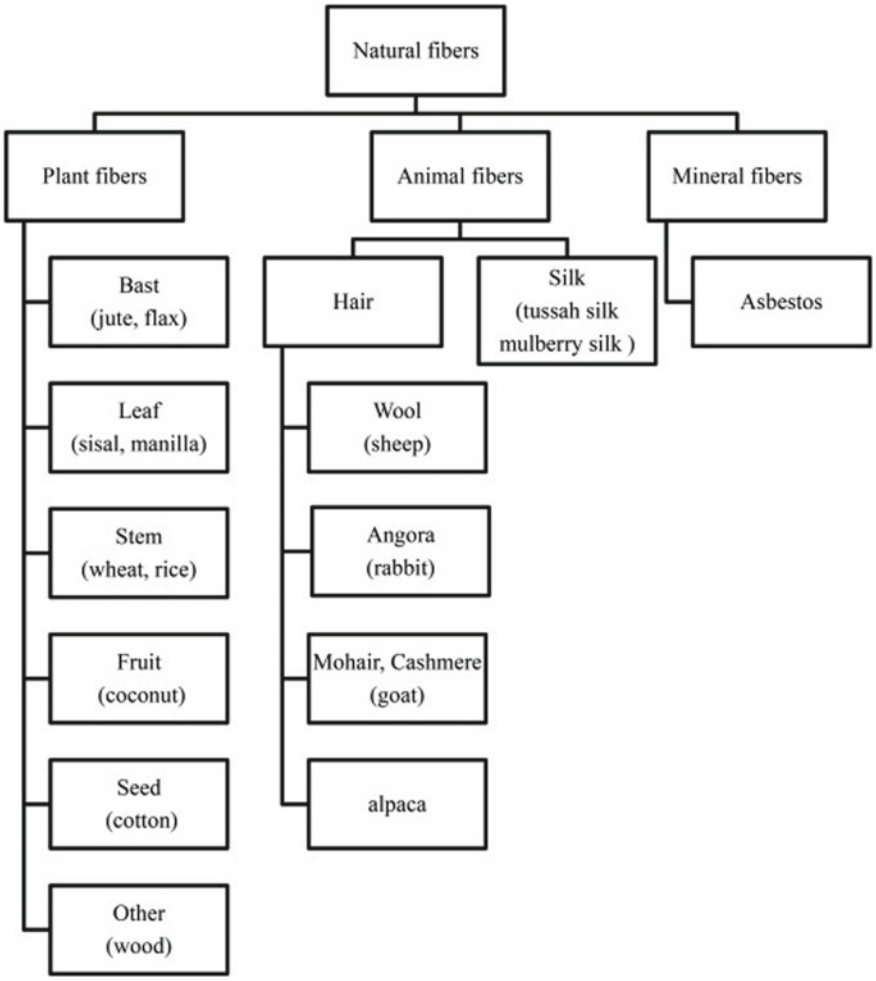

Fig. 1 Classification of natural fibers [22]

analyzed and processed by a data acquisition system (DAS) and stored in a computer [24] (Fig. 2).

4 Factors Affecting the Natural Frequency of Natural Fiber Composites

Composites reinforced with natural fibers exhibit variations in their natural frequency and damping ratio based on various factors. The primary factor comprises the proper

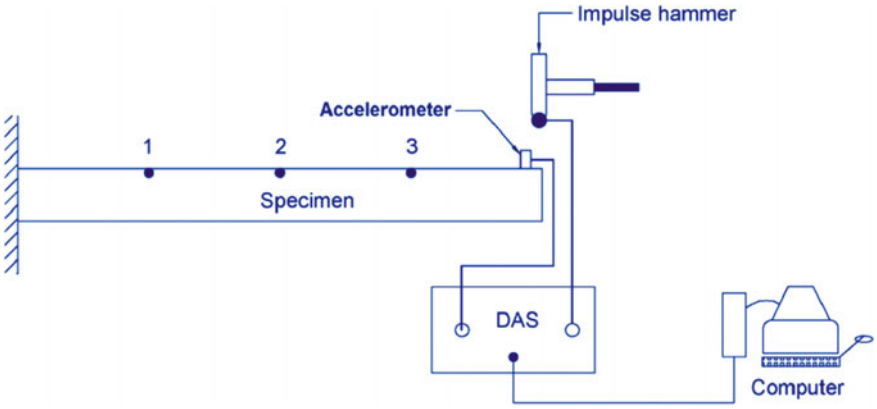

Fig. 2 Experimental modal analysis—a line sketch [25]

selection of matrix and reinforcement materials. As with any material, the natural frequency depends on the stiffness and mass of the material. Hence, the greater the stiffness and lower the mass of the composite, the greater the natural frequency of the composites. The stiffness, mass and quality of fibers depend on growth conditions, dimensions, age, extraction and processing. The polymer, natural fiber and any filler used must be compatible with each other [26, 27].

Fiber-matrix adhesion plays an important role in deciding the vibration behavior of the composites. Due to the hydrophilic nature of fibers, they generally have poor adhesion with polymer matrices. Chemical treatment of the fiber enhances fiber-matrix adhesion and positively affects the stiffness. Chemical treatment usually involves mercerizing the fibers in alkali solutions like NaOH, $KMnO_4$ and so on. Also, the curing temperature plays role in determining stiffness. The lower the curing temperature, the better the stiffness [28, 29]. Fiber weight percentage influences the fiber-matrix interface and hence the natural frequency of the composite. Fiber length also affects the stiffness of composite as it increases/decreases surface to contact area ratio between interfaces [24]. Furthermore, the aspect ratio, layering pattern and orientations of fibers in the composites determine its natural frequency [25, 30].

5 Free Vibration Characteristics of Bast Fiber-Based Composites

5.1 Jute Fiber-Based Composites

Jute is a type of bast fiber that is primarily cultivated in Asian countries like India, Bangladesh, etc. Jute is a very cheap alternative to synthetic materials, and also it has high strength and is eco-friendly [31, 32]. Researchers have found that the chemical

Table 1 Natural frequency of jute fiber based composites

Matrix	Reinforcement (s)	Natural frequency (Hz)	References
Epoxy	Untreated jute	36	[13]
Polyester	Untreated jute	24.34	[10]
Polyester	Untreated jute	38.08	[37]
Epoxy	5% NaOH treated jute	45.6	[13]
Epoxy	5% NaOH treated jute + 5% nano-clay	50	[38]
Epoxy	Jute + 4% Zno	37.67	[36]
Epoxy	Jute + 4% TiO_2	38.99	[36]
Polyester	Jute + Sisal	24.07	[10]
Polyester	25% NaOH treated jute + Glass	19.17 ± 0.64	[39]
Polyester	Jute + 2 wt.% Nano-clay	44	[38]

treatment of jute increases the mechanical properties [33]. Jute fiber is mostly used in the textile industry and also usage of jute is growing in the industries like automobile, civil, and sports [34]. Automobile companies like Mercedes Benz have used jute fiber composite in manufacturing door panels [35]. Researchers and industries have shown interest in jute fiber as an alternative to synthetic fiber.

Epoxy is reinforced with jute fiber which shows the natural frequency of 36 Hz, to increase the natural frequency of the composite the jute fiber is treated with 5% NaOH. The natural frequency of the composite increases to 45.6 Hz due to the removal of pectin, cellulose, waxy substance and improved interfacial adhesion between the layers of the composite. To further increase the frequency researchers have introduced the secondary reinforcement (nano-clay) which increases the stiffness of the composite which results in the frequency of 50 Hz [13]. Another work reported that the natural frequency of the composite depends upon the temperature and percentage of filler materials. The natural frequency and stiffness are high at 4% filler materials due to the fiber-matrix interaction. Beyond the addition of 4% of filler material the stiffness of the composite decreases [36]. The natural frequency value of various jute fiber-based composites is given in Table 1.

5.2 Flax Fiber-Based Composites

Flax is one of the most widely used bio-fibers in various domains such as marine, aerospace, sports, etc. [40]. The use of flax as reinforcement has increased dramatically due to the requirement for the development of sustainable materials as a part of increasing environmental concerns. The researchers have observed an increase in vibrational properties of composites when flax is used as a reinforcement in the polymers. The flax-epoxy composite material dissipates more strain energy than the

Table 2 Natural frequency of flax fiber based composites

Matrix	Reinforcement(s)	Natural frequency (Hz)	Damping ratio	References
Epoxy	Flax	–	0.014	[44]
Polypropylene	Flax	26.95	–	[16]
Neoprene rubber	Flax	15.85	–	[45]
PLA	Flax	238	4	[46]

carbon-epoxy laminate [40]. The flax fiber composites compared to glass fiber epoxy composites and carbon fiber composites have much better damping properties, particularly in the frequency range of 60–100 Hz [41]. The addition of flax fibers in the polypropylene can increase the frequency from 7.59 Hz to 26.95 Hz and it occurs in 0° because the fibers are stiffer. These composites have higher damping capability and thus these can be used in applications where low weight and high damping are the major requirements [16]. Flax fiber-reinforced composites have exceptionally excellent vibration and damping behavior at low frequencies [42]. These damping factors of flax are due to the internal friction produced by cellulose and hemicellulose present in the fibers [40–42]. The results from Table 2 yield that the flax-reinforced composites can be used commercially in places where noise and vibrations are major issues and significant damping is required [43].

5.3 Hemp Fiber-Based Composites

Hemp fibers are being used in automotive, furniture, building industries as a substitute for glass and carbon fibers for the reinforcement of thermoplastic matrices. These are used as reinforcement because they are cheap and have low density, high strength-weight ratios [47, 48]. Hemp natural fiber reinforced polypropylene (PP) composites are used in the automotive industry to lessen the dependence on petroleum-based fuels and products and replace existing materials with higher specific strength and lower cost [46]. A study showed usage of 40% hemp in a composite has a higher damping capacity than 100% polypropylene, and hence it is used as a reinforcement and an addition of 5% PP-MAH gives faster decay than the latter. The 30% hemp-added composites can be used in vehicles where vibration is a major concern. The 30 wt.% hemp fiber-reinforced polypropylene having 2.5 wt% POE–MAH has the highest damping ratio among coupled composites (Table 3).

Table 3 Natural frequency of hemp fiber-based composites [49]

Matrix	Reinforcement(s)	Natural frequency (Hz)	Damping ratio
Polypropylene	30 wt.% hemp	11.68 (±0.41)	0.140 (±0.006)
Polypropylene	60 wt.% hemp	11.81 (±0.10)	0.046 (±0.003)
Polypropylene	40 wt.% hemp + 2.5 wt.% PP-MAH	13.21 (±0.26)	0.053 (±0.017)
Polypropylene	30 wt.% hemp + 2.5 wt.% PP-MAH	11.72 (±0.15)	0.083 (±0.006)
Polypropylene	30 wt.% hemp + 5 wt.% PP-MAH	13.88 (±0.18)	0.055 (±0.008)

5.4 Kenaf Fiber Based Composites

Kenaf (Hibiscus cannabinus L.), a member of the Malvaceae family can grow up to 4 m with stem diameter ranging from 10 to 20 mm. Its characteristics have made it desirable in many applications. Kenaf bast fibers have the lowest amount of cellulose, the highest proportion of lignin among other plant bast fibers. Kenaf has a very low density (1200 kg-m^{-3}) compared to other plant bast fibers (approximately 1500 kg-m^{-3}) and has good fiber fineness. It has good specific tensile strength and low variability of Young's modulus (fibers with low variability in mechanical properties are desirable). On account of these properties, kenaf composites have found wide application in automobile parts such as door panels, trays, mats and many more in companies such as General Motors and Toyota; in medical applications like orthodontic brackets and dental posts; and in geotextile applications [50].

Composites consisting of kenaf also have good vibration damping properties. Natural frequencies at Mode 1–3 of various composites containing kenaf as reinforcement are listed in Table 4. Khandai et al. [51] in their experiments assessed the properties of flax/kenaf/glass/carbon and found Mode 1 natural frequency of kenaf to be 23.6 Hz. Ismail et al. [52] observed improved free vibration characteristics in the hybrid composites produced with kenaf (primary reinforcement) and bamboo (secondary reinforcement) fibers. The hybrid composites performed better than the composites with kenaf alone as reinforcement. Among the various compositions of kenaf and bamboo, the specimen with 30% kenaf and 70% bamboo showed optimal natural frequencies at Modes 1–3 and also the damping factor. Similarly, another work reported that increasing kenaf fiber composition increased the natural frequency and internal damping of the composite due to increased stiffness [53].

5.5 Ramie Fiber-Based Composites

Ramie fiber is native to China and is also called China grass. It can be woven into textiles, easily laundered and has an increase in strength when it is wet and does not

Table 4 Natural frequency of Kenaf fiber based composites

Matrix	Reinforcement(s)	Natural frequency (Hz)	References
Epoxy	60% of Kenaf	23.6	[51]
Epoxy	40% of Kenaf	65.92	[52]
Epoxy	60% of 6% NaOH treated Kenaf	82.84	[53]
Epoxy	Kenaf + bamboo (70:30)	78.13	[52]

lose its shape. It finds its applications in the making of industrial sewing thread and is also made into fabrics for household furnishings and clothing, frequently in blends. The viscoelastic behavior of ramie fiber added composites has a positive effect on the damping ratio [54]. As a result, the damping ratio increases with the increase in weight percentage of ramie fiber. With the increase in the weight percentage (%) of ramie fiber in polypropylene composites, the natural frequency increases [55].

5.6 Roselle Fiber-Based Composites

Rossele fiber is extracted from the rossele plant which is found in Borneo, Guyana, Malaysia, and Sri Lanka, etc. [56]. This type of fiber has high tensile strength due to the high content of cellulose. Rossele fiber can be used in industries like civil, marine and consumer goods. Low cost, availability, high strength are some of the advantages of this fiber [57]. In addition to this, the roselle fiber composite is used in Orthopaedic applications [58]. A study proved that the stacking sequence of the fiber influences the natural frequency and damping ratio of the composites. Rossele fiber has a naturally high damping property compared to jute, rossele absorbs a good amount of energy during vibration [59].

5.7 Banana Fiber-Based Composites

Bananas are one of the most globally exported fruits and are the fourth most important food crop produced (Banana Market—Growth, Trends, COVID-19 Impact, and Forecasts (2022–2027)). Hence, Banana plant fibers are abundantly available. Many studies have shown that bast fibers from the banana plant have good mechanical properties and their incorporation in synthetic composites improves both biodegradability and various other properties. Vibrational behavior is a very important property sought after in these fiber composites due to their need in various applications. Work done by some authors on banana fiber composites: untreated, treated and hybrid are detailed below.

Rajesh et al. [29] studied the free vibration characteristics of banana/sisal fibers reinforced hybrid composite and inferred that the properties were enhanced by increasing the fiber volume to 50% by weight in the epoxy-fiber composite. Increasing further would only depreciate the properties because of poor bonding between fiber and matrix. However, the hybrid composites without surface treatment performed poorly compared to sisal fiber composite or banana fiber composite amid banana fibers being weaker than sisal fibers. After treating the composites with 1% NaOH, good improvements in properties were observed as a result of increased adhesion between fiber and matrix.

Dodiya and Venkatachalam [60] performed dynamic analysis experimentally and using ANSYS on banana fiber reinforced hybrid composites. They treated the fibers with three solutions: NaOH, KOH and $KMnO_4$, respectively and tested their vibration characteristics. They found that stiffness improved notably and this was attributed to an increase in fiber volume and fiber treatment. Also, the stiffness decreased on increasing the curing temperature. The best natural frequency was obtained when the fiber is treated with NaOH and cured at room temperature. Chandrasekar et al. [30] investigated the effect of banana fiber inter-ply orientation on the free vibration properties of composites and found that both Mode 1 natural frequency and damping changes with fiber orientation. The [0º/90º/0º] orientation had maximum Mode 1 natural frequency. Sumesh and Kanthavel [61] investigated the effect of TiO_2 nano-fillers on the vibrational behavior of hybrid composites consists banana (primary reinforcement), sisal and pineapple fibers. In both these studies, they observed that the addition of nano-fillers up to 3% improved vibrational properties due to reduced voids and adhesion between fiber–fiber and fiber-matrix interface, while above 3% decreased these properties due to void content and agglomeration.

Arumuga Prabu et al. [27] observed the influence of red mud on banana reinforced composites and reported that untreated fiber composites incorporated with red mud had better vibrational properties than those treated with NaOH and Silane. This is attributed to low compatibility between treated fiber and red mud. Senthil Kumar et al. [25] determined that not only stiffness of the material, but layering patterns also affects the vibrational behavior of hybrid fiber composites by testing the banana fiber (primary reinforcement) and coconut sheath hybrid composites. Various layering patterns have been experimented, and it was found that the CBC (coconut sheath-banana fiber-coconut sheath) arrangement had the maximum natural frequency.

6 Conclusion

The natural fiber is a promising alternative to synthetic fibers because it is biodegradable, easy to manufacture, and strong. These fiber-added composites have a high natural frequency which is further increased by chemical treatment and secondary reinforcements. Notable improvements in both natural frequency and damping have been observed by many authors on varying the composite parameters. The bast fiber added composites showed favorable natural frequencies and these composites could

be used in automotive, aerospace and other industrial applications where the materials are subjected to vibrations. However, vibration-related studies are limited, and they could be explored in greater depth to aid in the selection of appropriate materials for vibration-related applications.

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Characterization of Bast Fiber Composites Added with Nano Reinforcements

Ayushi Kushwaha, Deeksha Jaiswal, G. L. Devnani, and Manash Protim Mudoi

Abstract Natural fiber reinforced composites (NFRC's) are novel materials whose demand is increasing day by day due to their unique advantages like sustainability, ecofriendly nature, availability of raw material, diverse applications, etc. Bast fiber composites are sub-category of these NFRC's in which natural fiber is extracted from the stem of the plant. Flax, kenaf, hemp, jute, and ramie are the major bast fibre plants. Extensive work has been done on the composites developed with these bast fibers during the last decade. Being hydrophilic in nature the adhesion of bast with hydrophobic matrix is the key issue that has drawn the attention of the scientific community. Surface treatment methodologies of bast fibers like chemical treatment (alkali, silane, acetylation, etc.), physical treatment (corona, plasma), and biological treatment (enzyme, algae) have been applied to these bast fibers to overcome these issues [5]. Nano reinforcements are also providing phenomenal changes in the field of composite as well as material science. In nano reinforcement a little quantity of nano material is mixed with a variety of bast fiber composites which can enhance the performance and quality of bast fiber composites drastically. Scientists have used different types of Nano clays like montmorillonite, closite, garamite, and various nano fillers mainly Nano SIO_2, CNTs, etc., and other nano reinforcements in bast fiber reinforced composites for enhancement in performance. Characterization with sophisticated analysis equipment for the performance evaluation after the addition of these nano reinforcements is a very important aspect of commercialization and comparison of these novel materials with synthetic fiber reinforced composites. Present compilation reviews of state of art characterization techniques by advanced analysis equipment like mechanical testing, dynamic, water absorption, Thermogravimetric analysis (TGA), XRD, FTIR, etc.

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1 Introduction

In the last few decades, composite materials such as thermoplastics, thermosetting, and nanofillers are now becoming the predominant materials when reinforced with natural fibers, a promising substitute for synthetic fibers [10, 11, 15, 27]. The extensive use of lignocellulosic fibers to make composite has acquired an important phase recently. The use of lignocellulosic fiber over synthetic fiber has several advantages due to its lower density and less cost per volume [22]. Many natural bast fibers [13] such as hemp, kenaf, ramie, and more are used in composites with the matrix. With the evolution in green technology, the demand for biodegradable substances is leading to research on their utilization in various sectors such as packaging, manufacturing, medicines, automobiles, and many more. The forest and agriculture residues are now utilized as a promising reinforcement material in polymer-based composites. The lignocellulosic fibers have drawn the attention of material scientists, researchers due to their easy availability, low cost, specific mechanical and thermal properties, eco-friendly aspect. Bast fiber [18] is sub-category of natural fiber in which fiber is extracted from the stem of the plant [17]. Though bast fibers have some disadvantages like poor matrix-fiber adhesion and poor moisture resistance. However many approaches have been taken to reduce their drawbacks and also modify their surfaces. There are various chemical treatments such as alkali treatment, acetylation, benzoylation, and more that are approached to modify the surface for better compatibility between fiber and matrix.

At present, nanofillers are high potential and promising filler material to improve the physical and mechanical properties [19] of the composite [2]. Due to the homogeneous distribution of nanofillers, they result in a better filler-matrix surface area [6, 9, 29]. Nanofillers are prone to defects hence their utilization in various fields of material science applications and polymer composites are setting up the new gravitate. Nanocomposites are high-performance materials that possess high fatigue strength, unique design possibilities, and better impact energy. These nanofillers can be organic and inorganic such as titanium dioxide, silica, cellulosic nanofillers, and carbon nanomaterials. Their application in construction and building is a perfect substitute for traditional materials.

Composite reinforcement is a technique that improves the various characteristics of the composite such as mechanical properties, thermal properties, and structural properties. The composites contain a matrix of polymer and various fibers such as natural and synthetic. The reinforcement of the fiber can be oriented, random, or layered. Composites are made up of reinforcement materials such as particulates, fibers, and fillers incorporated with the matrix of polymers, metals, or ceramics. The major function of the matrix is to hold the reinforced substance to produce the structural identity of the composites. The literature work suggests the importance of

BFCs (Bast fiber composites) incorporated with different nanofillers possess better characteristics for their applications in various fields.

1.1 Nano Fillers

Nanofillers are predominantly used due to their significant performance to improve comparisons properties of the matrix when incorporated with bast fibre. The mechanical, electrical, thermal, and morphological properties can be improved with the aid of fusing nanofillers in natural fibre-based composites. Nanocomposites are termed as one of the members of the family of nanomaterials where nanofiller is in the dispersed medium. They are classified as one-dimensional, two-dimensional, and three-dimensional nanocomposites on the basis of the type of nanofillers (Fig. 1).

i. One-dimensional (1-D) nanofiller: As plates, laminas;
ii. Two-dimensional (2-D) nanofiller: As nanofibres (Diameter < 0.1 μm), Nanotubes
iii. Three-dimensional (3-D) nanofiller: As isodimensional nanoparticles

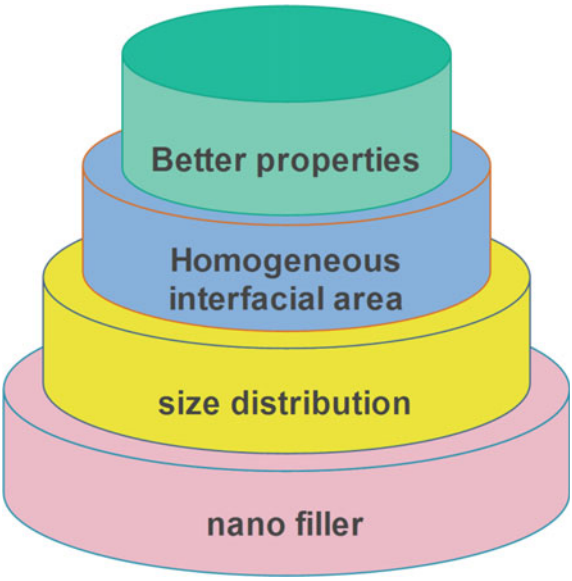

Fig. 1 Impact of incorporation of nanofillers with bast fibers

1.2 Some Important Nanofillers

1.2.1 Nanoclays

Nanoclay is one of the most predominant nanoparticles to make bast fibre-based polymer nanocomposites. They are used majorly due to their abundant availability in nature. They are layered silicate. The nanoclays can be classified into the following four groups:

(a) Keolite group (zeolite or halloysite),
(b) Illite group,
(c) Montmorillonite group,
(d) Chlorite group.

The major purpose of nanoclays is classified on the following basis, First is to reduce the cost of composite and another is to enhance certain properties. A small amount of nanofiller is able to alter the property of the composite to the greater. Nanoclays can be utilized for various purposes in agriculture as nano fertilisers. The rapid application of nanoclays in agro biotechnology has beneficial results. They can be fused with bast fibers to prepare high mechanical strength composites having a great physical appearance. Nanoclay incorporated natural fiber composites have better interfacial surface finishing. Nanoclays are grouped in accordance with their crystalline structure and also the position of elementary ions wit in the mesh. These can be natural clays, synthetic, and also as the transition metal phosphates. Koo (2006) explained that organic silicates have a modified layered structure that has a better compatibility of fiber and matrix hence, organic silicates are preferred over inorganic ones. The most commonly used polysilicate is Magadiite ($Na_2Si_{14}O_{29}H_2O$).

1.2.2 Nano Oxides

The major nano oxides which are used as nanofillers are titania (TiO_2), silica (SiO_2), and alumina (Al_2O_3). These nanoparticles have small range dimensions but higher surface area.

(a) **Nano Titanium Dioxide (TiO_2) or nano titania:**

The nanoparticles of titanium dioxide can be generated by the process of hydrolyzation of titanium tetrachloride. They are also called fumed titania. They can vary with the structure depending on the type of fiber is fused with. Titania has a better photocataytic effect that aids to improve the optical properties. When Titania is fused with bast fibers, they result in the enhancement of certain mechanical properties of BFCs (Bast fiber composites).

(b) **Nano Silicon Dioxide (SiO_2) or nano Silica:**

Silica nanoparticles are generated by the process at higher temperatures by hydrolysis of $SiCl_4$ (Silicon Tetraoxide). Unmodified silica nanoparticles contain silanol groups

that possess (-OH) Hydroxyl groups can result in the degradation of mechanical properties; hence to improve this factor, we can use thermal treatment of silica. After the eradication of (-OH) groups by the heating medium, these can result in better compatibility between fiber and nanofillers with no hydrophilic character. The excessively used nano-silica is diatomite that occurs naturally.

(c) **Nano Alumina (Al_2O_3):**

Alumina nanoparticles can be produced by the hydrolyzation of Aluminium trichloride ($AlCl_3$) at high temperature. They can be either hydrophilic or hydrophobic in nature. Their hydrophilic nature can be eradicated by reacting them with silanes. They can be used as inert fillers in the BFCs and they are in spherical structure.

(d) **Carbon Nanotubes (CNTs):**

CNTs are the allotropes of carbon having cylindrical nano structure. They are the members of fullerene family. CNTs can be obtained by the process of vapor deposition at approx. 1000 °C over methane (CH_4).

There are mainly three kinds of carbon nanotubes on the basis of their diameter.

i. Single-walled CNTs or called [SWCNTs] (size range 1–2 nm dia)
ii. Double-walled CNTs or called [DWCNTs] (size range 2–4 nm dia)
iii. Multi-walled CNTs or called [MWCNTs] (size range 4–150 nm dia)

CNTs owe remarkable mechanical, thermal, and electronic properties. CNTs are one of the best nanofillers used as an additive in the polymer composites. CNTs can vary in the different classes like length, number of layers, and thickness. For the growth of CNTs, the presence of a catalyst is essential. The catalyst is prior deposited on the surface of CNTs to be grown. The CNTs have extraordinary properties due to their young's modulus that ranges till 1 TPa. One difficulty with the CNTs to be used as dispersed medium is its dispersion in the polymer matrix but after being dispersed, their interfacial interaction is excellent with better mechanical properties. The small quantity of CNTs coupled with silica can result in excellent mechanical properties of the NFCs (Natural Fiber Composites).

(e) **Nanographene:**

Graphene is the single-layered two-dimensional honeycomb-like structure where each carbon atom is sp^2 hybridised. This structure of graphene provides it with better mechanical, thermal, and electrical conductivity as well as high stiffness. The major drawback of graphene is its extraction from graphite. However, by exfoliation, nanographene can be obtained using intercalation agents under low heat. The intercalation agents used are of two types:

i. Ionic compounds (Graphite-Bromine and graphite-Lithium)
ii. Covalent compounds (Graphite oxides)

Intercalation is possible only in the graphitic carbon compounds. The direct dispersion of nanographene is effective in the polymeric matrix when the polymer and nanographene are soluble in same solvent. This can be achieved by the process

of sonication. nano graphenes have higher young's modulus value (1 TPa), hence exhibit excellent properties and also enhances the specific properties of the polymer composites when added.

2 Bast Fibers/Nanofiller-Based Hybrid Composites

Bast fibers are excellent alternative of manmade synthetic fibers for reinforcement in polymers to develop composite materials. Nanotechnology is a field that is affecting almost all the fields of engineering and science by its advantageous outcomes. Composite manufacture is also largely affected [6, 8, 27, 29]. With the advent of nano materials [23]. The properties were inferior due to poor interfacial adhesion between bast fibers and polymer matrix as compared to synthetic fiber-based polymer composites. This shortcoming was overcome by the incorporation of nano clays/fillers which has an ability to improve the adhesion and provide stiffness and rigidity to the composite material [14]. developed hemp fiber-based polyester composites and compared the performance after incorporation of Closite 30 B nano clay material. Improvement of 6% in tensile modulus was observed with the inclusion of nano clay. The percentage of closite was 1.5% and fiber content was 21%. [24] developed epoxy composites with the infusion of laboratory-made natural nano filler oil palm empty fruit bunch fibre. The percentage of nano filler was kept in the range of 1–5% and was mixed with high-speed stirrer. Maximum value of thermomechanical properties was observed at the filler loading of 3% loading. Enhancement in performance was due to good interlocking and adhesion between filler and matrix [30] fabricated flax-based epoxy composites. They used nano sized TiO_2 as nano filler for the improvement in the performance of composites. The content of the nano-filler was kept from 0.89 wt.% to 7.14 wt.%. Measurement of tensile strength and interfacial strength was done to optimize the nano filler loading. An increase in tensile strength and interfacial shear strength of 23.1 and 40.5% was observed at an optimized filler loading of 2.34%. Significant improvement in properties is possible if composites are prepared efficiently with optimized loading of fiber/filler content.

2.1 Interfacial Adhesion of Bast Fiber and Nanofiller-Based Hybrid Composites

The Bast fiber and polymer matrix adhesion is harmed by the incompatible nature of fiber and matrix as the fiber is hydrophilic and the matrix is hydrophobic which ultimately gives the poor mechanical characteristics of fabricated composites. Significant work is done on solving this issue like loading optimization, length of fiber, chemical treatments, physical and biological treatments [4, 5, 15, 27, 29]. In spite of

huge benefits bast fibres are still facing issues like low values of modulus, deterioration high water absorption, and non-uniformity in physical and mechanical behavior compared with synthetic fibre-based composite materials. Nano-materials are extensively applied nowadays as reinforcement in bast fiber-based composites because of their high form factor. The incorporation of nano-fillers enhances the overall performance of bast fiber-based composites [23]. The mechanical performance of nano-filler-loaded bast fiber composites largely depends on the interaction at interfacial among the components. Better dispersion of nano-filler in the various polymer matrix is responsible for better stress and load transfer among them.

3 Characterization of Bast Fiber Composites Added with Nano Reinforcements

With the growing importance of nano reinforcement in bast fiber composites and their potential in performance improvement it is essential to characterize these novel materials extensively to use them in diverse fields of applications. The traditional and advanced characterisation techniques are discussed as follows.

3.1 Mechanical Characterization

The tensile testing analysis is used to measure the amount of tensile force that is required to pull the sample to the point where it breaks down. Tensile tests of various nano reinforced bast composites are done in a Universal testing machine (UTM). For fiber testing, a gauge length of 30 mm is used with a crosshead speed of 1 mm/min as per following the standards like ASTM D3822-07. In the case of composite materials the tensile testing is conducted as per standard ASTM D3039-14. The tests are carried out on a flat specimen (140 × 15 × 2.6 mm^3) at a crosshead speed of 2 mm/min following the guidelines as per standards. The flexural testing is done to evaluate the force required to bend the sample under three-point loading conditions. Flexural strength tests are also performed on a flat specimen (127 × 12.7 × 2.6 mm) following the standards like ASTM D790-10 using the same UTM. An impact test is performed to measure the ability of a material to bear shock and impact energy without breaking. The impact (Izod) strength use to be determined using un-notch samples (64 × 10 × 2.6 mm) according to the ASTM D256 standard using pendulum impact testing machine. The average value of measurements was taken for the test results of three different specimens of each composite specimen. In Fig. 2 tensile property values of unmodified and flax fibre modified with nano TiO_2 Epoxy composites are shown. The tensile strength value of the modified bast fiber composite increased by 14.70% from 68 MPa for unmodified fibre composite to 78 MPa by addition of 0.4 wt% nano TiO2 coated flax fibre composite. Tensile modulus values also increased with

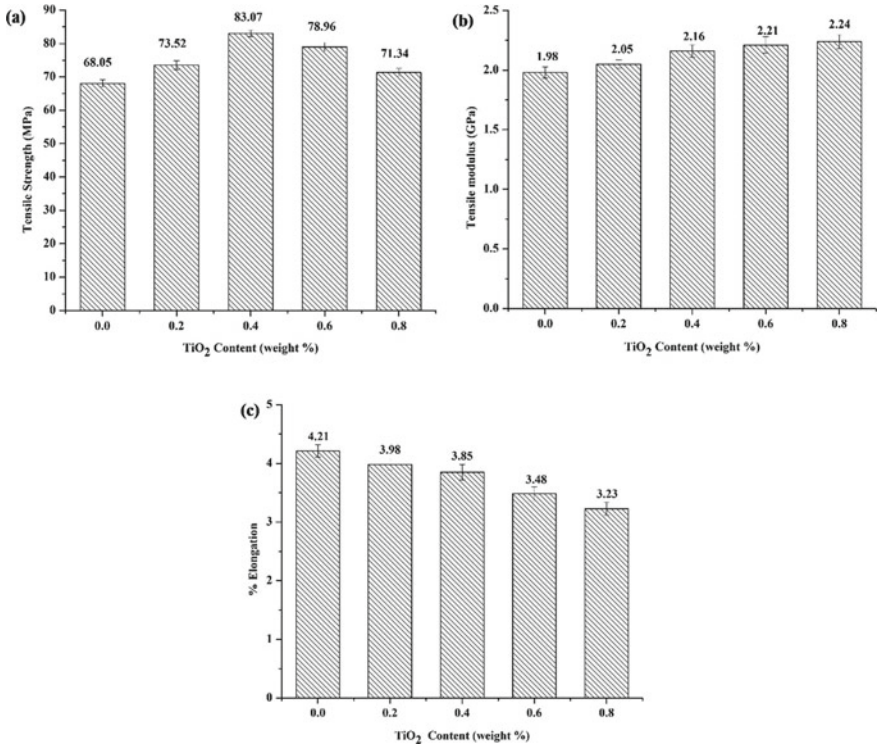

Fig. 2 **a** Tensile strength **b** tensile modulus and **c** percentage elongation values on the effect of nano TiO_2 coating on composites fibre modification [21]

the nanomaterial coating on the flax fibre surface, depicting the improvement in the stiffness values of the composite with modified fibres. It can be concluded that fiber modification by nano particles significantly improved the tensile properties of flax (Bast) fiber reinforced epoxy composites. Similar improvements were also observed in flexural property values shown in Fig. 3 [21].

3.2 Water Absorption

Water diffusion/absorption analysis of bast fiber composite materials use to be conducted in agreement with (ASTM D570-98) standards. Samples of dimension 76.2 × 24.6 × 2.6 mm are dried in oven at 50 °C until an unchanging weight of sample is obtained. After that composite samples are dipped in the beaker/vessel of distilled water and brought out at regular intervals, wiped with the help of tissue paper, and weighed in a very high precision weight balance of 0.001 g.

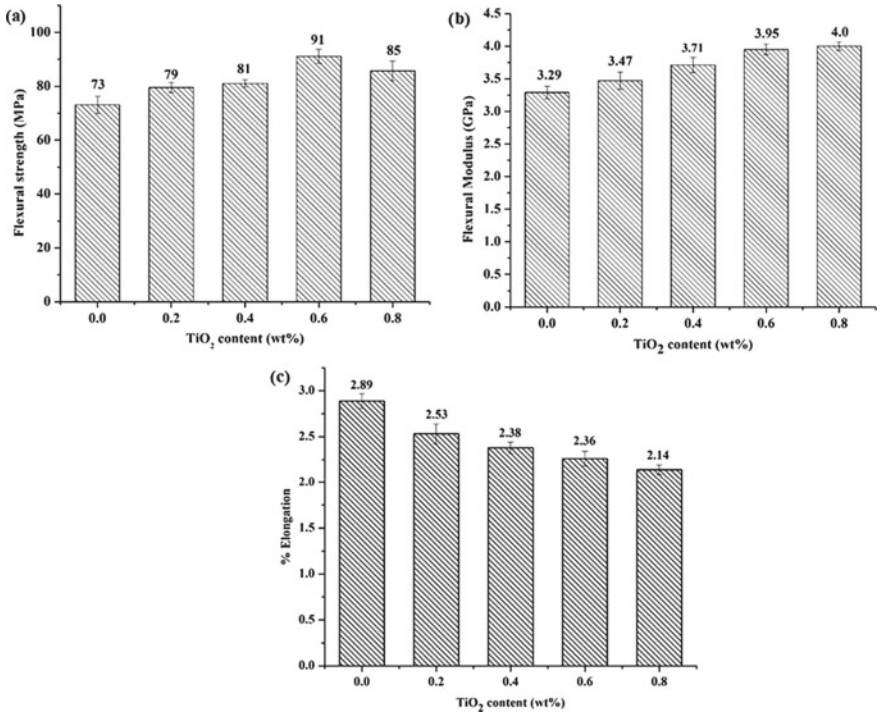

Fig. 3 **a** Flexural Strength **b** flexural Modulus **c** percentage elongation values for nano TiO_2 coated flax fibre composites [21]

The water diffusion/absorption tendency in bast fiber composites follows both Fickian as well as non Fickian behavior. The coefficient of diffusion is one of most important parameter for the understanding of water absorption behavior represents ability of the water molecules to penetrate inside the specimen structure. Following is the fundamental equation [7]

$$\frac{M_t}{M_m} = kt^n$$

where M_t is the % of the water absorbed in the sample at time t, M_m is the maximum % of the water absorbed, and k and n are the kinetic constants. Taking the natural logarithm of above equation we have

$$log\left(\frac{M_t}{M_m}\right) = log(k) + nlog(t)$$

where the value k signifies the interaction behavior between the bast fiber composite sample and the water and n represents the nature of diffusion behavior as Case I (Fickian diffusion $n = 0.5$) and Case II (non-Fickian diffusion $n > 1$). The diffusion coefficient or diffusivity for the water absorption can be calculated from the following mathematical equation:

$$\frac{M_t}{M_m} = \left(\frac{Dt}{\pi h^2}\right)^{1/2}$$

where h represents the thickness of the composite sample. The value of D gives the rate of water absorption and can be calculated from the slope/gradient of the linear part of the graph of M_t versus the square root of time t.

Prasad et al. [21] showed the comparison of water behavior of modified and unmodified flax fiber-based epoxy composites at different nano filler (nano TiO_2) fraction and it can be easily analysed that the chemical (silane) treatment and nanofiller reinforcement application significantly reduces the water absorption (saturated) value of the bast fiber-based composites. The tendency shows an appreciable decrease in water absorption due to fibre modification. 0.6 wt% nanofiller (TiO_2) coated flax fibre composites showed considerably reduced amount of water absorbed at the similar analysis conditions. In case of fibre modified composites it is concluded that the number of hydroxyl groups which were free on the fibre surface is diminished substantially by the action of SCA and nano TiO_2 reinforcements. Reducing water absorption is highly desirable to upgrade the quality of campsite material (Fig. 4).

3.3 Thermogravimetric Analysis

Thermogravimetric analysis (TGA) method is applied to study the thermal stabilities of bast fibers and their reinforced composites at higher temperature. In TGA, the mass and the rate of change in the weight of a specimen is used to measure as a function of time or temperature under controlled atmosphere. Thermogravimetric analyzer method is used to study the thermal degradation behavior of the untreated and treated bast fibers and their composites. Samples weighing between 9 to 10 mg use to be placed in an alumina pan and tests are performed at different heating rate normally 10 °C/min in programmed temperature range of room temperature − 700 °C under inert gas (nitrogen) atmosphere at pre-decided flow rate [31] performed thermogravimetric analysis (TGA) to analyse the thermal stability of PP/Kenaf/ PP-g-MA/MWCNTs nano-biocomposites. The results shows the values of the different weight loss temperatures and second degradation peak temperatures increased after addition of multi walled carbon nano tubes because of barrier effect of the MWCNTs. Table 1 is showing the summary of thermogravimetric analysis and corresponding diagrams are shown in Fig. 5

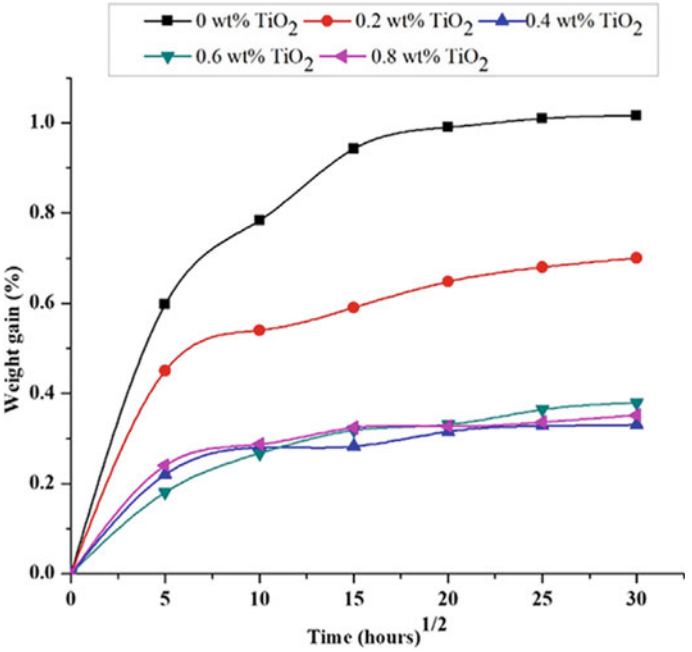

Fig. 4 Water absorption behavior unmodified and fibre modified composites at varying nano TiO_2 weight percentage [21]

Table 1 Peak temperatures from DTG curves and TGA results for PP/kenaf/PP-g-MA/MWCNTnano-biocomposite [31]

Samples	T30% (°C)	T60% (°C)	T90% (°C)	First peak (°C)	Second peak (°C)	Residual weight at 600 °C
PP/K30/M5	415.00	453.33	475.00	360.00	458.33	6.05
PP/K30/M5/N0.5	420.41	456.21	477.02	360.00	460.83	6.57
PP/K30/M5/N1.0	424.98	458.25	478.10	359.28	462.61	7.09
PPlK30/M5/N1.5	427.50	459.16	479.16	360.00	463.33	7.13
PPlK30/M5/N2.0	425.00	460.83	482.50	361.66	465.00	8.60

3.4 X-ray Diffraction

It is a non-destructive analysis procedure for delineating crystalline materials. It gives the analysis about phases, and different structural characteristics such like crystallinity, medium grain size, and crystal cracks. The peak intensities are attributed the atomic positioning inside the lattice planes. The atomic and molecular structure of crystalline material can be examined by X-ray diffraction (XRD, in this analysis the crystalline atoms of the test material cause a beam of incident X-ray to diffract into

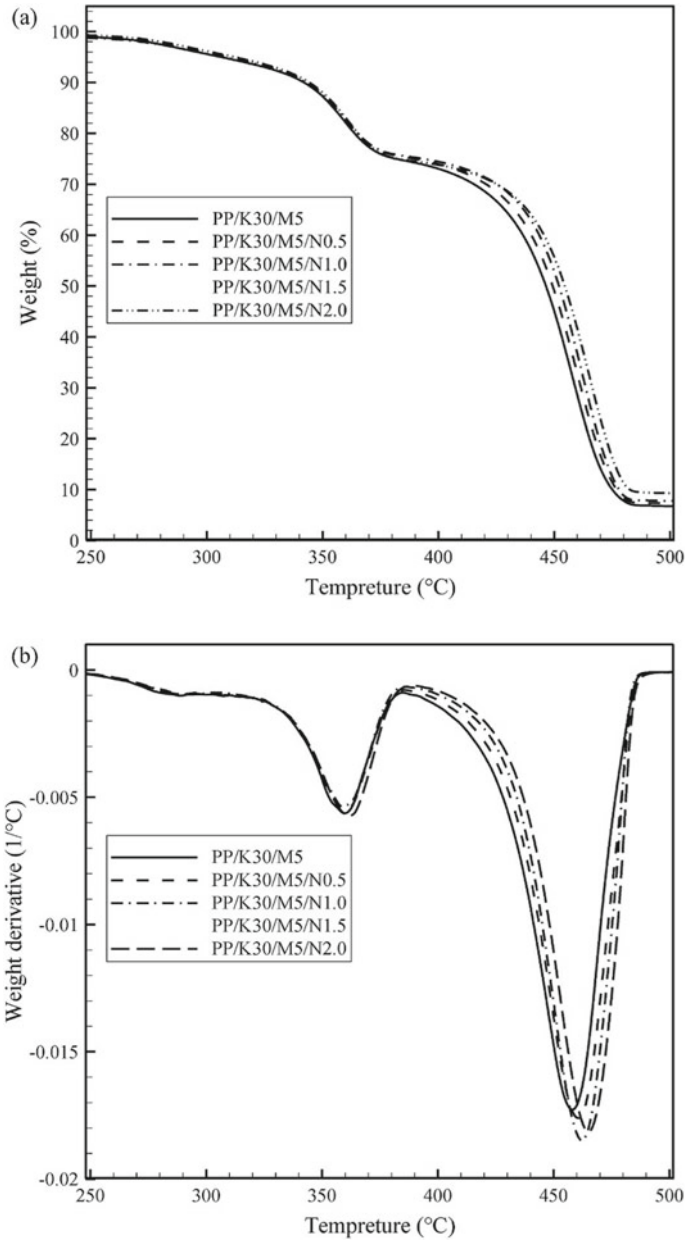

Fig. 5 **a** TGA and **b** DTG curves of PP/kenaf/PP-g-MA biocomposites as a function of MWCNT content [31]

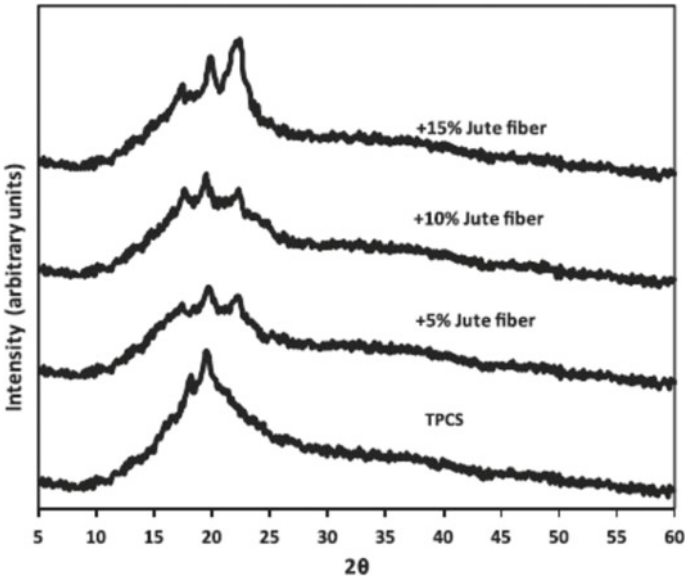

Fig. 6 X-ray diffraction pattern of TPCS/jute fiber composites with different jute fiber contents [20]

many specific directions. XRD is an important tool to evaluate the crystallinity of the untreated and treated bast fibers and composite with and without nano reinforcements containing these fibers. The XRD patterns can be recorded on a diffractometer. The crystallite size (L) can be calculated according to the Scherrer's equation:

$$L = \frac{K\lambda}{\beta cos\theta}$$

Prachayawarakorn et al. [20] Showed the XRD patterns of TPCS/jute fiber composite.

It was observed from Fig. 5 increased in loading of jute fiber upto 15% increased the XRD intensity of composites and ultimately having improved crystallinity.

3.5 Fourier Transform-Infrared Spectroscopy

In FTIR analysis technique, wavelength absorbed by the material in Infrared region is measured. This is obtained by exposure of material in Infrared radiation. The composition and structure of the material is analysed by the tendency to absorb different wavelengths. Unknown materials are analysed by matching the spectrum with the reference spectra database. This Analysis can be applied to evaluate unidentified

material, additives present in polymers and their composites. The analysis gives idea about the molecular composition and structure of the material [12].

Interferometer, a simple device is utilized to identify materials by involving optical signal contains all the wavelengths in IR zone. The signal can be measured and analysed quickly. Fourier Transform, a mathematical technique is used for the decoding of the signal. This automated process generated the spectral information of the material. The obtained graph is the spectrum which matched against reference information for evaluation and identification. It is a qualitative analysis technique to identify the chemical bonds present in untreated and chemically treated bast fibers and their composites with nano-reinforcements by producing an IR spectrum.

Figure 7 is showing the variations due to MWCNT incorporation in the nano-biocomposite, of kenaf fiber and poly propylene with the help of FTIR spectroscopy [31]. In the case of nano reinforcement samples all in all, the Infra-red spectra shows almost identical changes to those of PP/kenaf/PP-g-MA. This analysis tells that interactions occurring between MWCNTs and PP/kenaf/PP-g-MA bio composite are primarily physical.

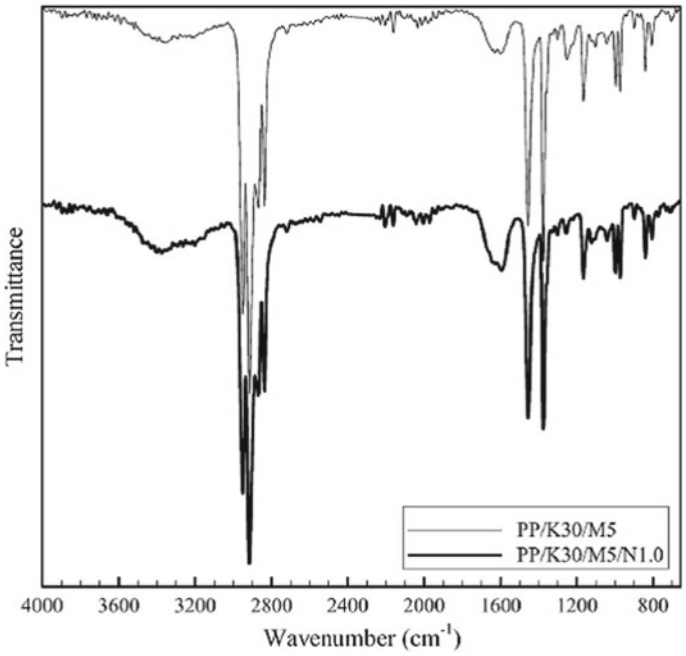

Fig. 7 FTIR spectra of PP/kenaf/PP-g-MA and PP/kenaf/PP-g-MA/MWCNT [31]

3.6 Scanning Electron Microscopy

Focused beam of high energy electrons are used to produce signals at the surface of material in scanning electron microscopy. These signals give idea about the morphological structure and chemical composition of the material. In most of the observation a small area (1 cm to 5 micron) of the sample is exposed and a 2D micrograph is produced which displays spatial variations. Magnification in this imaging varies from 20 X to 30,000 X along with spatial resolution in the range of 50 to 100 nm. With the help of EDS, chemical composition can also be analysed. It is an important analysis technique to understand the morphology of the untreated and treated fiber bast fibers surface as well as the interface between the bast fiber and the matrix. It also examines the effect of nano reinforcement on the morphology of bast fiber composites.

Figure 8 is showing the effect of nano TiO2 on the morphology of flax fiber reinforced epoxy composites.

Improvement in properties due to nano reinforcements is summarized in Table 1 taking consideration of work done on these materials in Bast fiber composites.

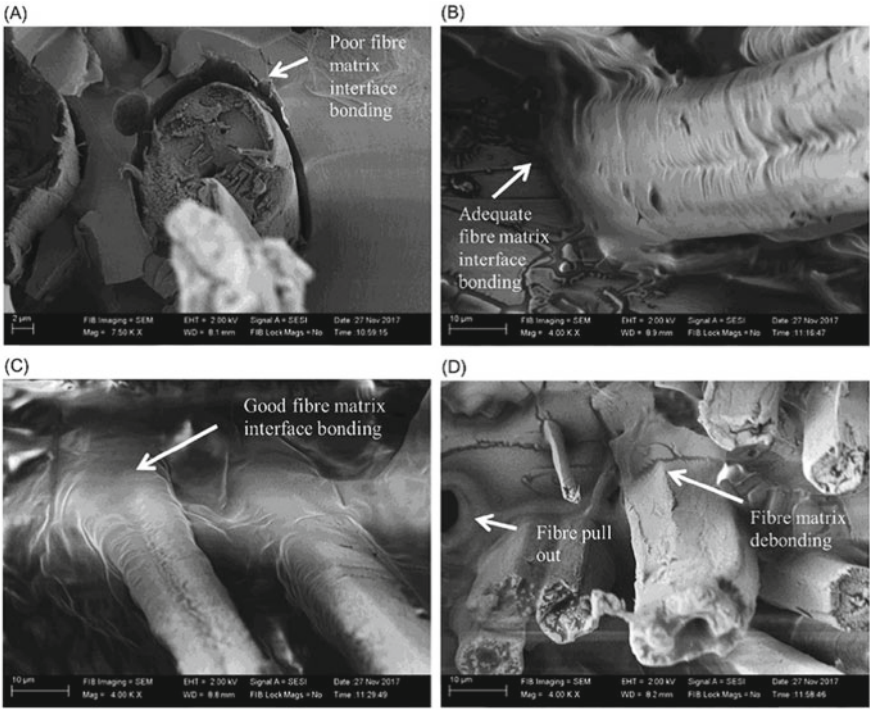

Fig. 8 SEM images exhibiting flax fiber and matrix interface of composites with TiO_2 addition by (**a**) 0 wt.%, (**b**) 0.5 wt.%, (**c**) 0.7 wt.%, and (**d**) 0.9 wt.% [3]

Table 2 Improvement in properties of bast fiber reinforced composites due to nano reinforcements [9]

Matrix	Fiber	Nanofiller used	Loading percentage of fiber and clay	Method	Improvement in mechanical and water absorption properties property as compare to absence of nanofiller	Reference
Polypropylene	Wood flour	Montmorillonite (MMT)-based nanoclayCloisite 20A	30% fiber loading and clay loading is from 0 to 6% and coupling agent maleated polypropylene from 0 to 7.5%	Injection molding	(a) Improvement in tensile strength by 20% and flexural strength by 13% (b) Composites prepared with 3% nanoclay exhibited lower water absorption	Ashori and Nourbakhsh [1]
Unsaturated polyester resin partially substituted by epoxidized soybean oil	Hemp	Cloisite 30B	21–22% fiber loading and clay loading is 0 to 1.5%	Compression molding	(a) Decrease in ultimate tensile strength by 20% (b) Moisture absorption reduced by 8%	Haq et al. [14]
Urea Formaldehyde	Wood	Nano -SiO_2	20% wood and 1% nano SiO_2	Pressurized impregnation	(a) Most of the properties have been improved (b) Decreased water absorption	Jiang et al. [16]

(continued)

Table 2 (continued)

Matrix	Fiber	Nanofiller used	Loading percentage of fiber and clay	Method	Improvement in mechanical and water absorption properties property as compare to absence of nanofiller	Reference
Epoxy	Ramie	Carbon nano tubes (CNT)	Carbon nano tube addition from 0 to 0.6%	Hand lay up	Flexural strength and flexural modulus improved by 34% and 37%	Shen et al. [26]
Epoxy	Jute	Graphene	25% fiber and 0.3,1,3% graphene	Hand lay up	Graphene as nanofiller enhanced the machinability of composite material	Sridharan et al. [28]
Epoxy	Kenaf	Oil palm nano filler, Montmorillonite	3% nano filler	Hand lay up	By addition of oil palm nano filler 24.9% increase in tensile strength and 28.3% increase in impact strength	Saba et al. [25]
Epoxy	Flax	Nano TiO2	2.34% nano filler		Interfacial shear strength to an epoxy resin were enhanced by 23.1% and 40.5%, respectively	Wang et al. [30]

4 Conclusion

Nano reinforcements like nanoclays, nano TiO2, and other nano fillers like CNTs are bringing exemplary improvements in the performance of bast fiber-based composites. State of art analysis techniques are very important for the performance evaluation of these composite materials. The positive effect due to these nano reinforcements on the tensile, flexural, impact, morphological, and thermal stability of bast fiber composites can be analysed clearly with the help of these characterization techniques. This will also facilitate the enhancement in the commercialization of these novel materials.

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