

Chapter 2

Literature Review

Abstract Due to specific advantages over synthetic counterparts, plant-based natural fibers are considered promising candidates for reinforcement in polymer composites for certain applications. This chapter deals with the classification, structure, and chemical composition of plant-based natural fibers. Some important aspects of jute fiber and its use as reinforcement in polymer composites are discussed. The use of cellulose nano fibrils as a filler in polymer composites is overviewed. Different chemical, physical, and biological methods for surface modification of natural fibers are discussed in detail. At the end, the importance of creep property in polymer composites and some important creep models have also been dealt with.

Keywords Plant fibers • Cellulose nano-fibers • Natural fiber composites • Surface modification

2.1 Natural Fiber Composites

The use of natural fibers as reinforcement in polymer composites is constantly increasing. Currently, the use of natural fiber composites is limited to interior and non-structural applications due to their poor moisture resistance and low mechanical properties (Dittenber and GangaRao 2012). These are being used in architectural, furniture, and automotive industries (Araujo et al. 2008). However, the research is underway to expand their applications by encountering the challenges associated with the use of natural fibers in polymer composites. A brief understanding of the nature, classification, and composition of natural fibers is presented in the following sections.

2.2 Natural Fibers and Their Classification

Fibers can be classified into two groups on the basis of production of fibrous polymers and production of fibers: natural fibers and man-made fibers. Natural fibers are those which occur in nature in the form of fibers, whereas man-made

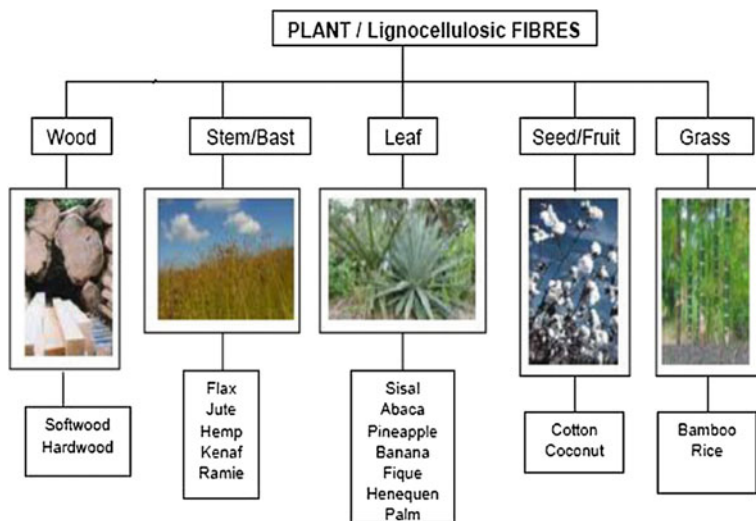


Fig. 2.1 Classification of plant-based natural fibers used as reinforcement in composites (Chandramohan and Marimuthu 2011)

fibers are those which are produced by spinning from polymer prepared by humans (synthetic fibers) or occurring naturally (chemical fibers). Natural fibers are further classified according to the nature of their source into vegetable/plant, animal, and mineral fibers. Plant-based natural fibers are mostly used as a reinforcing element in composites which are further classified as shown in Fig. 2.1, on the basis of their origin. Synthetic fibers whose feedstock are fossil fuel are the leading causes of environmental degradation due to the toxicity of the emitted fumes and non-biodegradability, whereas natural fibers have advantages such as biodegradability, renewability, low cost, and non-toxicity.

2.3 Chemical Composition and Structure of Plant-Based Natural Fibers

The elementary plant fiber is a single cell having length ranging from 1 to 50 mm and diameter from 10 to 50 μm . Plant fibers are like microscopic tubes, i.e., cell walls surrounding the central lumen. The lumen is usually responsible for the water uptake behavior of plant fibers (Tsoumis 1991). The fiber contains several cell walls. These cell walls are formed from oriented semicrystalline reinforcing cellulose microfibrils embedded in a matrix of pectines, hemicellulose, and lignin of varying composition. Such microfibrils have typically a diameter in the range of 10–30 nm and are made up of 30–100 cellulose molecules in extended chain conformation and provide mechanical strength to the fiber. The typical arrangement of fibrils, microfibrils, and cellulose in the cell walls of a plant fiber is shown in Fig. 2.2.

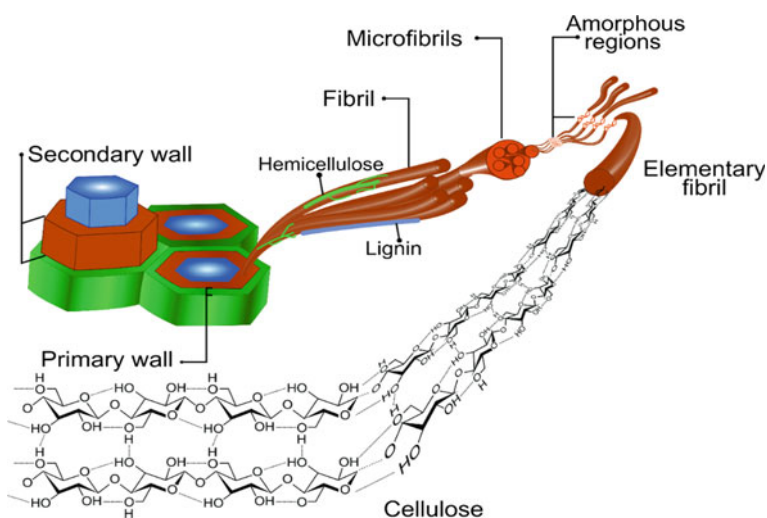


Fig. 2.2 Arrangement of fibrils, microfibrils, and cellulose in the plant cell wall (Rojas et al. 2015)

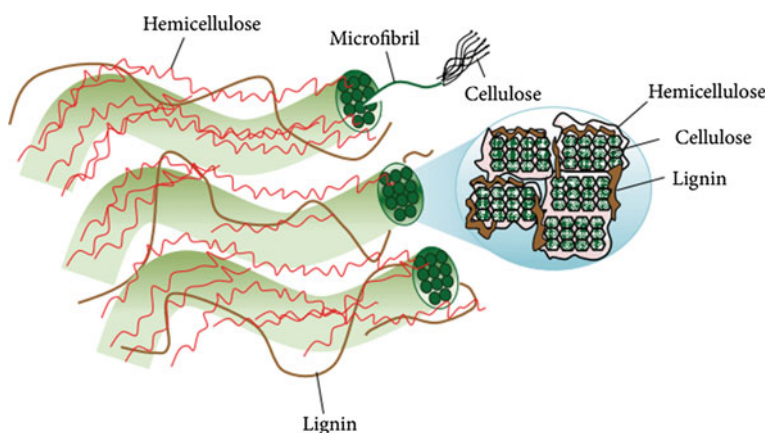


Fig. 2.3 Cellulose fibrils embedded in a matrix of hemicellulose and lignin (Lee et al. 2014)

The typical cell wall structure of plant fiber is shown in Fig. 2.3. The cellulose is hydrogen bonded to hemicellulose molecules of the matrix phase in a cell wall. Hemicelluloses are characterized by irregularity in cellulose chains composed from low molecular chains containing five member rings, open rings, and acidic parts. They are strongly hydrophilic and act as a component of cementing matrix between the cellulose microfibrils, forming the cellulose/hemicellulose network, which is considered to be the main structural component of the fiber cell. The lignin is hydrophobic on the other hand, acts as a cementing agent and increases the stiffness of the cellulose/hemicellulose composite.

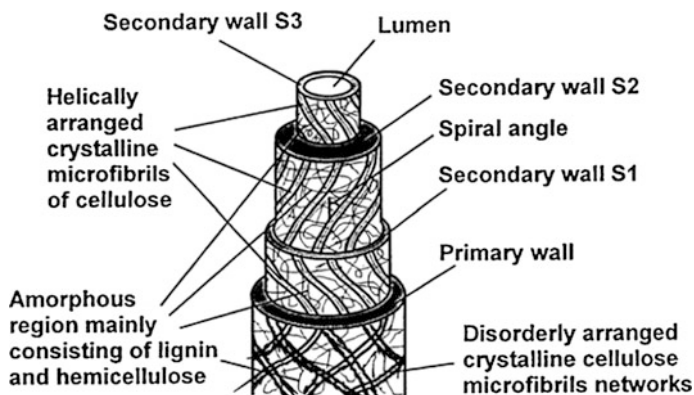


Fig. 2.4 Structural constitution of a natural cellulose fiber cell (Rong et al. 2001)

The plant fiber cell walls are divided into two main sections: a primary cell wall and a secondary cell wall. The primary cell wall consists of a loose irregular network of closely packed cellulose microfibrils, whereas the secondary wall is made up of three separate and distinct layers—S1 (outer layer), S2 (middle layer), and S3 (inner layer). S2 is the most important and thickest layer which determines the mechanical properties of fiber (Tsoumis 1991). Schematic illustration of the fine structure of a lignocellulosic fiber is presented in Fig. 2.4.

These fiber cell walls not only differ in the composition of cellulose, pectines, hemicellulose, and lignin but also in the orientation or microfibrillar/spiral angle of the cellulose microfibrils (Lee et al. 2014). Chemical composition, moisture content, and microfibrillar angle of some plant fibers are given in Table 2.1. The microfibrillar angle is the angle that the helical spirals of cellulose microfibrils form with the fiber axis. The microfibrillar angle varies from one plant fiber to another. The cellulose content in the fiber, microfibrillar angle, and the mean degree of polymerization of cellulose molecules are responsible for the mechanical properties of the fiber. Mean

Table 2.1 Chemical composition, moisture content, and microfibrillar angle of cellulose fibers (Mohanty et al. 2000c)

Fiber	Cellulose [wt%]	Hemicelluloses [wt%]	Lignin [wt%]	Pectin [wt%]	Moisture Content [wt%]	Waxes [wt%]	Microfibrillar Angle [°]
Flax	71	18.6–20.6	2.2	2.3	8–12	1.7	5–10
Hemp	70–74	17.9–22.4	3.7–5.7	0.9	6.2–12	0.8	2–6.2
Jute	61–71.5	13.6–20.4	12–13	0.2	12.5–13.7	0.5	8
Kenaf	45–57	21.5	8–13	3–5	–	–	–
Ramie	68.6–76.2	13.1–16.7	0.6–0.7	1.9	7.5–17	0.3	7.5
Sisal	66–78	10–14	10–14	10	10–22	2	10–22
Banana	63–64	10	5	–	10–12	–	–
Cotton	85–95	5.7	–	0–1	7.85–8.5	0.6	–
Coir	32–43	0.15–0.25	40–45	3–4	8		30–49

degree of polymerization also depends on the part of the plant from where the fibers are extracted. Fibers having higher cellulose content, higher mean degree of polymerization, and a lower microfibrillar angle display higher tensile strength and modulus.

Cellulosic fibers have both crystalline and amorphous domains. The crystallinity degree depends on the type and origin of the material. Cotton, flax, ramie, sisal, and jute have high degrees of crystallinity (65–70%), but the crystallinity of regenerated cellulose is only 35–40%. Progressive elimination of the less organized parts, i.e., amorphous domains, leads to fibrils with increasing crystallinity which can be almost 100% for cellulose whiskers. Crystallinity of cellulose results from the ordered arrangement of cellulose chains and from hydrogen bonding between them, but some hydrogen bonding also exists in the amorphous phase, although its organization is low (Lee et al. 2014). There are many hydroxyl (–OH) groups available in cellulose chains for interaction with water by hydrogen bonding. They interact with water at the surface as well as in the bulk. The quantity of water absorbed by the fiber depends on the relative humidity of the atmosphere. The sorption isotherm of cellulosic material depends on the degree of crystallinity and the purity of cellulose. All –OH groups in the amorphous region are easily accessible to water, whereas only a small amount of water interacts with the surface –OH groups of the crystalline region. The main components of plant-based natural fibers are cellulose (α -cellulose), hemicellulose, lignin, pectins, and waxes.

2.3.1 Cellulose

Cellulose is the major constituent of all plant fibers. Cellulose exists in polymer form of its β D-glucopyranose monomers that make a strong, rigid chain structure through polymerization of 1, 4- β glycosidic linkages. The monomers are linked in a state that one is turn over than other in each repeating unit. This gives the ability to the cellulose structure to make strong intramolecular and intermolecular hydrogen bonding due to the presence of hydroxyl groups as shown in Fig. 2.5. This produces a very compact and coherent structure that is responsible for the highly crystalline cellulose microfibrils. The strength of hydrogen bonds is very less as compared to the strength of covalent bonds but their presence in enormous amount in cellulose structure accounts for cellulose high structural strength. Although the chemical structure of cellulose for different plant fibers is same, the degree of polymerization and orientation of cellulose microfibrils varies considerably. The mechanical properties of a fiber are significantly dependent on the degree of polymerization and orientation of cellulose microfibrils.

2.3.2 Hemicelluloses

Hemicellulose like cellulose is a chain molecular substance but is distinguishable from the latter in having irregularities, branched chains containing pendant side groups, and

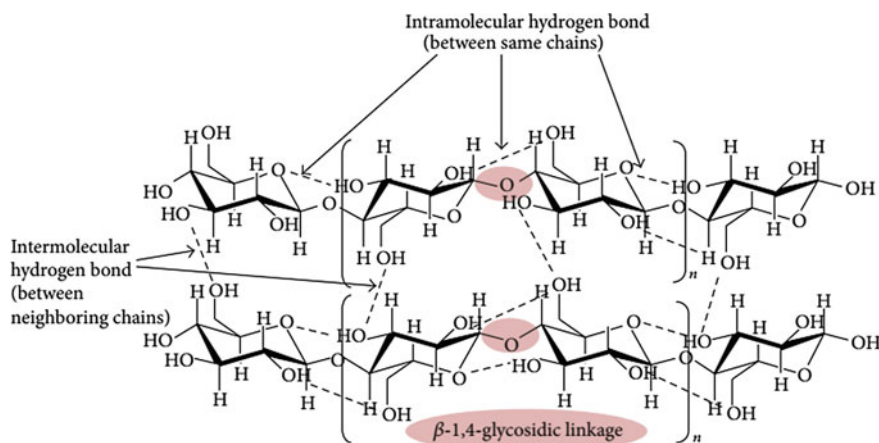


Fig. 2.5 Chemical structure of cellulose chains (Lee et al. 2014)

a relatively short chain length (low degree of polymerization) giving rise to amorphous nature. Hemicelluloses form the part of supportive matrix for cellulose microfibrils and are believed to be a compatibilizer between cellulose and lignin. Hemicellulose is very hydrophilic and soluble in alkali and easily hydrolyzed in acids. Hemicellulose occurs mainly in the primary cell wall and consists of polysaccharides of comparatively low molecular weight and built up from hexoses, pentoses, and uronic acid residues. It is mainly responsible for the biodegradation, moisture absorption, and thermal degradation of the fiber (Mohanty et al. 2000a). Figure 2.6 depicts the cell wall polymers, responsible for the properties of plant fibers in a better way.

2.3.3 Lignin

Lignin is a complex polymer which functions as the structural material and gives rigidity to the plant fibers. It is thought to be a complex, three-dimensional copolymer of aliphatic and aromatic constituents with very high molecular weight. Its chemistry has not yet been precisely established, but most of its functional groups and building units of the macromolecule have been identified. It is characterized by high carbon, but low hydrogen content. Hydroxyl, methoxyl, and carbonyl groups have been identified. Lignin has been found to contain five hydroxyl and five methoxyl groups per building unit. It is believed that the structural units of a lignin molecule are derivatives of 3-(4-hydroxy phenyl) prop-2-eneol. Lignin is amorphous and hydrophobic in nature. It is a thermoplastic polymer having a very slow thermal degradation which extends over the temperature range, starting from melting point 170 °C (Jonoobi et al. 2009). It is not hydrolyzed by acids, but soluble in hot alkali, readily oxidized and easily condensable with phenol. Lignin is thermally stable but is highly susceptible to

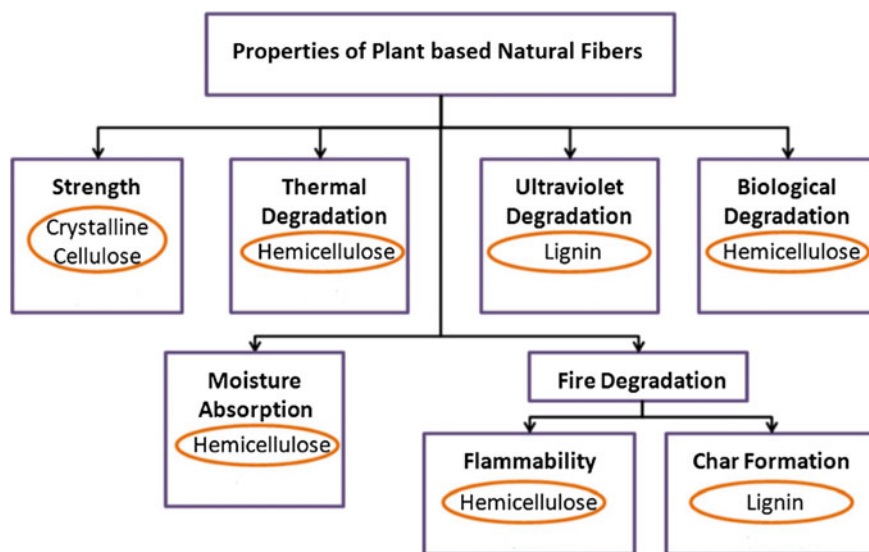


Fig. 2.6 Cell wall polymers responsible for the properties of plant fibers (Azwa et al. 2013)

ultraviolet light. Therefore, lignin is responsible for the ultraviolet light degradation of the fiber (Sedan et al. 2008) (Fig. 2.6).

2.3.4 Pectin

Pectin is a linear polysaccharide and mainly consists of D-galacturonic acid and corresponding methylester units joined in chains by means of 1, 4- α glycosidic linkage. The composition and structure of pectin are still not completely understood. Its structure is very difficult to determine because pectin can change during isolation from plant fibers, storage, and processing (Novosel'skaya et al. 2000). Additionally, impurities can accompany the main components.

2.4 Bast Fibers

Bast fibers constitute a significant share of the huge family of plant-based natural fibers. They are extracted from phloem/inner bark surrounding the stem of dicotyledonous plants. Figure 2.7 depicts the cross section of fibrous plant stem. Epidermis or skin protects the plant against moisture evaporation, sudden temperature changes and partly gives mechanical reinforcement to the stem of plant.

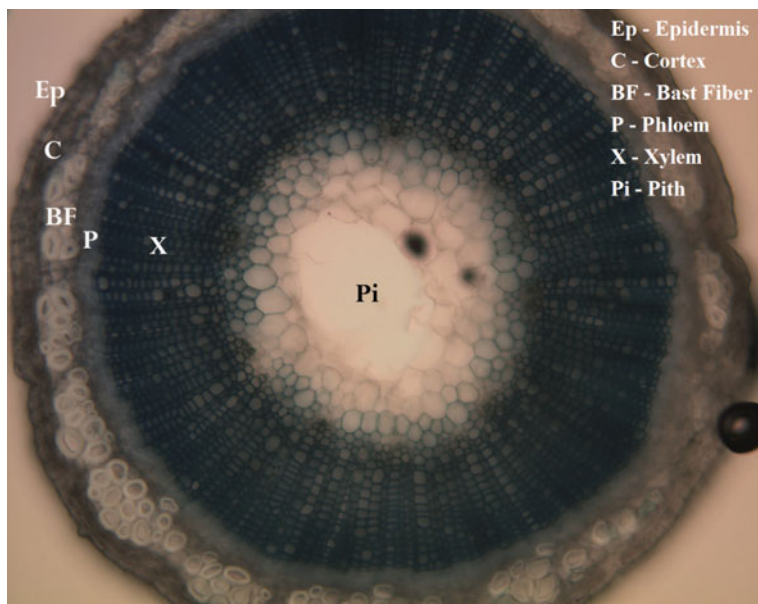



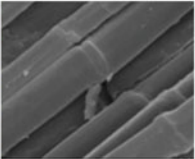
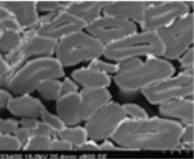

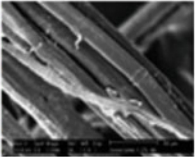
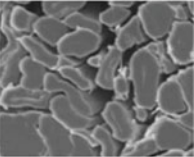

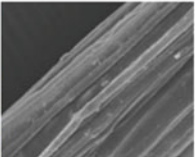
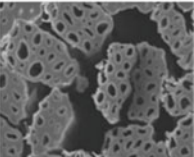

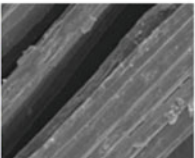
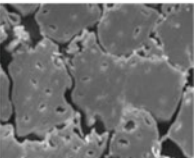

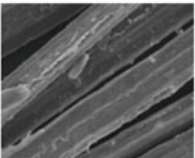
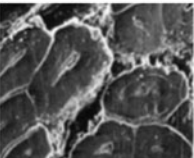
Fig. 2.7 Cross section depicting the position of bast fibers and other constituents of the bast fiber plant

Fibers are located in the phloem in the form of bundles under the skin. Xylem is the woody core in the middle part of the plant.

Bast fibers are separated from the woody core usually by water or dew retting and extracted by decortication. Retting is necessary to loosen the gummy substances which cement the fibers to the rest of the tissues in the stem and to each other. Decortication is the process of extraction of fiber bundles from retted stalk and is usually done manually. The fiber plants, longitudinal view and cross-sectional shapes of some important bast fibers are shown in Table 2.2. All bast fibers have lumen in their structure with different shapes and located in the central part parallel to fiber axis.

As bast fibers fall in the sub-category of plant/vegetable-based natural fibers, therefore, cellulose, hemicellulose, and lignin are the main constituents with 50–90% share of cellulose depending on the type of fiber and the part of stem from where the fiber is extracted. The properties of bast fibers are dependent on the conditions of cultivation and retting, varieties of fibrous plants as well as the condition of measurement. Bast fibers especially flax, hemp, jute, and kenaf have very good mechanical properties which are strongly related to the structure and composition.

Table 2.2 Typical bast fibers (Zimniewska et al. 2011)

Fiber	Botanical name	Plant photo	Longitudinal view of fibers	Image of fiber cross section
Flax	<i>Linum usitatissimum</i>			
Hemp	<i>Cannabis sativa</i>			
Kenaf	<i>Hibiscus cannabinus</i>			
Jute	<i>Corchorus capsularis</i>			
Ramie	<i>Boehmeria nivea</i>			

The structure, cell dimensions, microfibrillar angle, defects, and the chemical composition of fibers are the most important parameters that define the overall properties of the fibers (Satyanarayana et al. 1986). Generally, tensile strength and Young’s modulus increase with higher cellulose content of fibers, higher degree of polymerization of cellulose, longer cell length, and lower microfibrillar angle. The microfibrillar angle is related to the stiffness of the fibers. The fibers are more ductile if the microfibrils have more spiral orientation to the fiber axis. If the microfibrillar angle is less and microfibrils are oriented parallel to the fiber axis, the fibers will be stiff, rigid, inflexible and have high tensile strength. The important mechanical properties of bast fiber are presented in Table 2.3 (Kabir et al. 2012).

Table 2.3 Comparative mechanical properties of bast and E-glass fibers (Kabir et al. 2012)

Fiber	Density [g/cm ³]	Tensile strength [MPa]	Young's modulus [GPa]	Specific strength [* GPa/g cm ⁻³]	Specific modulus [* GPa/g cm ⁻³]	Elongation at break [%]
Jute	1.3–1.4	393–773	13–26.5	0.3–0.5	10–18.3	1.16–1.5
Flax	1.50	345–1100	27–6	0.2–0.7	18.4	2.7–3.2
Hemp	1.48	690	30–60	0.6	26.3–52.6	1.6
Ramie	1.50	400–938	61.4–128	0.3–0.6	40.9–85.3	1.2–3.8
E-glass	2.5	2000–3500	70	0.8–1.4	28	2.5

*Stress divided by fiber density

2.5 A Brief Overview of Jute Fiber

Jute, also known as golden fiber, is the cheapest bast fiber. Jute is the second only to cotton in world's production of natural fibers. India, Bangladesh, Nepal, China, and Thailand are the leading producers of jute. It is also produced in southwest Asia and Brazil. More than 98% of total world production of jute is grown in three South Asian countries, i.e., Bangladesh, India, and Nepal. It belongs to the genus *Corchorus* and family *Tiliaceae*. There are over thirty *Corchorus* species but only two of them are widely known, *Corchorus capsularis* (white jute) and *Corchorus olitorius* (tossa jute). Jute is an important crop in Bangladesh and India and has good socioeconomic importance in these countries (Ranganathan and Quayyum 1993).

Jute can be cultivated under quite a wide variety of conditions but for ideal growth it requires a high level of humidity (40–97%). The ideal temperature lies between 17 and 41 °C. The pure fiber content of the unretted plants lies between 4.5 and 7.5%. Generally, after about 90–120 days of sowing, the stems may be harvested and water retted. Retting takes around 10–20 days and jute fibers are decorticated subsequently in the form of fiber bundles and washed and dried (Pan et al. 2000).

The longitudinal and cross-sectional view of jute fiber is shown in Fig. 2.8. The cross section shows polygonal shape with the canal (lumen) of different size comprising about 10% of the cell area of cross section. The fibers are coarse, generally 20–25 µm in diameter; the length of the ultimate fibers is only 2–5 mm. The cellulose in jute fiber has an average molecular weight between 130,000 and 190,000 with an average degree of polymerization of approximately 800–1200. Jute is a fairly strong fiber exhibiting brittle fracture but small extension at break and poor elastic recovery. The mechanical properties recorded in the literature vary considerably, may be due to variation in the linear density of fibers and differences in the methods of measurement.

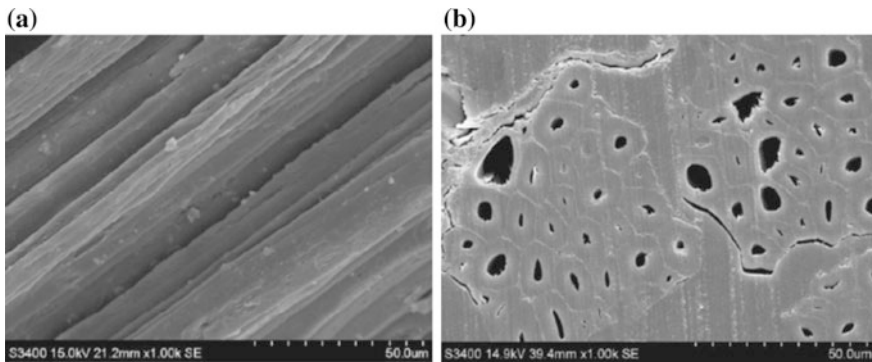


Fig. 2.8 **a** Longitudinal and **b** cross-sectional view of jute fiber (white jute) (Kicińska-Jakubowska et al. 2012)

2.6 Natural Fibers Reinforced Composites in Automotives

The concept of producing natural composite is about 3000 years old when clay reinforced with straw was used to build the walls of dwellings in ancient Egypt. However, natural materials emerged as a future possible material in early 1900s for use in the automotive sector (Suddell and Evans 2003). In 1941, during the World War II, natural fiber reinforced composites received considerable attention for making seats, bearings, and fuselages in aircraft due to shortage of aluminium at that time. The first example was “Gordon-Aerolite” a composite laminate of uni-directional flax yarn soaked with phenolic resin and cured under pressure, used as fuselages in aircraft. The other example was the cotton reinforced polymer composite, used by the military for aircraft radar (Piggott 1980). The first prototype composite car was developed from hemp fibers by Henry Ford in 1942 but unfortunately, the economic limitations hinder the general production of this car. Daimler-Benz has been working on the idea of replacing glass fibers with natural fibers in automotive components since 1991. In 1996, jute-based door panels were introduced by Mercedes-Benz into its E-class vehicles. The door trim panels developed from hybrid flax/sisal mate reinforced polyurethane composites were used in Audi A2 midrange car in 2000 (Mohanty et al. 2005). All body panels of a small prototype car were manufactured and assembled by a researcher in Brazil using jute fiber reinforced composites and hybrid composites (Al-Qureshi 2001) as depicted in Fig. 2.9. A remarkable 20% weight reduction is made in E-class Mercedes-Benz car using interior components made from a blend of flax and sisal fibers in an epoxy matrix (Jawaid and Khalil 2011) as shown in Fig. 2.10. Moreover, natural fiber reinforced composites with a total weight of 43 kg were used to manufacture 27 components in Mercedes S-class car (Pickering 2008).

Almost all well-known car manufacturers in the Europe are now using natural fiber composites in various interior components as those listed in Table 2.4. In reference to European Union (EU) guideline 2000/53/EG on the end of life vehicle



Fig. 2.9 A prototype car made from jute fiber reinforced composite and hybrid composite in Brazil (Al-Qureshi 2001)

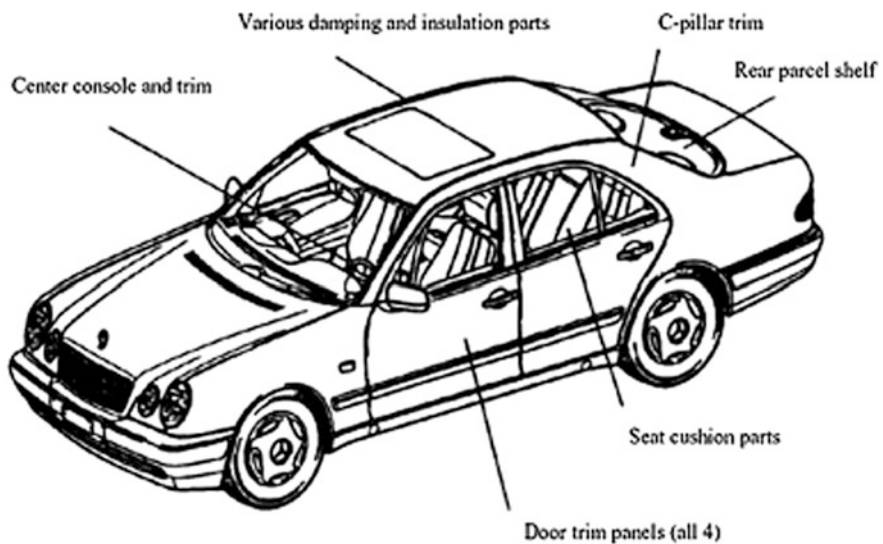


Fig. 2.10 Natural fiber composites application in the current E-Class Mercedes-Benz (Jawaid and Khalil 2011)

Table 2.4 Vehicle Manufacturers and use of natural fiber composites

Automotive manufacturer	Model	Applications
Audi	A2, A3, A4 (and Avant), A6, A8	Roadster, coupe, seat backs, side and back door panels, boot lining, hat racks, spare tyre lining
BMW	3, 5, 7 series	Door panels, headliner panel, boot lining, seat backs, noise insulation panels, molded foot well linings
Citroën	C5	Interior door panels
Daimler-Chrysler	A, C, E, and S-class;	Door panels, wind shield, dashboard, business table, pillar cover panel
Ford	Mondeo CD 162, Focus	Door panels, B-pillar, boot liner
Lotus	Eco Elise	Body panels, spoiler, seats, interior carpets
Mercedes-Benz	Trucks	Internal engine cover, engine insulation, sun visor, interior insulation, bumper, wheel box, roof cover
Peugeot	406	Seat backs, parcel shelf
Renault	Clio, Twingo	Rear parcel shelf
Vauxhall	Corsa, Astra, Vectra, Zafira	Headliner panel, interior door panels, pillar cover panel, instrument panel
Volkswagen	Golf, Passat, Bora	Door panel, seat back, boot lid finish panel, boot liner
Volvo	C70, V70	Seat padding, natural foams, cargo floor tray
Rover	2000 and others	Insulation, rear storage shelf/panel

(ELV) issued by the European Commission, 95% of the weight of a vehicle have to be recyclable by 2015 with 85% recoverable through reuse or mechanical recycling (Peijs 2003).

The car manufacturers in Germany are striving to make every component of their vehicle either recyclable or biodegradable (Hill 1997). In order to produce fuel efficient and low polluting vehicles, natural fiber composites are considered the ideal replacement of glass fiber reinforced plastics (GFRP) where appropriate, because of the main advantages of reduction in cost and weight. Currently, natural fibers account to over 14% share of reinforcement materials; however, the share is projected to rise to 28% by 2020 (HOBSON and CARUS 2011).

2.7 Polymer Matrices

Polymer matrix in a composite provides uniform load distribution to the reinforcing fibers and holds them together in place. They are usually of lower strength compared to the reinforcing fibers. Additionally, the matrix safeguards the composite surface against abrasion, mechanical damage, and environmental corrosion (Akovali 2001). The polymer matrix should be strong enough to withstand the load

but also good enough to transfer load to the reinforcing fibers. The major categories of polymer matrices are thermosets, thermoplastics, rubber matrices, and biobased polymer matrices.

2.7.1 Thermosets

Thermoset matrices are the most frequently used matrix materials in polymer-based composites industry, mainly because of their ease of processing. They are low molecular weight reactive oligomers at the beginning. Generally, they contain two (telechelic oligomer and curing agent) or more components and solidification begins when the components are mixed either at ambient or elevated cure temperatures. The subsequent reaction produces a rigid, highly crosslinked network or a vitrified system with exceptional strength (Saheb and Jog 1999). Epoxies, vinyl esters, polyesters, phenolic resins, and polyurethanes account for the majority of thermoset resins used in industry.

2.7.2 Thermoplastics

Thermoplastics are heat softenable, heat meltable, and reprocessable having one- or two-dimensional molecular structures as opposed to three-dimensional structures of thermosets. They usually come in the form of molding compounds that soften at high temperatures and consist of linear or branched chain molecules having strong intramolecular bonds, but weak intermolecular bonds (Pickering 2008). Their structure is either semicrystalline or amorphous. Melting and solidification of these polymers are reversible, and they can be reshaped by application of heat and pressure. Thermoplastic materials that currently dominate as matrices are polypropylene (PP), polyethylene (PE), polystyrene (PS), polyether-ether ketone (PEEK), and poly (vinyl chloride) (PVC). One of the limitations is the need to process the thermoplastic composites below the decomposition temperature of cellulose, which is ~ 190 °C. Only PP and PE matrices are amenable to natural fiber reinforcement. Of all the thermoplastics, PP shows the most potential benefits when combined with natural fibers for making biocomposites of industrial value. Among all the thermoplastics, the PP matrix natural fiber biocomposites show the most potential benefits of industrial value.

2.7.3 Rubber Matrices

The main classes of rubber matrices that have been used for the preparation of composites are: natural rubber (NR), butyl rubber (IIR), butadiene rubber (BR),

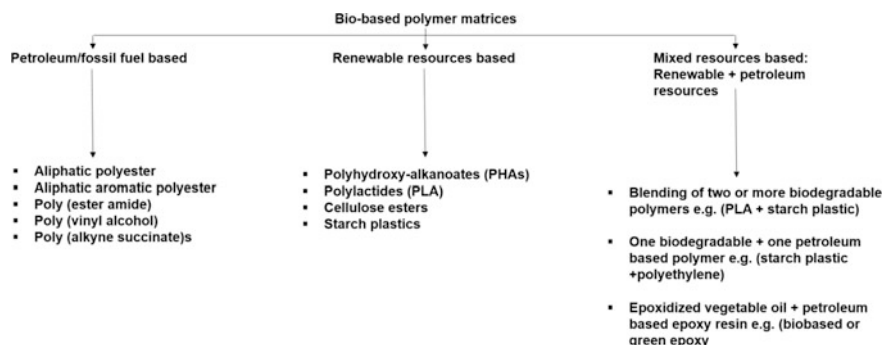


Fig. 2.11 Broad classification of biobased polymer matrices

styrene butadiene rubber (SBR), nitrile rubber (NBR), chloroprene rubber (CR), ethylene propylene diene rubber (EPDM), polyurethane rubber, and silicon rubbers but the most widely used rubber matrix is natural rubber.

2.7.4 Bio-based Polymer Matrices

The US Department of Agriculture and the US Department of Energy have set goals of having at least 10% of all basic chemical building blocks be created from renewable, plant-based sources in 2020, increasing to 50% by 2050 (Dittenber and GangaRao 2012). Currently, numerous researches are underway to develop a new class of composites, known as “green composites” by combining natural fibers with biodegradable/biobased resins. The classification of biobased polymers is presented in Fig. 2.11. Biobased polymers may or may not be fully biodegradable, depending on their structure, composition, and on the environment in which they are placed (Mohanty et al. 2005). Therefore, there is an ambiguity in the definition of biodegradable or green and biobased polymers. Most of the biodegradable epoxy polymers are not completely biobased nevertheless, there has been the development of oxidized green polymers from natural oils. As biobased green epoxy is used in the current study; it is the petroleum-derived epoxy resin blended with epoxidized vegetable oil in the presence of suitable curing agent.

2.8 Jute Fiber Reinforced Polymer Composites

Jute is considered the potential bast fiber for reinforcement in composites due to its good mechanical properties, cheaper availability, biodegradability, and large production relative to other bast fibers. Ray et al. (2002a, b) and (2004) (Sarkar and Ray 2004) extensively investigated the effect of alkali treatment on the mechanical,

dynamical mechanical, thermal, and impact fatigue properties of jute/vinyl ester composites. The results revealed that longer alkali treatment was more helpful to remove hemicelluloses and to improve the crystallinity of fibers thus enabling better fiber dispersion. The mechanical, dynamic mechanical, thermal, and impact properties were superior owing to the alkali treatment, which comprises treatment time, concentration, and conditions. In another study (Sudha and Thilagavathi 2016), the effect of alkali treatment on the jute fabrics and its influence on various mechanical properties such as tensile, flexural, and impact strength of jute/vinyl ester composites was studied. Alkali-treated samples exhibited the improvement in mechanical properties of composites which may be due to better adhesion between the fabric and the resin because of the removal of lignin and hemicellulose. Mechanical properties of alkali-treated nonwoven jute felt reinforced soy composites exhibited better tensile properties than those of raw jute felt composites (Avancha et al. 2013). Gassan and Bledzki (1999) also investigated the influence of alkali treatment on the mechanical properties of jute fibers as well as jute/epoxy composites. The strength and stiffness of composites were increased as a result of the improved mechanical properties of the fibers after alkali treatment.

The effect of surface modification of jute fabrics on the mechanical and biodegradability of jute/Biopol composites was studied (Mohanty et al. 2000b). The tensile strength was found to improve by more than 50%, bending strength by 30%, and impact strength by 90% in the composites as compared to values achieved for pure Biopol sheets. Degradation studies showed that more than 50% weight loss of the jute/Biopol composites occurs after 150 days of composite burial.

Gao and Mäder (2006) studied jute fiber reinforced polypropylene (PP) composites to evaluate the effect of matrix modification using maleic anhydride (MAH) graft copolymer and revealed the significant improvement in the adhesion strength with jute fibers and in turn the mechanical properties of composites. Gassan and Bledzki (1997) also studied the effectiveness of MAH graft copolymer on the mechanical properties of jute-PP composites. Flexural and dynamic strength of MAH-PP treated composites were increased due to improvement of fiber/matrix adhesion. Jute fiber reinforced polypropylene composites were evaluated regarding the influence of gamma radiation (Khan et al. 2009). Mechanical properties such as tensile strength, tensile modulus, bending strength, bending modulus, and impact strength of the gamma irradiated composites were found to be higher than that of untreated composites. The effect of interfacial adhesion on creep and dynamic mechanical behavior (Acha et al. 2007), the influence of silane coupling agent (Hong et al. 2008; Wang et al. 2010), the effect of natural rubber (Zaman et al. 2010) on the mechanical properties and the effect of potassium permanganate on the mechanical, thermal, and degradation properties (Khan et al. 2013) of jute-PP composites were also explored by different researchers.

Different thermoset plastic resins were used as matrices for jute fiber reinforced composites and properties including the thermal stability (Sarkar and Adhikari 2001), mechanical and thermomechanical behavior (Abdallah et al. 2010), durability (Singh et al. 2000), fiber orientation on frictional and wear behavior (Dwivedi

and Chand 2009), eco-design of automotive components (Alves et al. 2010), and alkylation effect on tensile, flexural and interlaminar shear strength (ILSS) (Sarikanat 2009) were examined.

The properties of jute fiber reinforced polyester composites were studied, including the relationship between water absorption and dielectric behavior (Fraga et al. 2006), impact damage characterization (Santulli 2001), weathering and thermal behavior (Dash et al. 2000a), effect of silane treatment on mechanical properties (Sever et al. 2010), effect of enzyme treatment on dynamic mechanical and thermal behavior (Karaduman and Onal 2013), and influence of copper incorporation on the mechanical and thermal behavior (Biswas et al. 2016).

The mechanical properties of PLA were improved significantly with jute reinforcement (Plackett et al. 2003). A 40 wt% composite of jute-PLA had doubled the strength of a pure PLA sample, though the impact resistance between the samples did not differ. The increase in tensile strength was temperature dependent and was contingent on the heating stage during composite formation not exceeding 210–220 °C. Tao et al. (2009) found that the mechanical properties of jute-PLA composites were optimum with 30% fiber volume fraction, and thermogravimetric analysis of composites showed that the addition of fiber to the composite improved the degradation temperature. Hongwei et al. (Ma and Joo 2011) reported that the optimum tensile properties of jute-PLA composites were obtained at 15 wt% fiber content and a processing temperature of 210 °C, whereas the maximum flexural strength and modulus of composites were obtained at 220 °C and 15 wt% fiber contents. The effect of different fiber surface treatments such as alkali, permanganate, peroxide, and silane on mechanical and abrasive wear performance of jute-PLA composites was studied (Goriparthi et al. 2012). Surface treatments resulted in enhancement of tensile and flexural properties and reduction in Izod impact strength of composites. The dynamic mechanical behavior exhibited higher storage modulus and lower tangent delta of treated composites than that of untreated composite and silane-treated composite showed higher thermal stability.

2.9 Micro-/Nanocellulose Filler Composites

Cellulose is an abundant low cost and renewable natural polymer exists in nature and is a major structural component of plant cells. The two types of nanofiller for composites, obtained from cellulose, are cellulose microfibrils and cellulose nanocrystals (CNC) or cellulose nanowhiskers (CNW) (Azizi Samir et al. 2005). Both chemical and mechanical means are usually adopted to extract cellulose fibrils and nanowhiskers from cellulosic resources. The wood of forest resources, ligno-cellulosic fibers (hemp, flax, jute, ramie, kenaf, etc.), and agricultural residues or by-products (corn cob, risk husk, sugarcane bagasse, etc.) are the abundant, cheaper, and readily available cellulose plant resources around the world (Ng et al. 2015). The extracted cellulose fillers have a perfect crystalline structure (about 65–95% crystallinities) with considerable characteristics such as their abundant hydroxyl

groups, high aspect ratio, large specific surface area, good mechanical properties, and high thermal stability which make them a good choice as polymer reinforcing filler in composites (Ng et al. 2015). It has been observed that cellulose crystals extracted from cellulose resources have tensile strength around 10 GPa and modulus around 150 GPa (Helbert et al. 1996) which suggest that cellulose can replace single-walled carbon nanotubes (SWCNTs) in many applications.

There are numerous methods that can be used to produce large quantities of cellulose fibrils/crystals in the laboratories as reviewed in the study by Siró and Plackett (2010). The aspect ratio, orientation, distribution, and loading of cellulose fillers in the polymer matrix decide the properties of composites (Jiang et al. 2007; Kvien and Oksman 2007). A lot of work can be found in the literature on nanocellulose-filled polymer composites, but only a few of them are discussed here. Xu et al. (2013) reported the mechanical and thermomechanical properties of waterborne epoxy reinforced with cellulose nanocrystals. The tensile strength, tensile modulus, glass transition temperature (T_g), and storage modulus increased with increasing filler content, indicating good reinforcement of the epoxy resin matrix. Isora nanofibrils (INFs) reinforced unsaturated polyester nanocomposites were studied for their mechanical and viscoelastic behavior (Chirayil et al. 2014). The improved network in the polyester matrix due to higher aspect ratio of isora fibrils resulted in the improvement of mechanical, T_g , and water barrier properties of prepared composites. Cellulose nanowhisker prepared by acid hydrolysis of microcrystalline cellulose (MCC) were used as filler to reinforce poly(vinyl alcohol) (PVA) with 1, 3, 5, 7 wt% loadings in order to evaluate the mechanical and thermal properties of nanocomposites (Cho and Park 2011). The tensile strength, modulus, and thermal stability were found to increase with the increase in nanocellulose content. However, DMA result showed a significant increase of the storage modulus of the nanocomposite at the 3 wt% of nanocellulose. Jonoobi et al. (2010) used kenaf pulp to isolate cellulose nanofibers which were reinforced with polylactic acid at 1, 3, 5 wt% loadings of filler. The results showed the improvement in tensile strength and modulus of composites with the increase in loading of cellulose fillers. The storage modulus was also increased for all samples as compared to neat PLA and tan delta peak shifted to higher temperature. The work on hybrid carbon woven fabric/epoxy composites reinforced with two types fillers, namely microfibrillated cellulose (MFC) and carboxylated nitrile-butadiene rubber nanoparticles (XNBR), has been reported recently to evaluate their fatigue performance (Carvelli et al. 2016). The best fatigue life was shown by the composites with maximum content of MFC in the system.

2.10 Surface Treatments of Natural Fibers

The fiber/matrix interphase plays an important role to characterize the mechanical properties of the composites. Strong interfacial adhesion is responsible for the good mechanical properties of composites. Several problems occur at the interphase

when natural fibers are used as reinforcement in polymer composites due to the presence of hydrophilic hydroxyl groups at the surface of natural fibers as this hydrophilic nature hinders the effective reaction with the matrix. Additionally, the presence of pectin and waxy substances cover the reactive functional groups of the fiber and act as a barrier to interlock with the matrix. Therefore, different chemical, physical, and biological methods (which are discussed briefly in the following sections) are used for surface modification of natural fibers in order to enhance the fiber/matrix interracial interaction.

2.10.1 Chemical Treatment of Natural Fibers

Chemical methods of fiber treatment help to expose more reactive groups on the fiber surface thus facilitating efficient coupling with the matrix and hence better mechanical properties of composites (Dash et al. 2000b). These methods actually reduce the hydrophilic tendency of natural fibers and thus improve the compatibility with the matrix. The following are some of the most commonly used methods for chemical modification of natural fibers.

2.10.1.1 Alkali Treatment

Alkali treatment of natural fibers is a widely used method to modify the cellulosic molecular structure. It changes the orientation of highly packed crystalline cellulose. In fact, alkali has a swelling reaction on a cellulosic fiber, during which the natural crystalline structure of the cellulose relaxes. The native cellulose in natural fibers has a monoclinic crystalline lattice of cellulose-I which can be changed into different polymorphous forms through chemical or thermal treatments. The important transformations are alkali-cellulose and cellulose-II as shown in Fig. 2.12. The type of alkali (KOH, LiOH, NaOH) and its concentration influence the degree of swelling and hence the degree of lattice transformation into cellulose-II (Fengel and Wegener 1983). Sodium hydroxide is more effective in cellulose swelling due to favorable diameter of Na^+ which is able to penetrate the smallest pores in between the lattice planes. Cellulose micromolecules are separated at large distances due to swelling and alkali sensitive hydroxyl (OH) groups present among the molecules are broken down, react with water molecules and then move out from the fiber structure.

The remaining reactive molecules form fiber-cell-O-Na groups between the cellulose molecular chains (John and Anandjiwala 2008). Hence, the hydrophilic hydroxyl groups are reduced and the fibers moisture resistance property is increased. Afterward, rinsing with water removes the linked Na-ions and converts the native cellulose to a new crystalline structure, i.e., cellulose-II.

This treatment also removes a certain portion of hemicelluloses, lignin, pectin, wax, and oil covering materials. The surface roughness is increased and fiber

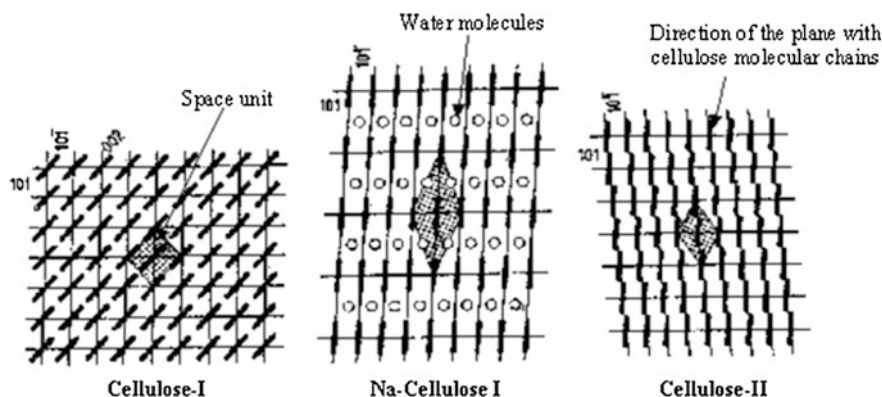


Fig. 2.12 A schematic representation of the transformations of crystalline lattices of cellulose-I, Na-cellulose-I, and cellulose-II by alkali treatment (Van de Weyenberg et al. 2006)

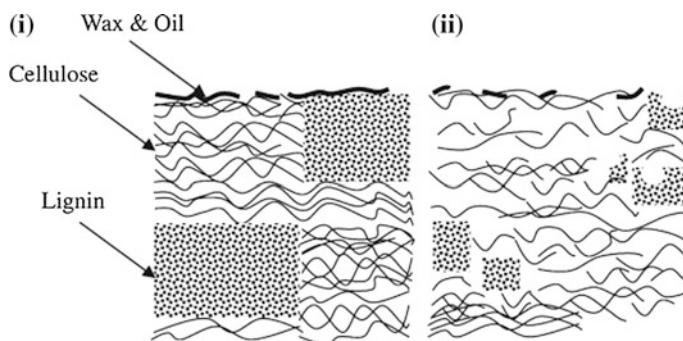


Fig. 2.13 Typical structure of (i) untreated and (ii) alkali-treated cellulose fiber (Kabir et al. 2012)

diameter is reduced, thereby aspect ratio (length/diameter) is increased. Hence, effective fiber surface area becomes larger for good adhesion with matrix. The alkali concentration higher than the optimum value can also damage the cellulose structure which can adversely affect the fiber and composite properties. A schematic view of natural fiber structure before and after alkali treatment is shown in Fig. 2.13 (Mwaikambo and Ansell 2002).

2.10.1.2 Silane Treatment

Silane is a multifunctional chemical compound and used as a coupling agent to modify the surface of fibers. Silane helps to form a chemical linkage between the fiber surface and the matrix through a siloxane bridge. Silanes may reduce the number of hydroxyl groups in the fiber structure. In the presence of moisture, silanols form due to hydrolysable alkoxy groups (Sreekala et al. 2000). One end of

the silanol reacts with the hydroxyl groups of fiber cell and other end reacts with the matrix functional group thus providing molecular continuity across the interphase of the composite.

2.10.1.3 Acetylation Treatment

Acetylation treatment describes an esterification method in which an acetyl functional group (CH_3COO^-) is introduced into an organic compound. In case of natural fibers, this acetyl functional group passivates hydroxyl groups of fiber and takes hydrophobic nature. The hydrophilic nature of fiber results in the dimensional stability of composites. Usually it is not done independently, but is preceded by pretreatment with alkali.

2.10.1.4 Benzoylation Treatment

Benzoyl chloride is mostly used in benzoylation treatment of natural fibers in order to reduce their hydrophilic nature and to improve interfacial adhesion in composites. Alkali pretreated fibers are treated with benzoyl chloride. During the reaction, benzoyl group is attached to the cellulose backbone by replacing hydroxyl groups resulting in a more hydrophobic nature of fiber (Joseph et al. 2003).

2.10.1.5 Peroxide Treatment

The functional group of peroxide can be represented as ROOR. Most commonly used peroxides for the treatment of natural fibers are benzoyl peroxide and dicumyl peroxide. The main advantage of peroxide treatment is the quick decomposition of a peroxide yielding free radical that can react with the hydroxyl group of the fiber and with the matrix resulting in good fiber/matrix adhesion along the composite interphase. Like acetylation and benzoylation treatments, fibers are pretreated with alkali before treating with peroxides. Higher temperature is more suitable for achieving the complete decomposition of peroxide (Sreekala et al. 2000).

2.10.1.6 Permanganate Treatment

Potassium permanganate (KMnO_4) in acetone solution is mostly used for permanganate treatment of natural fibers. The fibers are soaked in solution and concentration is carefully controlled to form highly reactive Mn^{2+} ions that react with the cellulose hydroxyl groups and form cellulose–manganate for initiating graft copolymerization. The hydrophilic tendency of fibers after permanganate treatment is reduced and chemical interlocking at the interphase is enhanced providing better fiber/matrix adhesion (Rahman et al. 2007).

2.10.1.7 Isocyanate Treatment

The isocyanate functional group ($-N=C=O$) of isocyanate compound reacts with the hydroxyl group of cellulose and lignin constituents of the fiber thus forming a urethane linkage which provides strong covalent bonds between fiber and the matrix. Additionally, isocyanate reacts with moisture present in the fiber and forms urea which reacts further with hydroxyl groups of fiber constituents, thus reducing the hydrophilic tendency of fiber (George et al. 2001).

2.10.1.8 Maleated Coupling Agents

Maleic anhydride coupling agent is frequently used to modify the fiber surfaces and provides efficient interaction with the functional surface of the fiber and matrix. This method is mostly used to modify fiber surfaces destined for pairing with a polypropylene matrix. Maleic anhydride reacts with the hydroxyl groups in the amorphous region of fiber structure. This reaction produces brush like long chain polymer coating on the fiber surface and reduces its hydrophilic nature (George et al. 2001). The covalent bonds between the hydroxyl groups of the fiber and the anhydride groups of maleic anhydride make bridge interface for efficient interlocking (Keener et al. 2004).

2.10.1.9 Graft Copolymerization

Acrylic acid ($CH_2=CHCOOH$), acrylonitrile ($CH_2=CH-C\equiv N$), and vinyl monomers are mostly used for graft copolymerization of natural fibers. Free radicals, initiated on cellulose molecule during grafting, interact with monomer of matrix thus enhancing the interlocking efficiency at the interphase (Kalia et al. 2009).

2.10.2 Physical Treatments of Natural Fibers

Natural fibers can be surface modified by physical methods which include corona, plasma, steam explosion, and high energy irradiation (laser, UV and gamma rays etc.). The ultimate purpose of all these methods is to improve the fiber/matrix adhesion by changing the structural and surface properties of the fiber. All physical treatments are considered the eco-friendly ones. The chemical composition of the fibers is not extensively changed by physical treatments. Therefore, the mechanical bonding between the fiber and the matrix is mainly responsible to enhance the interphase properties of composites. A brief description of corona, plasma, and steam explosion treatments is given below.

2.10.2.1 Plasma Treatment

Plasma is defined as a gaseous environment composed of charged and neutral species with an overall zero charge density (Kalia et al. 2013). Plasma treatment changes the surface properties of the fibers through the formation of free radicals, ions, and electrons in the plasma stream which can result in surface cleaning, etching, roughness or activation depending on the type and nature of used gases like helium, oxygen, air, and nitrogen. Low temperature, low pressure, atmospheric pressure plasma, and atmospheric glow discharge treatments are mostly used to modify the surface of natural fibers (Kalia et al. 2013). A schematic representation of atmospheric air pressure plasma is shown in Fig. 2.14 in which a high frequency electric current excites a feeding gas usually compressed air, into relatively low temperature plasma (Baltazar-y-Jimenez et al. 2008).

2.10.2.2 Corona Treatment

Corona discharge treatment (air plasma treatment) is an effective method of surface modification to enhance the fiber/matrix interaction in composites. This process changes the surface energy of the cellulose fibers by surface oxidation activation (Faruk et al. 2012). The corona plasma is generated by the application of high voltage to an electrode. The principle of corona discharge system is shown in Fig. 2.15.

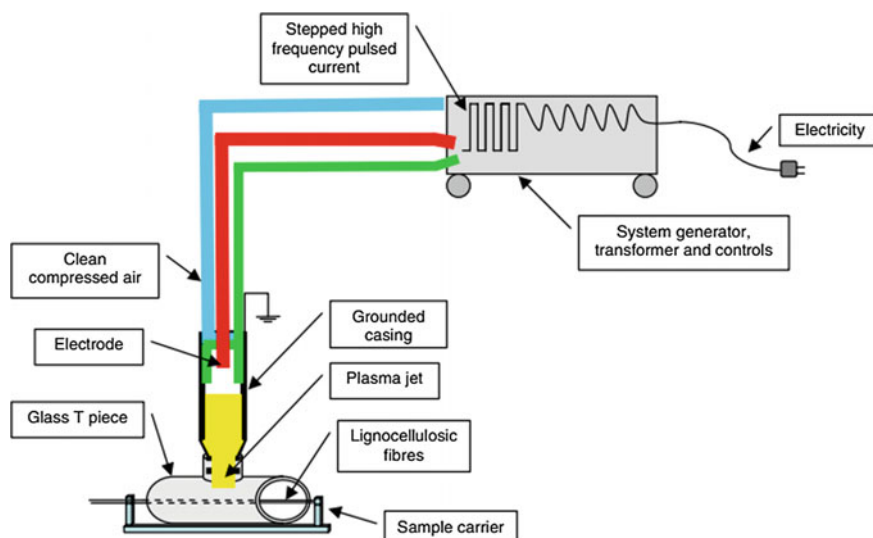


Fig. 2.14 A schematic view of atmospheric pressure plasma treatment (Baltazar-y-Jimenez et al. 2008)

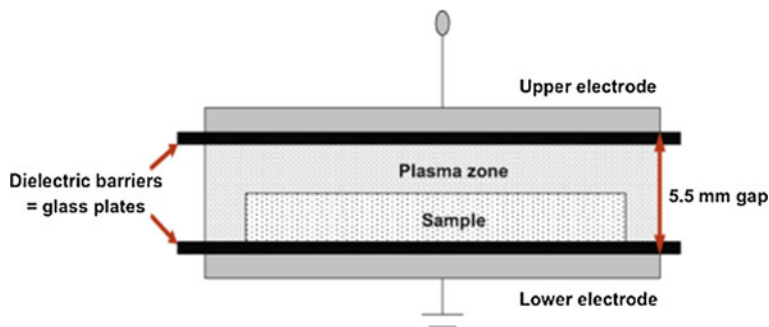


Fig. 2.15 Principle of corona discharge system (Ragoubi et al. 2010)

2.10.2.3 Steam Explosion

In steam explosion process, a high-pressure shock steaming of lignocellulosic materials at high temperature and pressure is involved followed by mechanical disruption of the pretreated material by violent discharge (explosion) into a collecting tank. This process has been applied to many lignocellulosic materials including plant fibers, to enhance dispersibility and adhesion with the polymer matrix (Satyanarayana 2004).

2.10.3 Biological Treatments of Natural Fibers

Biological agents such as enzymes, bacterial cellulose, and fungi provide an alternative way to chemical and physical methods for surface modification of natural fibers. These methods are also eco-friendly; therefore, the use of these methods is rapidly expanding. Biological methods offer several advantages over chemical and physical ones. They can selectively remove pectin and the hemicelluloses while requires less energy input (Gurunathan et al. 2015).

2.10.3.1 Enzyme Treatment

Enzymes offer an environment-friendly alternative to chemical methods for surface modification of natural fibers and their use is becoming increasingly substantial. Surface modification by the enzyme is safer and more advantageous as compared to chemical methods because of high reaction specificity of enzymes, milder reaction conditions, and non-destructive transformations on the surface of fiber (Pallesen 1996). The action of enzyme on the fiber cell is presented in Fig. 2.16 (Kalia et al. 2013). Surface modification by enzyme (usually by cellulase) results in the

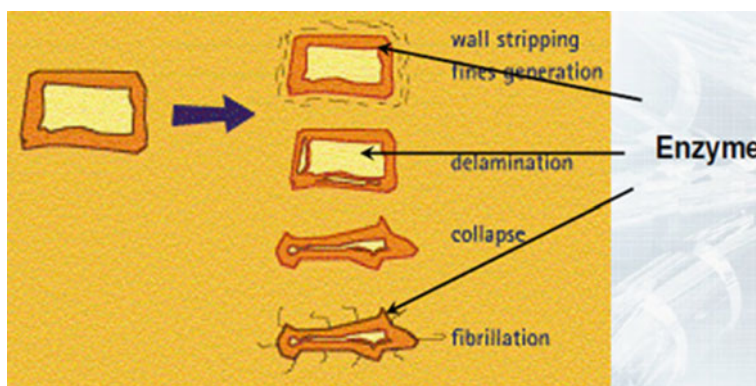


Fig. 2.16 Action of enzymes on the plant cell (Kalia et al. 2013)

degradation of cellulose in the fiber cell wall structure thus initiating the wall stripping, causing the generation of fine fibrils, and leaving the fibers less hydrophilic.

2.10.3.2 Fungi Treatment

Treatment of natural fibers with the fungi is also an eco-friendly and efficient alternative method to the chemical methods. Fungi increase the solubility of hemicelluloses thus reducing the hydrophilicity of the fiber. Fungal treatment also helps to remove lignin from natural fiber and causes the formation of holes (pits) on fiber surface, which provides roughness to the fiber surface and ultimately increases the interfacial adhesion between fiber and matrix (Jafari et al. 2007; Pickering et al. 2007; Kabir et al. 2012). Before treatment with fungi, the fibers are sterilized in an autoclave at 120 °C for 15 min. Then, fungi are added to the fibers with certain proportion and incubated at 27 °C for 2 weeks. Afterward, the fibers are sterilized, washed, and oven dried. The use of white rot fungus can be found in the literature for the treatment of plant fibers (Pickering et al. 2007).

2.10.3.3 Treatment with Bacterial Nanocellulose

The coating of natural fibers with bacterial cellulose involves the deposition of nanosized cellulosic materials onto the surface of fibers to enhance the interfacial adhesion between the fiber and the matrix (Lee et al. 2011). The bacterial cellulose is favorably deposited in situ onto the surface of natural fibers when cellulose producing bacteria such as *A. xylinum* is cultured in the presence of fibers in an appropriate culture medium. This deposition of bacterial cellulose onto fibers provides a new way of controlling the interaction between the fiber and the matrix.

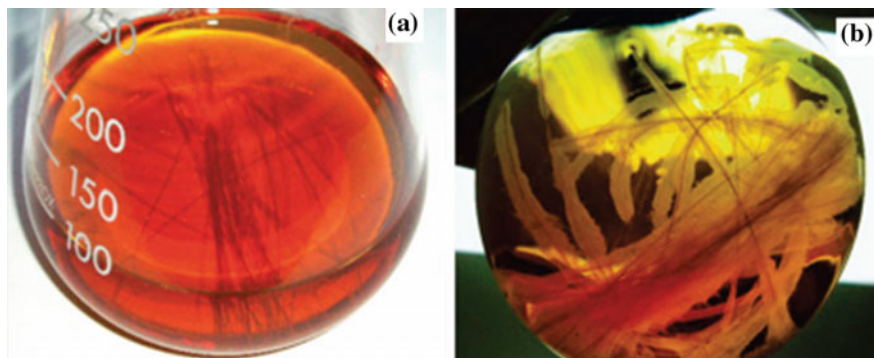


Fig. 2.17 Natural fibers **a** before and **b** after 2 days of bacterial treatment (Kalia et al. 2013)

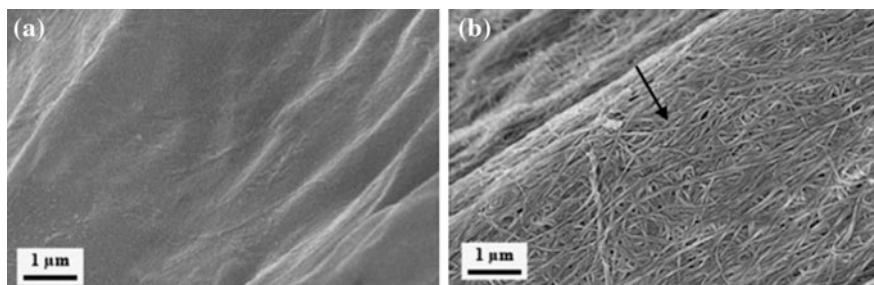


Fig. 2.18 Microscopic images showing **a** neat sisal fiber **b** sisal fiber coated with bacterial cellulose (Lee et al. 2011)

This method facilitates good distribution of bacterial cellulose within the matrix which results in an improved interfacial adhesion between fibers and the matrix through mechanical interlocking.

Figure 2.17 shows the culture medium and natural fibers immersed in the culture medium before and 2 days after culturing (Kalia et al. 2013). A layer of bacterial cellulose pellicles can be seen growing away from the surface of the natural fibers. The surface of sisal fibers was successfully modified by culturing cellulose producing bacteria in the presence of fibers in an appropriate culture medium as shown in Fig. 2.18 (Lee et al. 2011).

2.11 Creep in Natural Fiber Composites

Creep is defined as “a progressive deformation in a material under constant applied load”. All polymeric materials including polymer composites undergo creep deformation even at room temperature. Creep is unwanted phenomena especially in

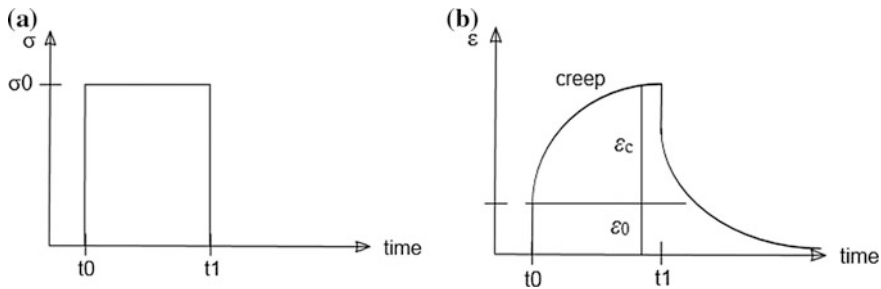


Fig. 2.19 Creep **a** application of constant stress; **b** strain response

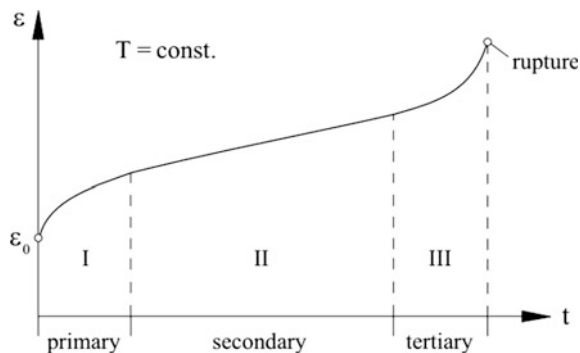
composites because it is source of time dependent form of instability in loaded structures. The creep deformation is realized under a constant applied load as shown in Fig. 2.19. An instantaneous strain (ϵ_0), proportional to the applied stress (σ_0), is observed after the application of constant stress (σ_0), followed by a continuous increase in strain as shown in Fig. 2.19b. The total strain (ϵ_t) at any instant of time t is represented as the sum of the instantaneous elastic strain (ϵ_0) and the creep strain (ϵ_c), i.e.,

$$\epsilon(t) = \epsilon_0 + \epsilon_c. \quad (2.1)$$

Generally, creep can be described in three stages: primary, secondary, and tertiary. During the primary stage, the material undergoes deformation at a decreasing rate, followed by a region where deformation occurs at a nearly constant rate, whereas in the tertiary stage, it occurs at an increasing rate and ends with fracture as depicted in Fig. 2.20.

Creep phenomenon is a very complex and depends on many material parameters. For example, in case of fiber reinforced composites, creep behavior depends not only on the viscoelastic response of the matrix and the fiber but also on the elastic and fracture behavior of the fibers, geometry and arrangement of the fibers, and the fiber/matrix interfacial properties. Fiber reinforced polymer composites

Fig. 2.20 Stages of creep (Betten 2008)



used in engineering applications are often subjected to stresses for a long time and at high temperatures in some situations as well. Creep is therefore a very important parameter in engineering design and manufacturing as this can lead to dimensional instability of the end product or even failure at applied constant stresses that are significantly lower than the ultimate tensile strength of material.

Considerable studies can be found in the literature on the creep behavior of natural fiber polymer composites and nanofiller composites. Nuñez et al. (2004) investigated the short-term and long-term creep behavior of woodflour/polypropylene (PP) composites at different temperatures using polypropylene maleic anhydride copolymer (PPMAN) as compatibilizer between the filler and matrix. The results were discussed with reference to effects of filler content, addition of compatibilizing agent and temperature. The study resulted in a reduction of creep deformation with the addition of woodflour and PPMAN in the matrix and decrease in temperature. Acha et al. (2007) also studied the effect of interfacial adhesion between jute fabric and polypropylene on the creep behavior of composites by chemically treating the jute and using two commercial maleated coupling agents. The time-temperature superposition principle (TTSP) was used to predict the long-term creep performance. The creep deformation was directly related to the interfacial properties of composites. The effect of temperature on the creep and recovery behavior of kenaf nonwoven fabric reinforced polypropylene composites was investigated in another study (Hao et al. 2014), and long-term creep performance was predicted by TTSP. Similarly, Jia et al. (2011) studied the effect of filler content and temperature on the creep and recovery behavior of multi-walled carbon nanotube (MWCNT)/polypropylene composites. TTSP was also applied to predict the long-term creep behavior. The creep strain was found to reduce with the increase in filler content and decrease in temperature. The creep behavior of alkali-treated jute reinforced PP composites (Chandekar and Chaudhari 2016) and starch grafted kenaf/PP composites (Hamma et al. 2014) was also explored by some researchers.

Xu et al. (2010) studied the creep behavior of bagasse fiber reinforced composites with virgin and recycled polyvinyl chloride (B/PVC), high density polyethylene (B/HDPE), and a commercial wood fiber/HDPE composite. The creep deformation of all of the composites was affected more significantly by the increase in temperature. The creep resistance of B/PVC was better than B/HDPE at low temperature, but they showed higher temperature dependence. The TTSP better predicted long-term creep behavior of the PVC composites than the HDPE composites. The effect of fiber size on the creep resistance of wood fiber/HDPE composites was explored in another study (Wang et al. 2015). The large sized wood fiber composites exhibited better creep resistance at all temperatures as compared to short wood fiber composites. The creep deformation of fique/LDPE composites was decreased by treating reinforcing fique fibers with alkali, silane, and pre-impregnation with polyethylene due to increase in fiber/matrix interfacial adhesion (Hidalgo-Salazar et al. 2013). However, the effect of interfacial adhesion due to silane and pre-impregnation with polyethylene on the creep response of composites was better.

The creep behavior was investigated using different thermoset matrices including, MWCNT/epoxy (Starkova et al. 2012), biofiber face honeycomb core sandwich/epoxy (Du et al. 2013), kenaf/unsaturated polyester (Osman and Mutasher 2014), flax/vinylester (Amiri et al. 2015), and bark cloth/epoxy (Rwawiire et al. 2016) composites.

The effect of nanofiller type on the creep response of latex modified polyamide-6 nanocomposites was investigated by Siengchin and Kocsis (Siengchin and Karger-Kocsis 2009). The study revealed that the creep resistance of composites was dependent on the type of nanofiller mainly due to fundamental morphological differences. Similarly, some researchers also discovered the influence of nanofiller (Zhang et al. 2004), the effect of nanofiller size and type (Yang et al. 2006a) on the creep response of polyamide-66 nanocomposites.

The creep resistance of starch films by incorporating starch nanoparticles (Shi et al. 2013) and cellulose nanofibrils (CNFs) (Li et al. 2015) was found to increase, but CNFs concentration above 20% resulted in decrease in creep resistance due to poor dispersion of nanofibrils. TTSP was successfully used to predict the long-term creep performance of these films.

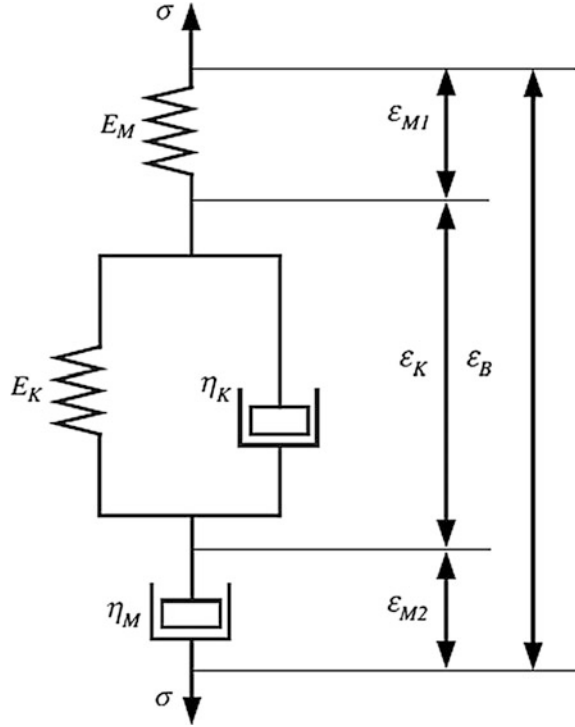
Yang et al. (2015) studied the creep behavior of bamboo fiber reinforced recycled PLA composites (BFRPCs) with fiber loading in the range of 0–80 wt%. The results exhibited the best creep resistance of BFRPC with 60 wt% fiber among all the composites. The effect of fiber type, stress, and temperature on the creep response of wood fiber reinforced biodegradable composites with two fiber loadings (20 wt% and 30 wt%) and containing matrix with a blend of poly(butylene adipate-terephthalate) (PBAT) copolyester and polylactic acid (PLA) was explored (Georgiopoulous et al. 2015). The creep deformation of composites showed a significant dependence on fiber type, temperature, and stress. The best creep resistance was presented by Lignocel[®] wood fibers at fiber loading of 20 wt%, probably due to the different nature of the fiber or/and its higher length.

2.12 Creep Models

Four parameters (or the Burger's) model is one of the mostly used physical models to give the relationship between the morphology of polymer composites and their creep behavior (Findley et al. 1989; Ward and Sweeney 2012). It is based on a series combination of a Maxwell element with a Kelvin–Voigt element as shown in Fig. 2.21 (Yang et al. 2006b). The total creep strain is divided into three separate parts: ε_M the instantaneous elastic deformation (Maxwell spring), ε_K viscoelastic deformation (Kelvin unit), and ε_∞ viscous deformation (Maxwell dash-pot). Thus, total strain as a function of time can be represented by Eqs. 2.2 and 2.3:

$$\varepsilon(t) = \varepsilon_M + \varepsilon_K + \varepsilon_\infty, \quad (2.2)$$

Fig. 2.21 A schematic representation of Burger's model (Yang et al. 2006b)



$$\varepsilon(t) = \frac{\sigma_0}{E_M} + \frac{\sigma_0}{E_K} (1 - e^{-E_K t / \eta_K}) + \frac{\sigma_0}{\eta_M} t, \quad (2.3)$$

where $\varepsilon(t)$ is the creep strain, σ_0 is the stress, t is the time, E_M and E_K are the elastic moduli of Maxwell and Kelvin springs, and η_M and η_K are the viscosities of Maxwell and Kelvin dashpots. η_K/E_K is usually denoted as τ , the retardation time required to generate 63.2% deformation in the Kelvin unit (Yang et al. 2006b). ε_M is a constant value and does not change with time. ε_K represents the earliest stage of creep and attains a saturation value in short time, and ε_∞ represents the trend in the creep strain at sufficiently long time and appears similar to the deformation of a viscous liquid obeying Newton's law of viscosity.

The four parameters E_M , E_K , η_M , η_K can be obtained by fitting Eq. 2.3 to the experimental data and can be used to describe the creep behavior of composites. The creep rate of viscoelastic materials can be obtained by taking the derivative of Eq. 2.3.

$$\frac{d\varepsilon(t)}{dt} = \frac{\sigma_0}{E_K} (e^{-E_K t / \eta_K}) + \frac{\sigma_0}{\eta_M}. \quad (2.4)$$

The Findley's power law model is an empirical mathematical model used to simulate the creep behavior of polymer composites. The model can be represented Eq. 2.5 (Findley et al. 1989);

$$\varepsilon(t) = at^b + \varepsilon_0, \quad (2.5)$$

where a and b are the material constants and ε_0 is the instantaneous strain. The ability of Findley's power law model to simulate the creep data has been found to be satisfactory in several studies (Yang et al. 2006b; Plaseied and Fatemi 2008; Jia et al. 2011). However, this model is not able to explain the creep mechanism of material. A two parameter empirical power law model has also been used in some studies (Tajvidi et al. 2005; Xu et al. 2010) to simulate the creep date. It has the form;

$$\varepsilon(t) = at^b, \quad (2.6)$$

where a and b are the material constants. The long-term creep is an important parameter to evaluate the end-use performance of composites, but it is often impractical to perform a creep test for an extremely long period of time. Time-temperature superposition principle (TTSP) is one of the common estimation techniques to predict the long-term creep behavior by shifting the curves from tests at different temperatures horizontally along the logarithmic time axis to generate a single curve known as master curve (Ward and Sweeney 2012). The shifting distance is called shift factor. According to this principle, the viscoelastic response of a material at a higher temperature is identical with the response of same material at the low temperature for a longer time.

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