

Part One

Introduction to Polymer Composites

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Advances in Polymer Composites: Macro- and Microcomposites – State of the Art, New Challenges, and Opportunities

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1.1

Introduction

Composites can be defined as materials that consist of two or more chemically and physically different phases separated by a distinct interface. The different systems are combined judiciously to achieve a system with more useful structural or functional properties nonattainable by any of the constituent alone. Composites, the wonder materials are becoming an essential part of today's materials due to the advantages such as low weight, corrosion resistance, high fatigue strength, and faster assembly. They are extensively used as materials in making aircraft structures, electronic packaging to medical equipment, and space vehicle to home building [1]. The basic difference between blends and composites is that the two main constituents in the composites remain recognizable while these may not be recognizable in blends. The predominant useful materials used in our day-to-day life are wood, concrete, ceramics, and so on. Surprisingly, the most important polymeric composites are found in nature and these are known as natural composites. The connective tissues in mammals belong to the most advanced polymer composites known to mankind where the fibrous protein, collagen is the reinforcement. It functions both as soft and hard connective tissue.

Composites are combinations of materials differing in composition, where the individual constituents retain their separate identities. These separate constituents act together to give the necessary mechanical strength or stiffness to the composite part. Composite material is a material composed of two or more distinct phases (matrix phase and dispersed phase) and having bulk properties significantly different from those of any of the constituents. Matrix phase is the primary phase having a continuous character. Matrix is usually more ductile and less hard phase. It holds the dispersed phase and shares a load with it. Dispersed (reinforcing) phase is embedded in the matrix in a discontinuous form. This secondary phase is called the dispersed phase. Dispersed phase is usually stronger than the matrix, therefore, it is sometimes called reinforcing phase.

Composites in structural applications have the following characteristics:

- They generally consist of two or more physically distinct and mechanically separable materials.
- They are made by mixing the separate materials in such a way as to achieve controlled and uniform dispersion of the constituents.
- They have superior mechanical properties and in some cases uniquely different from the properties of their constituents [2].

Wood is a natural composite of cellulose fibers in a matrix of lignin. Most primitive man-made composite materials were straw and mud combined to form bricks for building construction. Most visible applications pave our roadways in the form of either steel and aggregate reinforced Portland cement or asphalt concrete. Reinforced concrete is another example of composite material. The steel and concrete retain their individual identities in the finished structure. However, because they work together, the steel carries the tension loads and concrete carries the compression loads.

Most advanced examples perform routinely on spacecraft in demanding environments. Advanced composites have high-performance fiber reinforcements in a polymer matrix material such as epoxy. Examples are graphite/epoxy, Kevlar/epoxy, and boron/epoxy composites. Advanced composites are traditionally used in the aerospace industries, but these materials have now found applications in commercial industries as well.

1.2

Classification of Composites

On the basis of matrix phase, composites can be classified into metal matrix composites (MMCs), ceramic matrix composites (CMCs), and polymer matrix composites (PMCs) (Figure 1.1) [3]. The classifications according to types of reinforcement are particulate composites (composed of particles), fibrous composites (composed of fibers), and laminate composites (composed of laminates). Fibrous composites can be further subdivided on the basis of natural/biofiber or synthetic fiber. Biofiber encompassing composites are referred to as biofiber composites. They can be again divided on the basis of matrix, that is, nonbiodegradable matrix and biodegradable matrix [4]. Bio-based composites made from natural/biofiber and biodegradable polymers are referred to as green composites. These can be further subdivided as hybrid composites and textile composites. Hybrid composites comprise of a combination of two or more types of fibers.

1.2.1

Polymer Matrix Composites

Most commercially produced composites use a polymer matrix material often called a resin solution. There are many different polymers available depending upon the

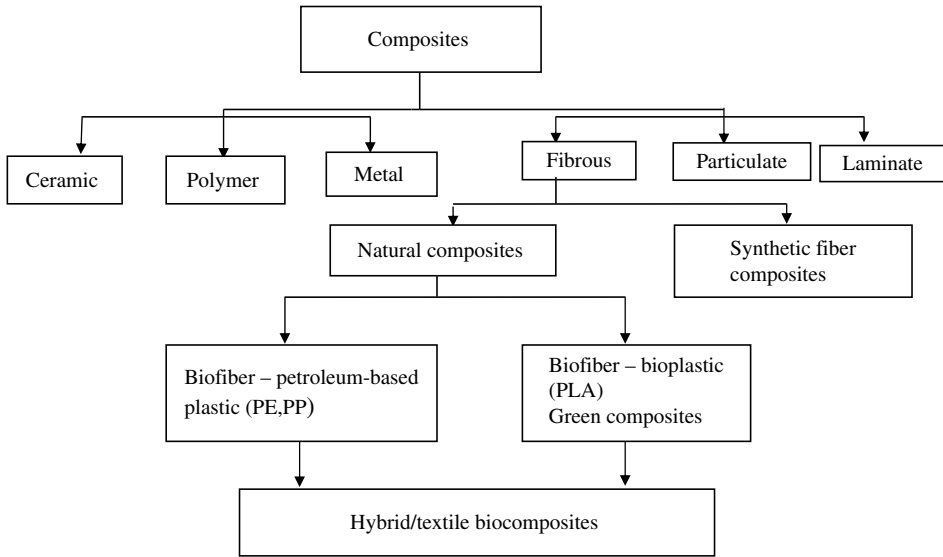


Figure 1.1 Classification of composites [5].

starting raw ingredients. There are several broad categories, each with numerous variations. The most common are known as polyester, vinyl ester, epoxy, phenolic, polyimide, polyamide, polypropylene, polyether ether ketone (PEEK), and others. The reinforcement materials are often fibers but can also be common ground minerals [6]. The various methods described below have been developed to reduce the resin content of the final product. As a rule of thumb, hand lay up results in a product containing 60% resin and 40% fiber, whereas vacuum infusion gives a final product with 40% resin and 60% fiber content. The strength of the product is greatly dependent on this ratio.

PMCs are very popular due to their low cost and simple fabrication methods. Use of nonreinforced polymers as structure materials is limited by low level of their mechanical properties, namely strength, modulus, and impact resistance. Reinforcement of polymers by strong fibrous network permits fabrication of PMCs, which is characterized by the following:

- a) High specific strength
- b) High specific stiffness
- c) High fracture resistance
- d) Good abrasion resistance
- e) Good impact resistance
- f) Good corrosion resistance
- g) Good fatigue resistance
- h) Low cost

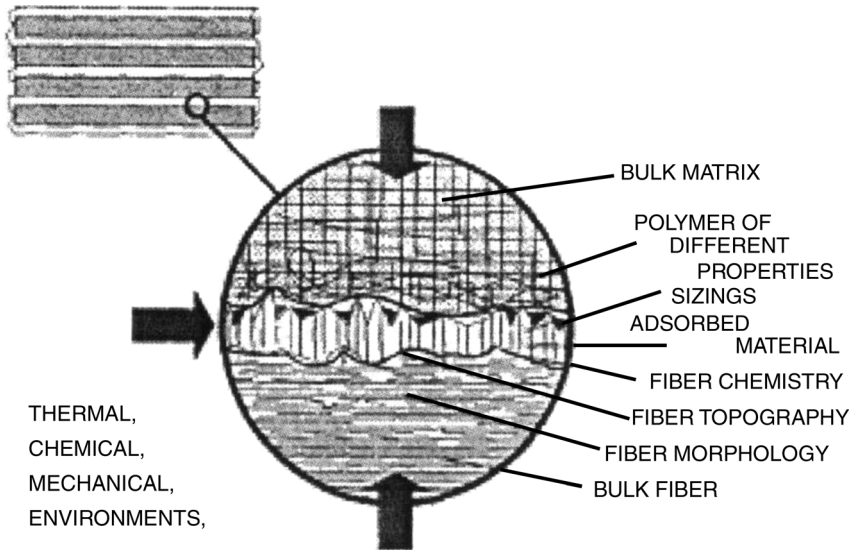


Figure 1.2 Schematic model of interphase [7].

The main disadvantages of PMCs are

- a) low thermal resistance and
- b) high coefficient of thermal expansion.

1.2.1.1 Factors Affecting Properties of PMCs

1.2.1.1.1 Interfacial Adhesion The behavior of a composite material is explained on the basis of the combined behavior of the reinforcing element, polymer matrix, and the fiber/matrix interface (Figure 1.2). To attain superior mechanical properties the interfacial adhesion should be strong. Matrix molecules can be anchored to the fiber surface by chemical reaction or adsorption, which determine the extent of interfacial adhesion. The developments in atomic force microscopy (AFM) and nano indentation devices have facilitated the investigation of the interface. The interface is also known as the mesophase.

1.2.1.1.2 Shape and Orientation of Dispersed Phase Inclusions (Particles, Flakes, Fibers, and Laminates) Particles have no preferred directions and are mainly used to improve properties or lower the cost of isotropic materials [8]. The shape of the reinforcing particles can be spherical, cubic, platelet, or regular or irregular geometry. Particulate reinforcements have dimensions that are approximately equal in all directions. Large particle and dispersion-strengthened composites are the two subclasses of particle-reinforced composites. A laminar composite is composed of two dimensional sheets or panels, which have a preferred high strength direction as found in wood. The layers are stacked and subsequently cemented together so that the orientation of the high strength direction varies with each successive layer [9].

1.2.1.1.3 Properties of the Matrix Properties of different polymers will determine the application to which it is appropriate. The chief advantages of polymers as matrix are low cost, easy processability, good chemical resistance, and low specific gravity. On the other hand, low strength, low modulus, and low operating temperatures limit their use [10]. Varieties of polymers for composites are thermoplastic polymers, thermosetting polymers, elastomers, and their blends.

Thermoplastic polymers: Thermoplastics consists of linear or branched chain molecules having strong intramolecular bonds but weak intermolecular bonds. They can be reshaped by application of heat and pressure and are either semicrystalline or amorphous in structure. Examples include polyethylene, polypropylene, polystyrene, nylons, polycarbonate, polyacetals, polyamide-imides, polyether ether ketone, poly-sulfone, polyphenylene sulfide, polyether imide, and so on.

Thermosetting polymers: Thermosets have cross-linked or network structures with covalent bonds with all molecules. They do not soften but decompose on heating. Once solidified by cross-linking process they cannot be reshaped. Common examples are epoxies, polyesters, phenolics, ureas, melamine, silicone, and polyimides.

Elastomers: An elastomer is a polymer with the property of viscoelasticity, generally having notably low Young's modulus and high yield strain compared with other materials. The term, which is derived from elastic polymer, is often used interchangeably with the term rubber, although the latter is preferred when referring to vulcanizates. Each of the monomers that link to form the polymer is usually made of carbon, hydrogen, oxygen, and silicon. Elastomers are amorphous polymers existing above their glass transition temperature, so that considerable segmental motion is possible. At ambient temperatures, rubbers are relatively soft ($E \sim 3$ MPa) and deformable; their primary uses are for seals, adhesives, and molded flexible parts. Natural rubber, synthetic polyisoprene, polybutadiene, chloroprene rubber, butyl rubber, ethylene propylene rubber, epichlorohydrin rubber, silicone rubber, fluoroelastomers, thermoplastic elastomers, polysulfide rubber, and so on are some of the examples of elastomers.

1.2.1.2 Fabrication of Composites

The fabrication and shaping of composites into finished products often combines the formation of the material itself during the fabrication process [11]. The important processing methods are hand lay-up, bag molding process, filament winding, pultrusion, bulk molding, sheet molding, resin transfer molding, injection molding, and so on.

1.2.1.2.1 Hand Lay-Up The oldest, simplest, and the most commonly used method for the manufacture of both small and large reinforced products is the hand lay-up technique. A flat surface, a cavity or a positive-shaped mold, made from wood, metal, plastic, or a combination of these materials may be used for the hand lay-up method.

1.2.1.2.2 Bag Molding Process It is one of the most versatile processes used in manufacturing composite parts. In bag molding process, the lamina is laid up in a mold and resin is spread or coated, covered with a flexible diaphragm or bag, and cured with heat and pressure. After the required curing cycle, the materials become an integrated molded part shaped to the desired configuration [12]. Three basic molding methods involved are pressure bag, vacuum bag, and autoclave.

1.2.1.2.3 Pultrusion It is an automated process for manufacturing composite materials into continuous, constant cross-section profiles. In this technique, the product is pulled from the die rather than forced out by pressure. A large number of profiles such as rods, tubes, and various structural shapes can be produced using appropriate dies.

1.2.1.2.4 Filament Winding Filament winding is a technique used for the manufacture of surfaces of revolution such as pipes, tubes, cylinders, and spheres and is frequently used for the construction of large tanks and pipe work for the chemical industry. High-speed precise lay down of continuous reinforcement in predescribed patterns is the basis of the filament winding method.

1.2.1.2.5 Preformed Molding Compounds A large number of reinforced thermosetting resin products are made by matched die molding processes such as hot press compression molding, injection molding, and transfer molding. Matched die molding can be a wet process but it is most convenient to use a preformed molding compound or premix to which all necessary ingredients are added [13]. This enables the attainment of faster production rate. Molding compounds can be divided into three broad categories: dough molding, sheet molding, and prepregs.

1.2.1.2.6 Resin Transfer Molding Resin transfer molding (RTM) has the potential of becoming a dominant low-cost process for the fabrication of large, integrated, high performance products. In this process, a dry reinforced material that has been cut and shaped into a preformed piece, generally called a perform, is placed in a prepared mold cavity. The resin is often injected at the lowest point and fills the mold upward to reduce the entrapping of air. When the resin starts to leak into the resin trap, the tube is clamped to minimize resin loss. When excess resin begins to flow from the vent areas of the mold, the resin flow is stopped and the mold component begins to cure. Once the composite develops sufficient green strength it can be removed from the tool and postcured (Figure 1.3).

1.2.1.2.7 Injection Molding Injection molding is a manufacturing process for both thermoplastic and thermosetting plastic materials. Composites is fed into a heated barrel, mixed, and forced into a mold cavity where it cools and hardens to the configuration of the mold cavity. Injection molding is used to create many things such as wire spools, packaging, bottle caps, automotive dashboards, pocket combs, and most other plastic products available today. It is ideal for producing high volumes of the same object [15]. Some advantages of injection molding are high production rates, repeatable high tolerances, and the ability to use a wide range of

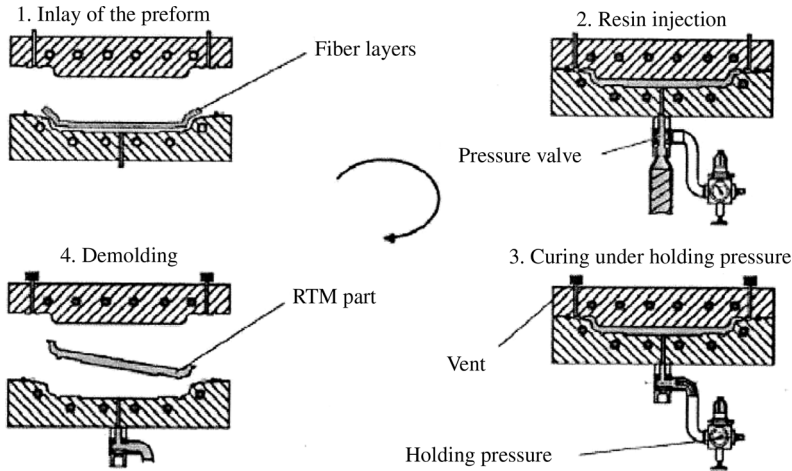


Figure 1.3 Schematic representation of RTM technique [14].

materials, low labor cost, minimal scrap losses, and little need to finish parts after molding. Some disadvantages of this process are expensive equipment investment, potentially high running costs, and the need to design moldable parts.

1.2.1.2.8 Reaction Injection Molding (RIM) RIM is similar to injection molding except that thermosetting polymers are used, which requires a curing reaction to occur within the mold. Common items made via RIM include automotive bumpers, air spoilers, and fenders. First, the two parts of the polymer are mixed together. The mixture is then injected into the mold under high pressure using an impinging mixer. The most common RIM processable material is polyurethane (generally known as PU-RIM), but others include polyureas, polyisocyanurates, polyesters, polyepoxides, and nylon 6. For polyurethane, one component of the mixture is polyisocyanate and the other component is a blend of polyol, surfactant, catalyst, and blowing agent. Automotive applications comprise the largest area of use for RIM-produced products. Polymers have been developed specifically for exterior body panels for the automotive industry. Non-E-coat polymers offer an excellent combination of stiffness, impact resistance, and thermal resistance for body panel applications. These provide excellent paintability and solvent resistance with the ability to achieve high distinction of image (DOI) when painted.

1.2.1.2.9 Reinforced Reaction Injection Molding If reinforcing agents are added to the mixture of RIM setting then the process is known as reinforced reaction injection molding (RRIM). Common reinforcing agents include glass fibers and mica. This process is usually used to produce rigid foam automotive panels. A subset of RRIM is structural reaction injection molding (SRIM), which uses fiber meshes for the reinforcing agent. The fiber mesh is first arranged in the mold and then the polymer mixture is injection molded over it.

1.2.1.2.10 **Spray-Up** In spray-up process, liquid resin matrix and chopped reinforcing fibers are sprayed by two separate sprays onto the mold surface. The fibers are chopped into fibers of 1–2" (25–50 mm) length and then sprayed by an air jet simultaneously with a resin spray at a predetermined ratio between the reinforcing and matrix phase. The spray-up method permits rapid formation of uniform composite coating, however, the mechanical properties of the material are moderate since the method is unable to use continuous reinforcing fibers.

1.2.1.3 Applications

PMCs are used for manufacturing

- i) **Aerospace structures:** The military aircraft industry has mainly led the use of polymer composites. In commercial airlines, the use of composites is gradually increasing. Space shuttle and satellite systems use graphite/epoxy for many structural parts [16].
- ii) **Marine:** Boat bodies, canoes, kayaks, and so on.
- iii) **Automotive:** Body panels, leaf springs, drive shaft, bumpers, doors, racing car bodies, and so on.
- iv) **Sports goods:** Golf clubs, skis, fishing rods, tennis rackets, and so on.
- v) Bulletproof vests and other armor parts.
- vi) Chemical storage tanks, pressure vessels, piping, pump body, valves, and so on.
- vii) **Biomedical applications:** Medical implants, orthopedic devices, X-ray tables.
- viii) Bridges made of polymer composite materials are gaining wide acceptance due to their lower weight, corrosion resistance, longer life cycle, and limited earthquake damage.
- ix) **Electrical:** Panels, housing, switchgear, insulators, and connectors. And many more.

1.2.1.4 Recent Advances in Polymer Composites

1.2.1.4.1 **3-D FRP Composites** Fiber-reinforced polymer (FRP) composites are used in almost every type of advanced engineering structure, with their usage ranging from aircraft, helicopters, and spacecraft through to boats, ships, and offshore platforms and to automobiles, sports goods, chemical processing equipment, and civil infrastructure such as bridges and buildings. The usage of FRP composites continues to grow at an impressive rate as these materials are used more in their existing markets and become established in relatively new markets such as biomedical devices and civil structures. A key factor driving the increased applications of composites over the recent years is the development of new advanced forms of FRP materials. This includes developments in high performance resin systems and new styles of reinforcement, such as carbon nanotubes and nanoparticles [17].

Recent work on 3D FRP composites includes the following:

- a) Manufacturing of 3D preforms by weaving, braiding, knitting, and stitching.
- b) Fabrication of FRP composite products by preform consolidation followed by liquid molding.

Table 1.1 Mechanical properties of natural fibers compared with synthetic fibers [20].

Fiber	Density (10^3 kg/m^3)	Elongation (%)	Tensile strength (MPa)	Young's modulus (GPa)
Aramid	1.4	3.3–3.7	3000–3450	63–67
Carbon	1.4	1.4–1.8	4000	230–240
Kelvar 49	1.45	2.0	2800	124
Cotton	1.5	7.0–8.0	287–597	5.5–12.6
Jute	1.3	1.5–1.8	393–773	26.5
Flax	1.5	2.7–3.5	345–1035	27.6
Hemp	—	1.6	690	—
Ramie	—	3.6–3.8	400–938	61.1–128
Sisal	1.5	4–6	511–635	9.4–22
Coir	1.2	30	175	4.0–6.0
Banana	1.3	2–4	750	29–32
Pineapple	1.56	—	172	62
Oil palm	1.55	—	100–400	26.5
Soft wood craft	1.5	—	1000	40.0
E-glass	2.5	2.5	2000–3500	70.0
S-glass	2.5	2.8	4570	86.0
SiC	3.08	0.8	3440	400
Alumina	3.95	0.4	1900	379

- c) Micromechanics model for mechanical properties of 3D woven/braided/knitted/stitched fabric polymer composites.
- d) Designing microstructure of 3D FRP composite materials to obtain optimum performance (for both continuous and discontinuous fiber composites).

1.2.1.4.2 Natural Fiber Composites Glass, carbon, Kevlar, and boron fibers are being used as reinforcing materials in fiber-reinforced plastics, which have been widely accepted as materials for structural and nonstructural applications [18]. However, these materials are resistant to biodegradation and can pose environmental problems. Natural fibers from plants such as jute, bamboo, coir, sisal, and pineapple are known to have very high strength and hence can be utilized for many load-bearing applications. These fibers have special advantage in comparison to synthetic fibers in that they are abundantly available, from a renewable resource and are biodegradable. But all natural fibers are hydrophilic in nature and have high moisture content, which leads to poor interface between fiber and hydrophobic matrix. Several treatment methods are employed to improve the interface in natural fiber composite [19]. Automobile industry in Europe has started using natural fiber composites in a big way both for exterior and interior of car bodies because of stringent environmental requirements (Table 1.1).

Natural fibers are generally incompatible with the hydrophobic polymer matrix and have a tendency to form aggregates. Therefore, the surface of both (matrix and fibers) should be appropriately wetted to improve the interfacial adhesion and to remove any impurities. The surface of hydrophobic matrices should be modified by

the introduction of polar groups by treating them with oxidative chemicals such as chromic acid/acetic acid or chromic acid/sulfuric acid [21]. Cold plasma chemistry opens up new avenues for the surface modifications of materials for composites and other applications. Various oxidative and nonoxidative chemical treatments are available for natural and synthetic fibers to improve the bonding at the interface. Alkali treatment has been proved to be an effective method for fiber modification from as early as 1935. It has been reported that on treatment with alkali, some of the wax components at the fiber surface are saponified and thereby removed from the fiber surface. Increased fiber/matrix adhesion as a result of improved surface area and increase in availability of the hydroxyl groups have also been reported as a result of alkali treatment.

Compared to unmodified composites, all chemically modified fiber composites show higher tensile properties and lower water uptake. As chemical treatment reduces hydrophobicity of the fiber it favors the strong interfacial adhesion between fiber and PP matrix. Tensile properties decrease with water uptake and time of immersion. Figure 1.4 shows the effect of chemical treatments on the tensile strength of the sisal/PP composites after immersion in water.

Compared to other natural fibers, banana and sisal have good mechanical properties. In general, the strength of a fiber increases with increasing cellulose

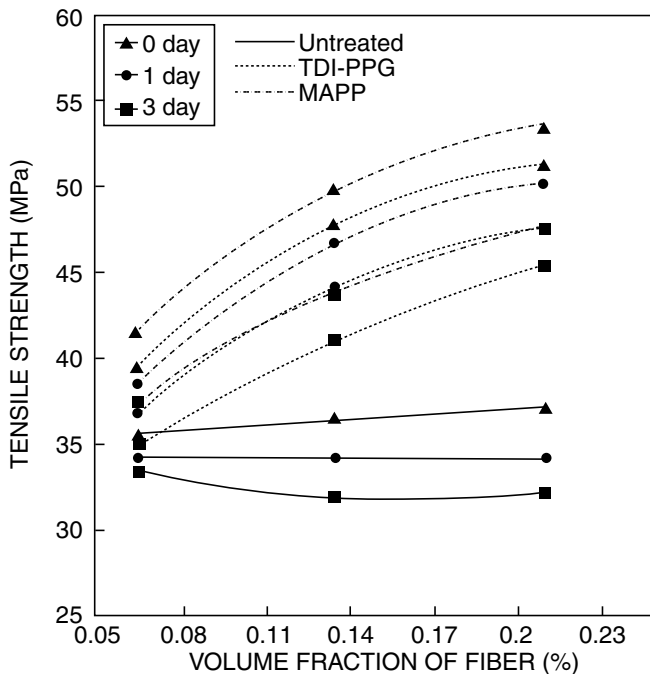


Figure 1.4 The effect of chemical treatments on the tensile strength of sisal/PP composites after immersion in water. Fiber loading 20%, temperature 20 °C [22].

Table 1.2 Properties of banana and sisal fiber [24].

	Banana	Sisal
Cellulose (%)	63–64	64–65
Hemicellulose (%)	19	12
Lignin (%)	5	9.9
Moisture content (%)	10–11	10
Microfibrillar angle (°)	11	20
Lumen size (µm)	5	11

content and decreasing spiral angle with respect to the fiber axis. The composition, microfibrillar angle, and lumen size of banana and sisal fibers are given in Table 1.2. The cellulose content of sisal and banana fibers is almost same, but the microfibrillar angle of banana fiber is much lower than sisal. Hence, the inherent tensile properties of banana fiber are higher than sisal fiber. The diameter of banana fiber is lower than sisal. As the surface area of banana fibers in unit area of the composite is higher, the stress transfer is increased in banana-reinforced composite compared to sisal-reinforced composites [23].

1.2.1.4.3 Fully Green Composites Research efforts are progressing in developing a new class of fully biodegradable green composites by combining fibers with biodegradable resins. The major attractions about green composites are that they are eco-friendly fully degradable and sustainable, that is, they are truly green in every way. The design and life cycle assessment of green composites have been exclusively dealt with by Baillie. Green composites may be used effectively in many applications such as mass-produced consumer products with short life cycles or products intended for one time or short time use before disposal. The important biodegradable matrices are polyamides, polyvinyl alcohol, polyvinyl acetate, polyglycolic acid, and polylactic acid, which are synthetic as well as polysaccharides, starch, chitin, cellulose, proteins, collagens/gelatin, lignin, and so on, which are natural [25]. Bio-based composites with their constituents developed from renewable resources are being developed and its application has extended to almost all fields. Natural fiber composites can be used as a substitute for timber and for a number of other applications. It can be molded into sheets, boards, gratings, pallets, frames, structural sections, and many other shapes. They can be used as a substitute for wood, metal, or masonry for partitions, false ceiling, facades, barricades, fences, railings, flooring, roofing, wall tiles, and so on [26]. It can also be used prefabricated housing, cubicles, kiosks, awnings, and sheds/shelters.

1.2.1.4.4 Other Emerging Areas

- Five-axis weaving technology for the next generation of aircraft and mechanical performance of multiaxis weave structures.
- Noncrimp fiber performs for helicopters composite parts.
- Noncrimp braided carbon fiber-reinforced plastics for aeronautic applications.

- d) Finite element modeling of textile-reinforced composites and comparison with real testing.
- e) Textile composites in ballistics: modeling the material and failure response.
- f) 3D textile composites: mechanical-progressive failure modeling and strength predictions.
- g) Long-term durability of plain weaves polymer composites.

1.3

Interface Characterization

The characterization of interface gives relevant information on interactions between fiber and matrix. The mechanical properties of fiber-reinforced composites are dependent upon the stability of interfacial region. Thus, the characterization of interface is of great importance. The various methods that are available for characterization of the interface are as follows.

1.3.1

Micromechanical Technique

The extent of fiber/matrix interface bonding can be tested by different micromechanical tests such as fiber pull-out (Figure 1.5), micro-debond test, microindentation test, and fiber fragmentation test.

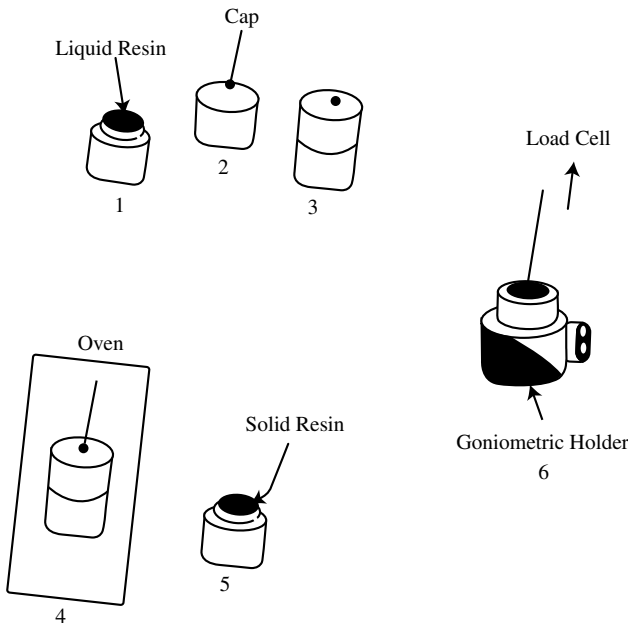


Figure 1.5 Schematic illustration of pull-out test preparation [27].

1.3.2

Spectroscopic Tests

Electron microscopy for chemical analysis/X-ray photoelectron spectroscopy, mass spectroscopy, X-ray diffraction studies, electron-induced vibration spectroscopy, and photoacoustic spectroscopy are successful in polymer surface and interfacial characterization.

1.3.3

Microscopic Techniques

Microscopic studies such as optical microscopy, scanning electron microscopy, transmission electron microscopy, and atomic force microscopy can be used to study the morphological changes on the surface and can predict the strength of mechanical bonding at the interface. The adhesive strength of fiber to various matrices can be determined by AFM studies.

1.3.4

Thermodynamic Methods

The frequently used thermodynamic methods for characterization in reinforced polymers are wettability study, inverse gas chromatography measurement, zeta potential measurement, and so on. Contact angle measurements have been used to characterize the thermodynamic work of adhesion between solids and liquids and surface of solids.

1.4

New Challenges and Opportunities

- In the context of eco-friendly materials, recyclability of the composites is one of the major problems. Recyclability of the composites will lead to the cost-effective products at the same time this is the remedy for the increased amount of waste materials. Green composites can replace all hazardous and waste-producing counterparts.
- Life cycle analysis should be done for all newly synthesized materials and thus the biodegradability can be measured. This will help us to select eco-friendly and acceptable materials.
- Microfibrillar composites, their properties and applications created a lot of interest in research because of its special properties and applications.
- Composite materials having long-term durability for continuous purposes are desirable and cost-effective.
- Since the interface has a significant role in property enhancement, new characterization techniques for interface will bring new opportunities.
- Online monitoring of morphology of composites during processing is another area, which requires a lot of attention of researchers.

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