

# Chapter 12

## Bio-nanocomposites from Natural Fibre Derivatives: Manufacturing and Properties

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**Abstract** Bionano-materials are playing an important role in a number of applications due to their inherent eco-friendly advantages since the last few decades. These materials are being explored as the potential alternatives to traditional synthetic materials for diverse applications with particular emphasis as green reinforcement and offer a number of advantages including considerable toughness, flexibility, easy processing, and recyclability. Indeed, nano-sized materials often display an outstanding equilibrium between toughness and strength and frequently enhanced characteristics of their individual components. The effect of different manufacturing conditions and different surface modification techniques for the bionano-materials as well as their polymer composites is discussed in details. Indeed, bionano-material-reinforced polymer composites are emerging very rapidly as the potential substitute to the metal- or ceramic-based materials in applications including automotive, aerospace, marine, sporting goods, and electronic industries.

**Keywords** Nanotechnology • Biomass • Biodegradable • Renewable resources

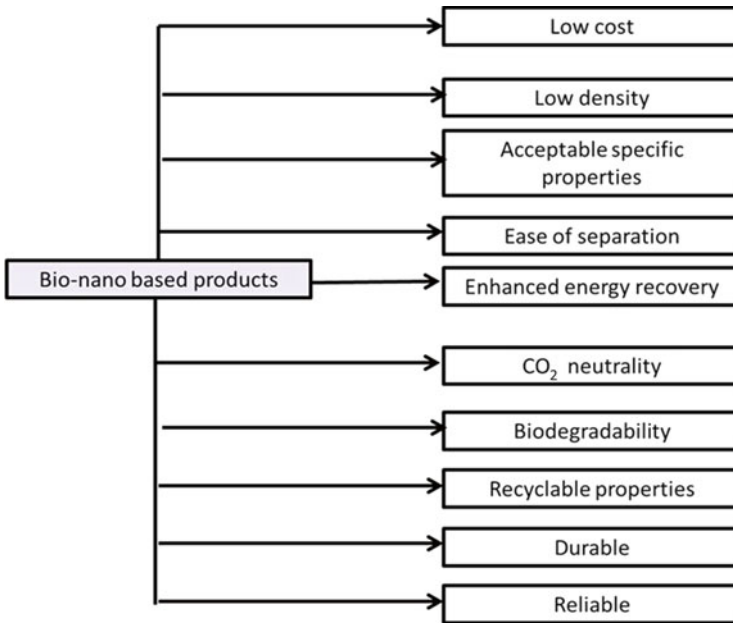
### 12.1 Introduction

Because of the growing environmental awareness, nano materials derived from natural sources (bionano-materials) are becoming more predominant in use. This, in turn, also increases the application of bionano-materials in the polymeric composite system, gaining preference over carbon fibre and glass fibre (Zheng-Ming et al. 2003;

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**Fig. 12.1** Advantages of bionano-based products

Hao et al. 2002; Ioannis 2005) (Fig. 12.1). Bionano-materials signify a traditional category of renewable material which, nowadays, are experiencing a great renewal (Hao et al. 2002).

### 12.1.1 *Natural Fibre Technology in Composite Materials*

The application of natural fibre in composites was expected to be a growing market. Natural fibre only costs as little as USD 0.50 kg<sup>-1</sup> and can be increased in a few months. Therefore, according to the natural fibre composite market trend and forecast Trend, Forecast, and Opportunity Analysis, the global natural fibre composite market touched USD 289.3 million with a compound annual growth rate of 15 % from 2005 (Farzana et al. 2006). This figure is expected to reach USD 531.3 million with an annual growth rate of 11 % for the next 10 years (Richard and John 2007).

The performance of natural fibres depends on several elements, such as the crystalline cell dimensions, composition, micro-fibrillar angle, structure, defects, physical/mechanical properties, hydrophilicity/hydrophobicity, and surface functionality of natural fibres (Joshia et al. 2004; Sgricciaa et al. 2008). The natural fibres' physical properties are important in the composite system and included dimensions, defects, strength, and structure (O'Donnell et al. 2004). For example, a high aspect ratio

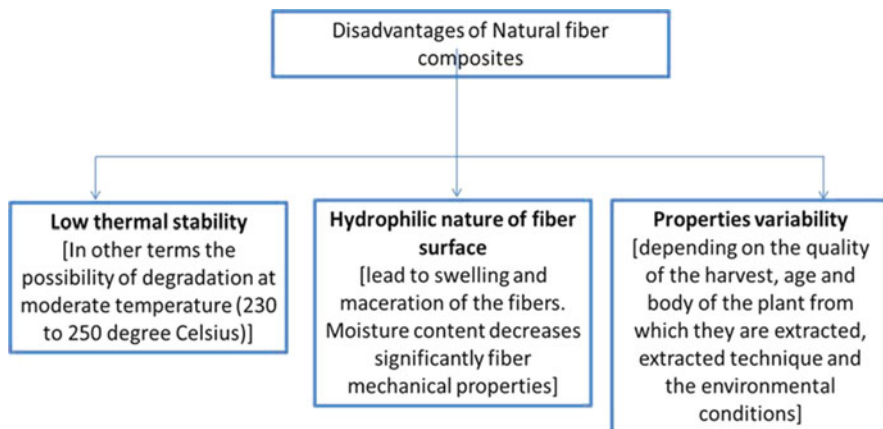


Fig. 12.2 Disadvantages of natural fibre composites

(length/width) which shows the possible strength properties carries a significant importance in natural fibre composites. The natural fibres potentially help in selecting natural fibres as a filler to use composites. Indeed, its dimensions, strength, defects, variability, structure, and crystallinity must also be considered (Muhammad and Mohini 2003).

The primary natural fibres' disadvantages in the particular composites are the poor adaptability of matrix and fibre and their comparatively high moisture adsorption (Fig. 12.2). Consequently, modifications of natural fibre are considered and result in a change in properties of fibre surface to enhance their adhesion with different matrices (Jayamol et al. 2001; Ming et al. 2005). A prototypical stiffness and strength could be attained with a strong interface which is very brittle in nature with easy crack propagation over the fibre and matrix. A huge number of researches have been done, displaying the significance of the interface and the effect of several kinds of surface modifications on the physical and mechanical properties of the natural fibre-reinforced composite system (Table 12.1).

### 12.1.2 Nanotechnology in Composite Materials

Replacement of traditional microcomposites of nanocomposite materials grew quickly in the last 20 years to overwhelm the restrictions of the micrometer scale, synthesizing novel structures and materials having extraordinary flexibility, enhanced physical performances, and noteworthy industry impact (Michael and Philippe 2000; Ramanathan et al. 2008). Generally, inorganic nanofillers utilized to generate nanocomposites, which compare to organic one, their biodegradability, processability, and biocompatibility, are highly restricted (Peter et al. 1999).

**Table 12.1** Surface modification of fibre-reinforced composite system

Surface modification	Examples	Advantages	References
Chemical modification	Mercerization, oxidation, cross-link, grafting, alkaline, benzylation, silane, maleated coupling, acetylation, acrylation and acrylonitrile grafting, permanganate, peroxide and isocyanate	<ul style="list-style-type: none"> <li>• Create more sites for chemical links</li> <li>• Significant performance enhancement of low-fibre content</li> <li>• Reduction in water absorption</li> </ul>	Xue et al. (2007)
Physical modification	Thermo-treatment, calendaring, stretching, corona and plasma treatment	<ul style="list-style-type: none"> <li>• Increasing the strength of the fibres</li> </ul>	Bledzki et al. (2009)
Biological modification	Enzymatic functionalization	<ul style="list-style-type: none"> <li>• Increase the compatibility and sensitivity</li> </ul>	Maya and Rajesh (2008)
Nanotechnology modification	Mechanical and chemical reduction process	<ul style="list-style-type: none"> <li>• Increase surface area and functionality</li> </ul>	István and David (2010)

All these necessities are connected by nanofibres as one category of bionanomaterial. On Earth, nanofibres are the most ample polymers due to its biodegradability property and high-strength nanocomposites generated using its structure (Mohammad and Karen 2006). An ample and increasing literature dedicated to nanofibres is attained either by a disintegration shearing action to gain micro-fibrillated fibres or by chemical acid hydrolysis treatment. In general, the nanofibres with elongated rodlike features and characteristics contain high-purity single fibre formed from different plant origins under controlled conditions (Xue et al. 2007). The nature of plant origin and hydrolysis conditions plays an important role on nanofibre dimensions. This allows a complete dispersion of nanofibres and maximizing the available matrix–filler interphase optimizing the organic–inorganic interactions, which are responsible for improving the final properties of the nanocomposites. The quality on the dispersion of nanocomposites components given by macroscopic processing factors, including equipment design, mixing velocity, residence time with very limited possibilities for process optimization (O’Donnell et al. 2004). Therefore, depending on the conditions of the procedure and affinity between matrices and nanofibres, various dispersion states can be attained, including the phase separated with agglomerate microcomposites, inserted where layers are uniformly dispersed, but remain orderly and exfoliated when nanofibres are fully and homogeneously dispersed and entirely disoriented (Richard et al. 1996). Exfoliated structures are continually the ultimate aim in nanocomposite compounding because the best performance, especially concerning barrier properties, has to be attained.

It is clear that polymer and hybrid inorganic–organic nanocomposites are focusing on filler functionalization to promote better polymer–filler interfaces. In particular, the processing approach provides a valid technological route to maximize the final properties of nanocomposites.

## 12.2 General Manufacturing and Fabrication Method of Bio-nanocomposite Materials

Generally, bio-nanocomposites are generated by utilizing conventional manufacturing methods (designed for traditional fibre-reinforced polymer composites and thermoplastic). The manufacturing methods contain compounding, mixing, extrusion, injection molding, and compression molding, which are appropriate for bionano-material-reinforced thermoplastic, thermoset, and elastomer composites. The resin transfer molding, compression molding, and pultrusion are generally implemented with the thermoset matrix. Mentioned manufacturing methods have well accumulated, and developed experience has confirmed their potential success for the production of bio-nanocomposites with controllable quality until now.

### 12.2.1 Powder Injection Molding

Powder injection molding is the combination of injection molding and powder metallurgy, which is suitable in the fabrication of small and complex parts in large quantities of bionano-composites (Chow et al. 2003; Won-Jun et al. 2006) (Fig. 12.3). In the powder injection molding process, combining two or more powders with different properties, including powder size, type of powder, and thermal expansion coefficient, is challenging (Kyriaki et al. 2007). For example, a difference in particle size increases the critical powder loading. In a high powder loading condition, a small quantity of binder is necessary. The binder is important in determining the success of the injection stage (Hezhou et al. 2008; Chow et al. 2004). Moreover, differences in thermal expansion coefficients induce residual stress and crack during and after the heating process.

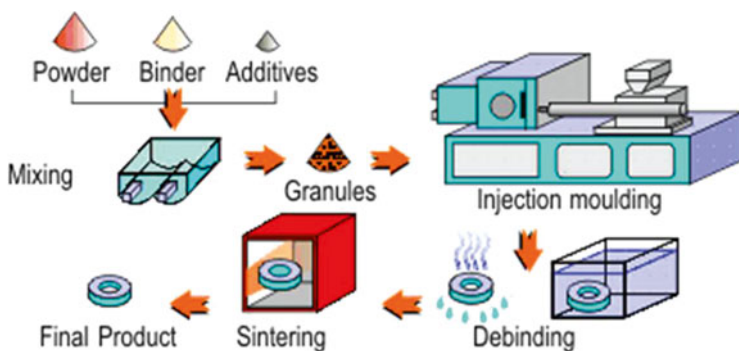


Fig. 12.3 Powder injection molding process

### 12.2.2 Micro-Cellular Injection Molding

The micro-cellular injection molding is an emerging process with several privileges, able to produce foamed portions. Applying  $N_2$  or  $CO_2$  in the supercritical fluid phase as a physical blowing and plasticizer agent generates lightweight foamed parts with reduced pressure, material, cycle time, and energy (Mingjun et al. 2004; Hrishikesh et al. 2003; Gong et al. 2005) (Fig. 12.4). The micro-cellular injection molding generates components with improved dimensional stability and less thermal degradation and material saving in comparison with the conventional injection molding method (Hrishikesh et al. 2003).

This will occur by exposing the gas/polymer solution to the thermodynamic instability by fast reduction of the solution pressure and increasing its temperature (Mingjun et al. 2005). The actual result is often a drop within the equilibrium solubility of the gas in the composite polymer, leading to the nucleation of frequent microvoids. Other than material formulation and processing parameters, nanofiller and polymer blends also have a robust effect on cell nucleation and growth (Pilla et al. 2007).

### 12.2.3 Liquid Composite Molding

Using liquid composite molding technique with nanofibre reinforcements offers additional economical, technological, environmental, and ecological advantages. Wide ranges of composite manufacturing processes were covered with the liquid composite molding, including resin transfer molding, resin infusion, and injection

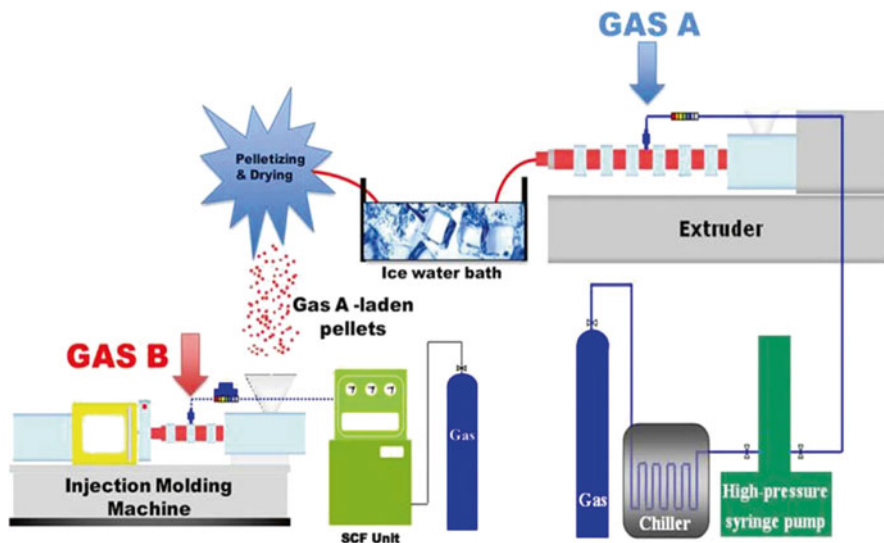


Fig. 12.4 Micro-cellular injection molding

compression molding (Mahrholz et al. 2009; Hammami et al. 1998; Pavel and Suresh 2004). In general, all liquid composite molding techniques involve placement of nanofibrous reinforcing material into some form of closed mold. Based on the manufacturing setup, different types of molds are applied for each liquid composite molding (Baoxing and Tsu-Wei 1999). It shows resistance to flow (quantified by permeability) which significantly influences the required time to fill mold and resin flow patterns. After filling the mold, the polymer matrix will be cured, and on sufficient rigidity, the portion removal from the mold occurs. Permeability and compaction behavior of reinforcement are two essential factors during mold-filling process modeling (Hammami et al. 1998).

In order to understand the through-thickness stress–strain behavior of the reinforcement, accurate general liquid composite molding filling and forces exerted on molds should be modeled (François et al. 2006). Due to compress technique apply towards the nanofibre reinforcement the clamping forces required, which supplied by both internal resin pressure and the stress. Some researchers reported stress relaxation behavior during compaction experiments on nanofibre reinforcements in the absence of a resin. Existence of fluid leads to the apparent lubricant effect, which highly decreases the mold clamping forces (Wang et al. 1994; Lin et al. 1993).

#### ***12.2.4 Electrospun Photon Exchange***

Electrospinning is a method which produces bio-nanocomposites, using the electricity to drive the spinning process. Therefore, it is a promising alternative to traditional bio-nanocomposite fabrication. Unlike traditional spinning techniques that produce fibre composites in the range of the micrometer (5–50  $\mu\text{m}$ ), electrospun photon exchange produces bio-nanocomposites with the range of nanometer size (10–1,000 nm) (Xiaobo et al. 2012; Biao et al. 2008; Dan and Younan 2003) (Fig. 12.5). The bio-nanocomposite synthesis, via a top-down nano-manufacturing process, is a money-saving process which leads to continuous nanofibres that are relatively easy to align, assemble, and process into applications (Biao et al. 2008). Therefore, this technique provides extraordinary properties such as small-sized dimension associated with large specific surface areas, a high degree of structural perfection, and the resultant extreme mechanical properties (Amit et al. 2005).

To date, two types of electrospun photon exchange process are fabricated, which are an uncharged polymer surrounding a three-dimensional interconnected mat of ionomer nanofibres and an ionomer surrounding an electrospun mat of uncharged polymer nanofibres (Thavasi et al. 2008). Electrospinning can fix a great degree of compositional and morphological control of volume fraction and the diameter of the charged or uncharged polymer nanofibres independently. Furthermore, fabrication of bio-nanocomposites via electrospun process occurs with solid-state “forced assembly” of nanofibre on a collector surface, in contrast with self-assembly in the solution, avoiding phase separation and poor dispersion problems causing large-sized domains of the minor composites components (Jih-Jen et al. 2010).

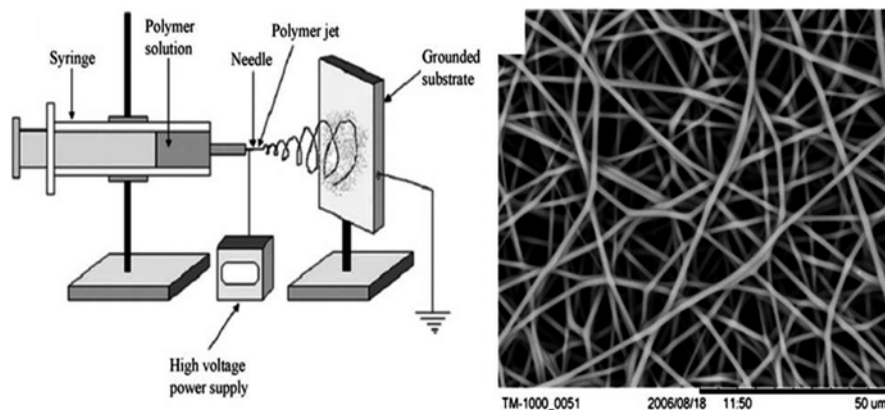


Fig. 12.5 Electrospun photon exchange (Xiaobo et al. 2012)

In general, bio-nanocomposites fabricated through the electrospun method exhibit impressive proton conductivity, water swelling, and mechanical properties.

In general, in the case of the electrospun photon exchange process, the biocompatibility of nanofibre advances with reducing the diameter (it is noteworthy that the electrospun polymer bio-nanocomposites have diameters one to three orders of magnitude smaller than their conventional counterparts). Therefore, electrospinning has a high potential to produce bio-nanocomposites in nanofibrous forms of medical materials, including hard tissue applications and composites of inorganic and polymer phases due to its mechanical and biological properties, which can be tuned appropriately for cellular function and bone formation (Priya et al. 2008; Kim et al. 2008). In addition, the combination of electrospinning with initiated chemical vapor deposition is an effective approach to preparing superhydrophobic bio-nanocomposites. However, this modification process requires the use of special equipments, including chemical vapor deposition reactor and plasma equipment (Amit et al. 2005).

In the meantime, dual electrospun photon exchange process has been introduced whereby the bio-nanocomposites are made by separate and simultaneous electrospinning of two polymers. To construct the first structure, a series of steps is followed, including mechanical mat compaction, solvent vapor exposure, thermal annealing, and acid/water washing (Chi-Ching et al. 2007; Seeram et al. 2006; Sujuan et al. 2010). Designed for the other structure, different series of steps are followed, including mat compaction with heating, thermal annealing, and acid/water washing. This dual process proposes numerous benefits compared to the conventional electrospun fabrication, namely, the elimination of a separate impregnation step (where complete void space filling is often problematic), the choice of making two distinct structures from the same initial dual nanofibre mat, the possibility of expanding the number of polymers that are spun simultaneously to three or more in order to add other properties to the bio-nanocomposites, and the ability to



control the spatial distribution of polymers in the bio-nanocomposite thickness direction by changing the relative flow rates of polymers during electrospinning (Sujuan et al. 2010). Therefore, the dual nanofibre electrospinning technique is considered as a robust platform for the fabrication of a wide variety of bio-nanocomposite structure through different processing approaches.

### 12.2.5 Spray-Up Method

The spray drying method is a direct technique to encapsulate the nanofibre into the polymer matrix homogenously (Yanjun et al. 2010). The preheated spray drier was filled with mixture. The preheating temperature is higher than the boiling point of the solvent (Fig. 12.6). This technique is limited to the high molecular weight and chemically stable polymer, while for those with small molecular weights, it tends to boil off in large quantities at the processing temperature (Kerang et al. 2000; Varun et al. 2006).

## 12.3 General Properties of Bio-nanocomposite Materials

It has been suggested that bio-nanocomposites compared to their neat polymer and traditional composites will enhance barrier properties and mechanical strength and improve heat resistance. Bio-nanocomposites are predicted to survive the thermal processing stress, transportation, and storage (Michael and Philippe 2000). Furthermore, bio-nanocomposites may allow down gauging due to improvement in their mechanical properties and reduce source material consequently.

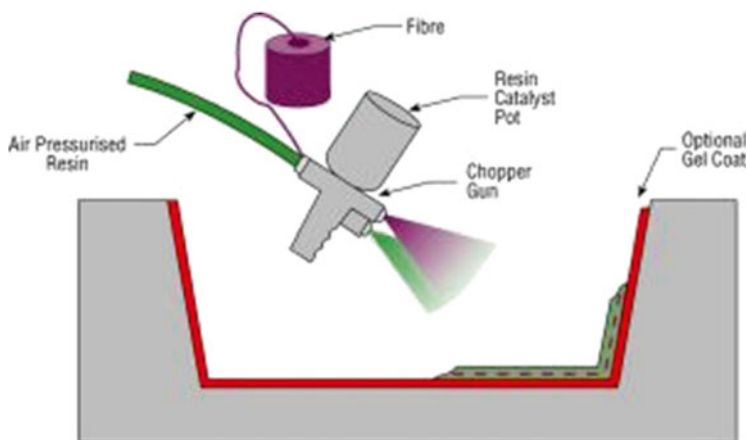


Fig. 12.6 Spray-up method

Indeed, one of the significant properties bio-nanocomposites have is their biodegradability capability. It may lead to fragmentation and loss of mechanical properties or may sometimes be followed by degradation via microorganisms (bacteria, fungi, and algae). This is a multistage procedure which applies enzymes and oxidation to the hydrolysis catalyzed (Mohammad and Karen 2006). The high hydrophilicity property of nanofibres helps easier water penetration into the polymer matrix and activation of the hydrolytic degradation process which leads to the biodegradation process (Richard et al. 1996). Meanwhile biodegradability is one of the main reasons to use nanofibres for the preparation of the bio-nanocomposite material, and the biodegradability of the produced bio-nanocomposites should not be sacrificed fabricating them.

## 12.4 Thermoset Bio-nanocomposites: Manufacturing and Properties

The application of thermoset polymer composites has been studied widespread by researchers all around the globe due to their enormous advantages (Fig. 12.7). In many applications, which necessitate high performance, including the automotive, nautical, and aeronautical constructions, in addition to those required in the civil and industrial sector, the need to satisfy resistance, stiffness, lifetime, and reliability requirements involves the use of the thermoset polymeric matrix reinforced with bionano-materials (Janis et al. 2000; Omid et al. 2012; Karger-Kocsis and Wu 2004). Therefore, in the case of thermoset bio-nanocomposites, it is important to

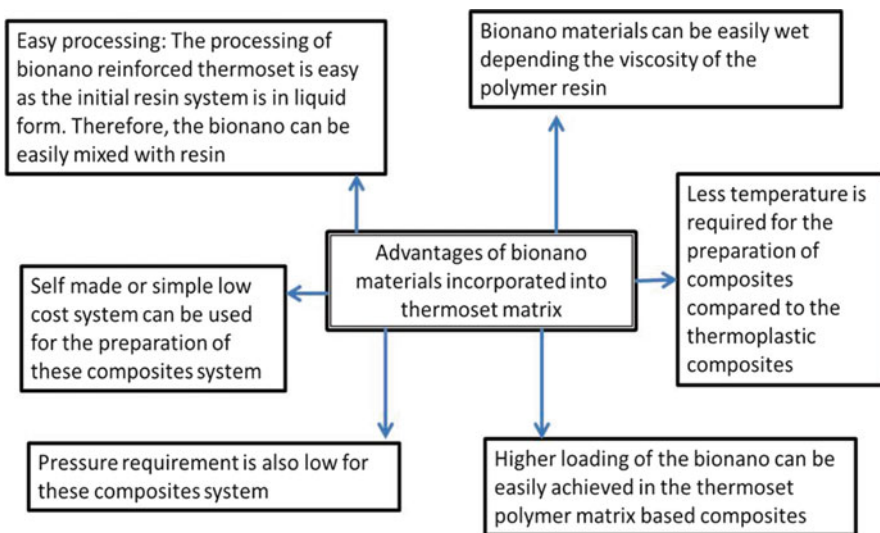


Fig. 12.7 Advantages of bionano-materials incorporated into the thermoset matrix

emphasize the types of nanofibres (including nano-hemp, nano-flax, nano-kenaf, etc.) which are common reinforcements in the thermoset composites for civil and industrial application (Carsten et al. 1999). The possibility to use thermoset bio-nanocomposites with lamination technique with lower temperature and low-pressure technologies significantly broadens their exploitation. Therefore, nanofibre-reinforced thermosets are processed by a relatively simple processing technique, including hand layup and spraying and the liquid composite molding techniques (Karger-Kocsis and Wu 2004). In addition, a variety of other techniques, including centrifugal casting, cold press molding, filament winding, pultrusion, reinforced reaction injection molding, rotational molding, and vacuum forming, were tried, but usage of nanofibre composites was seldom reported. Indeed, thermoset resin also allows the use of advanced close mold processing method including resin mold transfer, which involves a lower impact in the working environment and yet high reinforcement content and performance (Tia and David 2002; Matos Ruiz et al. 2000).

### 12.4.1 Epoxy Bio-nanocomposites

Epoxy matrix-based composites have been studied by a number of researchers for different applications using different types of nanofibres including nano-bacterial cellulose, nano-electrospun cellulose fibres, nano-recycled cellulose fibres, nano-cellulose whiskers, and nanocrystal cellulose (Rostamiyan et al. 2015; Yu et al. 2014; Oh et al. 2014). Nanofibre-based epoxy composites have been reported to be novel materials for different applications (Yu et al. 2014). Different types of surface functionalization of nanofibres have been carried out for further enhancing their use in epoxy-based polymer composites (Rafiq et al. 2014). For example, the nano palm tree fibre-reinforced epoxy was prepared, and it has been observed that at low frequency, the dielectric constant of the composites increases with the increase in temperature (Abdul Khalil et al. 2013a, b). It was summarized that in addition to the relaxation associated with the glass transition ( $T_g$ ) of the epoxy resin and ionic relaxation, the nano palm tree fibres also give rise to the other relaxation associated with the interfacial polarization of nano palm tree fibres and epoxy resin (Abdul Khalil et al. 2013b). Besides, the fracture toughness of the epoxy increased simultaneously with the addition of the nanofibre. This is due to the reinforcing effect that strongly depends on the aspect ratio of the nanofibre and its uniform dispersion.

Short-date nano palm tree fibre-reinforced polyepoxy composites were also prepared and studied in the different mechanical property analyses using resin transfer molding techniques (Nik Salleh et al. 2013). The interaction between nanofibres and epoxy matrix is enhanced by treating the nanofibres with chemical modification. The modification was carried out using TEMPO (2,2,6,6-tetramethylpiperidine-1-oxy radical) catalyst mediation (Abdul Khalil et al. 2013b). It is demonstrated that the surface modification assisted the fabrication of epoxy composites using resin transfer molding and increased the mechanical properties.

Nanofibre-reinforced epoxy composites/honeycomb core sandwich panels were also studied in detail for creep properties (Reis et al. 2013). It was observed that at ambient conditions (with relative humidity at 20–50 %), sandwich panels showed linear viscoelastic properties (stress level less than 30 % of their failure stress) (Yeh and Chiu 2011). However, higher relative humidity (65 %) produced a significant acceleration of creep strain. The test result of nano-flax fibre-reinforced epoxy resin produced by the resin transfer molding method was absorbed, and at the nanofibre volume fraction of 21–47 %, the nanofibre direction elastic modulus was 15–39 GPa, and the tensile strength was recorded at 132–280 MPa (Tsai and Chang 2011). Another study tested the similar nano-flax fibre/epoxy composites with 50 % of nanofibre volume fraction and presented 32 GPa as its nanofibre direction elastic modulus and 268 MPa as its tensile strength. Moreover, surface treatment to remove the moisture of nanofibre affects the bending strength in which after the surface treatment of 1–3 % NaOH, the surface treatment improved the strength of overall composites (Yeh and Lin 2009).

To test the real capability of nanofibre reinforced with epoxy composites, the water adsorption behavior of the carpet waste jute yarn reinforced with polymer matrix was studied. Along with the water absorption behavior, mechanical properties including flexural properties of nanofibre-reinforced composites and their impact were also studied after aging in distilled water (Wang et al. 2012; Zou et al. 2006). With the purpose of improving the nanofibre adhesion with the epoxy matrix, alkali treatment of the nanofibres was carried out and indicated that alkali treatment increases the mechanical properties and decreases the water adsorption of the resulting composites. This behavior was attributed to the strong interface improvement between the reinforcement and the nanofibres (Zou et al. 2006).

Hybrid nanofibre using nano-jute and nano-bagasse as the reinforcement was prepared (Jochen and Andrzej 1997, 1999; Bhosale et al. 2014). Both nanofibres were treated with furfuryl alcohol to increase the adhesion between the composite components. It was confirmed through different techniques that the surface modification decreased the composites' hydrophilicity and significantly increased the overall mechanical properties of the composites (Mishra and Biswas 2014).

### ***12.4.2 Polyester Bio-nanocomposites***

Polyester resin is commonly used as composite matrix because of its reasonable price, ease of handling, and balanced mechanical, electrical, and chemical properties as well as fire resistance properties (Godina et al. 2004; Navin et al. 2000; El-Tayeb 2008). However, polyester resin is unstable and degradable in the environment. Therefore, some studies focused on incorporating nanofibres in order to increase the modulus both below and above the glass transition temperature of polyester (Sami and Ridha 2007). It is generally accepted that comprising toughness would lead to the increase in stiffness. It has also been reported that the toughness of nanofibre/polyester composites is hooked on the morphology and composition of nanofibre in

polyester matrix. At low concentration, proper dispersion improves the toughness (Navin et al. 2000).

Nano-Luffa fibre-reinforced polyester composites have been used to tailor the polyester properties (Tanobe et al. 2014; Seki et al. 2012; Ghali et al. 2011). To improve the nanofibre–polyester interface, nano-Luffa fibre was alkali-treated at two temperatures, with varying alkali concentrations. It was observed that similar to other nano-natural fibres (Boynard et al. 2003), the nanofibres' external surface was completely removed and nanofibres with 5 % NaOH treatment exhibited the best flexural mechanical properties that attributed to mechanical interlock due to the increase of nanofibre roughness and the increase of the nanofibre contact area (Boynard and D'Almeida 2000).

Nano-sugarcane bagasse-reinforced polyester composites were also prepared and examined for tribological applications (Loh et al. 2013; Rodrigues et al. 2011). For that, compression molding along with hand layup techniques was used to prepare the composites with chopped 1.5 and 10 mm nanofibres randomly distributed and unidirectional mat nanofibres (Wu 2011). The SEM micrographs of composites showed a fairly good bonding between nano-sugarcane bagasse and matrix which can be served as the replacement to nano-glass fibre-reinforced composites (Brugno et al. 2011; Lee and Mariatti 2008).

The effect of chemical treatments on mechanical properties and water adsorption potential of nano-bamboo fibre-reinforced polyester also gained a lot of interest (Monteiro et al. 2014; Rassiah et al. 2014a). The chemical modification of nanofibres in mat form was carried out with maleic anhydride, permanganate, benzoyl chloride, benzyl chloride, and pre-impregnation (Rassiah et al. 2014b). It was summarized that the mentioned pretreatment significantly improved the mechanical properties and water adsorption potential of the resulting nanocomposites.

Nano-Brazilian sisal fibre-reinforced polyester composites were also prepared and studied for mechanical properties and pullout analysis (Ornaghi et al. 2014; Oladele et al. 2014). The surface functionalization of these nanofibres with NaOH and *N*-isopropyl acrylamide was subsequently carried out to get the polyester composites with optimum mechanical properties (Belaadi et al. 2014). Lignin content and density of nanofibres reduced with the chemical treatment and *N*-isopropyl acrylamide treatment caused a significant reduction in moisture absorption. The optimum result was achieved by treating the nanofibres with 2 % *N*-isopropyl acrylamide (Pereira et al. 2014).

Nano-kapok fibre-based polyester composites were prepared and subjected to different mechanical properties (Venkata Reddy et al. 2008, 2009). The surface modification of nano-Kapok fibres was carried out using alkali treatment, before being hybridized with glass and nano-sisal fabrics in polyester matrix (Mwaikambo and Bisanda 1999). It was recorded that the impact properties of composites were significantly increased with the loading of nanofibres. It enhanced the mechanical properties due to the excellent dispersion of the reinforcement in the polyester composites along with better load distribution (Venkata Reddy et al. 2008).

Currently, some research works paid attention to the fabrication and properties of nano-bacterial cellulose-reinforced polyester composites. The composites were

prepared using resin transfer molding, and to improve the interface, silane functionalization of nanofibres was carried out. It was revealed that silane-modified nanofibre-reinforced composites exhibit greater nanofibre–resin adhesion strength in comparison with untreated composites.

#### **12.4.2.1 Epoxy/Polyester Bio-nanocomposites**

Nano palm fibre-reinforced polyester and epoxy matrices were prepared using the short fibre form. Due to its hydrophobic matrices, epoxy/polyester owns repulsive forces leading to aggregation and poor interfacial contact of the nanofibre (Amor et al. 2014). Moreover, the greater presence of surface OH groups on reactive cellulose surfaces with respect to nanofibres may also permit further modification to alter the hydrophilicity (Ibrahim et al. 2013; Shinoj et al. 2011). Thus, surface modification of nanofibres was carried out to improve interface adhesion between the nanofibre and matrix (Amor et al. 2014). As an example, in the case of nanocellulose-reinforced epoxy/polyester bio-nanocomposites, sulfate ester groups were randomly introduced on the surface during H<sub>2</sub>SO<sub>4</sub> hydrolysis due to the formation of sulfate ester with high surface acidity, while HCl hydrolysis results in hydroxylated surfaces (Kaddami et al. 2006).

### **12.4.3 Polyurethane Bio-nanocomposites**

Polyurethane has commercial benefits as it can be molded, injected, extruded, and recycled (Jae et al. 2005; Khalid et al. 2007; Cynthia et al. 2005). One of the intended uses of polyurethane is the incorporation of bionano-material, including a nanofibre that is biocompatible and nontoxic. Polyurethane bio-nanocomposites are generally produced either using the solgel technique or the intercalation technique (Khalid et al. 2007).

In general process of polyurethane composites, the achieved reinforcement of nanofibre into polyurethane depending on the nanofibre intrinsic characteristics, including aspect ratio, shape, surface area, and rigidity (Saralegi et al. 2014; Pant et al. 2014). The dispersion and distribution state of nanofibres can be altered by the processing conditions, while compatibility/affinity with polyurethane can be improved by surface modification of nanofibres (Xiao et al. 2012; Auad et al. 2012).

### **12.4.4 Phenolic Bio-nanocomposites**

Phenol formaldehyde is the most commonly used resin systems, mostly thanks to its extreme temperature and fire resistance (Seena et al. 2002). The polymerization reaction between phenols and aldehydes (phenol and formaldehyde in specific)

generates phenolic resins (Sreekala et al. 2000; Richa et al. 2000). This polymer can be molded, and cure (cross-linking) may be affected by the temperature changes (Richa et al. 2000).

In phenolic composites, phenol formaldehyde is the most commonly used resin system. Lignocellulosic *Saccharum ciliare* nanofibre-reinforced polymer biocomposites were prepared using the compression molding technique employing phenol formaldehyde as a novel polymer matrix (Maiti et al. 2010; Kaith et al. 2010). It was found that different mechanical properties including tensile strength, flexural strength, compressive strength, and wear resistance increase up to 30 %, and beyond this loading, the properties decreased (Maiti et al. 2010). Surface modification of this bionano-material was carried out using silane treatment through aminopropyl triethoxysilane to evaluate the morphological and physicochemical properties (Singha and Thakur 2009).

Another type of bionano-material, which is nano-sisal fibre, is studied at different loading on phenolic composites. To further enhance the adhesion between nano-sisal fibres with the phenolic matrix, these bionano-materials were subsequently hydroxymethylated (Zeng et al. 2014; Bu et al. 2014). In other studies, lignin was extracted from nano-pinus-type wood and sugarcane bagasse (Wei et al. 2013). The lignin was further modified via a methylation process to produce methylated lignin. Another study has utilized chlorine dioxide as an oxidative to modify the nano-sisal fibre surface and subsequently the furfuryl alcohol and poly(furfuryl alcohol) treatment before introducing it to phenolic matrix (Li et al. 2014). Changes in the surface morphology of the nanofibres were confirmed by SEM analysis and demonstrated that nanofibre modification with methylated lignin induced significant changes in the morphology of the nanofibre surface. The modified nanofibre-reinforced composite also demonstrated greater impact strength due to the strong bonding between the composite components and was confirmed under SEM micrograph. Furthermore, the surface-modified nanofibres reinforced with phenolic matrix also exhibit low water absorption, and phenolic resin penetrated very well into the modified nanofibre (Da Silva et al. 2012; Zeng et al. 2010). This process resulted in the blocking of water passage through their channel.

#### ***12.4.5 Polyvinyl Ester Bio-nanocomposites***

Along with other esters used in polymer composite preparation, polyvinyl ester was also considerably investigated (Everson et al. 2006; Mallarino et al. 2005). Nano-bast fibres and various other types of nano-natural fibre-reinforced vinyl ester polymer composites have been prepared using resin transfer molding specifically for the automotive applications (Mallarino et al. 2005). For example, vinyl ester composites reinforced with natural nanofibres from *Luffa cylindrica* fruit were prepared after modification treatment. Different extraction treatment along with mercerization and esterification with dianhydride compounds was used to modify these nanofibres (Bazle et al. 2001; Sullivan et al. 1984). At the same time,

nano-pulp fibre-reinforced polyvinyl ester composites were studied to evaluate their different mechanical and thermal properties. It was observed that modification of nanofibre resulted in more compatibility with the polymer matrix, which indicated that the incorporation of nano-pulp fibres considerably improved the composite storage moduli compared to pristine polyvinyl ester (Thielemans et al. 2002; Liliana et al. 2009).

Another study on nano-flax fibre-reinforced polyvinyl ester composites manufactured using vacuum-assisted resin transfer molding method recorded that, at a nanofibre fraction of 25 %, the tensile strength was recorded at 122.4 MPa (Hongyan et al. 2008). In the case of nano-flax fabric composites, it was found that nanofibre volume fraction and the tensile strength are 25 % and 62 MPa, respectively (Roberts et al. 2003). Nano-flax fibre was selected to be incorporated into the polyvinyl ester due to its greater strength and better mechanical behavior than other nano-natural fibres, and the polyvinyl ester was selected as a resin due to its cheaper price, easier procurement, and better treatment for the resin injection (Goutianos et al. 2006).

Hybrid polymer composites of nano-oil palm empty fruit bunch fibre along with glass fibre were prepared using polyvinyl ester as polymer matrix. It was recorded that mechanical properties of the composites were significantly better than the mechanical and chemical board.

## 12.5 Thermoplastic Bio-nanocomposites: Manufacturing and Properties

The different thermoplastic composite systems based on the different types of thermoplastic reinforced with the number of nanofibres (nano-kenaf, nano-jute, nano-flax, nanocellulose) have been produced and characterized. In order to take full advantage of the nano-sized nanofibre reinforcement, a good dispersion in the thermoplastic matrix was required to enhance the reinforcement of hydrophilic phase and nonpolar matrix compatibility (Denault et al. 2014; Gates and Sun 1991).

The processing of nanofibre/thermoplastic composites most commonly involves extrusion of ingredients above the melt temperature of the matrix, followed by shaping of the operation including injection molding and thermoforming (Carneiro and Maia 2000).

### 12.5.1 Polypropylene Bio-nanocomposites

Some research works focused on developing the nano-kenaf fibre-reinforced polypropylene composites and concluded that it could be thermoformed for a wide variety of applications with properties that are comparable to existing synthetic composites (Pang and Ismail 2014; Hao et al. 2014; Sallih et al. 2014). Furthermore, the presence of other bio-fibres including garlic nano-powder or nisin usually



incorporated into polypropylene as antimicrobial agents leads to detriments in the tensile strength and elastic modulus values and leads to increase of the strain at break (Jirukkakul 2013). Another study found that a combination of nanocellulose derived from empty fruit bunch nanofibres and polypropylene matrix improved mechanical properties of polypropylene (Zainudin et al. 2014; Rivai et al. 2014; Ramli et al. 2013). The bonding effects of regenerated nanocellulose natural fibre were studied using a Raman technique, and the bonding was found to be dependent on the nanocellulose concentration, pH value, degree of crystallinity, and ionic strength of the oxidized nanocellulose aqueous suspension (Chee et al. 2013).

### **12.5.2 Polyethylene Bio-nanocomposites**

Polyethylene is a hydrophobic thermoplastic composite, widely used in everyday life. Due to polyethylene's cheap price, availability, and good processability, it is a promising matrix material for bio-nanocomposites (Kuruville et al. 1996; Tony et al. 2005). This cheap price plastic constitutes about 70 % of the whole amount of thermoplastic consumed by the plastic industries (Abdelmouleha et al. 2007). Therefore, utilizing nanofibres significantly reinforces the high-density polyethylene matrix and the bio-nanocomposites applied in lots of value-added products instead of those conventional applications (Kuruville et al. 1996).

Nanocellulose derived from cotton reinforced polyethylene biocomposites fabricated through an extrusion process (Fukuya et al. 2014; Iwamoto et al. 2014). Before the fabrication process, two separate methods were applied to overcome the nanocellulose dispersion challenge. Firstly, "the physical pretreatment method" was used to preblend polyethylene in a nanocellulose water suspension before extruding. Secondly, the mixture of the first step was dehydrated and freeze-dried before being compounded and extruded by HAAKE minilab (Abdelmouleha et al. 2007). This is a good technique to save time and the environment due to its dispersing agent-free nature. The "dispersion agent method" is another technique which uses polyoxyethylene as a dispersing agent. After applying polyoxyethylene dispersion as a coating agent, the nanofibres were mixed into the HAAKE minilab with polyethylene granules before being extruded. In order to increase nanocellulose and polyethylene matrix compatibility, maleic anhydride-grafted polyethylene was applied as a coupling agent (Pan et al. 2014; Lin and Dufresne 2013).

### **12.5.3 Polyvinyl Acetate Bio-nanocomposites**

There are some interests to study the impact on addition of nanocrystalline cellulose into polyvinyl acetate. For example, it reported the effectiveness of nanocrystalline cellulose extracted from *Phormium tenax* leaf natural fibre by acid hydrolysis as reinforcement phase in the polyvinyl acetate matrix (Kaboarani et al. 2012). The effect of nanocrystalline cellulose types and the content of polyvinyl acetate

structural properties were investigated deeply, and their effects on the water adsorption capacity have been evaluated in the case of the potential application in food packaging material (Nancy et al. 2006; Zhang and Zhu 2000).

#### **12.5.4 Polycaprolactone Bio-nanocomposites**

The efficiency of nano *L. cylindrica* fibre was investigated as a reinforcement agent for the polycaprolactone matrix (Siang et al. 2012). In general, nano *L. cylindrica* fibre was well known in the field of polymer composites because of its high cellulose content and low density ( $0.82\text{--}0.92\text{ g cm}^{-3}$ ), which is lower than the common nano-natural fibres including nano-sisal ( $1.26\text{ g cm}^{-3}$ ), nano-hemp ( $1.48\text{ g cm}^{-3}$ ), nano-ramie ( $1.5\text{ g cm}^{-3}$ ), and nano-cotton ( $1.51\text{ g cm}^{-3}$ ) (Libin et al. 2013; Gilberto et al. 2013). It was observed that polycaprolactone matrix mechanical and thermal properties enhance incorporate with nano *L. cylindrica*.

To exploit further on the potential of nano *L. cylindrica* fibre/polycaprolactone composites, its water barrier properties with varying content of nanofibres have been studied through complementary approaches including water sorption and water permeation kinetics (Arantzazu et al. 2014).

#### **12.5.5 Polyvinyl Alcohol Bio-nanocomposites**

Polyvinyl alcohol is the chief hydrophilic water-soluble polymer synthesis up to now. As a result of its outstanding chemical and physical properties, easy processing technique, low cytotoxicity, and biocompatible synthetic polymer, it has been widely used in different fields of biotechnology and biomedicine (Dutta et al. 2002). Polyvinyl alcohol is a highly versatile polymer offering a wide spectrum of property profiles, which opens the way of using it in a broad field of applications, including as a matrix for biodegradable composites (Lindsey and Street 1984). The nanocomposite approach has emerged in the last two decades as an efficient strategy to upgrade the structural and functional properties of polyvinyl alcohol (Mirmohsenia and Wallace 2003).

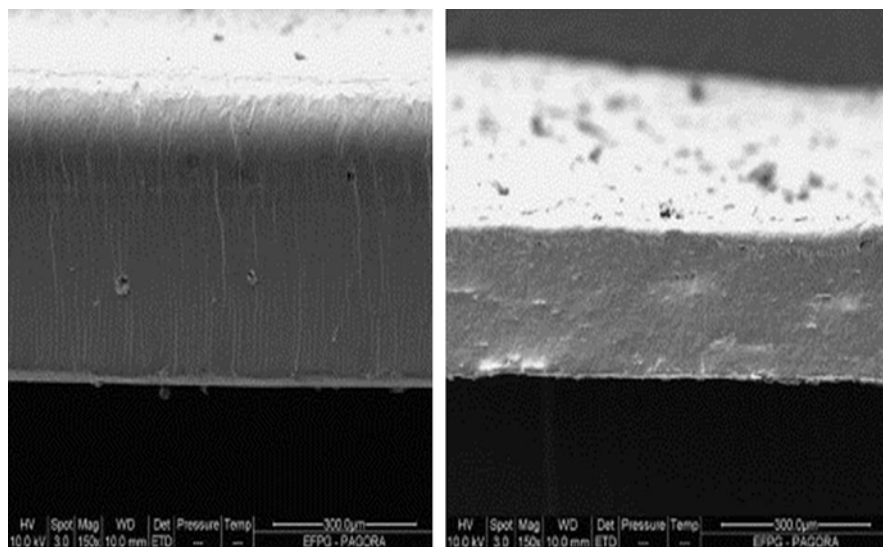
A new class of engineering-designed polyvinyl bio-nanocomposites was introduced by incorporating cellulose nanocrystal to overcome the limited biological performance and to enhance the mechanical properties of polyvinyl alcohol (Zhi-Gang et al. 2002; Li et al. 2013a, b, 2014). Indeed, the use of cellulose nanocrystal as bionano-materials in polyvinyl alcohol is to increase the load-bearing constituent in developing new and inexpensive biodegradable materials due to their high aspect ratio (Pereda et al. 2011). In this case, cellulose nanocrystal is extracted from a wide variety of natural sources available throughout the world, including ramie, potato, starch, cotton, wood, and others. The addition of cellulose nanocrystal was found to produce 100 % improvement in the tensile modulus of certain polyvinyl alcohol composites (Baheti and Militky 2013; Lee et al. 2009).

## 12.6 Elastomer Bio-nanocomposites: Manufacturing and Properties

Using nanofibres as reinforcing materials in elastomer bio-nanocomposites is considered as an innovative environment-saving application of natural materials by reducing CO<sub>2</sub> emissions in the atmosphere during their production, processing, and use (O'Connor 1977; Anongnuch et al. 1999; Coran et al. 1974). Nanofibre-reinforced elastomers have mechanical advantages over conventional ones, being 30 % lighter and 3–4 times stronger than the currently used material. They are also less susceptible to damage from heat, chemicals, and spilled gasoline (Tayaa et al. 1998). However, the strength of elastomer-reinforced nanofibres remains far below the theoretical properties. This is due to the limited compatibility between hydrophilic nanofibres and hydrophobic elastomer matrices (George 1971). Poor dispersion of nanofibres and the insufficient adhesion between the nanofibres and elastomer matrix also induce unsatisfactory mechanical properties of the bio-nanocomposites. To overcome the compatibility difficulties, nanofibres have been subjected to physical treatment (plasma), chemical modifications (esterification, etherification, oxidation, silylation, and polymer grafting by click chemistry), and direct silanol nanofibre condensation with prior hydrolysis treatment (Anongnuch et al. 1999). To solve the dispersion problem, the usage of an organic medium, including surfactant, is the most common way.

### 12.6.1 Natural Rubber Latex Bio-nanocomposites

Natural rubber latex is a colloidal dispersion of *cis*-1,4 Polyisoprene in a form of milky sap which is obtained from the incised bark of *Hevea brasiliensis* trees (Siby and Karger-Kocsic 2004; Kalaprasad and Alain 2003; Julien et al. 2010; Srisuwan et al. 2014). There are some limitations of natural rubber latex due to its hardly degraded solid waste upon disposal (Kalaprasad and Alain 2003). This is due to the presence of inorganic materials, including antioxidants, and sulfur cross-links that make natural rubber latex resistant to microbial infections. Even though its polyisoprene backbone chain degradation can occur in the natural environment, the process is time-consuming. Thus, the combination of natural rubber latex with nanofibre-based filler has been studied to accelerate the process (Phummor et al. 2014). This is used to make the latex-based products decomposable by microorganism this is brought by the several characteristics of nanofibre-based filler, including its cheapness, natural abundance, high susceptibility to biodegradation, and being a renewable resource. On the other hand, the incorporation of nanofibres largely increased the tensile strength, tensile modulus, and abrasion resistance of the natural rubber latex (Wongsorat et al. 2014; Yong and Mustafa 2014). This consequence resulted in a homogenous and well-dispersed bio-nanofibres in the natural rubber latex matrix (Fig. 12.8).



**Fig. 12.8** Scanning electron micrographs of (a) natural rubber latex and (b) natural rubber latex/cellulose whisker nanocomposites containing 10 % bagasse cellulose whiskers (Julien et al. 2010)

Initially, most of the nanofibre has no reinforcement ability of the natural rubber latex compound due to its hydrophilic characteristics. In order to achieve superior reinforcement, several techniques including the addition of coupling agent, ball milling, surface modification, acid hydrolysis, enzyme hydrolysis, and high-pressure homogenization have been applied (Tzounis et al. 2014; Manaila et al. 2014; Supri et al. 2014; Wisittanawat et al. 2014). For example, N<sub>2</sub>-pyrolyzed rice bran nanofibre at 900 °C has been used to improve the tribological, friction, thermal, vulcanization, and mechanical properties of the natural rubber latex (Supri et al. 2014). This surface modification of rice bran nanofibre exhibits various fascinating characteristics, including porous structure, high hardness, low density, low fraction, and high wear resistance (Xu and Kong 2014).

### **12.6.2 Hydrogenated Nitrile Butadiene Rubber Bio-nanocomposites**

Hydrogenated nitrile butadiene rubber shows the resistance against oil, chemical, heat, pressure, and ozone (Ding et al. 2015). They are broadly used in the automobile, aviation, oil field, and other industries for the high-performance rubber fittings. However, the crystallization of its ethylene group occurred at a temperature below -30 °C and deteriorated other physical properties (Rajesh et al. 2008, 2013; Lu et al. 2008). By incorporating nanofibre onto its rubber chains, the composite rubber possesses the balanced properties of high strength, elongation, and good oil resistance to a wide range of temperatures. This is brought by the formation of inter H<sub>2</sub> bonds

between the rubber and nanofibres (Rajesh et al. 2008). However, careful analysis discovers that it is not beneficial to the elasticity of rubber by adjusting the amount of nanofibres at a wide range, because high strain hysteresis (high loss factor) always brings slow response of its recovery. Therefore, maintaining the low content of nanofibres may be the key to tune the properties of produced composites (Konar et al. 2013; Threepopnatkul et al. 2011). Furthermore, it has been reported that the amount of nanofibres has important effects on the microstructure and nanofibre morphologies of the composites which may connect directly with the reinforcements of the bulk. It is believed that tuning the properties of hydrogenated nitrile butadiene rubber by nanofibres with H<sub>2</sub> bonds might be important to develop modification technologies in the sealing applications (Konar et al. 2013).

## 12.7 Biopolymer Bio-nanocomposites

### 12.7.1 Starch Bio-nanocomposites

As a result of its renewability, abundance, and biodegradability, starch has been widely studied as a structural or functional polymer, while its poor mechanical properties and its sensitivity to water are two important drawbacks that should be taken into account (Cristian et al. 2009; Nancy et al. 2009). Starch is the suitable candidate to be embedded in a matrix composed of the hydrophobic polymer when the moisture sensitivity has to be decreased. However, the addition of plasticizer decreases brittleness in the case of mechanical properties, and its mechanical strength degrades accordingly. Furthermore, to improve the low mechanical tensile properties, a classical strategy consists of including rigid nano-sized filler (Deeptangshu 2008). One possibility and factor in contributing the mechanical performance of starch based composites is utilizing nano sized filler (Zhanjun et al. 2011; Nayaka 2010).

Reinforcement of waxy maize nanofibres with filler content between 0 and 5 % into the starch matrix not only showed improvement of mechanical properties but also reduced the water vapor permeability by 40 % (Hélène et al. 2006; Anglès and Alain 2000).

## 12.8 Potential Applications of Bio-nanocomposites

### 12.8.1 Automobile

Automotive related industries area be the chief market for the bionano-composites since numerous automotive parts are produced using nanofibre at the present including flax, hemp, kenaf, and sisal. But nowadays, abaca, pineapple leaf, coir, oil palm,

**Table 12.2** Advantages of bionano-composites in automotive industries

Justification	Reasons	References
Raw material sources	Bionano-composites made with easily available renewable sources	Hartmut and Ulf (2003)
Properties	Lighter weight, low energy consumption, and low-cost product	Chae et al. (2005)
Volatility in oil prices	Impact substitute material markets and nano-natural fibre cost less	Jin-Hae et al. (2004)
Environmental advantages	Government policy	Marion et al. (2008)

bagasse, bamboo, wheat straw, curaua, and rice husk fibres are gaining interest and importance in both research and application in the automotive field (My Ahmed et al. 2005; James et al. 2011). The advantage of using bionano-composites in automotive industries could be money-saving, weight reduction, recycling, and marketing incentives rather than technical demand (Table 12.2).

### 12.8.2 Food Packaging

The main function of food packaging is to maintain the quality and safety of food products during storage and transportation and to extend the shelf life of food products. In order to achieve those benefits, unfavorable factors or conditions, including spoilage microorganisms, chemical contaminants, O<sub>2</sub>, moisture, light, and external force, should be controlled and prevented (Valapa et al. 2015; Reddy and Rhim 2014). Therefore, the good food package should have all these properties in addition to the common standard properties of packaging materials including mechanical, optical, and thermal (Sun et al. 2014).

With this respect, polymer bio-nanocomposites offer an ideal biodegradable food packaging material (Cunha et al. 2014; Ghaderi et al. 2014). These concerns include degradation rates under various conditions, changes in mechanical properties during storage, potential for microbial growth, and release of harmful compounds into packaged food products. In addition, polymer bio-nanocomposites are an excellent medium for incorporating a wide variety of additives including antioxidants, anti-fungal agents, antimicrobials, colors, and other nutrients (Khan et al. 2014).

### 12.8.3 Structural Applications

Structural parts are usually utilized in civil structures, and construction used to be extremely large. Due to the effect of the shape, size, and dimension of large structures on the polymer flow and wettability in bionano-composite production, controlling these elements seems very important (Aboshio et al. 2015; Fasahat et al. 2015;

Jölly et al. 2014). Nanofibre is a hydroscopic material, which brings the curing reaction of the resin for structural application into challenge. In order to overcome this issue, all of the moisture that could polymerize by reacting with the resin should be eliminated by treating the nanofibre (Sprenger et al. 2014).

#### **12.8.4 Tissue Engineering**

Bionano-filled polymer composites were utilized as matrices for tissue repairing and regenerating and drug delivery in a hydrogel form in the case of tissue engineering and regenerative medical field application (Kucinska-Lipka et al. 2015; Zhang et al. 2014; Kim et al. 2014). For example, crystal nanocellulose/polyvinyl alcohol composites accurately improve targeted controlled drug delivery to the preferred site of action (Zhang et al. 2014). The possibility to incorporate crystal nanocellulose into polyvinyl alcohol has numerous advantages, including the isolation of drug, slower drug release rates, and achievement of different drug release profiles, as well as the incorporation of multiple drugs in different microsphere populations (Bhaarathy et al. 2014; Shi et al. 2014).

### **12.9 Conclusion**

In the last decade, bionano-composites consisting of polymeric matrix materials and nanofibres have attracted scientific and industrial interest due to their improved properties. Their usages cover a varied range of applications such as high-barrier packaging for food and electronics and automotive and aerospace applications. At low nanofibre content as compared with the common micro and macro or neat counterparts, they reveal high property improvements in terms of lightweight, mechanical, corrosion resistance, lower density, lower weight components, thermal gas permeability resistance, and fire-retardant properties. Other advantages of bionanocomposites over traditional polymeric composites are economic viability, reduced tool wear during machining operation, improved energy recovery, reduced dermal and respiratory irritation, and biodegradability. Numerous life cycle assessment studies conducted with bio-nanocomposites prove these advantages.

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