

## Environment Friendly Composite Materials: Biocomposites and Green Composites

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### ABSTRACT

Biocomposites can supplement and eventually replace petroleum-based composite materials in several applications. Several critical issues related to bio-fiber surface treatments is to make it a more suitable matrix for composite application and promising techniques need to be solved to design biocomposite of interest. The main motivation for developing biocomposites has been and still is to create a new generation of fiber reinforced plastics material competitive with glass fiber reinforced ones which are environmentally compatible in terms of products, use and renewal. There is an immense opportunity in developing new biobased products, but the real challenge is to design suitable bio-based products through innovation ideas. Green materials are the wave of the future. Bio-nanocomposites have very strong future prospects, though the present low level of production, some deficiency in technology and high cost restrict them from a wide range of applications.

**Keywords:** Polymer, biocomposite, biodegradation, green technology

### 1. INTRODUCTION

Development of advanced polymer composite materials having superior mechanical properties opened up new horizons in the engineering field. Advantages such as corrosion resistance, electrical insulation, easy processability at relatively less energy requirement in tooling and assembly costs, higher stiffness and strength, fatigue resistance and lower weight than metals have made polymer composite widely acceptable in structural applications. The so-called advanced composites have replaced metals because of their excellent mechanical properties and low density giving them high specific strength and stiffness<sup>1</sup>. Such weight savings are highly desirable for applications in aerospace to transportation to reduce weight and associated fuel consumption. Another distinct advantage is their ability to be engineered to obtain required properties in different directions by appropriate fiber placing in different layers of the laminated structure.

Composite properties depend on the properties of the constituent materials i.e. the fibers and resins used. The strength and stiffness of the composites are directly a function of the reinforcing fiber properties which carry most of the load and their volume content. The resin helps to maintain the relative position of the fibers within the composite and, more importantly, transfers the load from the bottom fibers to the intact fibers. As a result, fiber/resin interfacial properties are also important and have a significant effect on composite properties including toughness and transverse fracture stress. To fabricate high strength composites, all three factors namely fiber properties, resin properties as well as fiber/resin interface characteristics are critical.

Currently most of the fibers and resins are derived from petroleum feed stocks and do not degrade for several decades under normal environmental conditions<sup>2</sup>. Composites made from thermosetting resins cannot be reprocessed or recycled, however, a small fraction of these thermosetting composites are crushed into powder used as filler or incinerated to obtain energy in the form of heat, most of them end up in land- fills at the end of their life. In anaerobic conditions, such as those in land - fills, the petroleum based composites may not degrade making that land unavailable for any other use. On the other hand, incineration produces toxic gases and requires expensive scrubbers. Both incineration and dumping in land- fills are environmentally undesirable as well as expensive. In the future, these methods of disposing of composites are expected to get even more expensive as the pollution laws all over the world get stricter and the number of land - fills decline. In addition, at the present rate of consumption, the world petroleum resources are estimated to last for the next 50 years or so<sup>2</sup>. Thus, there is a great interest generated in developing green composites using fully sustainable, biodegradable, environment friendly and annually renewable fibers and resins, particularly those derived from plants<sup>3</sup>. A variety of resins such as starch, cellulose, proteins, etc and fibers e.g. flax, ramie, kenaf, jute, sisal etc have been used to fabricate green composites for many applications.

### 2. BIO-COMPOSITES

Bio-composites are the combination of natural fibers (bio-fibers) such as wood fibers (hard wood and soft wood) or non-wood fibers (e.g. rice straw, hemp, banana, pine apple, sugar

cane, oil palm, jute, sisal and flax) with polymer matrices from both of the renewable and non-renewable resources. The term 'bio-composites' broadly covers composite materials where at least one constituent should be bio-based

- (i) bio-fiber reinforced petroleum derived polymers which are non-biodegradable e.g., polyolefins polyester, epoxy, vinyl ester, phenolics
- (ii) bio-polymers (e.g. PLA) reinforced by bio-fibers (jute) and
- (iii) bio-polymers reinforced synthetic fibers such as glass or carbon.

Bio-polymers reinforced with bio-fibers are generally considered to be called green composites. A variety of resins e.g. starch, proteins etc. and fibers e.g. flax, ramie, kenaf, jute etc. have been used to fabricate bio-composites for many applications. In many cases, only one component of the composites, either the fibers or the resin, has been biodegradable. Such semi-green composites have the same problem of disposal at the end of their life. Most such semi green composites have moderate tensile strengths and stiffness in the range of 100 MPa or 200 MPa and 1 GPa to 4 GPa respectively. As a result, their applications are restricted only non-critical and non-load bearing parts, such as packaging, casings etc. they cannot be used in load bearing applications where high strength and stiffness are required<sup>4-30</sup>. However, bio-composites are emerging as a realistic alternative to glass reinforced composites and because they are derived from renewable resources, material costs can be markedly reduced with their large scale usage. Several of them, 100 % renewable bio-composites are now available for example 'Polyflax' which has fibers and resins derived from sugarcane. Demands for new hi-tech materials in automotive and aerospace industries could provide an increasingly large market for bio-composites that use natural fibers and bio-based resins and polymers. Currently, it is difficult to replace petroleum based materials from cost and performance perspective. It is not necessary to make 100 % substitution for petroleum base materials immediately. A viable solution<sup>16</sup> is to combine different features and benefits of both petroleum and bio-resources to produce useful products having requisite cost performance properties for real world application

- (a) Low bio-based content product (20 % or less bio-based material)
- (b) Medium bio-based content product (21-50% bio-based content)
- (c) High bio-based content product (51-90% bio-based content).

### 3. BIOPOLYMERS

Biopolymers are generally polymers that are biodegradable. The input materials for the production of these polymers may be either renewable (based on agricultural plants or animal products) or synthetic. There are four main types of biopolymer based respectively on 1. Starch 2. Sugar 3. Cellulose 4. Synthetic materials. Another way of classification of natural bio-degradable polymers are based on the basis of sources a) Polysaccharides b) Proteins c) Bio-polyesters by direct fermentation d) other naturally occurring bio-

polymers<sup>31-33</sup>. These are presented in details in Table 1.

Current and future development in bio-degradable polymers and renewable input materials focus relate mainly to the scaling up of production and improvement of product properties. Larger scale production will increase availability and reduce prices. There are three principal ways to manufacture bio-base polymers

- (i) By making use of naturally occurring polymer through modification, for example cellulose derivatives, thermoplastic starch.
- (ii) By producing bio-base monomers by fermentation & polymerization example polylactic acid (PLA), bio-based nylon 6.
- (iii) By producing bio-based polymers with the help of genetically modified crop and/or microorganism, for example, poly hydroxyl alkanooates.

There are a great number of technologies and processes known for production of bio-based polymer articles, to name a few, extrusion, wet milling, drying, film blowing, thermoforming, injection molding, foaming, hydrolysis & fermentation. Although bio-polymers received much attention, a lot of such materials are not economically viable compared to petroleum based polymers. Although bio-polymer technology is now believed to be in the R & D and demonstration phase, some bio-polymer production processes as there are already commercially viable niche markets. For example, starch polymer are used as packaging material in agriculture and horticulture or as filler for car tyres. PLA that derives from corn are already in commercial production. Starch polymers have the greatest potential to replace polyolefins. Bio-based PLA have good potential for partially replacing PMMA, PA and PET whereas bio PTT's substitution potential for PET and nylon is very high. These suggest that there is a significant technological substitution potential for bio-base polymers which have wide application in medicine, textile packaging, agriculture, transport and several other sectors. Regarding the highly specific necessities of military medicine application, it may provide unique opportunities to absorb those advances at a rapid rate. Due to actual legislation and concerns the armies must consider the modification of ammunition composition in order to decrease or to eliminate toxicity ratio to human health and to the environment. The bio-polymers represent

**Table 1. Bio-degradable polymers**

Natural	Synthetic
1. Polysaccharides	Polyamide
Starch	Poly-anhydride
Cellulose	Poly vinyl alcohol
Chitin	Poly vinyl acetate
2. Proteins	Polyesters
Collagen Gelatin	Poly caprolactone
Casein, Albumin, Silk	Poly ethylene oxide
Protein from grains	Some Poly urethane
3. Polyesters	
Poly hydroxyl alkanooates	Some poly acrylates
4. Other polymers	
Lignin, Shellac, Natural Rubber,	
Cashew nut shell liquid (CNSL)-	

a potential alternative to old energetic materials. Therefore, ammunition items (explosive, propellants, pyrotechnics) will be designed and fabricated so that the items can be easily and safely recycled.

For those stake holders that are considering implementing bio-based polymer production in the chemical sector, the technology could contribute to

- (a) a reduction of petrol consumption
- (b) a relative reduction of fossil fuel import dependence
- (c) a reduction of greenhouse gas emission
- (d) a reduction of discharge of polymer production-associated waste system
- (e) a reduction of harmful solid waste deposition
- (f) an increase in employment in agriculture sector (i.e. harvesting).

#### 4. BIO-BASED FIBERS

Natural fibers can be defined as bio-base fibers or fibers from vegetable and animal origin. This includes all plant fibers (cotton, jute, hemp, kenaf, flax, coir, abaca, ramie, etc.) and protein base fibers (wool, silk). Natural fibers are grouped into three types: seed hair, bast fibers, and leaf fibers depending upon the source. Some examples are cotton, coir (seed/fruit hairs), hemp, flax, jute, kenaf (bast fibers), sisal, abaca (leaf fibers). Of these fibers, jute, flax, hemp and sisal are the most commonly used fibers for polymer composites<sup>19</sup>.

In terms of reinforcing polymeric materials, plant fibers are classified into two types of fibers such as long fiber and short fiber. Long and short fibers are identified by aspect ratio; if  $l/d > 100$ , it was identified as long fibers whereas  $l/d < 100$  was short fibers. Long fibers can be obtained from plant origins and have been used traditionally to produce high value composites for varied application. Short fibers are from plant origins or during fiber processing and are for non-wovens or yarns to be used as reinforcement. Natural fibers in the form of wood flour have also been often used for preparation of natural fiber composites.

Most cellulosic fibers are a renewable resource and the production requires little energy, and in the process, in a way  $\text{CO}_2$  is used while  $\text{O}_2$  is given back to environment. Additionally, thermal recycling is possible where glass and man-made fibers often cause problems in combustion processes. It is believed that the potential of using natural fibers in composite application has not been fully exploited as the full strength of fiber building blocks or micro-fibrils is not being utilized in the raw material.

Compared to synthetic fibers, natural plant-based fibers have several specific advantages. These natural fibers have low cost, high specific mechanical properties, good thermal and acoustic insulation and bio-degradability<sup>5,35-37</sup>. Table 2 shows that the tensile strength of glass fibers is substantially higher than that of natural fibers even though the modulus is of the same order. However, when the specific modulus of natural fibers (modulus/specific gravity) is considered, the natural fibers show values that are comparable to or better than those of glass fibers. These higher specific properties are one of the major advantages of natural fiber for making composites for applications wherein the desired properties also include weight

**Table 2. Mechanical properties of natural fibers<sup>19</sup>.**

Fiber	Specific Gravity	Tensile Strength (MPa)	Modulus (GPa)	Specific Modulus
Jute	1.3	393	55	38
Sisal	1.3	510	28	22
Flax	1.5	344	27	50
Hemp	1.07	389	35	32
Glass fiber (E-glass)	2.5	3400	72	28

reduction. Natural fiber composite has superior performance at a given price. Natural fiber composites have significant potential in transportation & construction market.

The chemical composition of natural fibers varies depending upon the type of fiber. Primarily, fiber contain cellulose, hemi-cellulose, pectin and lignin. The properties of each constituent contribute to the overall properties of the fibers. Hem-cellulose is responsible for the bio-degradation, moisture absorption and thermal degradation of the fiber as it shows least resistance whereas lignin is thermally stable but is responsible for the degradation on exposure to uv light<sup>19</sup>.

Since all plant-based fibers come in short length, they need to be twisted together to form yarns to obtain suitable continuous length forms. Yarns can be woven or knitted to tailor bi-directional properties. Staple fiber yarns are twisted during spinning to increase the cohesion (or friction) between fibers and localize the micro damages within the yarn leading to higher failure strength<sup>4,38-40</sup>. Woven fabrics have also been used as reinforcement for composites. Woven fabrics have two sets of yarns interlaced at right angles. The yarns running in longitudinal direction are referred to as warp or end and the yarns in the transverse direction are referred to as weft or fill. Balanced woven fabrics have the same number of yarns per cm, yarn count and spacing in both warp and weft directions and, as a result, give uniform tensile properties in both longitudinal and transverse directions. Woven fabrics offer several advantages such as impact resistance, damage tolerance, toughness and the ease of manufacturing<sup>38,39,41-43</sup>.

Flax fiber has lower density than glass fiber and as a result it affords to a comparable specific strength<sup>19</sup>. Spun yarns made from flax fibers have been used for fabricating unidirectional yarn reinforced green composites<sup>4</sup>. As far as composite applications are concerned, flax and hemp are two fibers that have replaced glass fibre automotive counterparts, especially prevalent in the German automotive industries. Sisal is often applied with flax in hybrid mats, to provide good permeability when the mat has to be impregnated with a resin. In some interior applications sisal is preferred because of its low level of smell compared to fibers like flax<sup>44</sup>.

#### 5. FIBER-MATRIX INTERFACE

The mechanical properties of fiber reinforced polymer composites are of great importance in deciding their end applications, Mechanical properties of composites depend on the properties of the constituent fibers, the matrix, and the fiber/matrix interfacial shear strength (IFSS). Fibers are the main load bearing components of a composite material and the fiber

strength directly transposed to the strength of composite structure whereas the matrix is responsible for holding the fibers in place and, more importantly, transferring the load from the broken fibers to their neighboring intact fibers through the interface. The IFSS is a critical factor that controls the toughness, transverse mechanical properties, and interlaminar shear strength (ILSS) of the composite materials. The tensile strength of the composite depends on how well the stress can be transferred from the broken to the surviving fibers through shear in the resin at the interface. Nardin and Ward<sup>45</sup> have suggested that the interfacial adhesion can be controlled by (a) physicochemical interaction (b) mechanical interaction (interlocking) and (c) chemical interaction or bonding. Good interfacial adhesion provides composites with structural integrity and good load transfer from broken fibers to intact fibers through the matrix, maintaining the integrity of the composites and resulting in strong composites<sup>46-49</sup>. Weak interfacial bonding, however, encourages energy absorption through interfacial cracks, thereby increasing the fracture toughness of the composites. The interfacial shear strength (IFSS) is measured using the single fiber composite (SFC) technique<sup>48-52</sup>. The microbead test has also been shown to be useful to measure the fiber/resin IFSS in the case of natural fibers<sup>53-56</sup>.

## 6. MODIFICATION OF NATURAL FIBERS

Natural fibers are incompatible with the hydrophobic polymer matrix and have a tendency to form aggregates. These are hydrophilic fibers and thus exhibit poor resistance to moisture. To eliminate the problems related to high moisture absorption, treatment of fibers with hydrophobic aliphatic and cyclic chemical structures has been attempted. These structures contain reactive functional groups that are capable of bonding to the reactive groups in the matrix polymer. Thus modification of natural fibers is attempted to make the fiber hydrophobic and to improve interfacial adhesion between the fiber and the matrix polymer<sup>57-71</sup>. Chemical treatment of natural fibers has been reviewed where it reported<sup>58</sup> that such as de-waxing (de-fatting), de-lignifications, bleaching, acetylation, cyanoethylation<sup>72</sup>, and chemical grafting were used for modifying the surface properties of the fibers for enhancing its performance. Delignification (de-waxing) is generally carried out by extracting with alcohol or benzene and treatment with caustic soda followed by drying at room temperature<sup>62</sup>. Many oxidative bleaching agents such as alkaline calcium or sodium hypochlorite and hydrogen peroxide are commercially used. Bleaching generally results in loss of weight and tensile strength<sup>63</sup>. These losses are mainly attributed to the action of the bleaching agent or alkali or alkaline reagent on the non-cellulosic constituents of fibers such as hemi-cellulose and lignin.

Acetylation of jute is reported to impart resistance to fungal attack and hydrophobicity. The change in properties is attributed to the decrease in moisture sorption in the cell walls and blocking of the hydroxyl group ( $\text{OH}^-$ ) of the wall components in such a way that enzymes of the wood-degrading micro-organisms cannot recognize them as attachable substrates. Acetylated jute is considerably more hydrophobic than unmodified jute<sup>64</sup>. Same observation is

shown in cyanoethylated jute by Saha<sup>65</sup>, *et al.* These modified jute shrink much less than unmodified jute when exposed to water and thus exhibits improved dimensional stability.

Chemical modification through graft copolymerization provides a potential route for significantly altering their physical and mechanical properties. Chemical grafting involves attaching to the surface of a fiber a suitable polymer with a solubility parameter similar to that of the polymer matrix, which acts as an interfacial agent and improves the bonding between the fiber and the matrix. Graft copolymerization of vinyl monomers such as methyl methacrylate, acrylamide, and acrylonitrile onto cellulose, cellulose derivatives, and lignocellulosic fibers has been well established and has been extensively studied over the past few decades<sup>66-68</sup>. Impregnation with monomer followed by its polymerization has also been one of the most common methods used for treatment of fibers. Samal and coworkers<sup>68</sup> have reviewed various methods of graft copolymerization onto cellulose fibers. Graft copolymerization onto cellulose takes place through an initiation reaction involving attack by macro-cellulosic radicals on the monomer to be grafted. The generation of the macro-cellulosic radicals is accomplished by a variety of methods such as (i) diazotization, (ii) chain transfer reactions, (iii) redox reactions, (iv) photochemical initiation and (v) radiation induced synthesis.

The effect of treatments with other chemicals, for example, sodium alginate and sodium hydroxide, has been reported for coir, banana, pine apple leaf fibers and sisal fibers<sup>70</sup>. The treatment resulted in an increase in de-bonding stress and thus improved the ultimate tensile strength up to 30%. Basak<sup>71</sup>, *et al.* have reported that treatment of jute with poly-condensates such as phenol-formaldehyde, melamine-formaldehyde, and cashew nut shell liquid-formaldehyde improved the wettability of jute fibers and reduced the water regain properties. Treatment with cardanol-formaldehyde was also found to reduce water absorption and improved the mechanical properties of jute/polypropylene composite<sup>73</sup> material. The chemical treatment of jute fibers with ethylene diamine and hydrazine results in formation of complexes with the hydroxyl group of the cellulose and thus reduces the moisture absorption of the fiber<sup>74,75</sup>. Samal and Ray<sup>76</sup> have studied the chemical modification of pine leaf fiber using alkali treatment, diazo coupling with aniline, and cross-linking with formaldehyde and p-phenylene diamine. These chemical treatments resulted in significant improvements in mechanical properties, chemical resistance, and reduced moisture regain. A study showed<sup>77</sup> that the treatment of wood with vinyl monomers improved the termite and fungal resistance and they also imparted flame retardancy to the polymer wood composites.

The thermal degradation of lingo-cellulosic fibers has been reviewed by Nguen, *et al.* in detail for modified and unmodified materials<sup>78,79</sup>. For improvement of thermal stability, attempts have been made to coat the fibers and/or to graft the fibers with monomers. Grafting is possible since the lignin can react with the monomers. Mohanty<sup>80</sup>, *et al.* have reported that grafting of acrylonitrile on jute improved the thermal stability as evidenced by the increase in degradation temperature from 170 °C to 280 °C. Sabaa<sup>81</sup> has also reported improved thermal stability for acrylonitrile-grafted sisal fibers as evidenced by

the increased initial degradation temperature, lowering of the rate of degradation and the total weight loss. In another study<sup>82</sup>, the polymer wood composites were prepared by *in situ* polymerization of various monomers and it was observed that the maximum rate of degradation was substantially reduced for phosphonate-treated wood flour. Natural fibers (lingo-cellulosics) are degraded by biological organisms since they can recognize carbohydrate polymers in the cell wall. Ligno cellulosics exposed outdoors undergo photo chemical degradation caused by ultra violet light. Resistance to biodegradation and UV radiation can be improved by bonding chemicals to the cell wall polymers or by adding polymer to the cell matrix.

In addition to the surface treatment of fibers, use of compatibilizer or coupling agent for effective stress transfer across the interface are also explored. The compatibilizer can be polymers with functional groups grafted onto the chain of the polymer<sup>34</sup>. The coupling agents are tetra-functional organo-metallic compounds based on silicon, titanium, and zirconium and are commonly known as silane, zirconate, or titanate coupling agents.

Physical treatments alter the structural and surface properties of the fibers without the use of chemical agent. Four methods can be classified as physical treatments such as (i) corona (ii) plasma (iii) heat treatment and (iv) steam pre-treatment. Corona treatment exploits the corona effect i.e. the function of high energy electro-magnetic fields with consequent ionization in their proximity even at atmospheric pressure & relatively low temperature. In the ionized region, excited species (ions, radicals etc.) are present and the latter are active in surface modification by the introduction of oxygen containing functional groups. The corona method has been applied by various research groups on natural fiber composites<sup>83</sup>.

Plasma treatment<sup>84</sup> induces changes on the surface of the material where an ionized region is formed depending on the gas feed which carries high energy photon, electrons, ions, radicals and excited species. Felix<sup>85</sup>, *et al.* modified cellulosic fibers with cold oxy plasma and characterized the interface on the system cellulose and linear low density polyethylene (LLDPE).

Heat treatment involves heating the fiber or processing the composite at temperature close to those at which the components of natural fiber begin to degrade. When cellulose is heated, it undergoes physical and chemical changes. Physical properties affected include enthalpy, weight, strength, color and crystalline stage. Chemical changes include reduction of degree of polymerization (DP) by bond scission, creation of free radicals, formation of carbonyl and/or carboxyl groups. The steam stabilization, another process<sup>86,87</sup>, which means permanent fixation of compressive deformation of lingo-cellulosic fiber through hydrothermal treatment using in-built steam from moisture of compressed fiber at high temperature and thereby stabilizing the fibers in normal configuration.

## 7. MATRIX IMPREGNATION

### 7.1 Thermosets

The degree of wetting during the production process

is important for a good adhesion between fiber and matrix. When applying thermosets the viscosity can be low which eases wetting. Problems that can be encountered are related to moisture and air. The fiber moisture can affect the chemical reaction. In order to prevent this, the fibers have to be dried before. In standard room conditions, the moisture contents often over 10 per cent.

Air is always present in the fibers and in the resin. The surface of the natural has a geometry and a chemical condition on which air bubble growth will be initiated especially in vacuum injection. In order to prevent many voids and a poor matrix interface during vacuum injection it is necessary to dry the fibers and to gas the resin.

### 7.2 Thermoplastics

Because of a higher processing viscosity of thermoplastic polymers, a proper wetting of fibers is difficult. High temperatures can also cause unwanted changes of the fiber surface or even destroy the fibers. Nevertheless, a low price, reasonable processing temperature and recyclability are the reason for a growing interest in polypropylene. Unmodified PP, however, will not have a proper adhesion with the fibers by applying consolidation forces alone. Natural fibers will only act as a reinforcement if compatibilizers are used. An often used compatibiliser is MAPP, a modification of a PP chain with maleic anhydride. A small amount of MAPP added to PP<sup>34</sup>, will lead to much higher strength properties of the material.

### 7.3 Thermoset Bio-composites

For thermoset composites, the bio-fibers are combined with petro-chemical based polymers like phenolic, epoxy, and polyester resins to form composite materials. These polymers contain reactive groups, which aid the interface development. For jute /polyester composites, an increase in modulus and strength was reported up to a volume fraction of 0.6 followed by a decrease which was attributed to insufficient wetting of the fiber<sup>88</sup>. In another study of jute/polyester composites, it was noted that the mechanical properties were dependent on the secondary chemical bonding between fiber and resin<sup>89</sup>. Sanadi<sup>90</sup>, *et al.* have studied the mechanical properties of sunhemp fiber reinforced polyester where the improved toughness was ascribed to the fiber pullout mechanism in the composite. In banana/polyester composites, the improvement in the properties was observed only when the fiber weight fraction was more than 19%. Jain<sup>91</sup>, *et al.* have studied bamboo/epoxy composites up to a volume fraction of 0.85. It was noted that the composites with banana fiber exhibited better tensile, impact, and flexural strength compared with the properties of other natural fiber composites. Thus the properties of natural fiber reinforced composites depend on a number of parameters such as volume fraction of the fibers, fiber aspect ratio, fiber-matrix adhesion, stress transfer at the interface and orientation. Other aspects include the prediction of modulus and strength using some well established models for two-phase systems and comparison with experimental data<sup>92</sup>. Both the matrix and fiber properties are important in improving mechanical properties of the composites. The tensile strength is more sensitive to the matrix properties, whereas the modulus is dependent on the fiber

properties. To improve the tensile strength, a strong interface, low stress concentration, fiber orientation required whereas fiber concentration fiber wetting in the matrix phase, and high fiber aspect ratio determine tensile modulus. The aspect ratio is very important for determining the fracture properties. Tobias and Ibarra<sup>93</sup> have studied the effect of cure temperature on polyester-based composites. The flexural strength increased with increasing cure temperature and maximum strength was obtained for abaca/polyester composites compared to banana/polyester and rice hull/polyester composites. The incorporation of pine apple leaf fiber in polyester resulted in an increase of 2.3 times in the specific flexural stiffness at 30 wt % content in the fiber. For sisal/polyester composites, the effect of treatment of fibers with silane, titanate, and zirconate coupling agents and N-substituted methacrylamide was investigated by Singh<sup>94</sup>, *et al.* The treatment resulted in improved strength retention properties of the composites and thereby the composites exhibited better properties in humid as well as dry conditions. Similar improved moisture resistance was reported by Bisinda<sup>95</sup>, *et al.*

#### 7.4 Thermoplastic Bio-composites

The mechanical properties of thermoplastic composites can be improved by interacting the compatibility between the fiber and matrix. Compatibilizers such as maleated ethylene, maleated propylene, and a few acrylic-grafted linear polymers are reported to enhance the adhesion between the fiber and polymer matrix<sup>34,96-103</sup>. For HDPE/cellulose fiber composites, the best improvement in tensile strength and tensile modulus was achieved with maleated ethylene<sup>98</sup>. The influence of coupling agents on the mechanical properties of HDPE/wood fibers have been studied by several authors<sup>96,104</sup>. It has been observed that incorporation of wood fibers in HDPE resulted in increase in the stiffness and decrease in tensile strength. Treatment of wood fibers with silane coupling agent and polyisocyanate resulted in an increase in tensile strength. Raj<sup>98</sup>, *et al.* have compared the tensile and impact properties of LLDPE/wood fiber composites with mica and glass fiber composites and have shown that the potential advantage of using wood fibers as reinforcement is in terms of material cost and specific properties. The influence of various chemical treatments on the properties of sisal/PE composites has been investigated by Joseph<sup>105</sup>, *et al.* The chemical treatments included sodium hydroxide, isocyanate and peroxide. Treatment with the cardanol derivative of toluene isocyanate was found to be better than other treatments as evidenced by the decrease in the hydrophilic nature of the composite. The composites exhibited better dimensional stability and retention of properties even after ageing<sup>106</sup>.

The addition of coupling agents and/or maleic anhydride PP improved the interfacial adhesion in PP/cellulose fiber composite, thereby leading to improved properties<sup>34</sup>. Sain<sup>107</sup>, *et al.* reported that the properties of PP/wood fiber composites were very poor due to absence of interface modifiers. The use of maleated PP, itaconic anhydride, and bismaleimide-modified PP resulted in a stable surface and thus improved tensile strength. Sun and Hawke<sup>108</sup> have studied the performance

of wood fiber composites using poly-isocyanate as bonding material. Tibor<sup>109</sup> has reported that the properties of electron beam processed PP/wood fiber composites were significantly better than those of conventionally processed composites. Significant improvement in properties was observed for PP/kenaf or jute fiber composites when maleated PP was used for modifying the fiber-matrix interface<sup>110-112</sup>. However, in short-fiber-reinforced composites, there exists a critical fiber length that is required to develop its full stressed condition in the polymer matrix. Fiber lengths shorter than this critical length lead to failure due to de-bonding at the interface at lower load. On the other hand, for fiber lengths greater than the critical length, the fiber is stressed under applied load and thus results in a higher strength of the composite. For good impact strength, an optimum bonding level is necessary. The degree of adhesion, fiber pull out, and a mechanism to absorb energy are some of the parameters that can influence the impact strength of a short-fiber-filled composite<sup>113</sup>. These developments are confined to polymer composites based on PE, PP, PS, and PVC, for which the processing temperature is about 200 °C. Reports on wood fiber composites with engineering plastics are scarce because of higher processing temperature for processing these polymers.

#### 7.5 Wood Plastic Composites<sup>114</sup>

In wood plastic composite (WPC) industry, virgin plastic materials are widely used. Similar to virgin plastics which can melt and can be processed below the degradation temperature of wood or other ligno-cellulosic fillers (200 °C) is usually suitable for manufacturing WPC. Plastics are one of the major volumes of global municipal solid waste (MSW) and present a promising raw material source for new value added products considering to their large amount of daily generation and low cost. HDPE plus PP were the largest component in MSW followed by PET, PS and LDPE. This creates a substantial amount of polyolefins that can be potentially recovered for WPC manufacturing. The study investigated the use of recycled high density polyethylene (RHDP) in WPC manufacturing. The addition of PE-g-MA has a positive effect on tensile strength as well as flexural properties because it strengthens the interfacial bonding between the fiber and the matrix polymer which resulted in good stress propagation & improved tensile strength. It is also noted that the addition of PE-g-MA can markedly reduce the water absorption.

#### 7.6 Green Composites

Green composites combine plant fibers with natural resins to create natural composite materials. Natural fibers emerge as low cost, light weight and apparently environmentally superior alternative to synthetic fibers. The resins and fibers used in green composites are biodegradable when they dumped, decomposed by the action of micro-organisms. They are converted to water and carbon dioxide. These are absorbed into the plant systems. Biodegradable polymers produced from renewable resources such as plants, animals and microbes through biochemical reactions offer a convenient and environment friendly solution to the problem of plastic wastes.

## 8. NATURAL FIBER- REINFORCED PLA GREEN COMPOSITES

The depletion of petroleum resources, plastic disposal problems and emission during incineration along with increasing environment regulations has led to increased interest in development of bio-composite materials that are compatible with environment and independent of fossil fuels. Considerable studies have been made on bio-composite materials with natural fibers and biopolymers such as starch, polylactide (PLA), polycaprolactone (PCL)<sup>115</sup>, Poly (3-hydroxy butyrate-co-3- hydroxyl valerate (PHBV). These materials find their way in commodity & non-structural applications such as casings of electronic products, interior parts of automobiles. Most of the bio-composites developed so far, has tensile & flexural strengths lower than 100 MPa which restricts their usage in high strength applications.

PLA is thermoplastic and its basic component is lactic acid and is derived from corn starch by fermentation. Lactic acid is either finally polymerized to poly lactic acid by stepwise poly-condensation or via ring opening polymerization of a di-lactide intermediate<sup>115-118</sup>. Numerous tests have shown that PLA is suitable as matrix for the embedding fibers in composites. Some products of natural fiber-reinforced PLA are already established at the market. Jacob Winter (Satzung, Germany) produces biodegradable urns from flax and PLA by compression molding. NEC corporation and UNITIKA LTD announced joint development of bioplastic composites for mobile phone shells consisting of PLA and 15-20 % kenaf fibers<sup>119,120</sup>. An example for the use of natural fiber- reinforced bioplastics in the automobile industry is the Toyota RAUM, which is equipped with a spare tire cover made of kenaf fiber –reinforced PLA<sup>121</sup>.

Several research papers deal with the optimization of natural and man- made cellulose fiber–reinforced PLA composites. Kimura<sup>121</sup>, *et al.* examined compression molded *ramie fibre* reinforced PLA composites in respect of tensile and bending strength as well as stiffness fiber volume content between 45% and 65%. Large proportions of ramie also increased the notched impact strength considerably. Ochi<sup>123</sup> investigated kenaf/PLA composites where tensile strength and bending strength as well as Young's modulus increase linearly up to a fiber content of 50%. Pan<sup>124</sup>, *et al.* produced kenaf/PLA composites by melt mixing and injection molding upto fiber content of 30% where tensile strength improved by 30%. Bax and Mussig<sup>125</sup> studied injection molded flax and man-made cordenka fiber reinforced PLA. While the impact strength of pure PLA could be multiplied by adding cordenka, the values of flax/PLA composites were inferior to the pure matrix. Ganster and Fink<sup>126</sup> investigated injection molded cordenka fiber reinforced PLA with a fiber mass content of 25%. Stiffness and strength of the composites could be approximately doubled compared to pure matrix while the impact strength could be tripled. Shibata<sup>127</sup>, *et al.* composed Lyocell fabric and PLA by compression molding. Tensile modulus and strength of Lyocell/ PLA composites improved with increasing fiber content where impact strength was considerably higher than pure PLA.

Some research papers with different fiber modification methods to optimize the composites' mechanical characteristics.

Tokoro<sup>128</sup>, *et al.* examined three kinds of injection molded bamboo fiber (short fiber bundles, alkali-treated bundles and steam exploded fiber bundles) reinforced PLA. The highest bending strength was obtained with steam exploded fibers. Mechanical, thermal and wear performance of jute fiber /PLA composites were studied as a function of fiber surface treatment. The treatments were done by alkali, permanganate, peroxide, and silane. Surface treatments resulted in improvement of tensile and flexural properties & reduction in impact strength. The TGA results showed a higher thermal stability for silane treated composites<sup>129</sup>.

Plasticizing PLA composites to optimize mechanical characteristics is also an important field of research. Masirek<sup>130</sup>, *et al.* prepared composites with hemp fibers, polyethylene glycol (PEG) and PLA by compression molding. Mechanical tests showed that the composites' Young's modulus markedly increased with the hemp content. Similar studies were also made by others using other chemicals. Okubo<sup>131</sup>, *et al.* experimented with compression molded bamboo fiber – reinforced PLA ,adding micro-fibrillated cellulose (MFC) as an enhancer for bending strength. They also found that tangled MFC fibers prevented micro-cracks along the interface between bamboo fiber and matrix. Improvement of mechanical properties, in particular, impact properties of hemp fiber –reinforced PLA composites by admixture of man-made cellulose fiber (Lyocell). The composites were investigated for their tensile & impact properties. The results show that combining hemp and Lyocell in a composite can improve the impact properties by 160% compared to hemp fiber reinforced composites. Thus, it is possible to adjust the properties of composites optimizing them as per end use requirements<sup>132,133</sup>.

### 8.1 Bio-polyester based Green Composites

Green composites ,in general, may not have high strength as in the case of advanced composites. However, there are many mass volume, non-critical applications where composites with moderate strength may be desirable. Poly(hydroxyl butyrate-co-valerates)(PHBV) are naturally occurring biodegradable polymers produced from a wide range of microorganisms<sup>134,135</sup>. Mechanical properties of PHBV polymers are comparable to those of traditional thermoplastics such as polyethylene and polypropylene<sup>136,137</sup>. Although PHBV polymers represent a new generation of biodegradable polymers, their applications have been limited because of their high cost<sup>138</sup>. Incorporation of fillers or fibers could not only make them more affordable but also improve their mechanical properties. There have been some studies on use of fillers, such as clay, calcium carbonate<sup>139</sup> and wood fibers<sup>140</sup> to modify properties of PHBV resins. Biodegradable fillers and fibers, such as strong natural cellulose fibers, would not only provide reinforcement for PHBVs but also keep the advantage of complete biodegradability. Even though many papers have been published on using natural cellulosic fibers including jute, sisal, etc. as reinforcements for general polymers<sup>141-144</sup>, much less research has been published on using natural cellulosic fibers as reinforcements for biodegradable polymers such as PHBVs. Fully degradable and environment friendly green composites were prepared by combining pineapple fiber and PHBV with 20 and 30% weight

content of fibers in a 0°/90°/0° fiber arrangement. Compared to PHBV virgin resin, both tensile and flexural strength and moduli of these 'green' composites were significantly higher<sup>145,146</sup>.

## 8.2 Green Composites based on Soy Protein Resin

Within the commercially available material, soy protein, perhaps, is the least expensive and most widely available material in the world. While many of the bio-degradable resins such as poly (lactic acid), poly (hydroxy alkanates) etc. are hydrophobic and do not bond well to plant-based fibers, others such as protein and soy-based resins which contain polar groups show good adhesion to them and form composites with good mechanical properties<sup>147-158</sup>. Soy protein, the major component of soybeans, has been used as resin and possesses several properties for such application. Soy protein molecules are ductile and can undergo bending, torsional and tensile deformations without visible damage<sup>159</sup>. Three main varieties of soy protein: soy protein isolate (SPI) containing ~90%; soy protein concentrate (SPC) with ~70 % protein, soy flour with ~50 % protein are commercially available. Paetau<sup>160</sup>, *et al.* and Liang<sup>159</sup>, *et al.* studied the suitability of soy proteins for manufacturing molded specimens. Both SPI and SPC resins displayed rigid and brittle polymer properties after curing, with tensile stress values comparable to that of polystyrene. Soy proteins have also been modified to improve the mechanical and thermal properties significantly and thus make the material more useful as resin<sup>147-151,153-157</sup>. Modified SPI by using stearic acid can improve the resistance to moisture absorption<sup>147</sup>. Modification of SPI with glutaraldehyde and poly(vinyl alcohol) followed by fabrication of composites with flax yarns and fabrics can give good properties<sup>154,155</sup>. SPI was modified using a poly-carboxylic acid, PHYTOGEL (PH) can give interpenetrating network (IPN) structure. The effects of different PH contents on the interfacial properties were characterized using single fiber composite (SFC) tests. The tensile strength of kenaf fibers at the critical lengths obtained from the single fiber composite (SFC) tests was predicted to be in the range of 450 - 750 MPa. The kenaf fiber /modified SPI/ Phytogel resin IFSS measured using the SFC technique with 20% of Phytogel content showed that IFSS is a function of the PH content which controls the resin shrinkage.

Soy protein offers several advantages compared with traditional petroleum based polymers. With improved mechanical and physical properties they have the potential for a wider application range such as computer casings, packaging and panels for auto interior. Flax yarn and fabrics were used to fabricate environment friendly green composites using SPC based resins. The modified SPC resin was made by blending SPC with nano clay particles and then cross-linked using glutaraldehyde. The unidirectional flax yarn reinforced SPC resin composites showed longitudinal tensile failure stress of 298 MPa and Young's Modulus of 4.3 GPa. Flax fabric reinforced composites had failure stress of 62 MPa and 82 MPa in the warp and weft directions respectively. The Young's Modulus values in both directions were around 1.2 GPa<sup>156</sup>. Both yarn and fabric reinforced composites have the potential to replace non-biodegradable materials in many fields due to their good mechanical properties.

Recent developments in both resins and high strength cellulosic fiber have been significant and has allowed the development of high strength advanced green composites with significantly better mechanical properties<sup>157-163</sup>. Many other properties such as physical and thermal properties as well as specific properties such as fire retardancy, vibration damping, impact strength, antimicrobial, gas barrier, water resistance, etc. can also be improved at the same time widen the application range of green composites. Modification of soy protein<sup>164</sup> carried out in two steps:

- (1) to form an interpenetrating network structure by blending with polysaccharides that cross-link themselves and
- (2) to further modify them using nano-clay and MFC to significantly improve their mechanical properties.

The mechanical properties of these resins were comparable or better than the most commonly used epoxy resins. These modified SPC resins were reinforced with high strength liquid crystalline (LC) cellulosic fibers to fabricate 'advanced green composites'. The same modified resins were also reinforced to fabricate composites using E-glass and KEVLAR fibers for comparison. Comparing the mechanical properties showed that the high strength green composites have excellent properties and could be used for many load bearing applications. Fully bio-degradable, environment-friendly green composites were fabricated using ramie fibers and SPC resin and their mechanical properties were investigated<sup>165</sup> where it showed that

- (i) as the curing temperature increased, the tensile strength and Young's modulus increased and the fracture strain decreased as a result of higher cross-linking
- (ii) addition of glycerin to SPC resin decreased tensile strength and modulus, while increasing the fracture strain due to plasticization and
- (iii) such composites had significantly higher tensile and flexural properties in the longitudinal direction than some of the common wood specimens.

However, in the transverse direction, the values were comparable to the wood specimens. The tensile properties of the composites in the longitudinal direction were controlled by the fibers, whereas, the properties in the transverse direction were dominated by resin, fiber/resin interface and longitudinal fiber splitting.

Plant-based fibers are composed of nano-and micro-fibrils<sup>166,167</sup>. Since the cellulose molecules in the fibrils are highly oriented and crystalline, micro-fibrils have excellent mechanical properties. Micro-fibrillated (MFC) can be obtained by mechanical shearing of cellulose fibers<sup>165-167</sup>. The MFC has high aspect ratio and high tensile properties and has been used for fabrication of composites. The composites fabricated using MFC and phenolic resin showed Young's modulus of 19 GPa<sup>168</sup>. MFC has also been shown to enhance the interfacial adhesion and toughness in bamboo composites by preventing the growth of micro-cracks<sup>169,170</sup>. Micro/nano-sized bamboo fibril (MBF) reinforced SPC resin composites were prepared. Addition of the MBF significantly increased the fracture stress and Young's modulus of the reinforced specimens. With the incorporation of 30 parts of MBF, 15 parts of glycerol in SPC showed fracture stress of 59.3 MPa and Young's modulus of 1816 MPa compared to the fracture stress of 20.2 MPa and

Young's modulus of 596 MPa obtained for SPC containing no MBF. The toughness of the specimen also increased significantly from 2.7 MPa to 6.0 MPa<sup>171</sup>.

## 9. BIO NANO-COMPOSITES

Recently, polymer-clay nano-composites have received significant attention as an alternative to conventional filled polymers. Because of their nanometer size dispersion, the polymer-clay nano-composites exhibit the large scale improvement in the mechanical and physical properties compared with pure polymer or conventional composites. These include increased modulus and strength, decreased gas permeability, increased solvent and heat resistance and decreased flammability<sup>172</sup>. Since Toyota researchers in the late 1980s demonstrated that mechanical and flame retardant properties of nylon-clay composite material increased dramatically by reinforcing with less than 5% of clay, extensive research work<sup>170</sup> has been performed in this area and several extensive review articles<sup>174-176</sup> regarding recent advances in the production of nano-composites with various polymers and their performance and application are available. Polymer-clay nano-composites are a class of hybrid materials composed of organic polymer materials and nano scale clay fillers. Among the nano-scale clays, Montmorillonite (MMT) is of particular interest and has been studied widely. MMT is a clay mineral consisting of stacked silicate sheets with a high aspect ratio and a plate like morphology. This high aspect ratio plays an important role for the enhancement of mechanical and physical properties of composite materials. In recent years numerous research activities on polymer-clay nano-composites have been performed. However, the matrices of polymer-clay nano-composites are mainly synthetic thermosetting<sup>177-179</sup> as well as thermoplastic polymers<sup>180-185</sup> but of late biodegradable plastics have found place even though limited, for example, PLA<sup>186</sup> and PCL<sup>187</sup> have also been used for the processing of nano-composites with a layered silicate. However, the materials prepared from natural polymers have poor performance compared with those prepared from synthetic polymers. Recent research<sup>188</sup> has indicated that organo-clays show much promise for starch based polymer nano-composites in terms of improvement in their mechanical properties and stability over those of the unfilled formulations.

### 9.1 Starch-based Nano-composites

Starch is one of the natural biopolymers most widely used to develop environmentally-friendly packaging materials to substitute for petrochemical-based non-biodegradable plastic materials. Native starch is not a true thermoplastic but it can be converted into a plastic-like material called thermoplastic starch (TPS)<sup>189</sup>. In the presence of plasticizers at high temperature (90 °C -180 °C) and under shear, starch readily melts and flows, allowing for its use as an injection, extrusion or blow molding material similar to most conventional synthetic thermoplastic polymers. Still it behaves like native starch and is mostly water-sensitive and has poor mechanical properties. In order to improve the properties, reinforcement of starch with nano-scale minerals has been considered without interfering in the biodegradability of the composites. De Carvalho<sup>191</sup>, *et*

*al.* Park<sup>192</sup>, *et al.* obtained well-ordered nano-composites by the melt intercalation method with plasticized starch. It was concluded that the better the dispersion of the clay in the TPS matrix, the better the mechanical, thermal, and barrier properties. Park<sup>193</sup>, *et al.* also tested the effect of the filler concentration of the TPS /clay hybrid with cloisite Na<sup>+</sup> and cloisite 30B. They found that both the tensile and water vapor barrier properties are generally increased with increasing clay content. Withelm<sup>194</sup>, *et al.* prepared nano-composite films with glycerol-plasticized starch reinforced with Ca<sup>2+</sup>-hectorite clay by the solution casting method. DMA results showed that the storage modulus with the starch /clay hybrid films increased considerably at temperatures greater than 25°C, which is further evidence for the reinforcing effect of clay particles. McGlashan and Halley<sup>188</sup> tested the use of nano scale MMT in thermoplastic starch/polyester blends and found excellent improvements in film blow-ability and tensile properties. Xu<sup>195</sup>, *et al.* prepared starch acetate nano-composites foam with four organo-clays by a melt intercalation method. The addition of organo-clays into a starch acetate matrix increased the glass transition temperature ( $T_g$ ) by 6 °C -14 °C depending on the type of clay. The nano-dispersion of the starch acetate molecules in silicate layers also restricted their thermal motion and this increased their thermal stability. The intercalation also influenced the mechanical properties of the hybrid. Huang and Yu<sup>196</sup> reported on preparation and properties of starch/MMT nano-composites prepared with thermoplastic corn starch and activated MMT by the melt intercalation method. TEM results indicated that MMT layers were exfoliated and uniformly dispersed in starch matrix at the nano meter level. Tensile strength and Young's modulus increased monotonously with increase in filler content up to 8%. Water resistance of the nano-composites also improved. The authors attributed such property improvement of the exfoliated nano-composite starch to MMT possessing a high aspect ratio and its homogeneous dispersion in the polymer matrix.

### 9.2 Cellulose-Based Nano-composites

Cellulose is the most abundant naturally occurring biopolymer. Naturally, cellulose is a very highly crystalline, high molecular weight polymer which is infusible. Because of its infusibility, cellulose is converted into derivatives to make it more processable such as cellulose ethers and cellulose esters. Cellulose acetate is currently used in large volume applications ranging from fibers to films, to injection molding thermoplastics. Recently, interest in developing nano-composites with cellulosic materials to obtain functional materials has been increased because of the improved mechanical and thermal properties and permeability of cellulose acetate (CA) films. Green nano-composites from cellulose acetate and organically modified clay could show the extent of swelling of the clay increased with increase in plasticizer<sup>197</sup>. Water vapor permeability of CA films decreased up to 100% by reinforcing with organo-clay. The same researchers prepared CA/organo-clay nano-composites with CA plasticized with triethylene citrate (TEC), organo-clay and cellulose acetate butyrate grafted with maleic anhydride (CAB-g-MA) as compatibilizer. They concluded that 5 wt % loading of the compatibilizer is

optimum for the best morphology and mechanical properties for the preparation of CA/TEC/clay composite. Cho<sup>198</sup>, *et al.* reported on the preparation and mechanical properties of nano-composite film prepared with cellulose diacetate and organo-clay using a solvent intercalation method. Ruan<sup>199</sup>, *et al.* reported interesting results on regenerated cellulose/tourmaline nano crystal composite films. Interestingly, the cellulose/tourmaline composite films showed anti-microbial action against staphylococcus aureus. This result implies potential use of the composites for functional packaging materials. Another type of utilization of cellulose as nano-composite feedstock is a cellulose whisker-reinforced nano-composite. Cellulose whiskers (micro-crystalline cellulose) have two dimensions in the nano meter scale, like carbon nanotubes<sup>191</sup> and have been successfully used for the reinforcing of biopolymers. Their effects on reinforcement are generally relatively low and other problems are realized in the commercial use of cellulose whiskers as structural materials such as a relatively high price and difficulty of cellulose micro-fibrils in a polymer matrix<sup>200</sup>.

### 9.3 Chitosan-Based Nano-composites

Chitosan is a partially deacetylated derivative of chitin, which is the second most abundant natural biopolymer next to cellulose. Because chitosan is biodegradable, non-toxic and readily bio-compatible, it has been studied extensively for various industrial and packaging applications. Its properties as a packaging material also need to be improved, as do other hydrophilic natural biopolymer based packaging materials. Lin<sup>201</sup>, *et al.* reported on a novel method for the preparation of a chitosan/montmorillonite (MMT) nano-composite using a solvent casting method. When chitosan was incorporated with potassium persulfate (KPS)-MMT, the diffraction peak of chitosan became broader, indicating that the incorporation of KPS-MMT had interfered with the crystalline structure of chitosan. The tensile properties of the nano-composite films depended on the amount of KPS incorporated in the MMT. The higher the quantity of the KPS incorporated into the MMT used, the more the MMT exfoliated along with the degradation of chitosan so that the Young's modulus increased but the tensile strength decreased. Xu<sup>202</sup>, *et al.* prepared chitosan-based nano-composite films with Na-MMT and Cloisite 30B using a solvent casting method. It has been found that the nano clay was exfoliated along with the chitosan matrix with small amounts of Na-MMT. They also confirmed that the tensile strength of a chitosan film was enhanced and the elongation at break decreased with the addition of clay into the chitosan matrix. Wang<sup>203</sup>, *et al.* also prepared a chitosan/MMT nano-composite using the solvent intercalation method and found that an intercalated/exfoliated nano structure was formed with low MMT content and the nano-dispersed clay improved the thermal stability and enhanced the hardness and elastic modulus of the matrix systematically with increase in clay loading. Darder<sup>204</sup>, *et al.* prepared a chitosan/MMT nano-composite<sup>205,206</sup> using a solution intercalation method with varying amounts of clay. They also reported on the potential application of a chitosan/MMT nano-composite as electrochemical sensors in the detection of different anions.

### 9.4 Protein-Based Nano-composites

Recently, composite soy protein with layered silicate clay materials has been used to improve film properties. Otaigbe and Adams<sup>205</sup> obtained better mechanical properties with improved water resistance for soy protein composites with polyphosphate fillers. Rhim<sup>208</sup>, *et al.* also demonstrated that soy protein isolate (SPI) films with organically modified MMT or bentonite increase the tensile strength with improved water vapor permeability. Dean and Yu<sup>209</sup> reported the effect of ultrasound treatment on inducing intercalation or exfoliation of unmodified Na-MMT (Cloisite Na<sup>+</sup>) dissolved in water with or without glycerol. The degree of intercalation increased as the time of sonification increased. In the case of the clay/glycerol/water mixture, more than 25 min of sonification caused a complete exfoliation of the clay. Dean and Yu also prepared soy protein-based nano-composite films and tested their microstructure and mechanical properties. The glass transition temperature ( $T_g$ ) determined by both DMA and DSC increased in composites. They postulated that this was due to the restriction in the molecular motion of the protein by the dispersion of the nano clay. The most significant improvement observed in the mechanical properties was for the elastic modulus. Rhim<sup>208</sup>, *et al.* prepared composite films of SPI and various clays. The tensile strength of the SPI hybrid films with O-MMT and bentonite, which are composed of mostly layered silicate clays increased significantly while those with talc and zeolite which are particle type fillers, decreased. The increase in gas barrier properties of nano-composite films is believed to be due to the presence of ordered dispersed silicate layers with large aspect ratios in the polymer matrix. Gelatin, an animal protein, was also tested for the preparation of a biocomposite with MMT clay in order to improve the mechanical and water resistance properties of the polymer<sup>208</sup>. The tensile strength and Young's modulus were improved which varied with the MMT content as well as the pH of the gelatin matrix.

### 9.5 Lipid Based Nano-Composites

Lipid materials such as beeswax, cadellila wax, carnauba wax, triglycerides, acetylated monoglycerides, fatty acids, and sucrose fatty acid esters, as well as resins such as shellac and terpene resins are used as edible film-forming materials. They can be used to coat a food or drug surface to provide a moisture-barrier component of a composite film. Composite films contain both lipid and hydrocolloid components (proteins or polysaccharide) in order to exploit the advantages of the lipid and hydrocolloid components. When a barrier to water vapor is desired, the lipid component can serve this function while the hydrocolloid component provides necessary mechanical strength.

Edible films from chitosan, cassava starch, and gelatin plasticized with glycerol have been developed by casting method and the effect of cassava starch, gelatin and glycerol from the film solution on various properties of chitosan based films have been studied using response surface methodology<sup>210</sup>. The results indicate that there was an interaction and molecular miscibility among the major components. The growth inhibition of phytopathogen on mango fruit surface indicated the efficiency

of these coatings and they can be applied for the conservation of fresh or minimally processed fruits and vegetables. Similarly the properties of biodegradable blend film from starch-chitosan with different plasticizers (sorbitol, glycerol and polyethylene glycol) were determined. Increasing concentration of these plasticizers resulted in decreased tensile strength with an increase in elongation at break, water vapor permeability and film solubility. One of the areas that is actively investigated is the development of biodegradable films from various biopolymers such as polysaccharides, proteins and lipids. The fabricated films were tested for transparency, glass transition temperature, tensile strength and water vapor permeability. It has been observed that the functional properties of rice starch film, such as its ductility and effectiveness as a barrier, are generally poor. Hence a rice starch based film, reinforced with starch nano-crystals (prepared by submitting native granules of rice starch to acid hydrolysis at 40 °C) was prepared by casting film solution on leveled trays. The results showed that the mechanical properties of the film were enhanced by the addition of starch nano-crystals. Furthermore, by increasing the starch nano-crystals content, the water barrier properties of the films were also improved. The addition of starch nano-crystals into rice starch films can improve the thermal stability of rice starch films. Whey protein isolate (WPI) based composite film with three different types of nano-clays (cloisite Na<sup>+</sup>, cloisite 20A, cloisite-30B) were prepared using a solution casting method and their physical and antimicrobial properties were determined in order to understand the effect of nano-clay on film properties. The type of nano-clay used significantly influenced the tensile and water vapor barrier properties of the composite films with the exception of cloisite30B, which had no negative effect. In addition, WPI/cloisite30b composite film showed a beneficially bacterio-static effect against gram-positive bacteria<sup>211-214</sup>.

## 10. CONCLUSION

Bio-composites that contain a significant content of bio-based material can presently achieve this at an affordable cost-performance ratio to compete with petroleum based materials and still maintain a positive balance among ecology, economy and technology. It has opened up an opportunity to supplement and may eventually replace petroleum-based composite materials thus offering new agricultural, environmental, manufacturing and consumer benefits. However, several critical issues in relation to finding suitable solution for application need to be solved by evolving promising techniques to design biocomposite of general as well as special interest. Development of new biocomposite materials with added functional properties such as in active and smart packaging system has created further scope for expansion of materials technology.

Bio-nano-composites have huge potential for the natural polymer-based nano-composites to enhance quality and safety of packaged foods by increasing barrier properties of packaging materials with antimicrobial activity. However, natural biopolymer based nano-composite film material with active packaging function is in its infancy and is now emerging because of environmental concerns and expectations for high quality food products. Much research is expected for such

biodegradable nano-composite materials to replace or reduce the use of the existing petro based products.

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