



OVERVIEW OF FLAX FIBER REINFORCED THERMOPLASTIC COMPOSITES

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Abstract

Flax fibres are often used for reinforcing thermoplastic to manufacture biocomposite materials exhibiting numerous advantages such as high mechanical properties, low density and biodegradability. The mechanical properties of a biocomposite material depends on the nature and orientation of the fibres, the nature of the matrix and mainly on the adhesion between fiber and the polymer matrix. The main problem with these natural fiber is poor compatability and hydrophilic nature. Therefore, the fiber is needed to be chemically treated to reduce water absorption, enhancing adhesion between fiber and polymer matrix. For selecting most suitable thermoplastic polymer for a certain application, the properties of the available polymers is needed to known. Since data tend to be widely scattered over many sources, it is the purpose of this paper to give an overview of the most relevant properties of flax, thermoplastic polymers and various chemical treatment and processing techniques adopted for developing a range of thermoplastic biocomposite.

Keywords: Thermoplastic, biocomposite, polymer matrix, chemical treatment, hydrophilic

Introduction

Polymers are long chain giant organic molecules are assembled from many smaller molecules called monomers. Another common name for many synthetic polymers is plastic which comes from the Greek word "plastikos", suitable for molding or shaping. Plastic has played an important role in our life in daily use from packing, wrapping, and building materials include half of all polymers synthesized. Other uses include textiles, electronic casing, automobiles, and many other all are made from polymers. However problem of recycling and limited petroleum resources gave rise to new material for new generation termed as "Biocomposite". Biocomposite consists of natural fiber, polymer matrix and additives in suitable proportions.

Plastics can be broadly classified in two groups, thermoplastic and thermoset. A thermoplastic is a material that can be processed or molded on application of heat many times without a chemical change in or bonding between the chains of the polymer. Because they can be processed many times, these materials are the backbone of the plastics recycling industry. Common examples of thermoplastics are polyethylene, polyvinyl chloride, and polypropylene, and some common uses are vinyl siding, plastic grocery bags, and milk bottles. Presently various thermoplastic are being used as matrix which is reinforced with natural fiber like flax, hemp, kenaf, jute etc. Some of the thermoplastics common used for manufacturing biocomposite are polyolefins (Polyethylene, Polypropylene, Polybutene), Vinyl Polymers (PVC), Polystyrene etc.

In North America, flax and hemp are two important fiber crop been grown. In recent years, considerable interests have been developed in natural fiber for reinforcing different thermoplastics. Since last few years, plant fiber like flax and hemp are gaining importance for manufacturing bio-composite, a new emerging industry in global market. Major portion of biocomposite market is dominated by automotive industry for making interior parts and bumpers. How it is now being using in packaging food and pharmaceuticals products (Bledski et al. 1999). Flax has less density than the glass fiber and has comparable tensile strength, therefore has a potential to replace synthetic fiber (Joffe et al. 2003).

Flax fiber

The natural fibres exhibit considerable variation in diameter along with the length of individual filaments. Quality as well as most of the other properties depends on factors like size, maturity as well as processing methods adopted for the extraction of fibres. The modulus of fibre decreases with increase in diameter. The dimension and arrangement of unit cells in a fiber determine the structure and also influence the properties of the fibers. The dimensions of individual in natural fibers are dependent on the species, maturity and location of the fibers in the plant and also on the fiber extraction conditions (Franck. 2005). When used for applications such as textiles and paper, the length to diameter ratio (l/d) of individual cells in a fiber affects the flexibility and resistance to rupture of the fibers and products made from them. (Reddy et al. 2005)

Transversally, unit cells in all of the natural-fibers (bio-fibers) have a central hollow cavity called the lumen. The shape (round, polygonal or elliptical) and size of the lumen depends on the source of the fiber and thickness of the cell wall. The presence of the hollow lumen decreases the bulk density of the fiber and acts as an acoustic and thermal insulator. These properties make bio-fibers preferable for light weight composites used as noise and thermal insulators in automobiles (Netravali et al. 2003)

Flax and other plant fibers mainly consist of cellulose, hemi-cellulose and lignin. Though apart from these, the plant fibers also consist of pectin, water solubles, moisture, fat and wax. The proportion of these components in a fiber depends on the age, source of the fiber and the extraction conditions used to obtain the fibers (Hearle et al. 1963).

A comparison of properties of flax with some other natural fibers and conventional manmade fibers can be obtained from Table 1.

Table 1 Comparative properties of Flax with some natural fibers and conventional manmade fibers

Fibre	Density (g/cm ³)	Tensile strength (MPa)	Young's modulus (GPa)	Elongation at break (%)
Cotton	1.5-1.6	287-800	5.5-12.6	7.0-8.0
Jute	1.3-1.45	393-773	13-26.5	1.16-1.5
Flax	1.50	345-1100	27.6	2.7-3.2
Hemp	-	690	-	1.6
Ramie	1.50	400-938	61.4-128	1.2-3.8
Sisal	1.45	468-640	9.4-22.01	3-7
Coir	1.15	131-175	4-6	15-40
E-glass	2.5	2000-3500	70	2.5
S-glass	2.5	4570	86	2.8
Aramid	1.4	3000-3150	63-67	3.3-3.7
Carbon	1.7	4000	230-240	1.4-1.8

Source: Mohanty, A.K. et al. (2000)

Again high tensile strength of flax may be attributed to its high cellulose content and comparatively low microfibrillar angle (Reddy et al. 2005). However, it is not possible to correlate the fiber strength exactly with cellulose content and microfibrillar angle because of the very complex structure of natural fibres. Filament and individual fibre properties can vary widely depending on the source, age, separating technique, moisture content, speed of testing, history of fibre, etc. The lignin content of the fibres influences its structure, properties and morphology. The waxy substances of natural fibres generally influence the fibre's adhesion and wettability characteristics (Mohanty et al. 2000).

Flax is widely preferred because of its higher strength over other natural fibres and hemp can be preferred based on its high amount of straw and fibres. Suitable matrix materials can be resin systems, thermoplastic starch and polyolefins such as Polyethylene and polypropylene. Polypropylene is more preferable because of higher economic, recycling and technical characteristics. The homogeneity of the fibre and matrix compound, the degree of elementarization and degumming, degree of polymerization and crystallization, good adhesion between the fibre and matrix, the moisture absorption and retention properties of the individual components and the compound itself, and the flame retardant properties of the resulting compound should be taken into consideration while choosing flax and hemp fibres for reinforcement/filler purposes in virgin/recycled polymer matrices. All these properties may depend, to some significant extent, upon the interfacial compatibility.

It was found out that flax and hemp fibers are more hydrophilic in nature than other green fibers, and that the applied separation methods affect the fiber surface morphology and consequently the surface area. The properties such as density, electrical resistivity, ultimate tensile strength, initial modulus, etc., are related to the internal structure and chemical composition of fibers (Reddy et al. 2005).

Chemical treatment of fiber and its effect on thermoplastic biocomposite

Before chemical extraction, in their natural state, fiber surfaces have waxes and other encrusting substances such as hemi-cellulose, lignin and pectin that form a thick outer layer to protect the cellulose inside. During fiber extraction, most of the surface waxes and other noncellulosic substances are removed. When these fibers are used as reinforcing fibers for composites, the removal of surface waxes and encrusting substances makes the fiber surface rough and improves the adhesion of fibers and polymer matrix. The presence of impurities on the surface of the fibers affects the appearance and processability of the fibers. Chemical, biological and physical treatments are used to improve the morphological features, processability and utility of the fiber.

The hydroxyl group in flax fibers contributes to a poor interface when combined with hydrophobic matrices. Physical and chemical methods can be used to optimize this interface. These modification methods are of different efficiency for the adhesion between the matrix and fiber (Cappelletto 2000). Different treatments of flax fibers aimed at improving the adhesion with a polymer matrix may alter not only the fiber surface properties but also fiber strength (Joffe 2003). Physical methods, such as stretching, calendaring, thermal treatment, and the production of hybrid yarns do not change the chemical composition of the fibers (Bledzki 1999). But fibers change structural and surface properties and thereby influence the mechanical bonding to polymers. The different surface chemical modifications of natural fibers aimed at improving the adhesion with a polymer matrix were performed by a number of researchers.

Alkaline treatment, also called mercerization, is one of the most used chemical treatments of natural fibers when used to reinforce thermoplastics and thermosets. The important modification resulting from alkaline treatment is the removal of the hydrogen bonding in the network structure, thereby, increasing surface roughness. This treatment also removes certain amount of lignin, wax and oils covering the external surface of the fiber cell wall, depolymerizes the native cellulose structure and exposes the short length crystallites (Mohanty et al. 2001). In the treatments researchers varied the concentration and time of treatment. Sodium hydroxide (NaOH) (5%) solution was used for time varied from 8 to 72 h by Ray et al. (2001) and Mishra et al. (2001). Morrison et al. (2000) and Jacob et al. (2004) used different concentrations (0.5, 1, 2, 4 and 10%). Weyenberg et al.(2005) has soaied flax fiber in the alkaline solution (1%, 2% and 3%) for 20 min at room temperature.

Jähn 2002 reported that this treatment has a lasting effect on the mechanical behavior of flax fibers, especially on fiber strength and stiffness. This treatment results in a better mechanical interlocking by increases the surface roughness and the amount of cellulose exposed on the fiber surface, thus increasing the number of possible reaction sites (Valadez et al 1999). This treatment also improves the mechanical, impact fatigue and dynamic mechanical behavior of the fiber-reinforced composite with saline treatment (Mohanty et al. 2001). It was found that alkali treatment of different natural fibers had significant effect on the mechanical, impact fatigue, and dynamic mechanical behavior (Sarkar and Ray 2004, Joseph and Thomas 1996, Weyenberg 2003 and Jacob et al. 2004).

Silane treatment mainly involves three processes namely hydrolysis, condensation and bond formation stage. In this treatment, in the presence of moisture, hydrolyzable alkoxy group leads to the formation of silanols. The silanol then reacts with the OH⁻ group of the fiber, forming stable covalent bonds to the cell wall that are chemisorbed onto the fiber surface (Agrawal 2000)..

Agrawal et al. (2000) treated oil palm fiber with 1% silane solution in a water-ethanol mixture (40:60) for about 3 h and observed that the treatment improved the thermal stability of the composites. Valadez et al. (1999) treated henequén fibers with a 0.033% w/w aqueous silane solution and tested their adsorption isotherms and tensile strength. They found that the effect of the silane treatment was higher than alkaline treatment. Some of researchers studied silane treatment in glass fiber composites (Kim 2001; Ishak 2001; Lee 2002; Debnath 2003).

Acetylation a well-known esterification method to introduce plasticization to cellulosic fibers . In this method the fiber is treated with pre-heated acetic anhydride and then with acetone (Hill et. al. 1998). In this process the ester bond between the plant cell wall polymeric material and the acetyl group is subject to hydrolysis with simultaneous loss of modifying agent as acetic acid. Hill et. al. (1998) reported that development in resistance to microbial degradation was observed.

In Acrylation treatment, Acrylic acid is also used in graft polymerization to modify fiber surface (Xu 2002; Karlsson 1999). This reaction is initiated by free radicals of the cellulose molecule. The cellulose is treated with an aqueous solution with selected ions and exposed to a high energy radiation. Then, the cellulose molecule cracks and radicals are formed (Bledzki 1999). Sreekala et al. (2002) reported that fibers were mixed with 10% NaOH for about 30 min and then treated with solution containing different concentrations of acrylic acid at 50°C for 1h. The fibers were washed with water/alcohol mixture and dried.

Several researchers investigated potassium permanganate (KMnO_4) solution (in acetone) with different concentrations to treat fiber for 1 to 3 min (Joseph 1996; Paul 1997; Joseph 1999; Sreekala 2002). This treatment leads to the formation of cellulose radical through MnO^{3-} ion formation. Paul (1997) reported that the highly reactive Mn^{3+} ions are responsible for initiating graft copolymerization.

The chemical coupling method is one the important chemical method, which improves the interfacial adhesion. In this method the fibre surface is treated with a compound that forms a bridge of chemical bonds between fibre and matrix (Bledski et al. 1999). The chemical composition of coupling agents allows them to react with the fiber surface forming a bridge of chemical bonds between the fiber and matrix. The development of a definitive theory for the mechanism of bonding by coupling agents in composites is a complex problem. Generally, coupling agents are molecules possessing two functions. The first function is to react with OH^- groups of cellulose and the second is to react with functional groups of the matrix. Bledzki and Gassan (1999) outlined several mechanisms of coupling in materials

Most researchers found these treatments were effective and showed better interfacial bonding. Some of the coupling agents like benzoyl peroxide (Joseph 1996; Sreekala 2000), acetic anhydride (Hill 1998; Sreekala 2003; Nair 2001), maleic acid anhydride (Gassan 1997; Oever 1998; Joseph 2003), isocyanates (George 1996; Maldas 1989), sodium chlorite (Mishra 2002; Mustata 1997), and stearic acid (Paul 1997; Zafeiropoulos 2002) etc. were also studied and used to modify the surface between fiber and matrix. Presently in industry, the most common process of bleaching the cellulose/jute uses oxidizing agents such as sodium hypochlorite, calcium hypochlorite, or hydrogen peroxide. It was also reported that nascent oxygen is involved the reaction process.

Apart from above discussed chemical treatments enzymes, chelators or both together can be used for improving fiber quality for biocomposite formulation. T . Stuart et. al. (2005) reported the effect of pectinolytic enzyme and Ethylene Diamine Tetraacetic Acid (EDTA) when used

separately or together on flax fiber for composite formulation. Both have positive effect on composite as pectinase clean the fiber surface and EDTA breakdowns the fiber bundles. Tensile strength is enhanced, however, Young's modulus remains independent of the treatment.

Properties of Thermoplastic

Though flax fiber pre-treatment is important, yet properties of polymers also vital role in biocomposite formulation. Most of the thermoplastic are amorphous and semi-crystalline. There are many aspects and properties to consider when choosing a thermoplastic resin for a product. Some of the properties considered generally while making a biocomposite are impact strength, flexibility, color (transparent vs. Opaque), chemical resistance, fatigue resistance. But the most important aspect of thermoplastic is its ease of bonding with natural or synthetic fiber and cost.

Table 2. Physical and Mechanical properties of various polymers

Properties	Limit	PP	LDPE	HDPE	PA-6	PA-66	PC	PBT	PET	PEEK	PPS	PEI	PAI
ρ (g/cm ³)	Upper	0.920	0.925	1.000	1.14	1.14	1.24	1.35	1.40	1.32	1.40	1.28	1.45
	Lower	0.899	0.910	0.941	1.09	1.08	1.19	1.23	1.30	1.264	1.30	1.27	1.38
W _{24h} (%)	Upper	0.02	<0.015	0.2	1.8	1.6	1.19	0.10	0.07		0.05	0.25	0.280.
	Lower	<0.01		<0.01	1.3	1.0	0.12	0.08			0.03		22
σ_{max} (MPa)	Upper	41.4	78.36	38	79	94	72	55.9	70	103.5	90	104.9	192
	Lower	26	4	14.5	43	12.4	53	51.8	50	70	65.6	103.5	90
E (GPa)	Upper	1.776	0.38	1.49	2.9	3.9	3	2.37	4.0	3.8	3.9	3.0	4.4
	Lower	0.95	0.055	0.413		2.5	2.3		2.7	3.1	2.6		2.8
σ_f (MPa)	Upper	55.2			117.3	131.1	93.2	96	112.3	110.4	151	151.8	240.8
	Lower				69	89.7	81.4	82.8	110.4	110	96	144.9	185.6
E _f (GPa)	Upper	1.73		1.07	2.8	3.5	2.38	2.6	2.8	3.9	4.1	3.5	6.6
	Lower	0.83		0.41	1.9	1.1	2.14	1.9		2.8	3.4	3.0	3.6
ϵ (%)	Upper	700	800	1000	150	>300	125	300	100	50	6	60	12
	Lower	15	90	12	20	35	90	100			1.1	6.0	
Izod, 1/8" (J/m)	Upper	267		1068	160	854	908	53.4	26.7	50.2	133	133	133
	Lower	21.4	>854	26.7	42.7	16	534	48.1			10.7	53.4	58.7

Source: Velde K. V. and Kiekens P. (2001)

PP: Polypropylene.

LDPE: Low-density polyethylene.

HDPE: High-density polyethylene.

PA-6: Polyamide 6.

PA-66: Polyamide 66.

PC: Polycarbonate

PBT: Polybutylene Terephthalate.

PET: Polyethylene Terephthalate.

PEEK: Polyether Ether Ketone.

PPS: Polyphenylene Sulfide.

PEI: Polyetherimide.

PAI: Polyamide Imide.

Glass transition temperature (T_g) of the polymer is an important factor for processing and desired mechanical properties of thermoplastic composite. It is found that amorphous thermoplastic performs well below the flexibility of amorphous polymers is reduced drastically when they are cooled below the T_g . At temperatures below T_g , there is no segmental motion and any

dimensional changes in the polymer are the result of temporary distortions of the primary valence bonds. For example, Polyvinyl Chloride has T_g of 81 °C and is therefore unsuitable for applications where the operating temperature is close to boiling point of water (Mascia, L. 1982). As flax is susceptible degradation when exposed to elevated temperatures for prolonged time and therefore in specific case of a flax reinforced plastic, the application temperature of the eventual product should remain relatively low (e.g. maximum 100°C). However, some polymers surpassed their T_g at this processing temperature but this is not necessarily a problem since the major part of the composite strength (unidirectional reinforced composite) is determined by its fibres (Velde K. V. and Kiekens P. 2001).

Other than T_g , melting point (T_m), is a more important parameter as above it the whole polymer chain is mobile and the mechanical properties are virtually reduced to zero. At the process temperature (T_p) should be higher than the melt pointing the viscosity of the polymer reduces drastically and the polymer is easy to process. However, sometimes degradation of polymer can occur, therefore additives are the most suitable alternative which can prevent thermal degradation of the polymer at these elevated temperatures. Therefore low process temperature (and melt point), may be advantageous as it reduces the energy cost of the production process and degradation of polymer will also not occur.

Thermoplastics can be used for manufacturing wide range of products depending of the properties of the particular polymer. LDPE has good electrical properties including low power factor and it is resistant to moisture and most chemicals other than oxidizers (Beadle 1971). LDPE is used to manufacture bottles and other containers, toys, kitchenware, water tanks, packaging film, coated materials for packaging, water and chemical pipe, wire insulation and sheathing, and film for agricultural and building applications (Beadle 1971). LLDPE has same density and melt index when compared to conventional low-density polyethylene products and is used for manufacturing films or flexible model products, is claimed to have better impact, tear, or puncture properties (Charrier 1991). High density polyethylene is harder and more brittle than LDPE and resistance to some of solvents which attack the low density product (Beadle 1971). Most of the HDPEs are formulated for extrusion and blow molding applications. Polypropylene has several outstanding properties like light weight, heat resistance, hardness, surface gloss, stain resistance, stiffness, chemical resistance, stress-crack resistance, dimensional stability. This make polypropylene and propylene copolymers excellent choices for molding items such as house wares, appliance parts, automobile parts and accessories, closures, laboratory ware, hospital ware, toys, sporting goods, and miscellaneous items for home and industry (Beadle 1971). Some of the expensive thermoplastics like PEEK, PPS are semi-crystalline in nature with higher glass transition temperature and better mechanical properties than conventional thermoplastics (Campbell 2004).

Processing of Thermoplastic Biocomposite

Thermoplastic fiber-reinforced composites can be processed by a wide variety of distinct methods or techniques. The techniques to process thermoplastic involving the continuous manufacture of a product having a uniform cross section, which include extrusion, extrusion covering, film blowing and calendaring, sheet thermoforming, blow molding, rotational molding, compression molding, transfer molding, injection molding and reaction injection molding casting (Charrier 1991).

The extrusion is an important process which basically involves melting and mixing of polymer continuously, shaping a fluid polymer through the orifice of a suitable tool (die), and subsequently solidifying into a product (Henson 1997). In the case of thermoplastics, the feed

material, in powder or pellet form, is now most commonly heated to a fluid state and pumped into the die, through a screw extruder; it is then solidified by cooling after exiting from the die. In order for the emerging extrudates to maintain their shape until they solidify, extrusion grades tend to have relatively high molecular weights associated with high viscosity and melt strength (Charrier 1991). Oladipo and Wichman (1999) investigated aspen wood fiber/HDPE composite and the components were fed at pre-determined mass flow rates, based on the desired wood fiber mass fraction in the composite, into an extruder having a 28 mm co-rotating twin screws operating at temperature of 150°C and a screw rpm of 100. This temperature ensured that while the polymer was fully melted (melting point is 120-135°C), the wood fibers were not burned. Short-flax-fibre reinforced compounds can be made by mixing flax fibres with a thermoplastic matrix at elevated temperature in an extruder or a kneader (Harri ette et. al. 2005).

Injection molding is one of the latest techniques adopted for manufacturing wide range of biocomposite for automotive and plastic industry. In the last few years, however, polypropylene/natural fibre-injection moulding, based on annual fibres such as flax or hemp, has been the subject of intensive research. This is especially the case in Germany, where the automotive industry forms a strong driving force for the development of these materials. The basic processing variables are time, temperature, pressure and flow rate. These variables have different values at different points in the process and depend on the different rates of throughput, polymer types, residence size of melt passages, position of mould cooling passages and so on. Thermoplastic have a very low thermal conductivity and hence are particularly susceptible to overheating. Thus prolonged exposure of polymer in barrel leads to thermal degradation of the polymer and it results in chain scission and reduction in molecular weight and melt viscosity. This effect can be seen in materials like polypropylene and polystyrene (Ogorkiewicz, R. M. 1969).

High thermal properties of some composites can be used to optimize the injection moulding process by controlling the cooling behaviour of polypropylene by the used magnetite, barite, strontium ferrite, glass fiber (Weidenfeller et. al. 2005). It was found that Injection temperature decrease elastic modulus to a point that the different composites showed statistically comparable rigidities (Ota et. al. 2005). The fibre length of both extruded and kneaded flax filled PP materials after injection moulding is significantly reduced. The extruded samples show, as expected, a slightly higher fibre length and broader length distribution and a slightly higher fibre thickness (Harri ette et. al. 2005).

Rotational molding is used to produce hollow objects with acceptable properties and natural fibers can be used efficiently as reinforcing agent for different thermoplastic matrices. It was reported by researchers that the properties of natural fibre-reinforced composites are superior to those of the unreinforced ones (Torres, F.G. and C.L. Aragon. 2006). In recent years various polymers like polyethylene, polypropylene, nylon styrene etc. are being used for manufacturing roto-molded products. Other processing techniques for thermoplastic biocomposite are compression thermoforming molding, , pultrusion etc.

Conclusions

It has been found that flax fiber can be used efficiently to reinforce different thermoplastic available. Thermoplastic are plastic which be re-melted and utilized again. Flax is one of the potential natural fibers having high tensile strength and low density which can replace glass fibres in performance polymer composites (Joffe et al 2003).The surface characteristics play an important role in manufacturing biocomposite as proper adhesion in required at fiber-polmer interface. The hydrophilic nature of the flax fiber accounts for the poor fiber-matrix adhesion and poor compatibility. The surface of fiber can modified by various chemical treatments (alkaline,

saline, acetylation, acrylation, potassium permanganate, bleaching, chemical grafting and coupling agents) to enhances the fiber-polymer matrix adhesion and other mechanical properties. These treatments can be used in combinations to obtain desirable results. Different thermoplastic has different properties and their usage should be done as per desired properties in the product. Thermoplastic properties (T_g & T_m) also play an important role in processing of desired thermoplastic biocomposite. Among various processing techniques available, extrusion and injection molding are widely used to manufacture a range of product. Rotational molding is used for making large hollow objects like water tank effectively. Major research has been done on Flax reinforced PP based biocomposite.

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