

Characterisation of Bast Fibres and their Composites: a review

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Abstract:

Bast fibres are defined as those "obtained from the cell layers surrounding the stems of various plants" [1] (Farnfield and Alvey, 1975). The fibres find use in textile applications and are increasingly being considered as reinforcements for polymer matrix composites as they are perceived to be "sustainable". The fibres are composed primarily of cellulose which potentially has a Young's modulus of ~140 GPa [2] (Vincent, 1982) (being a value comparable with man-made aramid [Kevlar/Twaron] fibres). The plants which are currently attracting most interest are flax and hemp (in temperate climates) or jute and kenaf (in tropical climates). This review paper will consider the growth, harvesting and fibre separation techniques suitable to yield fibre of appropriate quality. The text will then address characterisation of the fibre as, unlike man-made fibres, the cross section is neither circular nor uniform along the length. The choice of polymer matrix systems (both thermoplastic and thermoset) will be covered. The review will then consider the use of the basic rule-of-mixtures in the context of natural fibre reinforced composites and will address composite materials characterisation using microscopical, mechanical, chemical and thermal techniques. The review will close with a brief overview of the potential applications and the environmental considerations which might expedite or constrain the adoption of these composites.

Introduction

Chand *et al.* [3] (1988), Lilholt and Lawther [4] (2000), and Franck [5] (2005) have reviewed the technology of natural fibres. Bolton [6] (1994), Mohanty and Misra [7] (1995), Nabi Saheb and Jog [8] (1999), Bledzki *et al.* [9] (2002), Netravali [10] (2003), Baillie [11] (2004), Mohanty *et al.* [12] (2005) and Pickering [13] (2008) have reviewed the use of natural fibres in polymer composites. The intention of this paper is to review the use of bast (plant stem) fibres in the context of fibre-reinforced polymer-matrix composites. This paper will not address natural fibres from animals (*eg* silk [14] (Scheibel, 2004) or wool [15] (Blicblau, 1997)) or those resulting from wood, seeds (*eg* coir or cotton), leaves (*eg* abaca, pineapple [16] (Arib, 2004) or sisal [17] (Li *et al.*, 2000)) or grasses (bamboo, miscanthus or wheat) which have at various times been considered as reinforcements for composites. Given the broad scope of this article, it will inevitably be incomplete, but will hopefully provide a sensible overview of the topic.

The majority of plant fibres which are being considered as reinforcements for polymeric materials are bast fibres, defined as "Fibre obtained from the cell layers surrounding the stems of various plants" [1] (Farnfield and Alvey, 1975). All the important natural fibres were in textile use thousands of years ago with flax used in Egypt at least 7000 years ago. Archaeologists have found evidence that even Stone Age man knew how to twist short lengths of fibre together to form cords and yarns, using a spinning process not very different from that used in primitive parts of the world to this day [18] (McIntyre, 1971).

Temperate regions

Flax [19-23] (grown for fibre) and linseed (grown for seed oil) are cultivars: varieties of the same plant species *Linum usitatissimum* bred with an emphasis on the required product. In the UK the flax plant is normally sown in March-May and may grow to one-metre high dependent on the variety (there are 180 species [21] (Lewington, 2003)). The Flax Crop Production page of the Flax Council of Canada (FCC) website [23] is an especially useful resource giving comprehensive details of the husbandry of this plant. The life cycle of the plant consists of a 45 to 60 day vegetative period, a 15 to 25 day flowering period and a maturation period of 30 to 40 days with 12 distinct growth stages in the flax plant [19] (Turner, 1987). In 1941, flax fibres (and hemp) were used in resin matrix composites for the bodywork of a Henry Ford car [21] (Lewington, 2003). Flax is amongst the natural fibres now finding use in thermoplastic matrix composite panels for internal structures in the car industry (including car door panels, car roof and boot linings, and parcel shelves) [24] (Bledzki and Gassan, 1999); and [25] (Brahim and Cheikh, 2007).

Hemp *Cannabis sativa L* is an annual plant native to central Asia and known to have been grown in China over 4500 years ago [21] (Lewington, 2003). It probably reached central Europe in the Iron Age (circa 400 BC) and there is evidence of growth in the UK by the Anglo-Saxons (800-1000 AD). It does not require fertiliser, herbicides or pesticides to grow well (and hence is potentially of great interest in the context of sustainability). In suitable warm conditions, it can grow to 4 metres in just 12 weeks. True hemp is a fine, light-coloured, lustrous and strong bast fibre obtained by retting (see below). The colour and cleanliness vary considerably according to the method of preparation of the fibre. The lower grades are dark cream and contain much non-fibrous matter. The main producing areas are "Italy, Yugoslavia and Russia" [1] (Farnfield and Alvey, 1975). The fibre ranges in length from 1.0-2.5 m. The term hemp is often used incorrectly in a generic sense for fibres from different plants, *e.g.* abaca (manila hemp), sisal (sisal hemp) and sunn fibre (sunn hemp) [1] (Farnfield and Alvey, 1975). In 1941, hemp fibres (and flax) were used in resin matrix

composites for the bodywork of a Henry Ford car which was able to withstand ten-times the impact on an equivalent metal panel [21] (Lewington, 2003). Hemp, like flax, is amongst the natural fibres now finding use in thermoplastic matrix composites for internal structures in similar automotive applications to those for flax fibres.

Nettle. Nettle is another plant-stem fibre which may find application as a reinforcement, including (European) nettle *Urtica dioica* [26] (de Leo et al, 1993), [27] (Dreyer et al, 1996), [28] (Davies and Bruce, 1998) and Himalayan nettle *Girardinia diversifolia* [29] (Singh and Shrestha, 1987). The yield of nettle fibre ranges from 335 to 411 kg/ha in the second year and from 743 to 1016 kg/ha in the third year [30] (Hartl and Vogl, 2002). The fibres are far stronger than cotton but finer than other bast fibres such as hemp. They are a much more environmentally friendly fibre crop than cotton, which requires more irrigation and agrochemical input. Lewington [21] (2003) states that "during the Second World War ... Britain's Ministry of Aircraft Production experimented with the use of a very strong, high-grade paper made from nettle fibre for reinforcing plastic aircraft panels as well as gear wheels and other machine parts".

Tropical regions

Jute fibre is obtained from the bast layer of the plants *Corchorus capsularis* (white jute) and *Corchorus olitorius* (dark jute). Each of the above classes is further sub-divided into numerous grades denoting quality and other characteristics. Jute is the second most common natural fibre (after cotton) cultivated in the world. It is an annual plant that flourishes in monsoon climates and grows to 2.5-4.5 m [21] (Lewington, 2003). It is primarily grown in Bangladesh, Brazil, China, India and Indonesia. Jute-based thermoplastic matrix composites find a substantial market in the German automotive door-panel industry (growing from 4000 tons in 1996 to over 21000 tons in 1999 and rising) [21] (Lewington, 2003).

Kenaf, *Hibiscus cannabinus*, is a warm season, short-day, annual herbaceous fibre plant native to central Africa, and a common wild plant of tropical and subtropical Africa and Asia. It has been cultivated since around 4000 BC for food and fibre. It is known as *mesta* in India and Bengal, as *stockroot* in South Africa, as *java jute* in Indonesia and as *ambari* in Taiwan. The plant has a unique combination of long bast (about 35% of the stalk dry weight) with short core fibres in place of the hollow core [1] (Farnfield and Alvey, 1975) and [31] (aboutkenaf.htm). Strong interest is being shown in this plant in Malaysia as it is fast growing and can yield two crops/year in the local climate. Kenaf belongs to the Malvaceae, a family notable for both its economic and horticultural importance. Kenaf has a high growth rate, rising to heights of 4-6 m (12-18 feet) in about 4-5 months. It can yield of 6-10 tons (new varieties may reach 12 tons) of dry weight per acre per year. It is similar to jute in many of its properties and may be used either as an alternative to, or in admixture with, jute.

Fibre processing

The typical production cycle for flax fibres [1], which are probably the most labour and agrochemical intensive of the natural fibres, is:

Tillage: the preparation of land for cropping by ploughing, harrowing or similar operations. Conventional tillage is preferred in flax cultivation which is primary tillage followed by early spring tillage and planting. No-till methods have been trialled by some growers with no significant change in flax yield [32] (OMAFRA). Agricultural lime (CaCO₃) may be applied to maintain soil pH. The Henfaes Research Centre suggests that for flax grown in UK, the levels of fertiliser are 40 kg/ha of nitrogen (N), 50 kg/ha of phosphorus (P) as P₂O₃ and 50 kg/ha of potassium (K) as K₂O – [33] (Bangor). For flax in Northern Ireland (NI), the suggested levels of fertiliser are 20 kg/ha N, 20 kg/ha P₂O₅ and 80 kg/ha K₂O.

Drilling (planting) the seed usually occurs between the end of February and early April in Belgium, France and the Netherlands or in early April in NI. Flax is planted in narrow rows (150-200 mm apart) using similar equipment to that used for cereals. Optimum seed depth is 25-40 mm deep and optimum seeding rates are 35-50 kg/ha [32] (OMAFRA).

Weed control: Flax is a poor competitor with weeds which can contaminate the scutched (i.e. decorticated - see below) flax fibres. Herbicides are applied to achieve this.

Plant growth for flax consists of a 45 to 60 day vegetative period, a 15 to 25 day flowering period and a maturation period of 30 to 40 days and is well illustrated in Turner [19].

Desiccation: Chemical desiccation or drying of the crop has numerous advantages over field retting including earlier harvesting, elimination of the need for swathing, reduction in combining time and less wear and tear on machinery. Glyphosate is typically applied 10-14 days after full flower at about mid-July in NI. Glyphosate is only used where stand retting (see below) is adopted followed by direct combine harvesting of the crop.

Harvest: by either combine harvester or pulling, in August/September.

Rippling: the removal of flax seed capsules by drawing pulled stems through a coarse steel comb.

Retting is defined for flax as the "subjection of crop or deseeded straw to chemical or biological treatment to make the fibre bundles more easily separable from the woody part of the stem. Flax is described as water-retted, dew-retted or chemically-retted, etc., according to the process employed" [1]. Enzymes (e.g. pectinase digests pectin binder) may be used to assist the retting process, but termination of the retting process may be a problem and failure to achieve this can result in reduced fibre properties. Pre-harvest retting of flax, when glyphosate is applied at the mid-point of flowering, depends on uniform desiccation of the entire stem and is difficult to achieve during a dry season [34] (Sharma). As in dew-retting, stand-retting of the desiccated flax in the field relies on microorganisms and is dependent on the vagaries of the weather.

Decortication [35, 36] is the mechanical removal of non-fibrous material from retted stalks or from ribbons or strips of stem to extract the bast fibres. For flax, the process is usually referred to as “scutching”. This is usually achieved by a manual operation, hammer mill, inclined plane/fluted rollers or willower.

Hackling is the combing of long (line) flax fibres in order to remove short fibres, to parallelise the remaining fibres and also to remove any extraneous matter (shive).

Carding is defined as “the disentanglement of fibres by working them between two closely spaced, relatively moving surfaces clothed with pointed wire, pins, spikes or saw teeth” [1]. The product is known as **sliver**.

Spinning is “the drafting [decreasing the mass per unit length] and twisting of natural (or man-made) fibres”. The product is known as **yarn** or filaments. In the bast-fibre industry, the terms ‘wet spinning’ and ‘dry spinning’ refer to the spinning of fibres in the wet state, and in the dry state respectively.

A similar route to that described above is followed for the production of the other bast fibres.

Subsequent treatment of natural fibre textiles may include [1]:

Acetylation is defined as the “process of introducing an acetyl radical into an organic molecule” and the term “is used to describe the process of combining cellulose with acetic acid”. The reduced number of hydroxyl groups after acetylation confers a more hydrophobic character to the fibres.

Bleaching is defined as the “procedure, other than by scouring only, of improving the whiteness of textile material by decolorizing it from the grey state, with or without the removal of natural colouring and/or extraneous substances. *Note:* removal of colour from dyed or printed textiles is usually called ‘stripping’”.

Grafting is the incorporation of monomers (e.g. cyanoethylation: reaction with acrylonitrile) or oligomers (short chain polymers) by chemical reaction at the fibre surface.

Mercerisation is the “treatment of cellulosic textiles in yarn or fabric form with a concentrated solution of caustic alkali [soda], whereby the fibres are swollen, the strength and dye affinity of the materials are increased, and their handle is modified. The process takes its name from its discoverer, John Mercer (1844)”.

Scouring (solvent treatment) is defined as the “treatment of textile materials in aqueous or other solvents in order to remove natural fats, waxes, proteins and other constituents, as well as dirt, oil, and other impurities”.

Characteristics of the fibres

The principal components of the fibre cell walls are cellulose, hemicelluloses and lignin with pectin considered to be the main binder (Table 1).

Table 1: Proportions of the principal components of raw hemp bast – data from [37] (SK Batra, 1998).

<i>Component</i>	<i>Minimum proportion</i>	<i>Maximum proportion</i>
Cellulose	67.0%	78.3%
Hemicellulose	5.5%	16.1%
Lignin	2.9%	3.3%
Pectin	0.8%	2.5%

Cellulose is the most important structural component of nearly all green plant cell walls, especially in many natural fibres (flax, jute, hemp, cotton, *etc.*). The *cellulose* polymer is composed entirely of carbon, hydrogen and oxygen (the molecular formula can be written as if it consisted of only carbon and water, hence the name *carbohydrate*). Cellulose is a polysaccharide $(C_6H_{10}O_5)_n$ which can be degraded to give only glucose $(C_6H_{12}O_6)$. Note that the cellulose repeat unit is more correctly an *anhydroglucose* unit (glucose minus water). Cellulose is a strong, linear (crystalline) molecule with no branching. Cellulose has good resistance to hydrolysis although all chemical and solution treatments will degrade it to some extent. Resources on cellulose can be found at references [38-41].

Hemicelluloses are lower molecular weight polysaccharides, often copolymers of glucose, glucuronic acid, mannose, arabinose and xylose, which may form random, amorphous branched or nonlinear structures with little strength. Hemicellulose is easily hydrolyzed by dilute acids or bases, but nature provides an arsenal of hemicellulase enzymes for its hydrolysis. These enzymes are commercially important because they open up structure bound cellulose materials [42-44] (Bungay, 1996/97).

Lignin [44] is formed by non-reversible removal of water from sugars (primarily xylose) to create aromatic structures. Lignification progresses as the plant matures conferring mechanical stability to the plant and helping with the transport of water. Lignin resists attack by most microorganisms: the aromatic rings are resistant to anaerobic processes while aerobic breakdown of lignin is slow.

Vincent [2] (1982) states that the elementary fibril of cellulose is about 3.5 nm diameter and contains about 40 molecules. The cellulose elementary fibrils can be arranged into larger fibrils of 20-25 nm diameter. They are formed by neighbouring cellulose chains forming hydrogen bonds leading to partially crystalline regions (micelles). These form the structurally strong framework in the cell walls. Cellulose found in plants as microfibrils may be 2-20 nm diameter and 100 nm - 40 μ m long) [39] (Chaplin, 2002).

Cellulose has been estimated to have a modulus of 140 GPa when using X-ray diffraction to determine the strain [2] (Vincent, 1982). A slightly higher figure has been obtained by calculation from the chemical structure of the crystal with consideration of the straightening of the covalent bonds and stretching of the interchain hydrogen bonds. If hydrogen bonding is not included in the calculation, then the modulus drops by a factor of ~8. Experimentally measured moduli will inevitably be lower than theoretical values (due to <100% crystallinity and off-axis fibre orientation). Nevertheless, Vincent has reported values of 100 GPa for dry flax and ~80 GPa for wet flax. As water penetrates the amorphous regions of cellulose, the stiffness can drop by a factor of 2-4 as the contribution of the hydrogen bonding is progressively removed. Table 2 presents some mechanical property data for bast fibres. Table 3 presents the winding angle of the cellulose molecule relative to the principal axis for some natural fibres. Figure 1 presents reported elastic moduli against winding angle for several plant fibres.

Table 2: Typical properties of some bast fibres, with comparative values for E-glass

Properties	Fibre					
	E-glass[45]	Flax[46]	Hemp [45]	Jute[45]	Kenaf	Nettle [49]
Density kg/m ³ (d)	2550	1530	1520	1520	1193 [47]	-
E-modulus (GPa)	71	58 ± 15	70	60	14-38 [48]	87 ± 28
Tensile strength (MPa)	3400	1339 ± 486	920	860	240-600 [48]	1594 ± 640
Specific modulus (E/1000d)	28	38	46	39	12-32	-
Elongation at failure (%)	3.4	3.27 ± 0.4	1.7	2	-	2.11 ± 0.81
Moisture absorption (%) [50 via 51]	-	7	8	12	-	-

Table 3: The angle between the cellulose chains and cell length [52]
(extracted from RD Preston, *The Physical Biology of Plant Cell Walls*, 1974, page 293)

Plant/technique	θ° (range)	θ° (average)	Source
Cannabis sativa (hemp)			
optical microscopy: major extinction position	0.0 - 5.0	2.3 ± 0.3	[53] (Kundu and Preston, 1940)
striations	0.0 - 6.0	2.0 ± 0.3	[53] (Kundu and Preston, 1940)
Corchorus capsularis (jute)			
optical microscopy: major extinction position	0.0 - 23.0	7.9	[54] (Preston, 1941)
Agave sisalana (sisal leaf fibre)			
optical microscopy: major extinction position	8.0-32.3	20.4 ± 7.2	[55] (Preston and Middlebrook, 1949)
x-rays		18	[55] (Preston and Middlebrook, 1949)

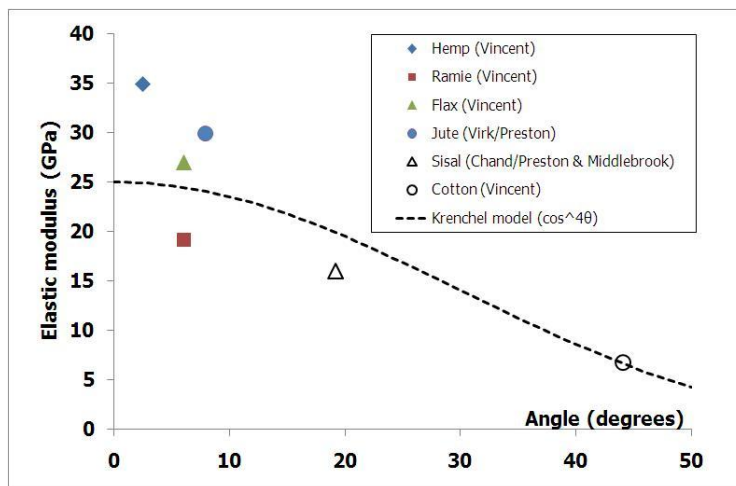


Figure 1: The variation of the elastic modulus of plant fibres with the angle between the cellulose molecules and the fibre principal axis for bast fibres (solid markers), leaf fibres (sisal) and seed fibres (cotton) combining data from Vincent [2], Chand *et al* [3], Preston [54] and Preston and Middlebrook [55] and Virk [56] with the Krenchel [57] model for $\cos^4\theta$ dependence on the fibre orientation forced though the cotton data.

Fibre Quality

Unlike synthetic reinforcement fibres, the natural fibres are perceived to have significantly greater variability in their mechanical properties as a consequence of the conditions experienced in the field and the potential damage arising from the above processes.

Polymer matrix

There are four key temperatures which affect the manufacture and performance of polymers and their composites. In ascending order they are:

- **T_g**: the glass transition temperature – where segmental motion of the polymer molecules is frozen out on cooling and the polymer undergoes a transition from tough/viscoelastic (when warm) to brittle/elastic (below T_g). This temperature should not be exceeded where the component will be subjected to maintained loads which could result in creep deformation.
- **T_m**: the crystalline melting temperature (range) – this is not applicable to amorphous polymers.
- **T_p**: the processing temperature.
- **T_d**: the decomposition temperature.

Synthetic Thermoplastic Polymers. Thermoplastic polymers are generally characterised by a high melt viscosity and a heat-form–cool manufacturing cycle. Typical thermoplastic matrix systems for glass-fibre reinforced composites are:

- polypropylene (PP, T_g = -20 to -5°C, T_m = 165-175°C),
- polyamide (PA, also known as nylon, T_g = 40-60°C, T_m = 160-265°C), and
- polyethyleneterephthalate (PET, saturated polyester, T_g = 70-80°C, T_m = 245-265°C).

There is a potential problem with degradation of the cellulosic fibres if extended process times are used with temperatures of $\geq 200^\circ\text{C}$. The glass transition temperature is normally $200 \pm 50^\circ\text{C}$ below T_m and this limits the application of natural fibre composites under moderate-to-high stress as creep deformation can occur above this temperature.

Biodegradable Thermoplastics based on Natural Feedstocks. The history of biopolymers and bioplastics has been reviewed by Scott [58] (1999) and Stevens [59] (2001). A number of biodegradable thermoplastic systems might be considered as matrix systems for fibre reinforced composites, including Cellulose Acetate Butyrate (CAB), Poly Lactic Acid (PLA), Poly Hydroxy Butyrate (PHB), Poly Hydroxy Valerate (PHV), Poly Hydroxy Butyrate-co-Valerate (PHBV) and Starch-CaproLactone blend (SCL).

PLA has been commercialised by Cargill Dow as NatureWorks™ PLA using corn-derived dextrose as the feedstock. It has an estimated energy consumption of 56 MJ/kg of polymer (50% more than for PET, 40% less than for polyamide) [60] (Gerngross and Slater, 2000) and hence this matrix material may not be the best option if carbon dioxide emissions are considered. Gupta et al [61] have reviewed the use of PLA (as a fibre) and identified the key thermal properties to be: T_g ~60-70°C, T_m ~150-170°C. This small difference between the two transition temperatures could allow extended process times without significant damage to the cellulose fibres while permitting use at ambient temperatures with resistance to creep deformation.

PHB, PHV and PHVB are in a class of materials known as Poly Hydroxy Alkanoates (PHA) in which a bacterium, initially *Ralstonia eutropha*, converts sugar directly into plastic. PHA naturally accumulates within the microbe as granules and can constitute up to 90% of a single cell's mass. Plants (*Arabidopsis thaliana*, *Brassica napus* (oilseed rape) and corn) have been genetically engineered to produce PHA [62] (Poirier et al, 1992), [63] (Slater et al, 1999), [60] (Gerngross and Slater, 2000). Hobbs and Barham [64-66] (1998) have undertaken a comprehensive study of the fracture and ageing behaviour of PHB.

Synthetic Thermosetting Resins. Thermosetting resins are usually supplied as a (relatively) low viscosity liquid which is combined with a curing agent (and heat) such that the mixture undergoes a chemical reaction (cross-linking) to form an insoluble, infusible solid material. Process temperatures are typically ambient to 200°C. The most common thermosets are:

- Unsaturated PolyEster resin (**UPE** diluted with styrene and cured with ~ 1% catalyst),
- Vinyl Ester resins (**VE** diluted with styrene and cured with ~ 1% catalyst),
- Phenolic Resins (cured with strong acid catalysts)
- Epoxy resins (**Ep** cured with hardener in stoichiometric proportions – typically a 1:3 to 1:5 mix ratio).

Thermosetting resins based on Natural Feedstocks. Thermosetting resins have been produced based on e.g. acrylated epoxidised soybean oil (AESO), cashew nut shell liquid (CNSL) [67, 68] (Jayabalan, 1985. Ikeda, 2002), norbornyl epoxidised linseed oil (NELO) [69] (Chen et al, 2002) and furan resins from furfuryl alcohol [70] (BioComp).

Composites Processing

The techniques for the manufacture of fibre-reinforced polymer matrix composites have been reviewed by Åström (1997), Gutowski (1997), Davé and Loos (1999) and Campbell (2003) [71-74], albeit that their emphasis is very much on synthetic fibres and thermosetting resins. Certain processes have been considered in greater detail:

- vacuum bagging, including autoclave cure [75-77] (Ciriscioli and Springer, 1990. Noakes, 1992, McBeath, 2000).
- Liquid Moulding Technologies (LMT) or Liquid Composite Moulding (LCM), including Resin Transfer Moulding (RTM) [78-84] (van Harten, 1993. Abraham and McIlhagger, 1996. K Potter, 1997. Rudd *et al*, 1997. Beckwith and Hyland, 1998. Kruckenberg and Paton, 1998. Parnas, 2000).
- Resin Infusion under Flexible Tooling (RIFT) [85-88] (Williams *et al*, 1996. Cripps *et al*, 2000, Summerscales and Searle, 2005, Beckwith, 2007).
- Filament winding [89] (Peters *et al*, 1991/1999).
- Pultrusion [90, 91] (Meyer, 1985. Starr, 2000).

Pinzelli [92] (1991) has reviewed the state-of-the-art in cutting and machining of composite materials based on aramid fibre reinforcements. He recommends that a band-saw with a fine tooth blade (14-22 raker-set or straight-set teeth/inch [\sim 5-9 teeth/cm]) operating at high surface speeds with the running blade teeth pointing upwards (reverse) should minimise the production of fuzz and keep the teeth from snagging fibres. Cullen [93] machined flax/jute epoxy composites using a band saw with 18 teeth/inch running in reverse. This configuration cut the fibres much more cleanly than when running with the teeth facing forwards.

Reinforcement Forms for Composites

The effective utilisation of the properties of a fibre as a reinforcement is a function of the fibre length and the fibre orientation with respect to the stress. These two fibre parameters are generally present with a statistical variation and hence are measured as the fibre length distribution factor (η_l) and the fibre orientation distribution factor (η_o). Both parameters range between 0 (short fibres or alignment transverse to the stress respectively) and 1 (continuous fibres or aligned with the stress respectively). Typical reinforcement forms are unidirectional ($\eta_l = 1$ and $\eta_o = 1$ when continuous fibres are aligned with the stress), woven fabrics ($\eta_l = 1$ and $\eta_o = 0.5$ when one set of fibres is aligned with the stress) and random chopped strand mats ($\eta_l < 1$ and $\eta_o = 3/8$).

Increasing alignment of the fibres enables more fibre to be incorporated into the composite. For practical purposes, the limiting fibre volume fractions are around 75% (unidirectional), 65% (woven) or 30% (random orientation). A major constraint on the effective use of natural fibre reinforcements is the lack of a cost-effective commercial methodology for the production of woven or otherwise aligned fabrics in the materials supply chain.

Interfacial bonding

The realisation of the full mechanical performance of the reinforcement is critically dependent on the effective load transfer by shear over the "half critical length" at each fibre end, which in turn is a function of the chemical and physical bonds between the fibre and the matrix. The main approaches to enhancement of the interaction between the fibre and the matrix are surface modification of the fibre (grafting or other chemical/physical treatments), application of coupling agents to the fibre surface or the use of compatibilisers in the matrix.

George *et al* [94] (2001) have critically reviewed the physical and chemical treatments that may improve the fibre-matrix adhesion. They conclude that good compatibility between cellulose fibres and non-polar matrices is achieved using polymeric chains that favour entanglement and interdiffusion with the matrix.

Bismarck *et al* [95] (2002) have used scanning electron microscopy (SEM), Brunauer-Emmett-Teller specific surface area (BET-SSA) determination, conventional water absorption measurements, electrokinetic zeta-potential (EZP) measurements and cross-polarised magic angle spinning nuclear magnetic resonance (CP-MAS NMR) spectroscopy to investigate the surface characteristics of flax, hemp and cellulose fibres. The effect of fibre separation methods and chemical treatments were studied. The flax-fibre separation processes were shown to affect the fibre surface morphology and, in turn, the specific fibre surface area. The EZP measurements clearly reflect differences in the hydrophilicity of the fibres. Green fibres (flax and hemp) are the most hydrophilic. The retting process results in fibres with a relatively high level of surface wax and hence reduced hydrophilicity.

Hassan *et al* [96] (2003) found improved tensile strength and elongation to break in mercerised jute fibres which had been grafted (with silanes and acrylamide under ultraviolet radiation) and improved resistance to weathering.

Zafeiropoulos *et al* [97] (2003) used three spectroscopic methods (XPS: x-ray photoelectron spectroscopy, ToF-SIMS: time-of-flight secondary ion mass spectrometry and ATR-FTIR: attenuated total reflectance Fourier transform infra-red spectroscopy) to investigate the effect of acetylation and stearic acid treatment on chemical and physical characteristics of flax fibre surfaces. They found no evidence of lignin at the fibre surface (although it could be in the bulk fibre). They noted that acetylation appeared to be unevenly distributed.

Materials characterisation

The determination of the parameters required for the rule-of-mixtures can be achieved in a variety of ways [98, 99] (Grafil, 1980. CRAG, 1988). Optical or electron microscopical techniques with image analysis [100-102] (Guild and Summerscales, 1993. Summerscales, 1998. Clarke and Eberhardt, 2002) may be used to determine η_d , η_b , η_o and V_f of composite materials. For natural fibres in a resin matrix, it will normally be necessary to enhance the contrast between the components (by *e.g.* polarising filters or staining techniques - Dubot [103] (2001) used methylene-blue as a stain for linseed fibres). Grafil Test Method 102.13 uses microscopy with an image splitting eyepiece to determine individual fibre diameters.

Optical coherence microscopy (OCM, also known as optical coherence tomography (OCT)) is a novel imaging technique which permits the acquisition of tomographic images with high resolution ($\sim 15 \mu\text{m}$ in three dimensions) and a high dynamic range ($>100 \text{ dB}$). It has found extensive use in biomedicine, notably for ophthalmic procedures. Reeves *et al* [104] (2002) have applied OCM to visualise the cellular and subcellular structures within intact *Arabidopsis* plants (including leaves, flowers, ovules and seeds).

Thumm [105] (2004) has used confocal microscopy to determine the interfacial behaviour and (non-) interactions in plant fibre composites. Labelled dyes were added to the polymer matrix to enhance the fluorescence. The extent of interaction was indicated by line profiles of the fluorescence for each component.

The determination of the fibre volume fraction is problematic. The textile industry, and many researchers in the composite field, normally assumes that fibres are of a regular circular cross-section and use microscopy transverse to the principal axis to determine a characteristic dimension (incorrectly referred to as the diameter). Figure 3 is a confocal scanning laser microscope image of a cluster of elementary jute fibres: these fibres (which are typical of bast fibres) are clearly not round.

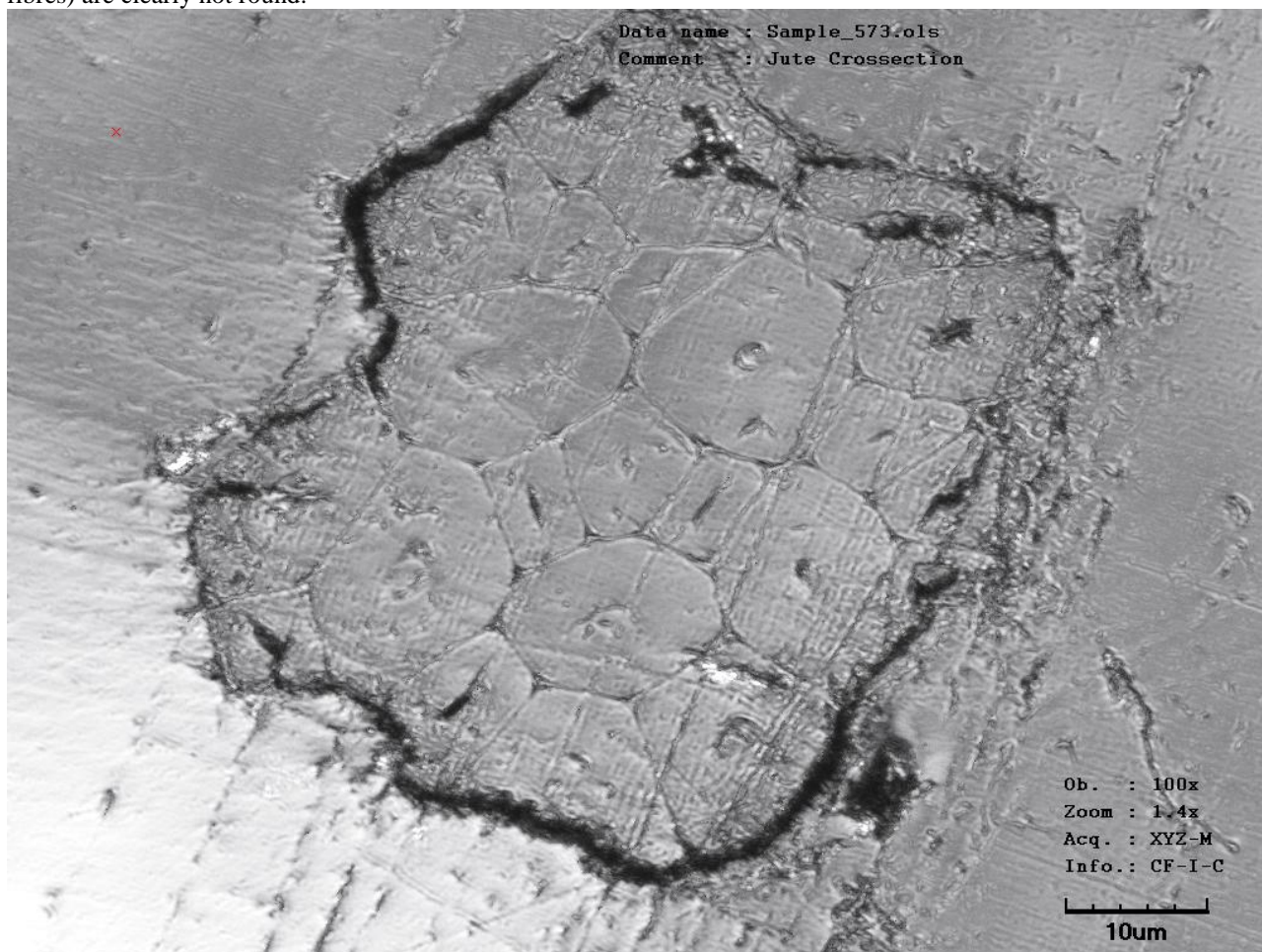


Figure 2: Confocal Scanning Laser Microscope (CSLM) image of a cluster of elementary jute fibres, courtesy of Amandeep Singh Virk.

Further, the fibres are hygroscopic so may swell and hence weight will be a function of the moisture content. The density of the fibre should be quoted at a specific moisture content (or ambient relative humidity) or ideally plotted against moisture content. The determination of volume fraction of the composite will be dependent on the moisture level and any consequent swelling of fibres. Subject to the above, the fibre volume fraction of composite materials may also be obtained by, *e.g.*:

- tow counting for unidirectional composites in an open-ended mould (Grafil Test Method 302.24) or from fabric areal weight in a moulding of known thickness (CRAG method 1000-2).
- direct weighing (Grafil Test Method 302.13) when a closed mould is used and no fibre is lost in the moulding flash. The mass fraction is then the mass of fibre divided by the mass of the composite after fabrication. Accurate values of the density of the components are necessary to convert the mass fraction to a fibre volume fraction.
- density gradient column (Grafil Test Method 301.12) which is based on observing the level to which the test specimen sinks in a column of liquid when the density of liquid changes uniformly with height. The absorption of liquid by the test specimen may complicate the analysis for this technique when natural fibres are under test.
- Archimedes principle (Grafil Test Method 301.21. CRAG methods 800/1000-1) using weight measurements in air and in water. The absorption of liquid by the test specimen may complicate the analysis for this technique when natural fibres are under test.
- resin burn-off (CRAG method 1000-3c) in an oven at 580-600°C. This method is inappropriate for natural fibre composites as both components of the composite will burn. It may be possible to use Thermo Gravimetric Analysis (TGA, possibly in combination with both normal and inert atmospheres) to determine the volume fraction if the two components have clearly differentiated decomposition temperatures. The technique has been used by Sharma *et al* [106-108] (1996, 1998, 2000) to characterise the components of flax fibres.
- chemical digestion using sulphuric acid and hydrogen peroxide (Grafil Test Method 302.56. CRAG method 1000-3a) or nitric acid (CRAG method 1000-3b). It may be necessary to select different chemicals for natural fibre composites. Green [109] (1991) has proposed the use of a microwave acid digestion bomb for the determination of fibre volume fraction of carbon-epoxy composites. This may be suitable for natural fibre composites. The "bomb" is a sealed chemically-inert vessel in which microwave heating can be used for rapid sample dissolution. These bombs can be placed directly in a microwave oven for specific, high speed heating to drastically reduce the time required to dissolve or digest an analytical sample [110].

Mechanical Properties

The elastic modulus (and ultimate tensile strength) of the individual filaments can be determined by ASTM D3379-75 (now withdrawn), Grafil Test Method 101.13 for 50 mm fibres or the French norm NFT25-704. A comprehensive study of jute fibres over a range of lengths using a similar methodology has been reported by Virk *et al* [56] (in press). The corresponding values for fibre tows can be determined by Grafil Test Method 103.22.

The elastic modulus of a composite material can normally be predicted using the standard rule of mixtures (Equation 1):

$$E_c = \eta_l \eta_o V_f E_f + V_m E_m \quad \text{Equation 1}$$

where η_l is the fibre length distribution factor, η_o is the fibre orientation distribution factor, E_f is the elastic modulus of the fibre, E_m is the elastic modulus of the matrix, V_f is the fibre volume fraction and V_m is the matrix volume fraction (assuming $V_f + V_m = 1$, i.e. no voids or other inclusions).

Lamy and Baley [111] (2000) conducted tensile tests on flax fibres of different diameters, d_i , and found that the Young's modulus for each class, E_i , decreased with increasing fibre diameter, where i is the class number (Table 4). They have proposed Equation 2 for the longitudinal elastic modulus, E_L , of a unidirectional flax-fibre composite material:

$$E_L = V_f \frac{\sum_{i=1}^n n_i d_i^2 E_i}{\sum_{i=1}^n n_i d_i^2} + V_m E_m \quad \text{Equation 2}$$

where n_i is the number of samples in each class and E_i is the Young's modulus of fibres in the range i . K_i is the contribution of fibres n_i of mean diameter d_i to the total elastic modulus E_f at 58643 MPa for the sample of fibres tested.

Table 4: Dependence of properties of flax fibre on fibre diameter (Tables 1/2 of Lamy and Baley [111] (2000))

Class i	1	2	3	4	5	6	7	8	9	10	11	12
n_i	6	44	84	136	170	143	121	83	45	25	7	5
d_i (µm)	6.8	8.8	11.2	13.9	16.2	18.8	21.2	23.6	26.2	28.6	32.1	34.5
E_i (MPa)	78680	75820	72320	68530	65220	61580	58130	54720	50960	47490	42560	39030
K_i (MPa)	73	860	2530	5990	9670	10350	10520	8400	5230	3230	1020	770

The dependence of the modulus of the composite, calculated using Equation 2, against fibre volume fraction correlated well with the experimental results. It was noted that selection of fibre diameters could be a route to improvement of the elastic properties of flax fibre reinforced epoxy resin composite materials. Baley [112] (2002) further reported a decrease in the Young's modulus with increasing fibre diameter for flax fibres (Fig.2a). Bodros and Baley [49] (2008) found that the Young's modulus and the stress at break of nettle fibres decreased when the fibre diameter increased (Fig. 2b).

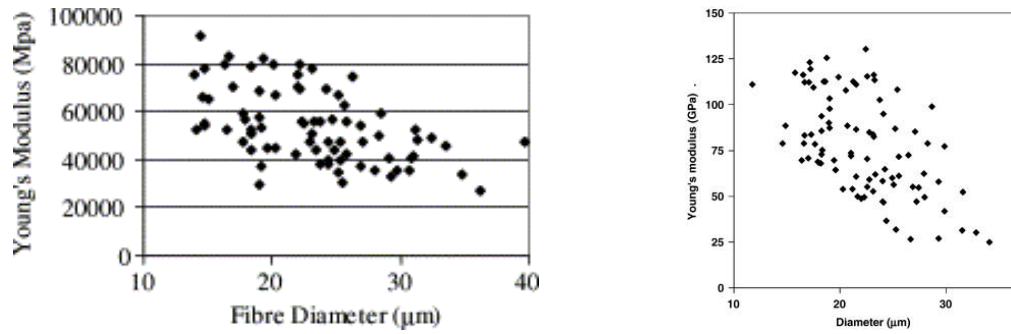


Figure 3: Young's modulus as a function of (a) flax fibre diameter (left) and (b) nettle fibre diameter (right) (reproduced from [111] and [49] respectively ... permission for use requested)

The authors of this review propose a modification of the rule of mixtures through the use of a fibre diameter distribution factor, η_d , (with values in the range 0-1) to produce Equation 3. This fibre diameter distribution factor will be related to the probability density function for the fibre diameter, which could be obtained from a comprehensive study of the chosen fibre and may well correlate to the factor given in Equation 2. Derivation of that parameter is beyond the scope of this review.

$$E_c = \eta_d \eta_l \eta_o E_f + V_m E_m \quad \text{Equation 3}$$

Typical mechanical properties for natural fibre reinforced polymer matrix composites are given in Table 4.

Table 5: Typical properties of natural fibre reinforced polymer matrix composites

(nb: specific results* reported here are for the composite with highest elastic modulus reported in each paper)

Fibre	Matrix	Configuration	% fibre	E (MPa)	σ' (MPa)	ϵ' (%)	Ref	Source
Flax	Epoxy	unidirectional	40	28000	133	n/a	113	Van der Wegenberg et al (2003)
Flax	PLLA	aligned roving	40 v/o	7300±500	44.1±7.2	0.9±0.2	114	Oksman et al (2003)*
Flax	PLLA	random mat	30 v/o	9519	99	1.5	115	Bodros et al (2007)*
Hemp	UP resin	mat	44 w/o	6200±600	53.0±6.0	1.39±0.26	116	Yuanjian & Isaac (2007)
Hemp	PP	injection moulded	40 w/o	5300	50.5	n/a	117	Beckerman & Pickering (2008)
Jute	PP	injection moulded	50 w/o	5480±330	32.0±0.5	n/a	118	Karmaker & Schneider (1996)
Jute	PP/MAPP	injection moulded	50 w/o	5420±420	57.9±0.4	n/a	118	Karmaker & Schneider (1996)
Nettle	Epoxy	unidirectional	24 v/o	9000	91	n/a	119	Merilä (2000)
Nettle	Phenolic	unidirectional	23 v/o	5000	13	n/a	119	Merilä (2000)

A potential problem with natural fibre reinforced polymer matrix composites is the hydrophilic nature of the cellulose fibres and hence the moisture sensitivity of the resulting composites. Khalil et al [120] have studied the acetylation of plant fibres in the context of improvement of the mechanical properties of composites was studied. Bast fibres from jute and flax were considered (along with oil palm empty fruit bunch (EFB), coconut fiber (coir) and oil palm frond (OPF)). The two bast fibres were found to be the least reactive of the five fibres studied.

Costa and D'Almeida [121] studied the effect of water absorption on the mechanical properties of jute or sisal fibre reinforced polyester or epoxy matrix composites. The diffusion behaviour in both composites could be described by the Fickian model. The jute-epoxy composites showed the best mechanical properties and were the least affected by the exposure of the composites to distilled water (Figure 4). This behaviour was attributed to a better fibre-matrix interface and better moisture resistance of jute fibres.

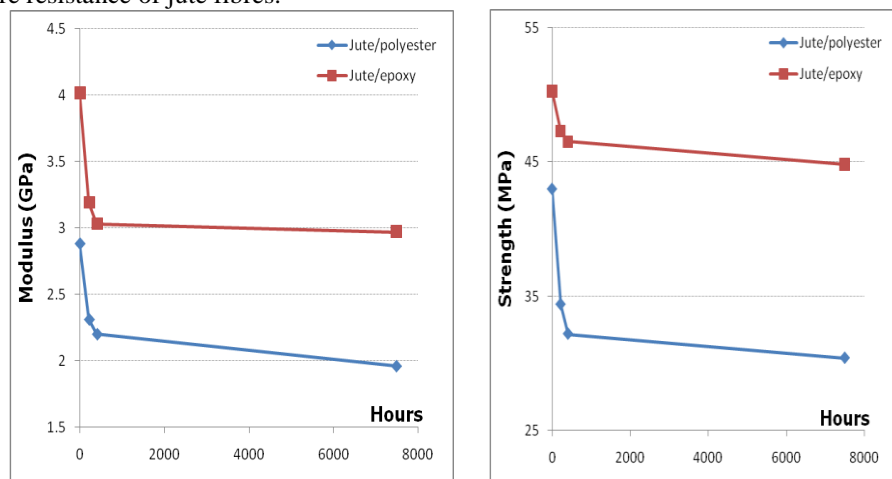


Figure 4: The deterioration of modulus and strength (loading mode not specified) for jute fibre composites exposed to distilled water for 0, 220, 410 or 7500 hours (data from Costa and D'Almeida [121])

The topic of moisture sensitivity in plant fibre composites warrants a review paper in its own right.

Markets and Current Applications

European production of fibre amounted to 60–70 thousand tons of flax and 25-30 thousand tons of hemp in 1999/2000 [122] (Karus et al, 2000). During the same year, world production of jute and kenaf was 2,610 thousand tons, concentrated in two main producer countries, India and Bangladesh. Production of jute and kenaf declined by 49% and 19% respectively from a peak of 3920 thousand tons in 1997/8 [123] (Aimin Liu, 2001).

The world market for composites was 7 million tons in 2000, and projected to reach 10 million tons in 2010 [124] (Nodal, 2001):

- the North American market accounts for nearly half of world-wide composites (3.4 million tons – 47%),
- Europe follows at 2 million tons – 28%,
- Asia is the third major market at 1.6 million tons – 23%.

The principal European producers are Germany, Italy, France, UK and Spain. Thermoset composites account for roughly 70% of the composites processing industry in Europe.

There is a wide range of user industries for composites in all the international markets, including mechanical structures, chemical plant and electrical insulators. It should be noted that although automotive and aerospace applications account for over half the value, the volume consumption is only 26%. Whilst composites for these industries (and medical and sports applications) are often based on high cost carbon and aramid fibres, there is a mass market for low cost composites – the GRP industry, based predominantly on glass fibre reinforcement and polyester resins.

By Western standards, the Indian composites industry is relatively small at 17.2 thousand tons in 2001 compared to France at 300 thousand tons [125] (Sundaram, 2002). After a period of exceptional growth up to 1999, the Indian industry has failed to live up its growth potential in recent years, and is relatively stagnant. The industry suffers from fragmentation (over 1,700 processors), weak demand from client sectors, under-utilisation of capacity, and quality problems.

The use of natural fibres as reinforcement for thermoplastic components is a relatively new phenomenon, dating back only to about 1995. The market has developed from pioneering work in the German automotive industry [24] (Bledzki and Gassan, 1999). In this market, jute is in competition with the indigenous European fibres, flax and hemp, and despite being used at the outset, has consistently fluctuated in relative market share. Figure 5 shows the total consumption of natural fibres increasing to 17,200 tons in 2002 [126] (Kaup et al, 2003): estimated as flax at 9,000 tons and hemp at about 2,200 tons with the balance of 6,000 tons split between jute, kenaf and sisal.

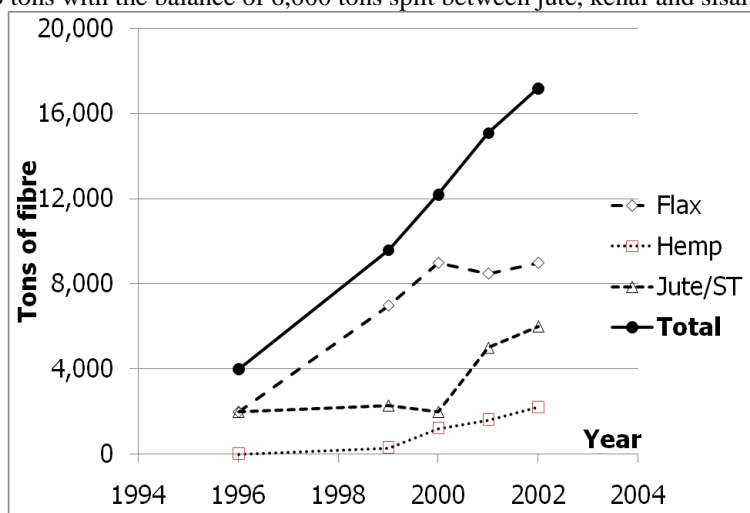


Figure 5: Use of natural fibres in the German automotive industry 1996 – 2002 (tons) (after Kaup et al, 2003 [126]).

The Status Report [126] scaled down the forecast of future consumption of natural fibres. Previous optimistic estimates of up to 35,000 tonnes/year have been reduced to just over 25,000 tonnes in 2005. An earlier report [127] (Ellison and McNaught, 2000) indicated that Germany alone was responsible for over two-thirds of the European production of natural fibre composites, and estimated the market size to be somewhat larger than the 2003 study. In Germany, the natural fibre composites market has created a dedicated infrastructure of secondary suppliers (mainly non-woven producers) and is still growing strongly. The phased withdrawal of EU Common Agricultural Policy (CAP) subsidy on flax and hemp fibre crops may retard the growth of this new industry.

Ellison and McNaught [127] (2000) have identified established commercial uses in:

- automotive interior components (Germany 70%). Natural fibre panels are now in common use as door and boot liners and parcel shelves. Every Mercedes and BMW model now features such components, and the technology has been taken into the Fiat group, Ford and the volume French marques by Tier One suppliers. Current use amounts to about 10kg per car, with a potential for double this consumption.
- domestic insulation (Germany 30%) – tow or sliver bound lightly with polymers.

Established benefits for the automotive industry include good mechanical properties, fewer occupational health issues in handling and lack of splintering in accidents. A review of relevant properties demonstrated the potential for competition with GRP (Glass Reinforced Plastics) [126] (Kaup et al, 2003). However, the principal drivers are the potential for weight reduction (10–30%) and the consequent cost advantage of natural fibre composites.

Further, they established that two processes were dominant:

- Compression moulding (thermoplastic and thermoset): thermoplastic mouldings usually comprise natural fibre/polypropylene blended needle-felt substrates, whilst thermosets consist of 100% natural fibre needle-felts impregnated with resin by processes such as RTM and S-RIM. The market is dominated by fibre carded sliver or tow chopped to a staple length of 80–90 mm.
- Co-extruded granulate for injection moulding is now under development on several fronts (Daimler Benz, Perstorp in Sweden, ATO/DLO), and already has an estimated 4.7% share of automotive processing technology in Germany [126] (Kaup et al, 2003). Short chopped sliver of 4–6 mm is now being used for co-injection.

Low fibre prices remain an obstacle to investment in the natural fibre supply chain. The MAFF study in early 2000 [127] (Ellison and McNaught, 2000) found benchmark prices for jute, flax or hemp to be in the range DM 0.90-1.20/kg, and three years later (after the introduction of the Euro), wholesale price levels remain in the EUR 0.55-0.62/kg range. Prices were set at a low level in the mid-1990s and the automotive industry remains a stringent taskmaster.

The major constraints on the application of natural fibres as reinforcements include

- batch-to-batch inconsistency and other fibre quality considerations.
- performance limitations, notably tensile strength and impact strength.
- susceptibility to moisture absorption
- odour and fogging.

Environmental Considerations

The End-of-Life Vehicle (ELV) Directive was enacted by the European Commission (EC) during 2002 to address pollution resulting from vehicles that have reached the end of their useful life. It aimed to significantly reduce the 8 million tonnes of waste generated each year by the 12 million cars that have reached their end of life. In phase one of the directive, car makers were responsible for the disposal of all new production that would eventually become ELV. In 2007, they became responsible for all the vehicles they had ever produced. The legislation also stipulates that car-makers must re-use or recover 85% of ELVs by weight. At least 80% of that weight must be re-used or recycled while up to 5% can be dealt with through other recovery operations such as incineration. In 2015, this target will rise to 95% of ELVs by weight, 85% of which must be re-used or recycled.

The new directives on landfill and ELV, encourage industry to move away from landfill and energy recovery towards mechanical recycling or reuse. Customers (especially in the automotive sector) are increasingly asking the composites industry to accept responsibility for recycling the end-of life waste. In the context of the EC directives, it will be necessary to make a strong case for disposal by incineration or by composting. Composites, based on natural fibre reinforcements, could prove to be more beneficial in the environment than “recyclable” materials but there is a need for quantitative life cycle analysis to clearly demonstrate that this is indeed the case.

Joshi et al [128] (2004) reviewed comparative life cycle assessment studies to conclude that natural fibres would be environmentally superior to glass fibre reinforced composites. The key drivers in favour of natural fibres were:

- natural fibre production has lower environmental impacts compared to glass fibre production,
- natural fibre composites have higher fibre content for equivalent performance, reducing the more-polluting polymer content
- the light-weight natural fibre composites improve fuel efficiency and reduce emissions in the use phase of the component (especially in automotive applications), and
- end-of-life incineration results in recovered energy and carbon credits.

However, the conclusions are tempered by two caveats:

- fertiliser use in natural fibre cultivation results in higher nitrate and phosphate emissions which can lead to increased eutrophication in local water bodies, and
- the environmental superiority of natural fibre composites may be negated if the operating lifetime is significantly reduced compared to the glass fibre composites.

Reed and Williams [129] (2004) have examined the potential for waste biomass (in the form of natural hemp, flax, jute, coir or abaca fibres) to produce activated carbon. After pyrolysis in a fixed bed reactor and steam activation, the yield of activated carbon was 20% by weight of the original biomass and surface areas were in the range 770-879 m²/g.

The environmental impact of natural fibres in industrial applications has been reviewed by van Dam and Bos [130]. They include quantitative data and suggest that:

- "natural fibre production requires less than 10 percent of the energy used for production of PP fibres (around 90 GJ/tonne)".
- the total energy input for jute fibre cultivation (excluding field labour, retting and decortication) was calculated at 3.75-8.02 GJ/tonne when grown by numerous small farmers utilising labour and animal power with limited use of agrochemicals and machinery.
- the energy input from inorganic fertilisers, based on the energy content of the substance and the energy required for production, transport, storage and application is 17 MJ/kg for potassium (K), 26 MJ/kg for phosphorous (P) and 128 MJ/kg for nitrogen (N).
- the energy input from pesticides, based on the energy content of the substance and the energy required for production, transport, storage and application is 320-476 MJ/kg for fungicides, 461-568 MJ/kg for insecticides and 467-622 MJ/kg for herbicides.

In an independent analysis, Khan [131] calculated that the total energy consumed, including the embodied energies of fertilisers and pesticides would be 18-20 GJ/tonne of jute fibre.

Dissanayake et al [132-135] have begun to undertake a Quantitative Life Cycle Assessment (QLCA) to compare flax fibres and E-glass fibres as the reinforcement for composites within an ISO 14040 framework. They are considering all eight environmental impact classification factors (EICF) identified by Azapagic [136, 137], ISO 14047 [138] and the European Environment Agency [139] (Table 6). The total energy required [140] using low energy agricultural processes was found to be 59.3GJ/tonne for flax sliver and 85.6GJ/tonne for yarn. Traditional mouldboard ploughing and bio retting was found to require 199GJ/tonne for sliver and 231GJ/tonne for yarn. Fibreglass and fibreglass reinforcement mats are reported to have embodied energies of 29.7 GJ/tonne [141] and 54.7 GJ/tonne [130] respectively! The analysis for the full set of EICF is on-going.

Table 6: A correlation of the eight environmental impact classification factors

Azapagic et al [136, 137]	ISO/TR 14047:2003(E) [138]	European Environment Agency [139]
Acidification Potential (AP)	Acidification	Acidification
Aquatic Toxicity Potential (ATP)	Ecotoxicity	Ecotoxicity
Eutrophication Potential (EP)	Eutrophication/Nitrification	Eutrophication
Global Warming Potential (GWP)	Climate change	Climate change and global warming
Human Toxicity Potential (HTP)	Human toxicity	Human toxicity
Non-Renewable/Abiotic Resource Depletion (NRADP)	Depletion of abiotic/biotic resources	
Ozone Depletion Potential (ODP)	Stratospheric ozone depletion	Stratospheric ozone depletion
Photochemical Oxidants Creation Potential (POCP)	Photo-oxidant formation	Photochemical ozone formation (summer smog)

SUMMARY

This review paper has considered the growth, harvesting and fibre separation techniques suitable to yield bast fibre of appropriate quality for use as the reinforcement of polymer-matrix composites. The text then addressed the characterisation of the fibre. Unlike man-made fibres, the cross section is neither circular nor uniform along the length which leads to increased complexity in the calculation of fibre mechanical properties. The choice of polymer matrix systems (both thermoplastic and thermoset) was covered. The review then considered the use of the basic rule-of-mixtures in the context of natural fibre reinforced composites and addressed the characterisation of composite materials using microscopical, mechanical, chemical and thermal techniques. The text closed with a brief overview of some potential applications and the environmental considerations which might expedite or constrain the adoption of these composites.

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