

1 INTRODUCTION

Composites originated as biomaterials employing plant fibres as reinforcements. References have been made to the use of linen and hemp textiles as reinforcements of ceramics as early as 6500 BC [1]. The Egyptians have also been known to use grass and straw as reinforcing fibres in mud and clay bricks for the building of walls over 3000 years ago [2]. While synthetic fibres, specifically E-glass, dominate today's FRP market [3], awareness of the scarcity of non-renewable resources and a demand for environmental sustainability have led to a renewed and ever-increasing interest in biocomposites. This is reflected by the increasing number of publications on biocomposites during recent years, including books [1, 2, 4-7] and review articles [8-21].

Plant fibres, such as flax, sisal and bamboo, offer several economical, technical and ecological advantages over synthetic fibres in reinforcing polymer composites (Table 1.1). The wide availability, low cost, low density, high specific properties and eco-friendly image of plant fibres has portrayed them as prospective substitutes to traditional composite reinforcements, specifically E-glass [8, 22-26]. As 87% of the 8.7 million tonne global FRP market is based on E-glass composites (GFRPs) [3], plant fibres and their composites have a great opportunity for market capture.

Although the use of plant fibres (non-wood and non-cotton) in reinforced plastics has tripled to 45,000 tonnes over the last decade [1, 11, 25], plant fibre composites (PFRPs) make up only ~1.9% of the 2.4 million tonne EU FRP market (Fig. 1.1) [25]. Notably, the use of carbon fibre composites, globally and in the EU, is lower than the use of biocomposites and on the same level as the use of PFRPs (Fig. 1.1) [3, 25]. It is of interest to note that while PFRPs were developed and are viewed as alternatives to GFRPs [8, 26], they have mainly replaced wood fibre reinforced thermosets in the EU automotive industry [27, 28]. Up to 30% of these PFRPs are based on thermoset matrices, while the rest are based on thermoplastic matrices (Fig. 1.1) [27].

Table 1.1. Comparison between plant and synthetic fibres [8, 23, 26, 29-31].

Properties		Plant Fibres ^a	Glass Fibres ^b	Carbon Fibres ^c
Economy	Annual global production [tonnes] ^d	31,000,000	4,000,000	55,000
	Distribution for FRPs in EU [tonnes] ^d	Moderate (~60,000)	Wide (600,000)	Low (15,000)
	Cost of raw fibre [£/kg]	Low (~0.5-1.5)	Low (~1.3-20.0)	High (>12.0)
Technical	Density [gcm⁻³]	Low (~1.35-1.55)	High (2.50-2.70)	Low (1.70-2.20)
	Tensile stiffness [GPa]	Moderate (~30-80)	Moderate (70-85)	High (150-500)
	Tensile strength [GPa]	Low (~0.4-1.5)	Moderate (2.0-3.7)	High (1.3-6.3)
	Tensile failure strain [%]	Low (~1.4-3.2)	High (2.5-5.3)	Low (0.3-2.2)
	Specific tensile stiffness [GPa/gcm⁻³]	Moderate (~20-60)	Low (27-34)	High (68-290)
	Specific tensile strength [GPa/gcm⁻³]	Moderate (~0.3-1.1)	Moderate (0.7-1.5)	High (0.6-3.7)
	Abrasive to machines	No	Yes	Yes
Ecological	Energy demand of raw fibre [MJ/kg]	Low (4-15) ^e	Moderate (30-50)	High (>130)
	Renewable source	Yes	No	No ^f
	Recyclable	Yes	Partly	Partly
	Biodegradable	Yes	No	No
	Hazardous/toxic (upon inhalation)	No	Yes	Yes

^a Includes bast, leaf and seed fibres, but does not include wood and grass/reed fibres.

^b Includes E- and S-glass fibres.

^c Includes PAN- and pitch-based carbon fibres.

^d Estimated values for the year 2010, from [32] for global fibre production values and from [24, 25, 27] for values on the distribution of fibres for FRPs in EU.

^e While the energy required in the cultivation of plant fibres is low (4-15 MJ/kg), further processing steps (*e.g.* retting and spinning) can significantly increase the cumulative energy demand, for instance, to up to 146 MJ/kg for flax yarn [10].

^f Carbon fibres based on cellulosic precursors currently account for only 1-2% of the total carbon fibre market [33].

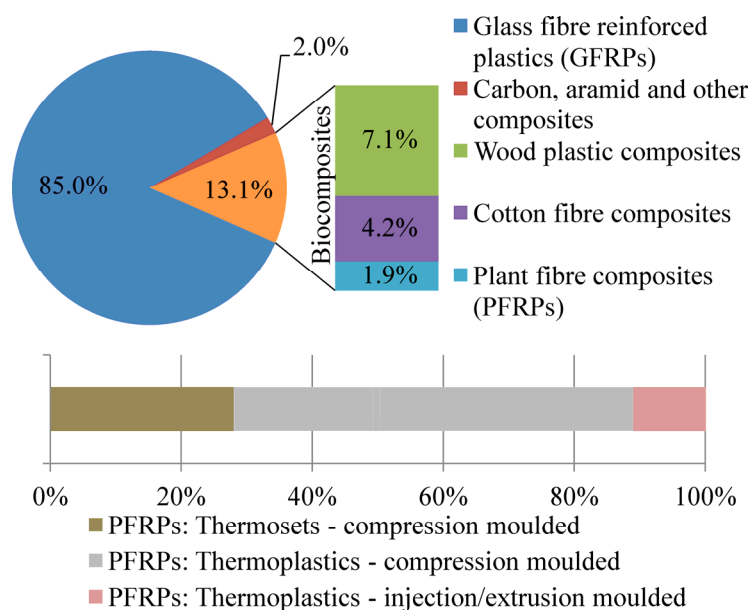


Fig. 1.1. PFRPs, primarily manufactured via compression moulding, account for only ~1.9% of the 2.4 million tonne EU FRP market in 2010 [25, 27].

By commercial application, over 95% of PFRPs produced in the EU are used for non-structural automotive components, which are manufactured primarily via compression moulding [10, 25, 27]. Other than automotive applications (for interior components such as door and instrumental panels) [1, 25, 31, 34], PFRPs are being considered for applications in:

- i)* construction and infrastructure (such as beams, roof panels, bridges) [1, 20, 21, 34-40],
- ii)* sports and leisure (for boat hulls, canoes, bicycle frames, tennis rackets) [1, 21, 27, 34, 36, 40],
- iii)* furniture and consumer goods (such as packaging, cases, urns, chairs, tables, helmets, ironing boards) [1, 21, 25, 27, 34, 36-40],
- iv)* pipes and tanks (for water drainage/transportation) [1, 14, 34, 35, 39-41], and
- v)* small-scale wind energy (as rotor blade materials) [42-46].

In many of these applications, plant fibres are being employed primarily as light, cheap and ‘green’ reinforcements, playing little or no structural role. Interestingly, this is different to what was envisaged in the mid-twentieth century, when the potential of plant fibres as structural reinforcing agents was acknowledged by

pioneers like Ford to manufacture the first ‘green car’ with an all-plastic-body using 70 wt% lignocellulosic fibres [47]. Ford was even able to demonstrate the strength and impact resistance of the material by famously taking a sledgehammer onto the car’s deck lid [47]. At the same time, Aero Research Ltd developed Gordon Aerolite, a flax/phenolic composite, to replace light-alloy sheets for building the structural members of Spitfire fuselages for British military aircrafts during the Second Great War [48]. With Britain facing potential shortages of aluminium, Gordon Aerolite was then the most promising material for aircraft [48]. Furthermore, the structural potential of plant fibres is revealed by the fact that bast fibres (like flax, hemp and jute) are high in cellulose content (~60-80% of the dry chemical composition [22]) and native cellulose has remarkable tensile stiffness (138 GPa) and strength (>2 GPa) [1, 49, 50]. Therefore, investigating and eventually promoting the potential use of plant fibres in load-bearing composite components, as a possible replacement to E-glass, is a natural step ahead.

1.1 DESIGNING PFRPs FOR STRUCTURAL APPLICATIONS

While structural composites are required to sustain external loads in addition to self-support (like the shear web of a wind turbine blade) or play a principal role in supporting the structure of the final component (like the airframe of an aircraft), non-structural composites are primarily for aesthetic purposes enduring minimal loads (like the interior panels of a car). Hence, the ‘make-up’, that is the design and construction, of structural and non-structural composites is different.

One of the many advantages of composite materials, in general, is the possibility of tailoring material properties to meet different requirements. It is well-known that the macro-mechanical behaviour of heterogeneous FRPs depends on many factors; including the stress-strain behaviour of each phase (that is, the fibre and the matrix), the volumetric composition, the geometrical structure and arrangement of the phases, and the interface properties [51].

While the EU automotive industry has principally focussed on three bast fibres, namely flax, jute and hemp, for PFRP production [10, 11, 25, 52], noting their regional availability, other fibres like sisal [13], bamboo [53], cotton [54], coir [55]

and even banana leaf [56] have been shown to be suitable for such non-structural applications. However, as bast fibres themselves play a structural role in a plant, their morphology and mechanical properties make them most suitable for load-bearing applications. Bast fibres are abundant [15], producible with low environmental footprint [5], and due to their textile applications are readily available in the forms of semi-finished products (yarns/rovings, mats and aligned fabrics) [5]. In addition, as the fibre extraction processes for bast fibres for composites applications have been the subject of several studies (such as [1, 57-59]), advancements have been made to produce fibres with high aspect ratios, fewer defects and better mechanical properties. Therefore, this study employs bast fibres for PFRP manufacture. Being of natural origin, the properties of plant fibres are variable and inconsistent. On the other hand, structural components need to have highly controlled properties. Hence, it is necessary to consider the effect of bast fibre type on PFRP mechanical properties and to specifically investigate whether employing such fibres in the forms of yarns and fabrics could enable the production of PFRPs with consistent quality.

Currently, both thermosets and thermoplastics are used with plant fibres [27]. However, there is a general trend, particularly in the automotive industry, of diminishing use of thermoset matrices and increased use of thermoplastic matrices [3, 25, 27]. This is primarily because the latter are faster to process, are fabricated by a cleaner process (dry systems with no toxic by-products), are easier to recycle, and are less expensive (for high volume production). However, thermosets are high-performance matrices (due to the formation of a large cross-linked rigid three-dimensional molecular structure upon curing), form a better interface with hydrophilic plant fibres, have low processing temperatures and have low viscosities allowing manufacture with liquid composite moulding (LCM) processes, which are more suitable for larger geometrically-intricate components (like wind turbine blades). It should be noted that in terms of end-of-life disposal, the use of thermosetting matrices, rather than thermoplastic matrices, does not necessarily lower the eco-performance of the PFRP produced. This is because the addition of plant fibres can significantly reduce the recyclability and reusability of a thermoplastic system [10, 15, 60]. All PFRPs can be incinerated for energy recovery

or re-used as fillers; the additional option with thermoplastic-based PFRPs is that they can also be granulated and re-processed into extrusion/injection moulded components [10]. Notably, thermoplastic-based PFRPs that are recycled by remoulding into new parts exhibit severely deteriorated mechanical properties due to repeated thermal exposure [60]. In fact, the ‘recyclability’ of PFRPs is an altogether different and unresolved issue.

Thermosets are selected as the matrix materials for this study. Firstly, the effect of matrix type on fibre/matrix compatibility and therefore PFRP mechanical properties needs to be studied. In addition, as porosity (particularly matrix-related) is an unavoidable part of composites produced via LCM processes, special attention must be paid to its effect on PFRP mechanical properties. The importance of studying porosity rises as PFRPs reinforced with twisted yarns are known to experience issues with impregnation and wettability [61, 62]. While Madsen *et al.* [63-65] have studied the effect of porosity on the properties of bast fibre compression moulded thermoplastics in detail, investigations on the effect of porosity on the properties of thermoset-based PFRPs are limited.

Today, for composites applications, plant fibres are typically used in the form of non-woven mats (for compression moulding) or granules/pellets (for injection/extrusion moulding) [15, 25, 27]. As the fibres are discontinuous (short ($< 3\text{-}30\text{ mm}$) [1, 15, 66] or even sub-critical length ($< 0.2\text{-}3\text{ mm}$) [1, 66-68]) and randomly oriented, the mechanical properties of the resulting non-structural composite are dominated by the polymer matrix, rather than the strong and stiff fibres [8, 62, 65]. According to Krenchel’s fibre orientation efficiency factor [69], employing randomly oriented fibres in two-dimension (non-woven mats) and three-dimension (granules/pellets) would reduce the reinforcing effect of the fibre (in terms of providing strength and stiffness) to 37.5% and 20.0% of its potential, respectively. Using short and sub-critical length fibres (with low aspect ratios) would slash the fibre length efficiency factor as well.

For load-bearing applications, the use of reinforcements in the form of continuous aligned fibres is essential as they preserve high efficiency factors (of length and

orientation), thus allowing the entire properties of the fibre to be exploited. As technical plant fibres are staple fibres with a discrete length, they need to be processed into yarns/rovings and then textile reinforcements; that is, a continuous product with highly controlled fibre orientation. As found by several researchers [65, 70, 71], including the author of this thesis, employing such plant fibre yarns/rovings for PFRP manufacture enables realising the true potential of plant fibres as reinforcing agents. However, specific considerations are necessary when utilising yarns for composites manufacture. Firstly, the conversion of plant stems to workable technical fibres, spun yarns and eventually fabrics introduces several degrees of defects, thus diminishing fibre mechanical properties [14, 22, 59]. Secondly, the intricate structure-property relations of a yarn have several direct implications on the resulting composites. In particular, the twist and compaction of the reinforcing yarn affect composite mechanical properties, resin impregnability, yarn permeability and wettability, and even void formation [61, 62]. These issues need to be systematically investigated for wide applicability of plant yarn reinforced composites.

The volumetric composition of a composite is known to have a significant and well-predicted effect on the composite properties. In many studies on PFRP mechanical properties, the volumetric composition of the composites is not well-characterised. While most researchers give estimates of fibre weight fraction, some state the fibre volume fraction assuming no porosity. While there are some well-documented studies on structure-property relationships in PFRPs, there have been no direct studies on determining the minimum, critical and maximum fibre volume fraction for PFRPs. In the automotive industry, random short fibre PFRPs are employed at fibre volume fractions ranging from 15 to 55% [25, 31]. Importantly, the critical fibre volume fraction, above which the reinforcing effect of the fibre is realised, for random short-fibre PFRPs is typically in excess of 25% and even up to 50% (interpreted from [72, 73]). In essence, the tensile strength of the matrix is higher than the tensile strength of the composite for many of these components. On the other hand, Madsen *et al.* [64, 74] have found that the maximum fibre volume fraction, above which poor impregnation and extensive void formation lead to reduced mechanical properties, is of the order of 50% for hemp yarn reinforced

composites but in the range of 33-46% for short random flax/jute reinforced composites. That is, the fibre volume fraction process window for PFRPs is much smaller than that for conventional FRPs. Hence, investigating structure-property relations in twisted yarn reinforced PFRPs for structural composites is imperative as it would enable identifying the range of fibre volume fractions that produce useful properties and provide models to predict the composite properties.

Generally, the measurement of uniaxial composite tensile properties is appropriate in analysing the reinforcing contribution of plant fibres. However, structural components may not only be subjected to uniaxial loads, but also to off-axis loads and even fatigue loads. Indeed, the mechanical behaviour of aligned PFRPs subjected to off-axis loads and cyclic loads has been only sparsely investigated. If PFRPs are to be seriously considered for structural applications, their response to off-axis loads and cyclic loads needs to be thoroughly investigated and documented. In addition, to predict the response of PFRPs exposed to such loads, models need to be developed.

Finally, while PFRPs are attractive for structural applications, studies have largely been based on lab-scale coupon testing and computational modelling. To date, there are only limited, if any, scientific studies that conclusively show the suitability of PFRPs over GFRPs for structural applications [75]. The performance of PFRPs in real full-scale structural applications needs to be ambitiously tested and analysed.

1.2 THESIS OBJECTIVES

The work described in this thesis has formed part of a Nottingham Innovative Manufacturing Research Centre (NIMRC) funded project entitled '*Sustainable manufacture of small wind turbine blades using natural fibre composites and optimal design tools*'.

The overall objectives of this thesis are to *i)* characterise, *ii)* optimise, and *iii)* achieve an improved understanding of, the mechanical properties of PFRPs for structural applications. Furthermore, using composite rotor blades as a case study, the question is addressed whether PFRPs are potential alternatives to GFRPs for structural applications.

Bast fibres (flax, hemp and jute) are selected as suitable reinforcements for this study, due to their mechanical properties and ready availability. To develop composites for load-bearing applications, this study concerns PFRPs based on continuous aligned reinforcements (unidirectional and multi-axial), fabricated from yarns/rovings, embedded in a thermoset matrix. A liquid composite moulding (LCM) process is employed for composite manufacture.

Components like small wind turbine (SWT) blades are subjected to various loading situations over their design life, including static and fatigue loading. Hence, the first logical step for the application of PFRPs in structural applications would be to experimentally investigate and characterise these several mechanical properties (particularly, static tensile and fatigue properties). Once the necessary benchmark is set, efforts can be made to optimise these properties by investigating the effects of a range of relevant composite parameters such as *i*) plant fibre yarn type and quality, *ii*) thermoset matrix type, *iii*) volumetric composition (fibre, matrix and void content), *iv*) reinforcing yarn structure, and *v*) textile architecture (ply orientation). This methodology would not only facilitate an improved understanding of the mechanical behaviour of PFRPs, but would also provide a systematic solution in developing simple mathematical and/or micro-mechanical models for predicting their properties. Eventually, the optimised material technologies would be applied in the manufacture of full-scale structural components and the developed predictive models could be employed to demonstrate the components' structural integrity.

1.3 THESIS OUTLINE

This thesis presents different aspects of the potential of plant fibres as reinforcements in structural polymer composites. The thesis consists of 8 chapters. This chapter, *Chapter 1*, gives a general introduction to the subject, in addition to the objectives and outline of the thesis. In providing a relevant background for the work described in this thesis, *Chapter 2* contains a general literature survey on technical plant fibres and their composites with useful mechanical properties for structural applications.

Chapters 3-6 form the central part of the thesis, with each chapter having a short introduction and literature review concerning the specific issue. In *Chapter 3*, the

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mechanical properties of plant yarn reinforced unidirectional thermoset matrix composites are studied and compared to that of GFRPs. Attention is also paid to the effect of plant fibre/yarn type and matrix type on the properties of the composites. In *Chapter 4*, structure-property relationships in plant yarn composites are investigated, specifically to determine the range of fibre volume fractions (that is, minimum, critical and maximum fibre content) that produce PFRPs with useful tensile properties. *Chapter 5* details the effect of orientation, in the form of *i*) reinforcing yarn twist and *ii*) ply orientation, on the tensile properties of flax yarn reinforced composites, with a focus on mathematical modelling of the experimental data. Furthermore, attention is paid to the non-linear tensile stress-strain response of plant fibres and their composites. *Chapter 6* evaluates the fatigue performance of various aligned PFRPs through lifetime (S-N) diagrams, comparing them to aligned GFRPs. The effect of *i*) plant fibre type/quality, *ii*) fibre content, *iii*) textile architecture, and *iv*) stress ratio, on PFRP cyclic loading behaviour is investigated. Constant life diagrams are produced to enable the fatigue life prediction of PFRP components subjected to cyclic loads.

To apply the results from *Chapters 3-6* (gathered through coupon testing) and demonstrate the potential of PFRPs for full-scale structural applications, *Chapter 7* details a case study on the structural integrity (static and fatigue) of a 3.5-meter SWT blade built from flax/polyester. A comparison of the manufacturing and mechanical properties of the flax blade and an identical E-glass blade is presented. Finally, *Chapter 8* presents the main conclusions and highlights topics for future work.

1.4 LIST OF PUBLICATIONS AND AWARDS

Several publications have arisen from the work described in this thesis. The dissemination has been in the form of peer-reviewed journal papers, conference papers and presentations, and through public engagement. A critical review article, derived from the literature survey in *Chapters 1 and 2*, and original research articles, based on *Chapters 4-7*, have been published in peer-reviewed journals. The novel research has also attracted numerous awards.

Peer-Reviewed Journal Papers

1. **Shah DU**, Schubel PJ, Licence P, Clifford MJ. Hydroxyethylcellulose surface treatment of natural fibres: the new ‘twist’ in yarn preparation and optimization for composites applicability. *Journal of Materials Science*, 2012, 47(6): p. 2700-2711.
2. **Shah DU**, Schubel PJ, Licence P, Clifford MJ. Determining the minimum, critical and maximum fibre content for twisted yarn reinforced plant fibre composites. *Composites Science and Technology*, 2012, 72: p. 1909-1917.
3. **Shah DU**, Schubel PJ, Clifford MJ. Modelling the effect of yarn twist on the tensile strength of unidirectional plant fibre yarn composites. *Journal of Composite Materials*, 2012, 47(4): p. 425-436.
4. **Shah DU**, Schubel PJ, Clifford MJ, Licence P. The tensile behaviour of off-axis loaded plant fibre composites: an insight on the non-linear stress-strain response. *Polymer Composites*, 2012, 33(9): p. 1494-1504.
5. **Shah DU**, Schubel PJ, Clifford MJ, Licence P. Fatigue life evaluation of aligned plant fibre composites through S-N curves and constant-life diagrams. *Composites Science and Technology*, 2013, 74: p. 139-149.
6. **Shah DU**, Schubel PJ, Clifford MJ. Can flax replace E-glass in structural composites? A small wind turbine blade case study. *Composites Part B: Engineering*, 2013, 52: p. 172-181.
7. **Shah DU**. Developing plant fibre composites for structural applications by optimising composite parameters: a critical review. *Journal of Materials Science*, 2013, 48 (18): p. 6083-6107.

Conference Papers/Presentations

1. **Shah DU**, Schubel PJ, Clifford MJ, Licence P, Warrior NA. Mechanical characterization of vacuum infused thermoset matrix composites reinforced with aligned hydroxyethylcellulose sized plant bast fibre yarns, in *4th International Conference on Sustainable Materials, Polymers and Composites (ECOCOMP 2011)*. 6-7 July 2011. Birmingham, UK.
2. **Shah DU**, Schubel PJ, Clifford MJ, Licence P, Warrior NA. Yarn optimisation and plant fibre surface treatment using hydroxyethylcellulose for the development of structural bio-based composites, in *18th International Conference on Composite Materials (ICCM-18)*. 21-27 August 2011. Jeju, South Korea.
3. **Shah DU**, Schubel PJ, Clifford MJ, Licence P. Fatigue characterisation of plant fibre composites for small-scale wind turbine blade applications, in *SAMPE*

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Europe Student Conference 2012 (in SAMPE Europe 33rd International Conference SEICO 12). 24-25 March 2012. Paris, France.

4. **Shah DU**, Schubel PJ, Clifford MJ, Licence P. Fatigue characterisation of plant fibre composites for small-scale wind turbine blade applications, in *5th Innovative Composites Summit (in JEC Asia 2012)*. 26-28 June 2012. Singapore.
5. **Shah DU**, Schubel PJ. Can flax replace E-glass in structural composites? A small wind turbine blade case study, in *5th International Conference on Sustainable Materials, Polymers and Composites (ECOCOMP 2013)*. 3-4 July 2013. Birmingham, UK.

Other Articles

1. **Shah DU**, Schubel PJ, Clifford MJ, Licence P. Fatigue characterisation of plant fibre composites for rotor blade applications, in *JEC Composites Magazine*. June 2012, No. 73: Special JEC Asia, p. 51-54.
2. **Shah DU**, Schubel PJ. Can flax replace E-glass in small wind turbine blades?, in *JEC Composites Magazine*, Jan-Feb 2013, No. 78: Feature Wind Energy, p. 29-33.

Awards

1. **Jan 2012: ‘Composites Sustainability Award’** at SAMPE/BCS/IOM3 Annual UK Student Seminar, London (UK).
2. **Mar 2012: ‘JEC Sustainable Solutions Award’** at SAMPE Europe Student Conference, Paris (France).
3. **May 2012: ‘Research Impact Prize’** at University of Nottingham Faculty Postgraduate Research Event, Nottingham (UK).
4. **June 2013: ‘JEC Asia 2013 Innovation Award’** in the Biocomposites category, at JEC Asia 2013, Singapore (Singapore). This award is for research and development on the first natural fibre reinforced composite small wind turbine blade.

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