

4 EFFECT OF FIBRE VOLUME FRACTION ON THE TENSILE PROPERTIES OF PLANT YARN REINFORCED COMPOSITES^{*}

4.1 INTRODUCTION

Fibre reinforced plastics (FRPs) are heterogeneous materials. They consist of reinforcing fibres embedded in a continuous matrix. While the fibres provide strength and stiffness to the composite, the matrix transmits externally applied loads to the fibres and protects the fibres from external damage. As described in *Chapter 2*, the properties of a composite material are generally a weighted linear combination of the properties of the fibre and the matrix; the fibre volume fraction being the critical parameter. In fact, micro-mechanical models, backed by experimental data, show that several composite properties including density, Poisson's ratio, stiffness (tensile, compressive, shear), and strength (tensile, compressive, shear, impact), increase proportionally with fibre volume fraction [1]. However, these composite properties are limited by the minimum, critical and maximum fibre volume fraction. Hence, knowledge of the processing window (in terms of range of fibre volume fractions) which produces composites with useful properties is essential.

Importantly, fibre and matrix volume fractions are not the only volumetric components of a composite. Porosity, defined as air-filled cavities, is often an unavoidable part in all composites. They develop during the mixing and consolidation of the fibre and the matrix, for instance during the injection of resin in a RTM process. Porosity is known to have highly detrimental effects on composite properties [1-4]. To reduce porosity in composites, several innovative techniques have been developed [1, 5]. Nonetheless, knowledge of the void content is essential for the reliable prediction of composite properties using micro-mechanical models.

^{*} This chapter is based on the peer-reviewed journal article:

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(15): p. 1909-1917.

4.1.1 Structure-property relationships in PFRPs

Renewable bio-based composite materials provide an exciting opportunity to develop sustainable materials. Plant fibres in particular are an attractive source of reinforcement for FRPs. As a result, they have been subjected to several characterisation and development studies. However, in many studies on plant fibre composite (PFRP) mechanical properties, the volumetric composition of the composites is not well-characterised [6]. While most researchers present the fibre weight fraction, some state the fibre volume fraction assuming no porosity. This is due to issues with the measurement of fibre and void volume fraction in PFRPs. Conventional methods, such as resin burn-off and acid/chemical digestion, prove unsuccessful with PFRPs, as the plant fibres degrade and get consumed alongside the resin upon high temperature exposure and chemical attack [7-9].

There are, however, some well-documented studies on structure-property relationships of PFRPs; be it for randomly-oriented short-fibre reinforcements [10-15], randomly-oriented long-fibre reinforcements [16], uniaxially-oriented long-fibre (slivers) reinforcements [17-20] or aligned staple fibre yarn reinforcements [6, 18, 21-25]. Most of these studies employ density measurement methods (described in [7]) to determine the volumetric composition of the composites. The aims of these studies have been to *i*) characterise the composite properties over a range of fibre volume fractions, *ii*) compare the results with predictive micro-mechanical models (such as the rule of mixtures or Halpin-Tsai equations) and *iii*) compare the performance with E-glass composites.

However, there have been no direct studies on determining the minimum $v_{f,min}$ and critical $v_{f,crit}$ fibre volume fraction for PFRPs. More recently, Sawpan *et al.* [26] did attempt to calculate the minimum fibre volume fraction $v_{f,min}$ for short-fibre hemp/polyester composites, but they could not verify this experimentally as their characterisation study was based on fibre weight fractions (neglecting porosity). The maximum obtainable fibre volume fraction for PFRPs has also not been studied; where a maximum fibre volume fraction has been quoted [6, 17, 21], it has been based on composite processing limitations and tensile test data. For aligned PFRPs, twisted plant fibre staple yarns are the readily available and widely used form of

continuous reinforcement. Such staple fibre yarns themselves have a fibre volume fraction (referred to as the packing fraction ϕ by textile engineers [27]). Hence, the use of such twisted yarn reinforcements has an (unfavourable) effect on the theoretical (geometrically-permissible) maximum fibre volume fraction $v_{f,max,theo}$, which needs to be investigated. Furthermore, the influence of increasing fibre volume fraction on porosity is disputed and needs more insight; while some reports suggest an increase in porosity with fibre content [6, 16, 23], others suggest no correlation [12, 17, 22].

The purpose of this chapter is to analyse the relationship between structure and properties of aligned PFRPs. Specifically, the effect of fibre volume fraction on PFRP physical properties (porosity and fibre packing arrangement) and tensile properties is discussed. Parameters such as minimum, critical and maximum obtainable fibre volume fraction are also determined to identify the range of fibre volume fractions that produce twisted yarn reinforced PFRPs with useful properties.

4.1.2 Theory: Minimum and critical fibre volume fraction

In composite theory [1], for brittle-fibres and a ductile-matrix the strength-fibre content relationship is well-understood (Fig. 4.1). If there are very few fibres present ($0 < v_f < v_{f,min}$), the stress on a composite may be high enough to break the fibres. The broken fibres, which carry no load, can be then regarded as an array of aligned holes. The net effect is that the composite tensile strength σ_c is even below that of the matrix σ_m . This defines a minimum fibre volume fraction $v_{f,min}$ below which the fibres weaken the material rather than strengthen it and composite failure is controlled by the matrix. The reinforcing action of the fibres is only observed once the fibre volume fraction exceeds the critical fibre volume fraction ($v_f > v_{f,crit}$). This is mathematically presented in Eq. 4.1 and diagrammatically illustrated in Fig. 4.1. Note that σ'_m is the matrix stress at the fibre failure strain ϵ_f (schematic in Fig. 4.2). Eq. 4.2 and Eq. 4.3 mathematically define the minimum $v_{f,min}$ and critical $v_{f,crit}$ fibre volume fractions, respectively.

$$\sigma_c = \begin{cases} \sigma_m(1 - v_f) & \text{for } 0 < v_f < v_{f,min} \\ \sigma_f v_f + \sigma'_m(1 - v_f) & \text{for } v_f > v_{f,min} \end{cases} \quad \text{Eq. 4.1}$$

$$v_{f,min} = \frac{\sigma_m - \sigma'_m}{\sigma_f + (\sigma_m - \sigma'_m)} \quad \text{Eq. 4.2}$$

$$v_{f,crit} = \frac{\sigma_m - \sigma'_m}{\sigma_f - \sigma'_m} \quad \text{Eq. 4.3}$$

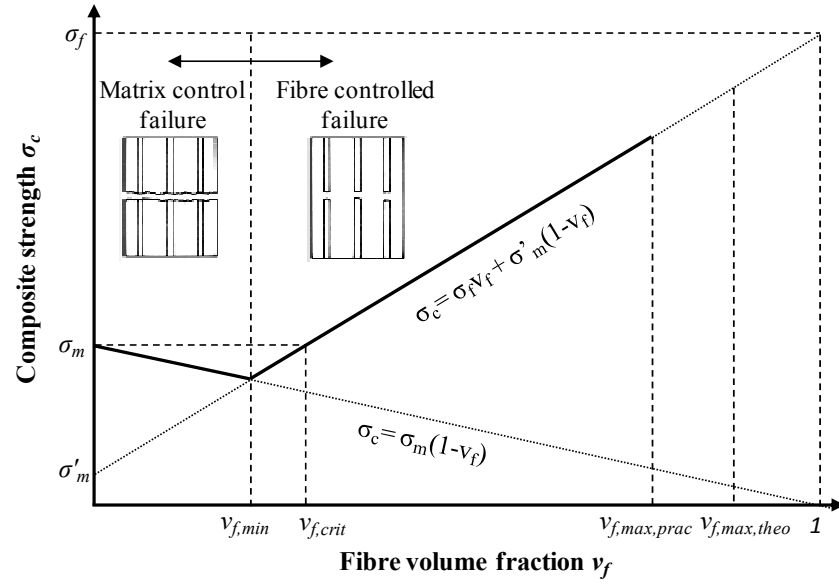


Fig. 4.1. Schematic illustration of the variation of the strength of a unidirectional (brittle-fibre ductile-matrix) composite with fibre volume fraction.

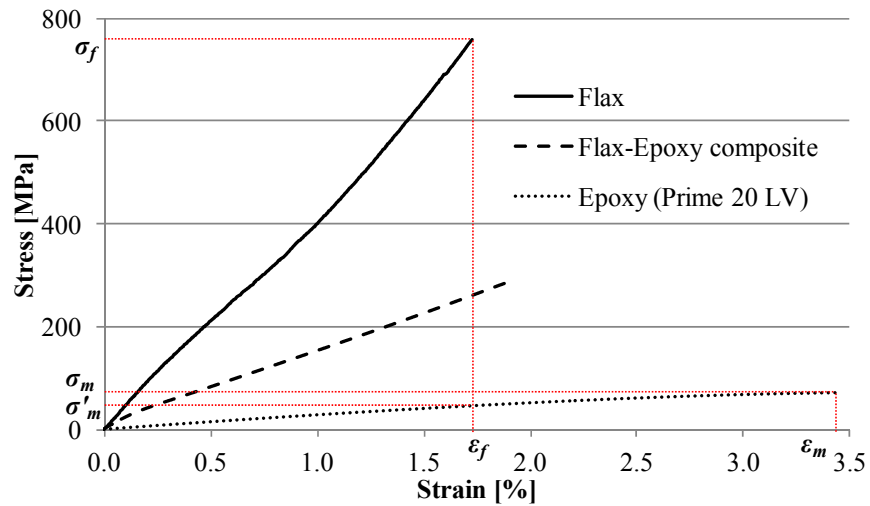


Fig. 4.2. Plant fibre thermoset composites are a brittle-fibre ductile-matrix system.

A thermoset bast fibre reinforced composite is also a brittle-fibre ductile-matrix system, where the fibre failure strain ε_f is lower than the matrix failure strain ε_m (Fig. 4.2). Hence, if plant fibres are to be used as reinforcements for structural composites, knowing the minimum and critical fibre volume fraction is paramount as the PFRP would be designed for $v_f > v_{f,crit}$. There is only one study (by Ghosh *et al.* [11]) which implicitly illustrates the minimum and critical fibre volume fractions (neglecting porosity) for short banana leaf fibre reinforced vinyl-ester composites to be $v_{f,min} \approx 15\%$ and $v_{f,crit} \approx 25\%$. Interpreting the results of Sawpan *et al.* [26], it is found that short fibre hemp/polyester composites have a minimum and critical fibre weight fraction of $\sim 20\%$ and $>30\%$ (up to 60%), respectively. While the automotive industry is by far the biggest consumer of PFRPs [28], the plant fibre reinforcements are short, randomly-oriented and employed at fibre volume fractions ranging from 15 to 55% (fibre weight fractions of 20-65%) [28, 29]. This implies that the tensile strength of the matrix is higher than the tensile strength of the composite for many of these PFRP components. In addition, the values of minimum and critical fibre volume fractions for PFRPs, found from Ghosh *et al.* [11], Sawpan *et al.* [26] and this study, are substantially higher than those of conventional FRPs; an aligned carbon/polyester composite would have $v_{f,min} = 2.3\%$ and $v_{f,crit} = 2.4\%$ [1].

4.1.3 Theory: Maximum achievable fibre volume fraction

Several studies (for instance [15, 17, 21, 30, 31]) have concluded that the generalised rule of mixtures (Eq. 2.1 and Eq. 2.2) is valid for PFRPs. As the fibre content exceeds $v_{f,crit}$, the strength of the composite increases proportionally (as in Fig. 4.1). However, there is a ‘practical’ maximum fibre content $v_{f,max,prac}$ above which composite properties deteriorate [12, 17], often due to a drastic increase in porosity [6, 16, 21] or increased fibre-fibre interactions [32, 33]. Madsen *et al.* [21] found that when aligned hemp/polypropylene laminates were fabricated at a nominal fibre volume content of 61%, the actual measured fibre volume content was only 51% with a larger porosity content of 17%. In essence, impregnation and wettability issues arise close to this maximum fibre volume fraction. Pan [34] has also suggested (according to Cox [35]) that at high fibre volume fractions, fibre-to-fibre spacing becomes so small that the stress transfer between fibre and matrix becomes

inefficient eventually causing premature failure due to increased shear stresses on all planes parallel to the axes of the fibres. The resulting delamination has been observed in jute/polyester composites at high fibre content [17].

The experimentally determined optimal (or practical) maximum fibre volume fractions $v_{f,max,prac}$ for PFRPs range from about 60% for aligned jute roving reinforced polyester [17], 46-54% for aligned hemp yarn reinforced polyethyleneterephthalate (PET) [6, 21], and between 33% and 46% for short random flax and jute reinforced polypropylene [6]. For synthetic fibre composites, $v_{f,max,prac}$ is much higher at 75-80%.

On the other hand, the theoretical maximum fibre volume fraction $v_{f,max,FRP}$ of a fibre reinforced composite is a function of fibre packing geometry. This is higher than the practical maximum fibre volume fraction $v_{f,max,prac}$. Based on ideal fibre packing geometry, quadratic arrangement of the fibres leads to a $v_{f,max,FRP}$ of $\pi/4$ (= 78.5%) while hexagonally-packed fibres generate a higher $v_{f,max,FRP}$ of $\pi/2\sqrt{3}$ (= 90.7%) [34]. Usefully, a more accurate value of the theoretical maximum fibre volume fraction $v_{f,max,FRP}$ can be determined through compaction studies, in which the evolution of fibre volume fraction with compaction pressure is studied. Generally, a power-law function (of the form $v_f = aP^b$, where P is compaction pressure and a and b are material constants) is a suitable fit for the compaction curves [25, 36]. As P increases, after a steep rise in v_f , the fibre volume fraction plateaus. The asymptotic value of fibre volume fraction can be regarded as $v_{f,max,FRP}$.

Madsen [25] and Xu *et al.* [36] have conducted such studies on plant and glass fibre preforms. Through such compaction studies, it is found that $v_{f,max,FRP}$ is different for synthetic fibre reinforced plastics and plant fibre reinforced plastics. This is because the packing ability of plant fibre assemblies is lower than that of synthetic fibre assemblies [21, 25, 37, 38]. It is suggested that fibre alignment and the degree of fibre separation affects the compact-ability of a preform. This was discussed previously in *Chapter 3.3.1*.

Importantly, for PFRPs that are reinforced with staple plant fibre yarns, the twist (and packing fraction) of the yarn would also affect preform compaction. The

packing fraction of a high twist yarn is absolute and will not usually change upon compaction (during composite processing) due to the transverse pressure in a yarn induced by the twisting process. However, if the yarn twist level (and thus the packing fraction) is very low, due to negligible transverse pressure in the yarn the yarn may be compacted further. Roe *et al.* [17] were able to produce higher fibre volume fractions in their jute/polyester composites (of up to 60%) as they were using slivers; these are compressible and thus the distance between fibres within the slivers can be reduced.

If the composite manufacturing technique relies on preform consolidation through press-moulding (*i.e.* application of positive pressure), as is typical in compression moulding, $v_{f,max,FRP}$ of preforms produced with higher twist yarns would be lower as the yarns will not spread upon compaction. Lower twist yarns, on the other hand, will spread out (like tows) under the application of pressure and will thus leave less resin rich zones and produce higher $v_{f,max,FRP}$.

The composite manufacturing utilised for studies in this thesis, however, is vacuum infusion in a rigid all-aluminium mould tool. The chosen manufacturing technique does not enable the utilization of a hydraulic press, for instance, to compact the preform. In such a manufacturing technique, the theoretical maximum fibre volume fraction of the plant fibre preform is dependent on the nominal packing fraction of the yarn. In fact, the theoretical maximum fibre content $v_{f,max,theo}$ would be a linear combination of the yarn packing geometry within a composite and fibre packing arrangement within the yarn ϕ (Eq. 4.4).

$$v_{f,max,theo} = v_{f,max,FRP} \cdot \phi \quad \text{Eq. 4.4}$$

The maximum packing fraction of a yarn ϕ_{max} is 75.0% assuming an open-packed structure [39] or 90.7% assuming hexagonal close-packed fibre arrangement [27, 40]. Hence, the absolute limit of the maximum fibre volume fraction of a PFRP reinforced with twisted yarns ranges from 58.9% to 82.2%, depending on the combination of packing assumed in the composite and the yarn. The lower value of this absolute limit (*i.e.* 58.9%) is comparable to the experimental values of $v_{f,max,prac}$ observed in literature (quoted previously).

Importantly, the yarn packing fraction ϕ is a function of the yarn twist level [41]. Pan [41] derived a semi-empirical relationship (Eq. 4.5) between twist level T (turns per meter or tpm) and packing fraction ϕ of such staple fibre yarns. In Eq. 4.5, ϕ_{max} is the maximum packing fraction of the yarn, and A and B are constants. While Pan [41] used ϕ_{max} , A and B as 0.7, 0.78 and 0.195, respectively, he suggested that these factors could be modified to suit other particular yarns. Conventional staple fibre ring-spun yarns have a packing fraction ϕ of 50-60% [42].

$$\phi = \phi_{max} (1 - Ae^{-BT}) \quad \text{Eq. 4.5}$$

Substituting Eq. 4.5 into Eq. 4.4 for ϕ results in a mathematical model (Eq. 4.6) for determining the maximum obtainable fibre volume fraction $v_{f,max,theo}$ in PFRPs reinforced with twisted staple plant fibre yarns, produced via vacuum infusion in a rigid mould (without the capacity to compact through application of pressure). The result of Eq. 4.6 is graphically presented in Fig. 4.3 for various combinations of parameter values ($v_{f,max,FRP}$, ϕ_{max} , A and B). Experimental data, based on calculated packing fractions ϕ of plant fibre yarns with twist level T have also been plotted. The experimental data is for yarns that have been used for PFRP manufacture. The data is from *Chapter 3 and Appendix A*. From Fig. 4.3, it is found that the packing fraction ϕ of staple fibre yarns used for PFRPs (particularly those used for the study described in this thesis), is well described by Eq. 4.5 with the factors ϕ_{max} , A and B of 0.6, 0.78 and 0.0195, respectively.

$$v_{f,max,theo} = v_{f,max,FRP} \cdot \phi_{max} (1 - Ae^{-BT}) \quad \text{Eq. 4.6}$$

In general, the results in Fig. 4.3 show that increasing yarn twist would lead to a composite with higher fibre volume fractions, before levelling off at a maximum obtainable volume fraction. This is in agreement with the study by Baets *et al.* [18] which reports that epoxy composites reinforced with flax slivers (0 tpm), rovings (41 tpm) and yarns (280 tpm), had fibre volume fractions of 42%, 48% and 50%, respectively. It is important to highlight here again that the above conclusion relates to the use of a manufacturing technique that doesn't enable preform compaction. Using composite manufacturing techniques which enable the application of

consolidation pressure (such as compression moulding and prepregging with autoclave consolidation) can enable the production of high fibre volume fraction composites if lower twist yarns (rovings/slivers) are used, due to their compactability and spread-ability.

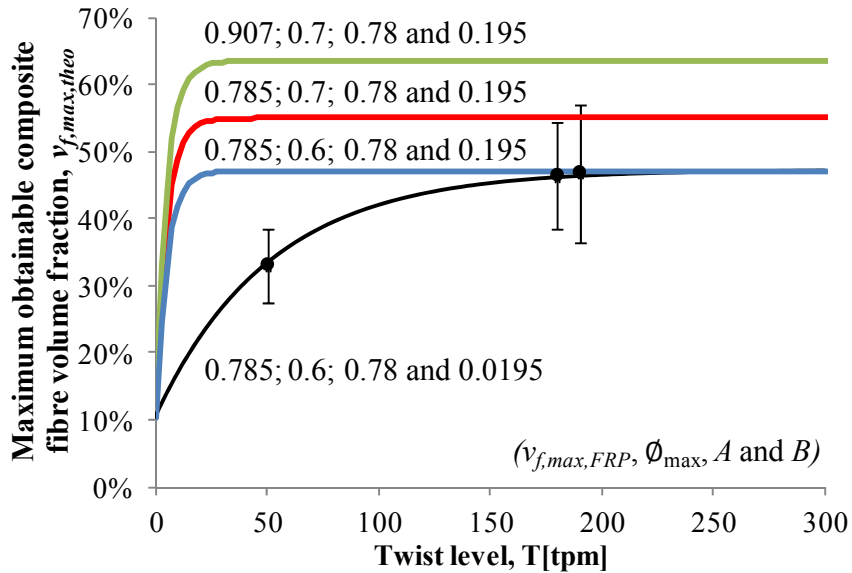


Fig. 4.3. The effect of yarn twist level on the maximum obtainable fibre volume fraction $v_{f,max,theo}$ for PFRPs reinforced with such twisted yarns. Experimental data is from *Chapter 3 and Appendix A*. Refer to Eq. 4.6 and text for details.

While the minimum and critical fibre volume fraction set the lower limit of effective reinforcing fibre volume fraction, the maximum fibre volume fraction sets the upper limit. These limits determine the fibre content design envelope for structural PFRPs employing twisted yarn reinforcements.

4.2 EXPERIMENTAL METHODOLOGY

4.2.1 Materials and composite manufacture

Unidirectional mats were prepared from two commercially available plant fibre yarns: a low twist (50 tpm) flax yarn from Composites Evolution (UK) and a high twist (190 tpm) jute yarn from Janata and Sadat Jute Ltd (Bangladesh). The flax yarn employs polyester as a binder yarn (13 wt% of yarn). These are the same yarns (F50 and J190) used for the study in *Chapter 3*. The aligned mats were prepared using a

drum-winding system and hydroxyethylcellulose binding agent (described in *Chapter 3*).

Unidirectional composite laminates (250 mm square 3-3.5 mm thick) of five different fibre volume fractions were fabricated. To generate different fibre volume fractions, an increasing number of unidirectional mat layers were used (*i.e.* 1, 2, 3, 4 and 5 layers). The reinforcement mats were used as-produced (without any preconditioning). While composite manufacturing processes like hot-pressing and compression moulding can produce much higher fibre content PFRPs, vacuum infusion is the chosen technique in this study as it readily enables the manufacture of large components, such as wind turbine blades. Vacuum infusion was carried out in an all-aluminium mould tool. Resin infusion was carried out at 70-80% vacuum (200-300 mbar absolute pressure). Line-gate resin injection was employed with the flow direction being perpendicular to the yarn axis. The composite manufacturing process has been described in detail in *Chapter 3*.

An unsaturated polyester (Reichhold Norpol type 420-100) matrix was used. The resin was mixed with 0.25 wt% NL49P accelerator (1% Cobalt solution) and 1 wt% Butanox M50 MEKP initiator. Post cure was carried out at 55 °C for 6 h after ambient cure for 16 h. From manufacturer datasheet, the resin has a cured density ρ_m of 1.202 gcm⁻³, tensile modulus E_m of 3.7 GPa, tensile strength σ_m of 70 MPa and failure strain ε_m of 3.5%.

4.2.2 Physical characterisation

The fibre weight fraction w_f of a laminate was calculated using the ratio of the mass of the preform and the resulting composite laminate. Composite and fibre density were determined using helium pycnometry (minimum of 5 samples). The fibre volume fraction v_f , matrix volume fraction v_m and void volume fraction v_p of the manufactured composites were then determined using equation Eq. 4.7, where w and ρ represent weight fraction and density, respectively while the subscripts f , m and c denote fibres, matrix and composite, respectively.

$$v_f = \frac{\rho_c}{\rho_f} w_f; \quad v_m = \frac{\rho_c}{\rho_m} (1 - w_f); \quad v_p = 1 - (v_f + v_m) \quad \text{Eq. 4.7}$$

Optical microscopy was also used to qualitatively image the fibre/yarn packing arrangement and porosity in the composites. Sample preparation has been previously discussed in *Chapter 3*.

4.2.3 Tensile testing

Tensile tests were conducted according to ISO 527-4:1997 using an Instron 5985 testing machine equipped with a 100 kN load cell and an extensometer. At least six 250 mm long and 15 mm wide specimens were tested for each type of composite at a cross-head speed of 2 mm/min. The ultimate tensile strength σ_c , tensile modulus E_c (in the strain range of 0.025-0.100%) and the strain at failure ε_c of the specimen were measured from the stress-strain curve. The fracture surfaces were also observed under a Philips XL30 Scanning Electron Microscope (SEM) at an acceleration voltage of 15 kV. The samples were sputter-coated with platinum.

4.3 RESULTS AND DISCUSSION

4.3.1 Volumetric composition

Flax and jute/polyester unidirectional composites have been produced with 5 different fibre volume fractions, by simply increasing the number of layers of unidirectional mats. The density of the composites is observed to increase with fibre volume fraction; the composite density approaches the density of the flax and jute fibres of $1.529 \pm 0.003 \text{ gcm}^{-3}$ and $1.433 \pm 0.005 \text{ gcm}^{-3}$, respectively. However, a drop in density is observed for the jute/polyester composites at $v_f = 31.7\%$ due to a relatively higher void content. The volumetric composition of fibre, matrix and void within the composites is tabulated in Table 4.1. Note the consistency in fibre/matrix volume fractions (indicated by the small standard deviations) for the composites; Madsen *et al.* [43] also observed such small variations in fibre volume fraction for their hemp and flax yarn reinforced composites.

Also note the low void content (typically in the range of 0.3-1.4%) of the PFRPs produced. Although the void content seems to be higher for greater fibre content, there is no clear correlation between composite fibre volume fraction and porosity (Fig. 4.4). Very low linear regression R^2 -values of 0.126 and 0.272 are obtained for

void content as a function of fibre content for flax and jute composites, respectively. This is in agreement with references [12, 17, 22] but disagreement with references [6, 16, 23].

Table 4.1. Density and volumetric composition (mean $\pm$ stdev.) of the fabricated laminates.

Fibre (# of layers)	Composite density ρ_c [gcm ⁻³]	Fibre volume fraction v_f [%]	Matrix volume fraction v_p [%]	Void volume fraction v_v [%]
Flax (5)	1.301 $\pm$ 0.009	32.5 $\pm$ 0.2	66.9 $\pm$ 0.5	0.6 $\pm$ 0.7
Flax (4)	1.282 $\pm$ 0.002	27.3 $\pm$ 0.1	72.0 $\pm$ 0.1	0.7 $\pm$ 0.2
Flax (3)	1.264 $\pm$ 0.001	24.0 $\pm$ 0.1	74.6 $\pm$ 0.0	1.4 $\pm$ 0.1
Flax (2)	1.245 $\pm$ 0.001	17.8 $\pm$ 0.0	80.9 $\pm$ 0.1	1.2 $\pm$ 0.1
Flax (1)	1.220 $\pm$ 0.001	6.1 $\pm$ 0.0	93.8 $\pm$ 0.1	0.1 $\pm$ 0.1
Jute (5)	1.276 $\pm$ 0.002	37.8 $\pm$ 0.1	61.1 $\pm$ 0.1	1.1 $\pm$ 0.2
Jute (4)	1.225 $\pm$ 0.002	31.7 $\pm$ 0.1	64.1 $\pm$ 0.1	4.2 $\pm$ 0.1
Jute (3)	1.251 $\pm$ 0.004	25.2 $\pm$ 0.1	74.1 $\pm$ 0.2	0.7 $\pm$ 0.3
Jute (2)	1.238 $\pm$ 0.003	17.1 $\pm$ 0.1	82.6 $\pm$ 0.2	0.3 $\pm$ 0.2
Jute (1)	1.215 $\pm$ 0.002	7.6 $\pm$ 0.0	92.0 $\pm$ 0.2	0.4 $\pm$ 0.2

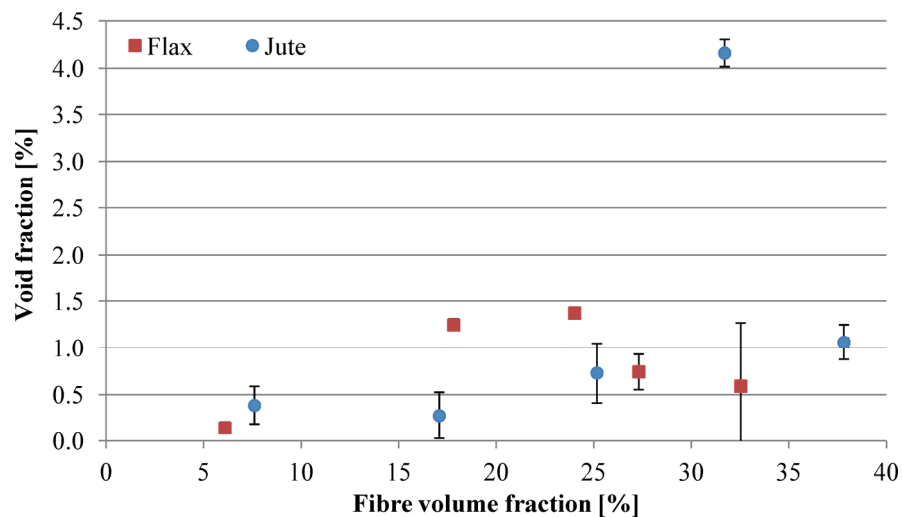


Fig. 4.4. There is no clear correlation between fibre content and void content.

The volumetric composition and the presence of voids in composites of different fibre volume fractions can be visually observed from the microscopic images in Fig. 4.5. It is observed that for low fibre volume fractions (up to 2 layers) voids generally form within the yarn bundle (intra-yarn voids). Increasing the fibre volume fraction further results in the formation of voids between adjacent yarns (inter-yarn voids), rather than within the yarn. This is possibly due to the changing resin flow dynamics with fibre content. At low fibre content, impregnation within the yarn is difficult as high overall permeability and low yarn permeability leads to fast infusion elsewhere and slow infusion within the yarn. Essentially, the resin moves faster in the channels than in the yarn; the ‘outrun’ produces intra-yarn voids (Fig. 4.6a). However, at high fibre content the yarns are much closer to each other and the overall permeability is comparable to the yarn permeability. However, capillary pressure is larger within the yarn. Hence, flow is faster through the yarn so that voids are formed between yarns, *i.e.* inter-yarn voids (Fig. 4.6b). In essence, fibre content does not have an obvious effect on void content, but it does influence the type of voids.

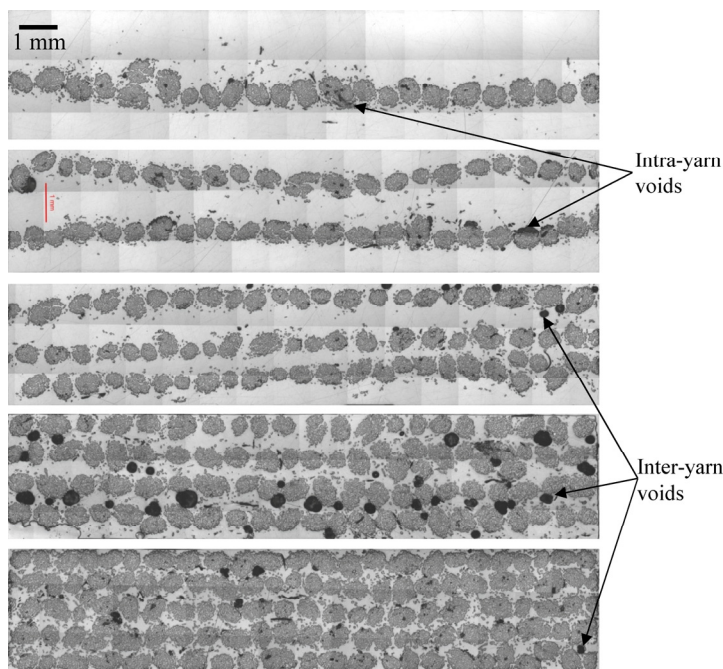


Fig. 4.5. Microscopic images of the cross-section of jute/polyester composites showing the composition and fibre/yarn packing arrangement for increasing fibre content.

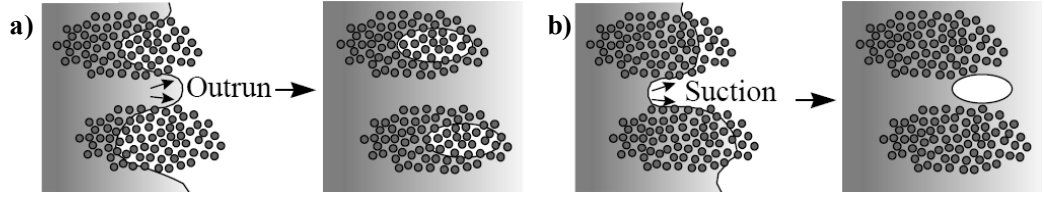


Fig. 4.6. The packing arrangement and fibre volume fraction affect the type of void formed. *a)* At low fibre content, due to low yarn permeability but high overall permeability, the yarn is not properly impregnated and thus intra-yarn voids are formed. *b)* at high fibre content, although yarn and overall permeability are similar, capillary flow in the yarn dominates and therefore inter-yarn voids are formed.

4.3.2 Maximum fibre volume fraction

The yarns used in this study are flax (50 tpm) and jute (190 tpm). The experimentally known packing fractions ϕ are 0.421 and 0.596 for the flax and jute yarn, respectively (from *Chapter 3*). From Fig. 4.5, it can be seen that the yarns follow a square packing arrangement hence $v_{f,max,FRP}$ is taken to be $\pi/4$ ($= 78.5\%$). Using Eq. 4.4, the derived maximum obtainable fibre volume fraction $v_{f,max,theo}$ is 33.1% for the flax composites and 46.8% for jute composites. The bottom-most image in Fig. 4.5 is of jute/polyester with $v_f = 37.8\%$ (5 layers). The yarns seem well-packed within the composite cross-section and thus a theoretical maximum fibre content $v_{f,max,theo}$ of 46.8% for the jute composites is realistic. The order of the values of the theoretical maximum fibre content $v_{f,max,theo}$ is similar to the order of values of the practical maximum fibre content $v_{f,max,prac}$ reported in literature (discussed in Section 4.1.3).

4.3.3 Tensile properties

The tensile stress-strain curves reveal the general changes in tensile properties of the composite for increasing fibre content. Fig. 4.7 presents stress-strain curves of representative specimens for jute/polyester composites of different fibre volume fractions. The curves are shifted upwards when fibre volume fraction increases, suggesting that the elastic modulus and tensile strength increase. It is also observed that the failure strain increases at first and then becomes fairly constant.

It is interesting to note from the composite stress-strain curves in Fig. 4.7 that for all fibre volume fractions PFRPs show a non-linear response. In fact, increasing fibre

content exaggerates the non-linear response. The stress-strain curve of a single plant fibre is also found to be non-linear (Fig. 4.2). This is different from synthetic fibres and their FRPs (particularly E-glass) whose stress-strain behaviour is entirely linear. The non-linear stress-strain response of plant fibres and their composites is discussed in detail in *Chapter 5*.

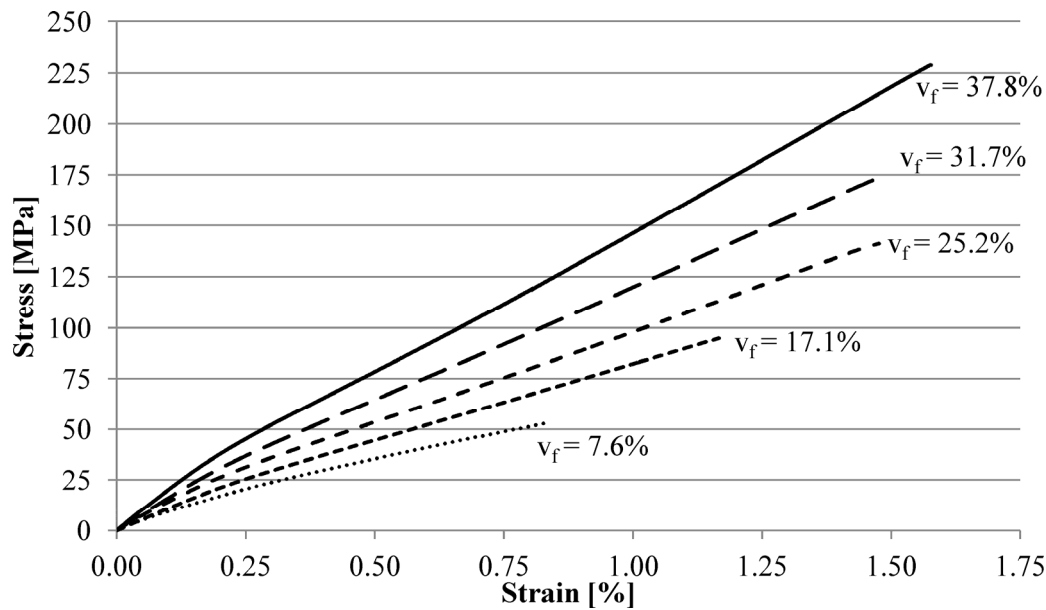


Fig. 4.7. Typical stress-strain curves of jute/polyester with variable fibre volume fraction.

The fracture surfaces of the tensile test specimen (Fig. 4.8) also give insight into the reinforcing effect of the plant fibres at different fibre volume fractions. It can be seen in Fig. 4.8 that for the first two specimen (up to $v_f \approx 18\%$), for both flax and jute composites, tensile fracture is macroscopically brittle with a flat fracture surface. The composite failure seems to be matrix-controlled. Little microscopic pull-out of the fibres is noticed in the SEM images (Fig. 4.9). This is also a sign of low impact strength, as fibre pull-out is more energy dissipative than fibre fracture [44].

Increasing the fibre content produces a more serrated and uneven fracture surface, as can be seen in Fig. 4.8. The composite failure is fibre-controlled. SEM images in Fig. 4.9 show that the fibre pull-out length is also increased implying an increase in toughness. For jute/polyester composites in particular, the fracture path becomes

longer and starts running along the length of the fibres/yarns. It can be seen in Fig. 4.8 that at high fibre volume fractions, delamination between adjacent yarns and layers occurs. It is interesting that no sign of delamination is noticed in the flax/polyester composites. This is possibly due to the difference in structure of the yarn (specifically, twist level).

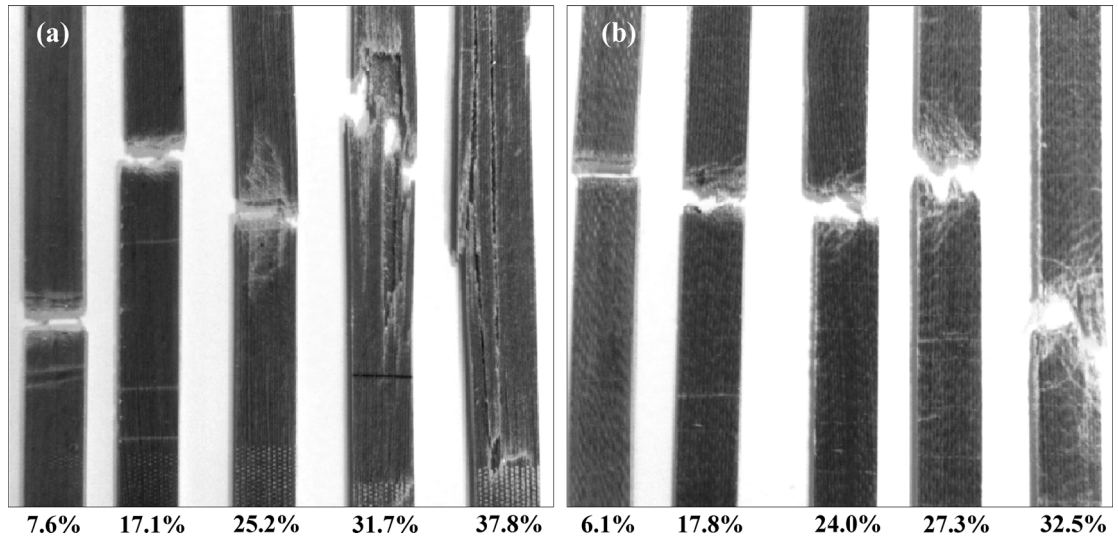


Fig. 4.8. The effect of fibre volume fraction of *a)* jute and *b)* flax on the fracture of tensile specimen. Increasing fibre content (left to right) produces a more serrated fracture surface and even delamination.

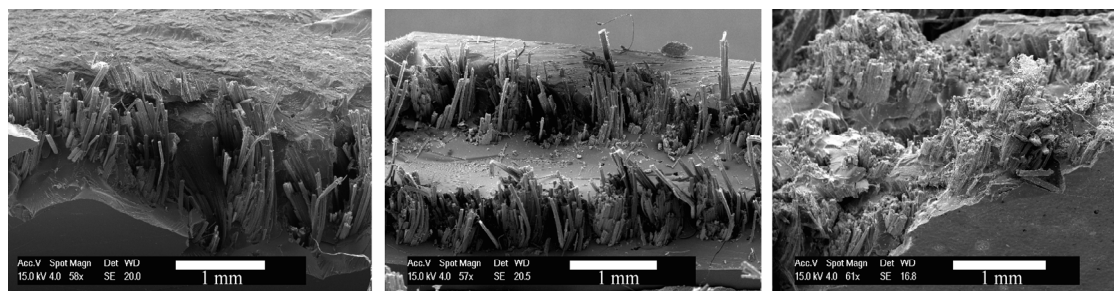


Fig. 4.9. SEM images of fracture surfaces of jute/polyester composites showing increasing fibre pull-out and serrated surface for increasing fibre content. From left to right: 1 layer, 2 layers and 4 layers of unidirectional reinforcement.

4.3.3.1 Tensile modulus

The variation of the tensile modulus with fibre volume fraction (Fig. 4.10) of flax ($R^2 = 0.993$) and jute ($R^2 = 0.992$) composites demonstrates that the rule of mixtures (Eq.

4.8) is followed closely. This is in agreement with several other studies (such as [13, 17, 19-21, 30, 31]).

$$E_c = \eta v_f E_f + (1 - v_f) E_m \quad \text{Eq. 4.8}$$

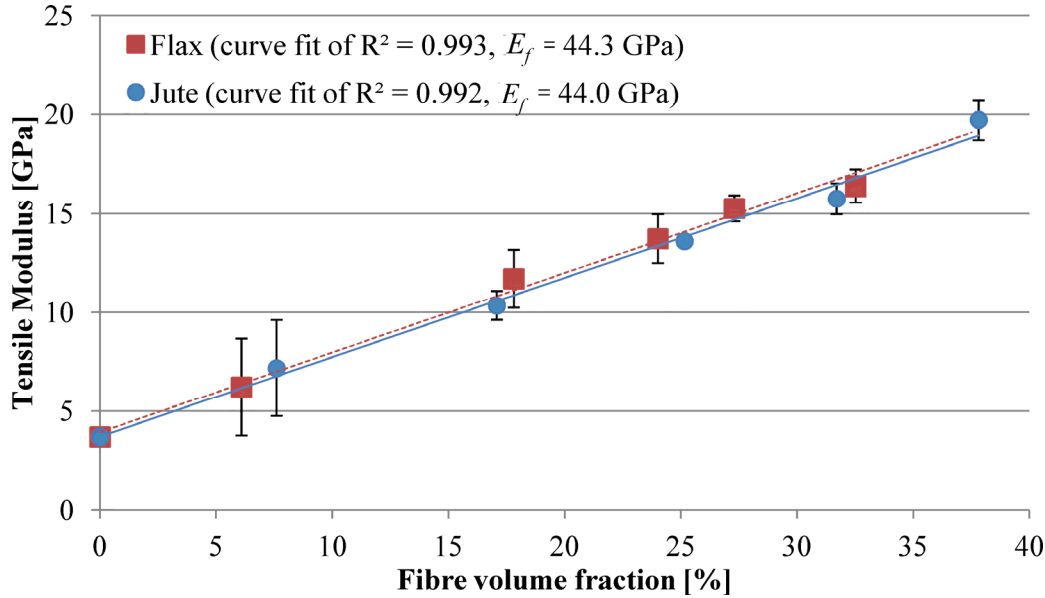


Fig. 4.10. Variation of tensile modulus with fibre volume fraction.

The back-calculated effective fibre modulus ηE_f for flax and jute is thus obtained as 44.3 GPa and 44.0 GPa, respectively. This is in the range of literature values [45, 46] generally quoted for flax and jute, although flax can achieve a much higher tensile modulus (of over 70 GPa), as shown in *Chapter 3*. Note that this effective fibre modulus ηE_f incorporates any effect of the various efficiency and correction factors in the modified rule of mixtures for PFRPs discussed in detail in *Chapter 2.4* (Eq. 2.3 and Eq. 2.4): *i)* fibre length efficiency, *ii)* fibre orientation distribution, *iii)* fibre diameter distribution, *iv)* fibre cross-sectional area correction factor and *v)* porosity correction factor. Note that it has been shown in *Chapter 3* that the length efficiency factor is almost unity for such yarn reinforced PFRPs.

As the theoretical maximum fibre volume fraction of flax and jute composites is known, the maximum theoretical tensile modulus achievable can be determined. This is found to be 17.3 GPa for flax/polyester (at $v_{f,max,theo} = 33.1\%$) and 22.6 GPa for

jute/polyester (at $v_{f,max,theo} = 46.8\%$). This compares to a tensile modulus of 33.7 GPa for E-glass/polyester (at $v_f = 44.0\%$) (measured in *Chapter 3*). Note that E-glass/polyester has a much higher $v_{f,max,theo}$ ($= v_{f,max,FRP}$), hence it can deliver much higher properties. The low $v_{f,max,theo}$ of PFRPs, in comparison to conventional FRPs, is a therefore a significant disadvantage. However, it should be noted that due to the low density of PFRPs, the high specific stiffness properties of PFRPs still make them attractive materials. Particularly, if high-quality plant fibre reinforcements (like F20) are used.

A note should be made here regarding the effect of porosity on the tensile modulus. The void content for flax and jute composites ranges between 0.1% and 1.4%, with no obvious increase with fibre content. Interestingly, despite the relatively high void content ($v_p = 4.2\%$) of jute/polyester with $v_f = 31.7\%$, no apparent drop in the elastic modulus (or tensile strength) is noticed (considering the standard deviation), despite a drop in density. Madsen *et al.* [6] show that for plant fibre thermoplastic composites, the effect of porosity on material stiffness is approximated by a multiplication factor of $(1 - v_p)^2$. In essence, a void content of 4.2% should reduce the potential composite stiffness (represented by the rule of mixtures) by 8.2%. An extensive study on the effect of void content on mechanical properties of E-glass thermoplastic composites was conducted by Gil [3]; it is observed that a void content of 4% would reduce the composite tensile strength or stiffness by 10-30%. However, Santulli *et al.* [2] suggest that no obvious reduction in mechanical properties is observed for void content below 3-4% for such E-glass thermoplastics. It is proposed that the same may be true for PFRPs. Reviewing the results of Madsen *et al.* [21] it is found that for hemp-PET composites, for up to 3.2% void content (at 40% v_f) there is negligible effect (considering the standard deviations) of void content on composite tensile strength and stiffness. Only at 50% fibre content, the void content jumps to 11.6% and is observed to reduce the stiffness and strength significantly. In essence, void content of up to 4% has minimal effect on PFRP properties.

4.3.3.2 Tensile strength

Fig. 4.11 shows the experimental data of tensile strength as a function of fibre content. The characteristic brittle-fibre ductile-matrix variation in composite tensile strength as a function of fibre volume fraction is noticed (as previously illustrated in Fig. 4.1). Again, good agreement with the rule of mixtures (Eq. 4.1) is noticed.

The back-calculated effective fibre tensile strength σ_f for flax and jute is obtained as 502.7 MPa and 615.2 MPa, respectively. This is in the range of literature values [45, 46] generally quoted for flax and jute, although flax can achieve a much higher tensile strength (of about 1100 MPa). Again, this effective fibre strength incorporates any effect of length and orientation efficiency factors. No drop in tensile strength is observed for jute/polyester with $v_f = 31.7\%$ with a relatively high void content ($v_v = 4.2\%$).

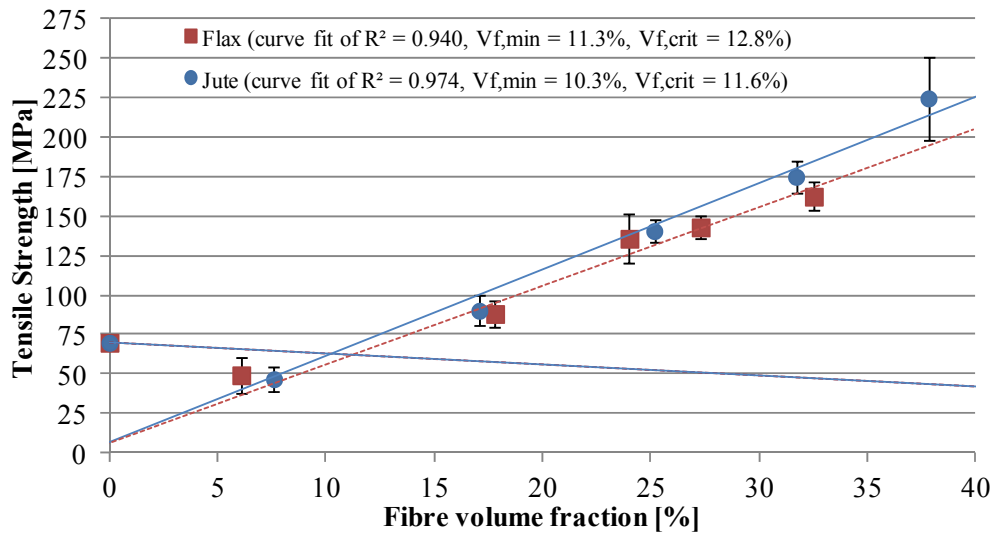


Fig. 4.11. Variation of tensile strength with fibre volume fraction.

The minimum and critical fibre volume fractions can also be determined from Fig. 4.11 and using Eq. 4.1-4.3, using $\sigma'_m = 6.2$ MPa (obtained from the curve fit of data points with $v_f > 15\%$). The minimum and critical fibre volume fractions are found to be $v_{f,min} = 11.3\%$ and $v_{f,crit} = 12.8\%$ for flax/polyester composites and $v_{f,min} = 10.3\%$ and $v_{f,crit} = 11.6\%$ for jute/polyester composites. Hence, for the design of useful

aligned PFRPs, where plant fibre twisted yarns are reinforcing the matrix, the fibre volume fraction needs to be in excess of ~10%.

Ghosh *et al.* [11] implicitly illustrate the minimum and critical fibre volume fractions for short banana fibre reinforced vinyl-ester composites to be $v_{f,min} \approx 15\%$ and $v_{f,crit} \approx 25\%$. The significantly higher minimum and critical fibre volume fraction of short fibre PFRPs compared to twisted yarn reinforced PFRPs is the direct result of higher critical load transfer lengths in short fibre PFRPs due to lower interfacial shear strength and lower fibre aspect ratios.

Notably, the minimum and critical fibre volume fractions for PFRPs are substantially larger than those observed in conventional unidirectional FRPs. For an aligned carbon-polyester composite [1], $v_{f,min} = 2.3\%$ and $v_{f,crit} = 2.4\%$. In addition, while the difference in $v_{f,min}$ and $v_{f,crit}$ for a carbon-polyester composite is only 0.1%, it is ~1% for twisted yarn reinforced PFRPs and ~10% for short fibre PFRPs [11]. σ'_m (or in fact $(\sigma_m - \sigma'_m)$) relates to the work-hardening efficiency of the matrix for the fibre reinforcement. A low σ'_m (and thus a high $(\sigma_m - \sigma'_m)$) correctly implies a higher minimum and critical fibre volume fraction (from Eq. 4.2 and Eq. 4.3). σ'_m , which defines the matrix stress at fibre failure strain, is about 22 MPa for the carbon-polyester system, but only 6.2 MPa for both the flax/polyester and the jute/polyester systems. From Ghosh *et al.* [11], it is observed that σ'_m is even lower at 5 MPa for short banana leaf fibre reinforced vinyl-ester composites. Interestingly, σ'_m is often estimated to be about 40-50 MPa (using $\sigma'_m = E_m \epsilon_f$) for a matrix reinforced with plant fibres [18, 21, 47]; this is clearly a gross over-estimation.

As the theoretical maximum fibre volume fraction of flax and jute composites is known, the maximum theoretical tensile strength can be determined. This is found to be 170.6 MPa for flax/polyester (at $v_{f,max} = 33.1\%$) and 263.1 MPa for jute/polyester (at $v_{f,max} = 46.8\%$). This compares to a tensile strength of 825.7 MPa for an E-glass/polyester (at $v_f = 44.0\%$) (measured in *Chapter 3*). The poor mechanical strength performance of PFRPs, in comparison to E-glass/polyester, is particularly due to *i)* the comparatively poor mechanical strength of plant fibres, and *ii)* the lower (achievable) fibre volume fractions of PFRPs.

It seems that vacuum-infused PFRPs utilising staple fibre twisted yarns have a small window of fibre volume fractions which produce useful composites. A high $v_{f,crit}$ (on the order of 10%), low $v_{f,max,prac}$ (on the order of 45-55%) [6, 21] and low $v_{f,max,theo}$ (on the order of 45-60%) implies that the possible range of employable fibre volume fractions for such PFRPs is only 35-50%. Importantly, short-fibre randomly-oriented PFRPs have a much higher $v_{f,crit}$ (on the order of 25%) and lower $v_{f,max,prac}$ (on the order of 30-45%) [6] implying that the useable range of fibre volume fractions is even lower (5-20%). This significantly limits the maximum exploitation of the mechanical properties of plant fibres in FRPs.

4.3.3.3 Strain at failure

The failure strain is observed to increase with increasing fibre volume fraction before levelling off to a value of about 1.62% for flax composites and 1.47% for jute composites (Fig. 4.12). The strain value corresponds to the effective strain at tensile failure of the fibres. This behaviour is similar to that observed in the literatures [17, 21].

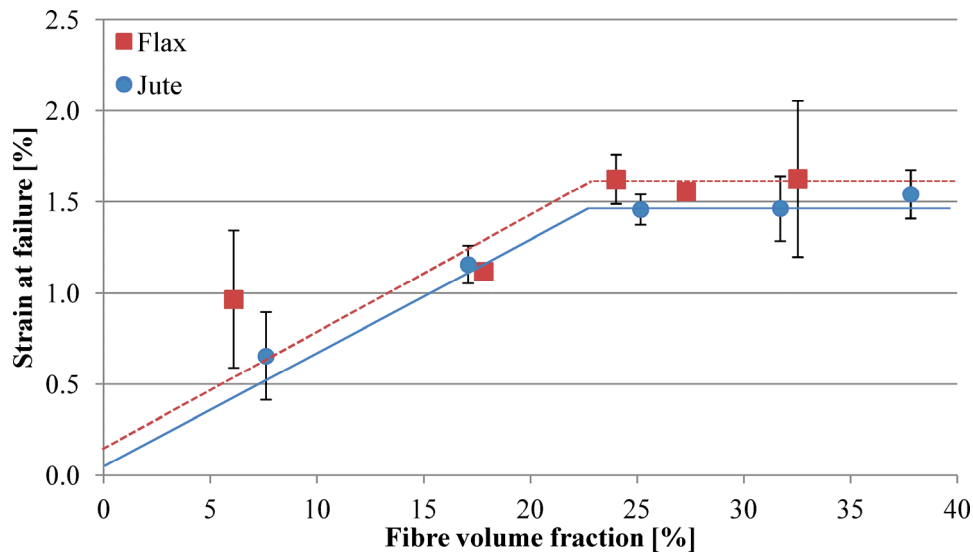


Fig. 4.12. Variation of tensile failure strain with fibre volume fraction.

4.4 CONCLUSIONS

The effect of fibre volume fraction on the physical and tensile properties of aligned PFRPs has been investigated. Yarn reinforced PFRPs are producible with low local variations in fibre/matrix volume fractions and low void content (typically in the range of 0.3-1.4%). There is no clear correlation between fibre volume fraction and porosity content. However, low fibre content PFRPs are prone to intra-yarn voids, while high fibre content PFRPs are prone to inter-yarn voids. This is possibly due to changing resin flow dynamics with increasing fibre volume fraction. Importantly, a void content of up to 4% is found to have minimal effect on the tensile properties of PFRPs.

The effect of fibre content on PFRP tensile properties is found to closely follow the rule of mixtures, similar to that of conventional FRPs. At low fibre content matrix-dominated brittle fracture occurs; increasing fibre content makes the fracture surface serrated and increases the occurrence and length of fibre pull-out.

A simple model has also been developed to approximate the theoretical maximum obtainable fibre volume fraction $v_{f,max,theo}$ of PFRPs reinforced with staple fibre yarns. The model is a linear combination of the yarn packing arrangement within the composite and the fibre packing arrangement within the yarn. The absolute limit of the theoretical maximum fibre volume fraction of a PFRP reinforced with twisted yarns ranges from 58.9% to 82.2%. The lower value of this absolute limit (*i.e.* 58.9%) is comparable to the experimental values of practical maximum fibre volume fraction $v_{f,max,prac}$ observed in literature. However, $v_{f,max,theo}$ for typical yarn reinforced PFRPs is in the range of 35-50%.

PFRPs utilising staple fibre twisted yarns have a small window of fibre volume fractions which produce composites with useful properties. A high $v_{f,crit}$ (on the order of 10%), low $v_{f,max,prac}$ (on the order of 45-55%) and low $v_{f,max,theo}$ (on the order of 45-60%) implies that the possible range of employable fibre volume fractions for such PFRPs is only 35-50%. Importantly, short-fibre randomly-oriented PFRPs have a much higher $v_{f,crit}$ (on the order of 25%) and lower $v_{f,max,prac}$ (on the order of 30-45%) implying that the useable range of fibre volume fractions is even lower (5-20%). This

significantly limits the maximum exploitation of the mechanical properties of plant fibres in FRPs. In comparison, aligned synthetic fibre reinforced composites have a lower $v_{f,crit}$ (2.4% for carbon/polyester) and much a higher $v_{f,max,prac}$ and $v_{f,max,theo}$ (on the order of 75-80%), implying that the range of fibre volume fractions that produce composites with useful properties is 70-75%.

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Chapter 4

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