Fibre Orientation of Novel Dynamically Sheet Formed Discontinuous Natural Fibre PLA Composites

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ABSTRACT

This paper describes work carried out to fabricate and to assess the fibre orientation in PLA reinforced by aligned discontinuous harakeke and hemp fibre mats produced using a dynamic sheet former (DSF). These mats were combined with PLA sheets to make composites with fibre contents of 5 – 40 wt% using a hot press. It was found that the fibre orientation factors (K_o) for both harakeke and hemp fibre composites were higher than those values seen in the literature for composites prepared using injection moulding and hot pressed using randomly oriented fibre mats, but slightly lower than the highest values obtained with aligned fibre nonwoven preform composites utilising more processing stages. The highest K_o values for harakeke and hemp fibres in this work were found to be 0.58 and 0.44 respectively. K_o decreased, reflecting increased fibre misalignment as fibre content increased, believed to be due to fibre agglomeration and the higher pressure required during processing.

KEYWORDS:

A: Discontinuous reinforcement; C: Analytical modelling; D: Mechanical testing; E: Compression moulding

1. INTRODUCTION

The mechanical properties of composites are generally governed by the mechanical properties of their constituents, fibre orientation and the fibre/matrix interface. Fibre orientation, which can be subdivided into three categories; aligned, partially aligned and randomly aligned, is generally represented using a fibre orientation factor (K_o), according to the degree of fibre alignment relative to the loading direction. Composites with fibres aligned parallel to the loading direction are found to have the greatest composite strength and stiffness as a result of effective stress transfer between fibre and matrix. Higher K_o also allows higher fibre volume fraction to be obtained [1]. Natural fibre composites with a high degree of fibre alignment can be produced using carding, where long fibres are carded and combined with polymer prior to compression moulding. A high degree of fibre alignment can also be obtained using continuous fibre yarn, which is normally produced by fibre spinning or wrapping fibres together. Fibre yarns can be made from the same type of fibre, a mixture of different fibres, or can be a mixture of fibres and thermoplastic filaments [2]. Production of these forms of fibres, however, are often time consuming and limited to certain types of fibres. Some degree of fibre alignment can be obtained during injection moulding, where discontinuous fibres are generally added to the matrix in a loose form during extrusion prior to injection moulding, dependent on matrix viscosity, mould design and size, length and fibre concentration [3-5]. The least fibre orientation occurs in randomly aligned fibre mat made using hot pressing with polymeric powder or sheet. Composites with partially and random fibre alignment made using injection and compression moulding have greater ease of fabrication and often require less time to

produce, but their strength and stiffness are much lower due to poor fibre alignment. The interfacial shear strength (τ) between fibres and matrix determines the extent of stress transferred to fibres. A strong interface provides composites that display good strength and stiffness but tend to be brittle, whereas a weaker interface reduces the stress transfer from the matrix to the fibre, hence the composite displays lower strength and stiffness, but could be higher in composite toughness. It is acknowledged that due to its hydrophilic nature, cellulose based natural fibre [6-8] has limited interaction with hydrophobic matrices commonly resulting in limited interfacial strength.

K_o and τ are difficult to measure experimentally for fibres with irregularly shaped fibre cross-sections, particularly with fibre aggregation (found in most natural fibre composites). Alternatively, the Bowyer-Bader model, based on a modification of the Kelly-Tyson model can be used [9, 10]. The Bowyer-Bader model defines composite strength in terms of strength contribution from fibres of different length and the matrix, but if composite strength and fibre length distribution are known, K_o as well as τ can be calculated. This model has been used to determine K_o and τ for composites reinforced with discontinuous fibre prepared mostly using injection moulding and found to give good approximations relative to the experimental values.

The main objective of this paper was to evaluate the fibre orientation factors (K_o) using the Bowyer-Bader model and compare with K_o values obtained for other discontinuous fibre composites. K_o values obtained here can also be used to better predict the composite strength and stiffness using theoretical modelling equations compared to commonly assumed values of K_o [11-16]. In this work, composites were reinforced with aligned discontinuous alkali treated harakeke and hemp fibre mats giving fibre contents for both

harakeke and hemp from 5 to 40 wt%, fibre orientation within mats was improved using a dynamic sheet former (DSF). τ values obtained using the same model are also reported.

2. THEORETICAL MODELLING - BOWYER - BADER MODEL

As previously mentioned, the Bowyer and Bader predicts the tensile strength of discontinuous fibre composites of varying fibre alignment by considering the sum of the contributions of different fibre lengths, as well as that of the matrix [9, 10]. The Bowyer-Bader model can be expressed as follows:

$$\sigma_c = K_\theta(X + Y) + Z \quad (1)$$

where σ_c is the composite strength, Z the is strength contribution of the matrix, X and Y are the strength contributions of fibres with subcritical and supercritical fibre lengths respectively, relative to a critical fibre length determined using the following equation:

$$L_c = \frac{\sigma_f D}{2\tau} \quad (2)$$

such that, L_c is the fibre length required for the maximum stress in the fibre to reach the fibre fracture stress (σ_f), i.e. fibre strength, and D is the average diameter of the fibre. As previously mentioned, this model can also be used to estimate fibre orientation factor (K_θ) and interfacial shear strength (τ) if the composite strength and fibre length distribution are known. This model is found to give a good approximation relative to the values determined experimentally [10, 12, 17-20]. For the calculation of K_θ and τ, subcritical and supercritical fibre lengths are defined as the length of fibre shorter or longer relative to the critical fibre length at any particular strain (L_ε), as given by [9]:

$$L_\varepsilon = \frac{E_f \varepsilon_c D}{2\tau} \quad (3)$$

such that, E_f is the Young's modulus of the fibre, L_ε is the fibre length, where, at particular strain, the fibre stress reaches a maximum only at the centre of the fibre. The average stress

in the fibres of length equal to or shorter than L_ε at a particular strain according to the Bowyer-Bader model is given by:

$$\sigma_{f\varepsilon} = \frac{L\tau}{D} \tag{4}$$

and for the fibres longer than L_ε is given by:

$$\sigma_{f\varepsilon} = E_f \varepsilon_c \left(1 - \frac{E_f \varepsilon_c D}{4L\tau}\right) \tag{5}$$

giving the overall composite stress at any strain level as:

$$\sigma_\varepsilon = K_\theta \left[\sum_i^{L_i<L_\varepsilon} \frac{\tau L_i V_i}{D} + \sum_j^{L_j>L_\varepsilon} E_f \varepsilon_c V_j \left(1 - \frac{E_f \varepsilon_c D}{4\tau L_j}\right)\right] + E_m \varepsilon_c (1 - V_f) \tag{6}$$

where E_m is the Young's modulus of the matrix, σ_ε is the composite stress at a particular strain, V_i and V_j are the volume fractions of the subcritical and supercritical fibre lengths, respectively, and L_i and L_j are the subcritical and supercritical fibre lengths, respectively, in reference to L_ε. In order to determine K_o and τ, the following steps were employed. Firstly, values of two strains, ε_{c1} and ε_{c2} were selected from tensile stress-strain curve and the corresponding composite stresses σ_{c1} and σ_{c2} were determined. Strength contributions of the matrix (Z) from a tensile test of neat PLA were also determined at the same strain levels. These parameters (σ_{c1}, σ_{c2} and Z) were then used to calculate R, the ratio of the fibre load bearing contribution using the following equation:

$$R = \frac{\sigma_{c1} - Z_1}{\sigma_{c2} - Z_2} \tag{7}$$

Then, an assumed value of τ was taken and the corresponding values of $L_{\varepsilon 1}$ and $L_{\varepsilon 2}$ calculated using Equation (3). Accordingly, the fibre contribution terms X_1, Y_1 and X_2, Y_2 were calculated from X and Y (Equation (6)) for the different strain levels respectively, and used to calculate R', the theoretical value of R using the following equation:

$$R' = \frac{X_1 + Y_1}{X_2 + Y_2} \quad (8)$$

The assumed value of τ was adjusted until R'=R and finally used in Equation (6) to obtain a value of K_o using strength and failure strain of the composite [21].

3. EXPERIMENTAL METHODS

3.1 Materials and Specimens

Harakeke fibre treated with 5 wt% sodium hydroxide (NaOH) and 2 wt% sodium sulphite (Na_2SO_3) and hemp fibre treated with 5 wt% sodium hydroxide (NaOH) were used to produce fibre mats [22]. Nature Works® 3052D injection moulding grade polylactide acid (PLA) with a density of 1250 kg/m^3 from Nature Works LLC, USA was used as a thermoplastic matrix.

Aligned fibre mats were produced using a Canpa automatic dynamic sheet former (DSF) as shown in Figure 1. In this study, fibre mats weighing 130 to 140 g/m^2 were produced using approximately 45 g treated discontinuous harakeke or hemp fibre. Firstly, fibre was dispersed in water (approximately 10 litres of water was used to disperse 10 g of fibre) in a mixing drum fitted with a disintegrator. Mats were then produced by spraying a controlled quantity of this low concentration suspension inside a spinning drum containing a porous medium to retain the fibre using a controlled pump and drum spinning speed.

Figure 1

The fibre mats were finally rinsed and compacted by allowing the drum to spin for a further 2 to 3 minutes. They were then dried in an oven at 80°C for 24 hours before being

cut to size (150mm x 90mm) to enable them to fit in a compression mould. Samples of dried harakeke and hemp fibre mats are shown in Figure 2.

Figure 2

Fibre mats and PLA sheets were dried for at least 4 hours at 105°C and 60°C respectively. They were weighed and arranged in a stack (in between Teflon sheets to prevent sticking to mould) with relative numbers of each based on the required fibre weight percentage. Stacks were heated and pressed in a hot press as for PLA samples (at 170°C and pressed for 3 minutes at 3 MPa). After hot pressing, the moulded composite materials were removed from the press and allowed to cool down to room temperature.

3.2 Methods

3.2.1 Scanning Electron Microscopy (SEM)

SEM was used to observe harakeke and hemp fibres. The observations were conducted using a Hitachi S-4100 field emission scanning electron microscope operated at 3 kV. Fibres were placed on aluminium stubs using double-sided adhesive tape and sputter coated with platinum and palladium to make them conductive.

3.2.2 Light Microscopy

Transverse and longitudinal sections of composite samples were obtained for examination. Moulds were initially wiped with a release agent to facilitate specimen removal and epoxy resin was poured into the mould followed by placement of composite sections. Specimens were demoulded after 24 hours of curing at room temperature and ground and polished using a series of coarse and fine abrasive papers starting from 320 followed by 500, 1000,

2000 and 4000 grit for approximately three to five minutes for each step. Optical images of fibre mats and composite sections were taken using an Olympus BX60F5 optical light microscope.

3.2.3 Tensile Testing of PLA and Composites

Prior to tensile testing, all specimens were placed in a conditioning chamber at 23°C ± 3°C and 50% ± 5% relative humidity for at least 48 hours. 5 replicate samples were tested for each batch and average tensile strength (TS) and Young's modulus (YM) were obtained using the results from all specimens. Tensile testing followed the procedures detailed in ASTM D 638-03; Standard Test Method for Tensile Properties of Plastics. An Instron 2630-112 extensometer was attached to the central part of the test specimen to measure the specimen extension. The specimens were tested at a constant rate of 2 mm/min. Stress versus strain graphs were constructed for obtaining orientation factor using the Bowyer-Bader model. Further details of mechanical performance are published separately.

4. RESULTS AND DISCUSSION

4.1 Assessment of Fibre Morphology

Figure 3 shows the surface of alkali treated harakeke and hemp fibres. Hemp fibre appeared to have smoother surface, a less rounded shape and have larger fibre diameter compared to harakeke fibre. From Figure 4, it is evident that the average diameter for hemp fibre is significantly larger with greater standard deviation relative to harakeke fibre. Although, the average tensile for hemp fibre is higher [22], the contribution of fibre strength towards composite tensile strength for hemp fibre is expected to be smaller as a result of smaller surface area.

Figure 3

Figure 4

4.2 Assessment of Fibre Orientation

Representative images of cross-sections of harakeke and hemp composites with 20 wt% fibre content sectioned parallel and perpendicular to the DSF rotation direction are shown respectively in Figures 5 and 6 (parallel (a), perpendicular (b)). Fibres sectioned longitudinally appear as dark lines whereas those sectioned transversely appear as light circles. It is clearly evident that the fibres have reasonable alignment in the DSF rotation direction as more transverse fibre cross-sections were observed within the sections cut perpendicular to DSF direction and more longitudinal fibre sections were observed within the sections cut parallel to the DSF rotation direction. It can also be observed that the fibres are well dispersed without obvious fibre agglomeration. Similar orientation was seen at 30 wt% content (Figures 7 and 8); however, at this fibre content, there are some fibre agglomerates and voids in the hemp composites. Comparison of fibre agglomerations for harakeke and hemp composites reinforced with 20 and 30 wt% are shown in Figure 9. It can be seen that, the fibre agglomeration for composites with 30 wt% fibre is more noticeable compared to that with 20 wt% fibre. Fibre agglomeration is attributed to insufficient polymer for adequate wetting increased possibility of fibre-fibre interaction. The formation of voids at high fibre content could be due to the evaporation of a greater amount of moisture from the fibre which would be expected to reduce the effect of reinforcement.

Figure 5

Figure 6

Figure 7

Figure 8

Figure 9

4.3 Determination of Fibre Orientation Factor (K_o) and Interfacial Shear Strength (τ) According to the Bowyer - Bader Model

In this work, as previously mentioned, the Bowyer-Bader model has been employed to calculate fibre orientation factor (K_o) and interfacial shear strength (τ). K_o and τ were determined using the experimental tensile stress-strain curves and the fibre length distributions, following the procedure detailed previously. Most harakeke and hemp fibre composites failed with failure strains of less than 0.02 and so composite stresses (σ_1 and σ_2) corresponding to strain values well away from final fracture strain, $\varepsilon_1 = 0.005$ and $\varepsilon_2 = 0.01$, such that $\varepsilon_2 = 2\varepsilon_1$ were selected (as required by the model) [23]. Analysis was also conducted using strains of $\varepsilon_1 = 0.0075$ and $\varepsilon_2 = 0.015$ for comparison. Using higher strain levels has been shown to give lower values of K_o, as reinforcement efficiency decreased due to matrix cracking and fibre debonding occurring closer to the composite failure strain [10]. Typical composite stress-strain curves for harakeke and hemp fibre composites obtained from tensile testing are shown in Figure 10. The curves correspond to the experimental results that were close to the composites mean tensile strength values. It is well accepted that stress-strain curves of many thermoplastic composites are non-linear

even at low strain levels. Due to this non-linearity, polynomial curve fitting parameters have been used to improve the accuracy of the analysis [12].

Figure 10

The relationship of composite stresses at strains of 0.005 and 0.01 versus fibre content is shown in Figure 11; stress can be seen to increase with increasing fibre content as expected from Equation (6). A linear regression line is used to fit the data and shows good agreement with the data. However, it may be observed that at 30 wt% fibre content, the reinforcement efficiency is slightly decreased.

Figure 11

The analysis was conducted by substituting the following parameters into the model: E_f = 21.2 GPa for harakeke and 26.4 GPa for hemp, E_m = 3.6 GPa for PLA, σ_f = 782.4 MPa and 911.3 MPa for harakeke and hemp respectively, as obtained in previous work [22]. It was found that at low strain levels, L_ε is small relative to the (measured) fibre lengths and so most of the length of fibres would be at the value of maximum stress. Therefore, the composite is tending towards behaving as a composite reinforced with continuous fibres [19]. K_o and τ and other related parameters obtained for composites at different fibre contents are tabulated in Table 1.

Table 1

It can be seen from the table that K_o for harakeke fibres were higher than for hemp fibres at all fibre contents. K_o for both harakeke and hemp fibre composites were higher than values

seen in the literature for those composites prepared using injection moulding and hot pressed using randomly oriented fibre mats (0.24 – 0.38) [17, 18, 20, 24, 25] and slightly lower compared to the highest values obtained with aligned flax/polypropylene nonwoven preforms (0.45 – 0.6) composites [26]. The highest K_θ values for harakeke and hemp in this work were found to be 0.58 (15 wt %) and 0.44 (20 wt %) respectively. Lower K_θ values for hemp fibre composites compared to harakeke fibre composites could be related to lower aspect ratio and fibre agglomeration influencing orientation. It should be noted that the contribution of fibres and matrix (X, Y and Z) are based on intrinsic properties while the experimental composite strength is based on numbers of mechanisms, i.e. fibre length, fibre orientation, interfacial bonding and fibre debonding. Fibres with length longer than critical fibre length as for harakeke contribute more towards the actual strength of composite, thus higher K_θ can be obtained. Similarly, if degree of fibre agglomeration is lower, thus the resulting experimental composite strength is higher, hence higher K_θ. A similar trend has been observed in previous work, where decreased fibre length for composites at high fibre fractions can cause a decreased K_θ in injection moulded samples [17, 25, 27]. Higher K_θ values for injected moulded glass and carbon fibre composites than for injection moulded natural fibre composites have been obtained, which fits with the expectation that a higher degree of fibre straightness along with less possibility of fibre agglomeration can also contribute to an increase in K_θ [9, 10, 12, 19, 28].

From our data, it was also found that the K_θ obtained was not entirely independent of strain, in contrast with the assumption used in the model [9, 10]; K_θ generally decreased at higher strain levels closer to composite failure, believed to be due to matrix cracking and fibre debonding as previously mentioned, particularly for hemp fibre composites for which the higher strain used was closer to the composite failure strain (indeed some had failed at

strains lower than those used in analysis as reflected by a "–" in table) [18]. From Figure 12, it is also evident that K_o decreased as fibre content increased. A similar trend has also been observed in previous work for injection moulded composites [17, 25, 27]. Reduction in K_o for harakeke and hemp composites with fibre contents of more than 30 wt% fibre is more evident, corresponding to a higher potential of fibre misalignment during processing due to fibre agglomeration as seen in Figures 7 and 8, which would be more likely at higher fibre contents due to fibre–fibre interaction from hydrogen bonding mentioned previously [29]. It should be noted that fibre alignment would also be expected to depend on matrix viscosity during processing, such that polymers with higher viscosity would cause a higher degree of fibre displacement as a result of higher pressure used during processing.

Figure 12

General trends for τ versus fibre content for harakeke and hemp fibre composites are graphically presented in Figure 13. τ values obtained were found to be comparable to values reported in previous studies [30-36]. Linear-trend lines fitted to the predicted τ values suggest a slight increase with increased fibre content. Increase in τ values could be explained by the increase in pressure used at higher fibre contents increasing interfacial bonding [19, 25]. Notably, τ values for harakeke and hemp composites with 20 wt% fibre were found to be the highest, thought to be due to the greatest fibre contribution towards composite strength as at this fibre content and degree of fibre distortion and agglomeration could be minimal.

Figure 13

It can be seen that τ for harakeke was slightly higher than for hemp fibre, except at 20 wt% fibre, which could be related to the greater surface area for harakeke fibre due to smaller fibre diameter (see Figure 4). A higher τ for harakeke fibre compared to hemp fibre could also be due to a higher degree of surface roughness for harakeke as can be seen in Figure 3.

5. CONCLUSIONS

The alignment of fibres within composites reinforced using fibre mats produced using a DSF has been evaluated. It was found that using a DSF to make fibre mats lead to better fibre alignment compared to that for composites made using injection moulding and composites reinforced with randomly oriented fibre mats. The orientation factors for harakeke and hemp fibre composites were found to be up to 0.58 and 0.44 respectively. Fibre orientation and interfacial shear strength for harakeke and hemp fibre composites were found to be slightly dependent on fibre content, believed to be due to fibre agglomeration and increased pressure within composite materials during processing.

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