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Interfacial shear strength of flax fibers in thermoset resins evaluated via tensile tests of UD composites

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ABSTRACT

A method of interfacial shear strength evaluation, based on the length distribution of fibers pulled out from the tensile fracture surface of an oriented flax-reinforced composite, is applied to composites with vinyl ester and acrylated epoxidized soy oil resin matrices. Two approaches for characterizing the strength of fibers with modified Weibull distribution, fiber fragmentation tests and fiber tension tests, are compared in the analysis of pull-out data. Interfacial shear strength is found to increase by a few percent when loading rate is increased from 1.33% to 8%/min.

1. Introduction

An issue hampering utilization of the outstanding specific properties of plant fibers in composites is their insufficient adhesion to most polymers, which can be improved by specific chemical and physical treatments [1,2]. The level of adhesion is frequently characterized by the interfacial shear strength (IFSS). The test methods applied for experimental determination of IFSS of bast fibers include fiber fragmentation [3–7], fiber pull-out [8–13], and microbond [14–16] tests.

Fiber-reinforced composite tests are considered preferable to single-fiber tests due to more realistic loading conditions of the interface [17]. Composite tests characterizing adhesion include fiber bundle pull-out, ball compression, transverse tension and shear of a unidirectionally reinforced composite etc. [18].

Recently, a method for IFSS estimation has been proposed using the pulled out fiber length distribution of oriented-flax-fiber reinforced composite [19]. The method assumes Weibull two-parameter distribution of fiber tensile strength. However, the strength–length scaling of bast fibers has been shown to deviate from that stipulated by the Weibull distribution in some cases (e.g. for elementary flax [20,21] and hemp fibers [22,23], technical jute fibers [24]). In such cases, the modified Weibull distribution [25,26] usually applies

$$P_{\Sigma}(\sigma) = 1 - \exp\left[-\left(\frac{l}{l_0}\right)^{\gamma}\left(\frac{\sigma}{\tilde{\beta}}\right)^{\tilde{\alpha}}\right] \quad (1)$$

where l stands for fiber length, $\tilde{\alpha}$ is the shape parameter, $\tilde{\beta}$ the scale parameter, γ denotes the length exponent ($0 \le \gamma \le 1$), and l_0 is a reference length. Note that, at $\gamma = 1$, the distribution function Eq. (1) coincides with the Weibull two-parameter distribution.

The origins of the modified Weibull distribution are related to the inter-fiber variability in strength characteristics, stemming from differences in their damage content [27,28]. For bast fibers, the most prominent damage mode is kink bands that do exhibit scatter among fibers in terms of their number [23,29] and extent [30,31]. It should be noted that inaccuracy in evaluation of the cross-sectional area of bast fibers may also contribute to an increase in the apparent strength scatter [31], particularly for technical fibers [32,33]. However, the variability of mechanical damage is apparently the main factor causing deviation from the Weibull scaling for elementary fibers since the modified Weibull distribution function is found to be preferable also for the ultimate strain distribution [20,34] free from artifacts related to characterization of fiber cross-sectional area.

In recent years thermosetting resins originating from renewable resources such as vegetable oils, starch, hemicellulose etc. have been developed. This development is driven mainly by (i) climate change concerns (i.e. renewable bio-based materials are CO_2 neutral) and (ii) the need to reduce dependency of fossil oil as raw material since this resource is not renewable and increasing demand for products and energy worldwide should not rely entirely on a limited resource. For these new resins to be accepted by the industries their properties need to be well understood and comparison with conventional resins such as vinyl esters and epoxies is highly interesting. If such resins are compatible with natural fibers, i.e. exhibit good interfacial properties, completely bio-based composites can be realized.

In the current study, we consider application of the model of fiber pull-out length distribution proposed in [19] for fibers with the modified Weibull strength distribution. Unidirectional (UD)

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flax-fiber-fabric reinforced composites with two different matrices: (i) a conventional petroleum based vinyl ester (VE) and (ii) an acrylated epoxidized soy oil resin (AESO) based on $>70\%$ renewable raw material are tested in tension at two loading rates and the lengths of fibers pulled out from the specimen fracture surfaces are measured by optical microscopy. IFSS estimates, obtained using either Eq. (1) or fiber fragmentation tests to characterize fiber strength, are obtained.

2. Experimental

Materials and manufacture procedure of flax/VE and flax/Tribest composites have been described in [35]. Below we briefly recapitulate the essential data.

A non-crimp flax fiber fabric produced by Engtex, Sweden, from unbleached 570 tex 58 t/m yarn (FinFlax Oy, Finland) was used as the reinforcement of a bio-based and a conventional petroleum based resin. The fabric had a directional oriented structure, with the aligned flax yarns transversely warp-knitted by a 16 tex polyester yarn. The bio-based matrix was acrylated epoxidized soy oil resin, Tribest S350-01 EXP from Cognis. The conventional matrix was vinyl ester Dion 9102 from Reichhold.

The composite laminates were manufactured by vacuum infusion technique on a metallic water-heated table. A stack of three aligned layers of flax fabric was infused with the respective resin using vacuum bag. In the case of VE, room-temperate cure with subsequent post-cure at 50 °C for 2 h was applied. Tribest laminates were left to cure overnight at 80 °C and 250 mbar vacuum pressure.

Rectangular specimens of 5 mm width and 25 mm gage length, aligned with the reinforcement direction, were cut out of the plates of the composite materials. The composite specimens were tested in tension up to failure by loading with stroke control. Two loading rates were employed, 1.33%/min and 8%/min. The tests were performed using Zwick/Roell electromechanical testing machine (capacity 2.5 kN). Cross-section of a specimen, used in strength evaluation from the failure load, was calculated using the average width of the specimen and the average thickness of the respective plate. The tensile strength is presented in Table 1. (Larger scatter in flax/VE strength stems from higher susceptibility to stress concentration at the grips of the brittle-resin composite, as discussed in [35]).

The fracture surfaces were inspected by scanning electron microscope (SEM) and optical microscope. SEM micrographs, shown in Fig. 1, reveal similar morphology of fracture surfaces for both matrices suggesting comparable level of adhesion.

Diameter and length of the pulled out elementary flax fibers were measured using the optical microscope in magnification range of 50–100 ×. The aspect ratio of each fiber was determined using its measured length and diameter values. The fiber pull-out measurement data of specimens with the same matrix and loading rate were pooled for subsequent analysis. Average and minimum pulled-out fiber aspect ratios, as well as the average fiber diameter, for each matrix and loading rate combination are presented in Table 1. The empirical distributions of fiber aspect ratios are shown in Fig. 2 for flax/VE and in Fig. 3 for flax/Tribest composites.

3. Model

3.1. Weibull strength distribution

The model derived in [19] relates pulled out fiber length distribution on the fracture surface of an oriented flax fiber-reinforced composite to fiber strength distribution and IFSS. It is based on the following assumptions:

- there is a minimum length, corresponding to aspect ratio λ_{min}, of pulled out fibers, limited by the clustering of short fibers, fracture surface roughness etc., that are amenable of measurement by optical microscopy;

a

b

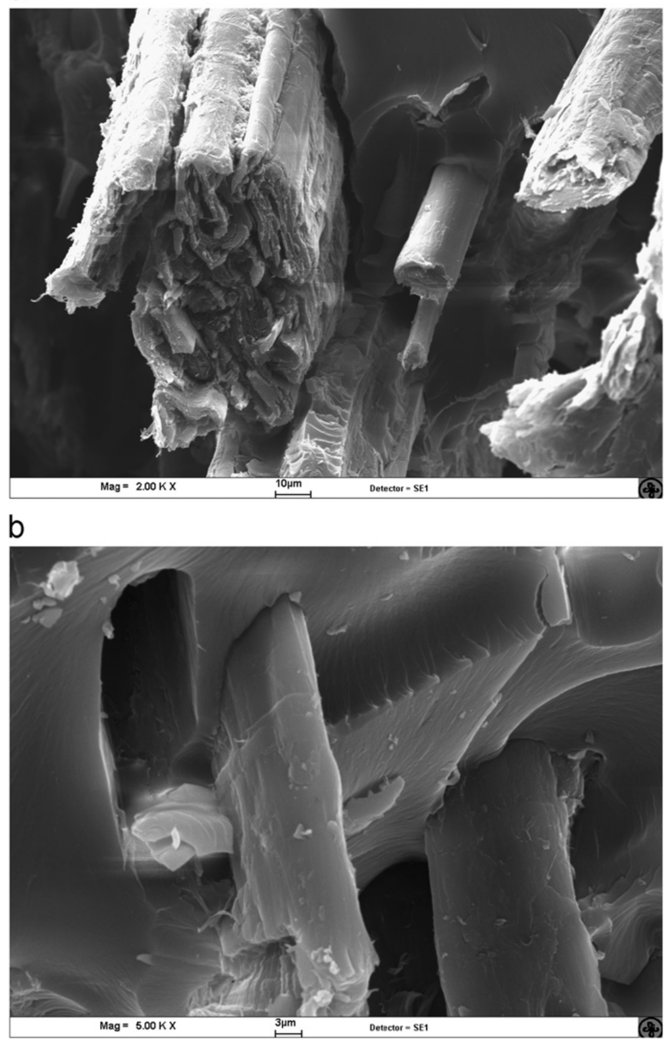

Fig. 1. SEM micrographies of flax/VE (a) and flax/Tribest (b) fracture surfaces displaying pulled out fibers.

Table 1
Tensile strength of flax/VE and flax/Tribest composites and geometrical characteristics of the fibers pulled out from the facture surfaces.

Matrix	VE		Tribest	
Loading rate (%/min)	8	1.33	8	1.33
Average tensile strength (standard deviation) (MPa)	174 (12)	166 (19)	159 (6.5)	150 (6.2)
Average pulled out fiber aspect ratio $\langle \lambda \rangle$	18.7	19.8	16.9	21.2
Smallest pulled out fiber aspect ratio λ_{min}	5.3	6.8	3.5	6.7
Average pulled out fiber diameter d (μm)	17.8	16.9	17.5	17.7

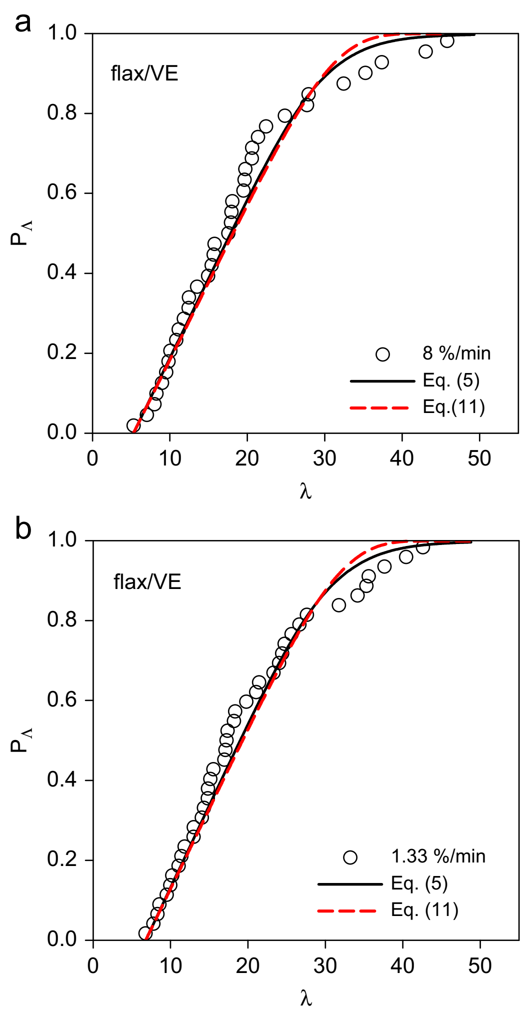

Fig. 2. Distribution of pulled-out fiber aspect ratio in flax/VE composites at 8%/min (a) and 1.33%/min (b) loading rate.

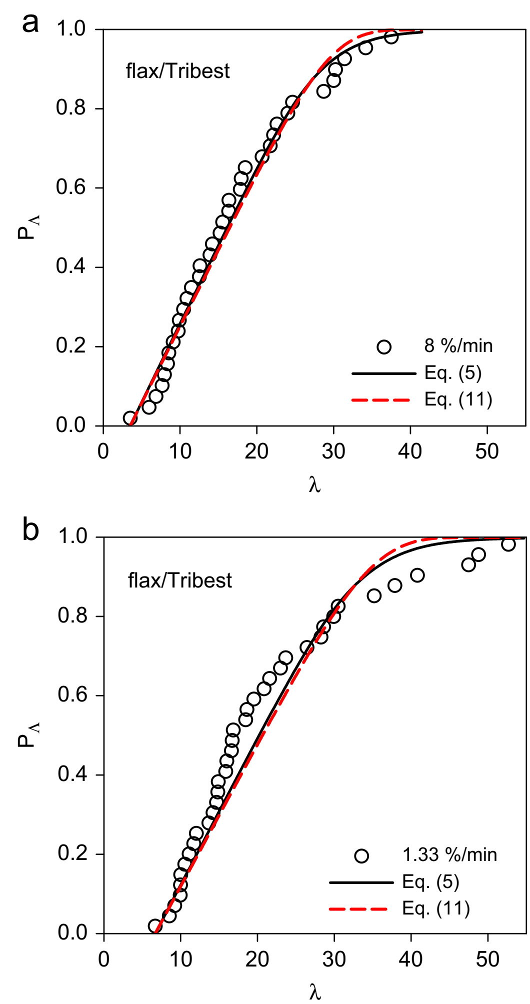

Fig. 3. Distribution of pulled out fiber aspect ratio in flax/Tribest composites at 8%/min (a) and 1.33%/min (b) loading rate.

– the relatively long ($\lambda > \lambda_{min}$) fibers have been pulled out from the fracture surface without breaking;
– the distribution of embedded fiber lengths with respect to the fracture surface is uniform;
– fiber strength follows a two-parameter Weibull distribution:

$$P_\Sigma(\sigma) = 1 - \exp\left[-\frac{l}{l_0}\left(\frac{\sigma}{\beta_\sigma}\right)^{\alpha_\sigma}\right]. \quad (2)$$

Then the aspect ratio distribution of the pulled out fibers is given by

$$P_\Lambda(\lambda) = 1 - \frac{\Gamma[(1/\alpha_\sigma+1), f(\alpha_\sigma,\beta_\sigma,\tau)\lambda^{\alpha_\sigma+1}]}{\Gamma[(1/\alpha_\sigma+1), f(\alpha_\sigma,\beta_\sigma,\tau)\lambda_{min}^{\alpha_\sigma+1}]} \quad (3)$$

where

$$f(\alpha_\sigma,\beta_\sigma,\tau) = \frac{d}{l_0(\alpha_\sigma+1)}\left(\frac{4\tau}{\beta_\sigma}\right)^{\alpha_\sigma}.$$

and τ is IFSS, λ denotes fiber aspect ratio, and d is fiber diameter.

It follows from Eq. (3) that the average aspect ratio is expressed as

$$\langle \lambda \rangle = f(\alpha_\sigma,\beta_\sigma,\tau)^{-1/\alpha_\sigma+1}\frac{\Gamma((2/\alpha_\sigma+1), f(\alpha_\sigma,\beta_\sigma,\tau)\lambda_{min}^{\alpha_\sigma+1})}{\Gamma((1/\alpha_\sigma+1), f(\alpha_\sigma,\beta_\sigma,\tau)\lambda_{min}^{\alpha_\sigma+1})}. \quad (4)$$

3.2. *Modified Weibull strength distribution*

Fiber strength distribution may deviate from the Weibull distribution Eq. (2) in that, although strength scatter at a fixed gage length agrees with Eq. (2), the variation of average strength with gage length is slower than that implied by Eq. (2). Then the modified distribution function Eq. (1) applies. It has been argued [27,28] that Eq. (1) for fiber batch strength distribution arises if the strength of each individual fiber follows the Weibull distribution, but the parameters of Eq. (2) vary among fibers in the batch. In the following, we extend the pull-out model to fibers with the modified Weibull strength distribution. Two cases are considered: when fragmentation test results of the reinforcing fibers are available, and when fiber tension tests, yielding Eq. (1) parameters, have been performed for fiber characterization.

3.2.1. *Fiber fragmentation tests*

Fiber fragmentation test [36] yields the number of fiber breaks as a function of applied load, from which Weibull distribution Eq. (2) parameters $\alpha_{\sigma i}$, $\beta_{\sigma i}$ for individual fibers can be determined. If the number of fragmentation tests n is sufficiently large, the shape and scale parameter dataset obtained can be assumed to characterize the whole fiber batch. The pulled out fiber length characteristics for such a fiber batch can then be obtained by averaging the respective relations Eqs. (3) and (4) as follows [19]:

$$P_\Lambda(\lambda)=1-\frac{1}{n}\sum_{i=1}^{n}\frac{\Gamma[(1/\alpha_{\sigma i}+1),f(\alpha_{\sigma i},\beta_{\sigma i},\tau)\lambda^{\alpha_{\sigma i}+1}]}{\Gamma[(1/\alpha_{\sigma i}+1),f(\alpha_{\sigma i},\beta_{\sigma i},\tau)\lambda_{min}^{\alpha_{\sigma i}+1}]} \quad (5)$$

$$\langle\lambda\rangle=\frac{1}{n}\sum_{i=1}^{n}f(\alpha_{\sigma i},\beta_{\sigma i},\tau)^{-1/\alpha_{\sigma i}+1}\frac{\Gamma((2/\alpha_{\sigma i}+1),f(\alpha_{\sigma i},\beta_{\sigma i},\tau)\lambda_{min}^{\alpha_{\sigma i}+1})}{\Gamma((1/\alpha_{\sigma i}+1),f(\alpha_{\sigma i},\beta_{\sigma i},\tau)\lambda_{min}^{\alpha_{\sigma i}+1})}. \quad (6)$$

It should be noted though that fragmentation testing yields directly the Weibull distribution parameters $\alpha_{\varepsilon i}$, $\beta_{\varepsilon i}$ of the limit strain of a fiber. In the case of linear elastic fibers, their conversion to the respective strength distribution parameters is elementary (see e.g. [37]): $\alpha_{\sigma i}=\alpha_{\varepsilon i}$, $\beta_{\sigma i}=E_f\beta_{\varepsilon i}$, where E_f is the fiber Young's modulus. For flax fibers, there is an initial non-linear deformation stage [38], characterized by the non-linear strain increment ε_n. Hence the limit strain is related to fiber strength as follows [20,34]:

$$\varepsilon=\frac{\sigma}{E_f}+\varepsilon_n. \quad (7)$$

Substituting the variables in Eq. (7) by their average values and expressing the mean strength and limit strain via the respective Weibull distribution parameters, allowing $\alpha_{\sigma i}=\alpha_{\varepsilon i}$, we obtain the following estimate for the scale parameter of fiber strength distribution:

$$\beta_{\sigma i}=E_f\left(\beta_{\varepsilon i}-\frac{\varepsilon_n}{\Gamma(1+1/\alpha_{\sigma i})}\right). \quad (8)$$

3.2.2. *Fiber tension tests*

Conventionally, fiber strength scatter is characterized by fiber tension tests. Then distribution Eq. (1) parameters are determined from the test results for at least two fiber gage lengths.

According to the interpretation proposed in [27], Eq. (1) applies to fiber batch where each of the fibers possesses strength distribution, Eq. (2), the shape parameters α_σ of Eq. (2) for all fibers coincide, but the scale parameters β_σ vary randomly among fibers according to the distribution function:

$$P_B(\beta_\sigma)=1-\exp\left[-\left(\frac{\beta_\sigma}{\overline{\sigma}_0}\right)^m\right]. \quad (9)$$

The average value of the scale parameter is

$$\langle\beta_\sigma\rangle=\overline{\sigma}_0\Gamma\left(1+\frac{1}{m}\right). \quad (10)$$

As a first approximation, we obtain pull-out length characteristics by substituting $\langle\beta_\sigma\rangle$ given by Eq. (10) into Eqs. (3) and (4), arriving at

$$P_\Lambda(\lambda)=1-\frac{\Gamma[(1/\alpha_\sigma+1),f(\alpha_\sigma,\langle\beta_\sigma\rangle,\tau)\lambda^{\alpha_\sigma+1}]}{\Gamma[(1/\alpha_\sigma+1),f(\alpha_\sigma,\langle\beta_\sigma\rangle,\tau)\lambda_{min}^{\alpha_\sigma+1}]} \quad (11)$$

$$\langle\lambda\rangle=f(\alpha_\sigma,\langle\beta_\sigma\rangle,\tau)^{-1/\alpha_\sigma+1}\frac{\Gamma(2/\alpha_\sigma+1,f(\alpha_\sigma,\langle\beta_\sigma\rangle,\tau)\lambda_{min}^{\alpha_\sigma+1})}{\Gamma(1/\alpha_\sigma+1,f(\alpha_\sigma,\langle\beta_\sigma\rangle,\tau)\lambda_{min}^{\alpha_\sigma+1})}. \quad (12)$$

Analytical relations, linking the parameters of Eq. (1) (characterizing the whole fiber batch) with those of Eqs. (2) and (3) (describing individual fiber strength), were established in [27] by extensive numerical simulations. Solving these relations for the shape parameter α_σ of Eq. (2), one obtains

$$\alpha_\sigma=\frac{\tilde{\alpha}}{\gamma}. \quad (13)$$

whereas the parameters of scale parameter β_σ distribution, Eq. (9), are given by

$$m=\frac{\tilde{\alpha}}{\sqrt{1-\gamma^2}}$$

$$\overline{\sigma}_0=\frac{\tilde{\beta}}{1-(m^2+\alpha_\sigma^2)^{-0.75}}. \quad (14)$$

4. Results and discussion

The strength of flax fibers produced by FinFlax has been characterized both by elementary fiber fragmentation tests and by single fiber tension tests [20]. Using average fiber modulus E_f=69 GPa and non-linear strain increment ε_n=0.32% values and Eq. (8), the set of individual fiber strength distribution parameters $\alpha_{\sigma i}$, $\beta_{\sigma i}$ for n=21 fibers was obtained from the respective limit strain distribution parameters [20]. (The number of fragmentation tests appeared sufficient to characterize fiber properties as suggested by the reasonable agreement of the relevant distributions derived from single-fiber tension and fragmentation test results reported in [20]. The applicability of Eq. (7) with the average E_f and ε_n values for relating fiber strength and limit strain distributions has been demonstrated in [20,34].) Then Eq. (6) was solved numerically for IFSS τ, using the measured average and minimum pulled out fiber aspect ratios and average fiber diameters (Table 1). The IFSS values obtained are presented in Table 2. Having established IFSS estimates, theoretical aspect ratio distributions according to Eq. (5) were determined and plotted in Figs. 2 and 3 by solid lines. It is seen that the theoretical distributions are in reasonable agreement with the experimental measurements.

Alternatively, the parametric dependence on the fiber diameter d of the theoretical distribution functions, Eqs. (5) and (11), can be eliminated by integration over the experimental diameter range, using their distribution density, as suggested in [19]. However, this would complicate the resulting expressions while providing only a minor correction to the obtained results due to their relatively weak dependence on diameter (e.g., for fiber and interface characteristics reported in Table 2, the predicted average fiber aspect ratio varied by ca. 8% upon changing the fiber diameter value entering the theoretical relations by a factor of two).

The parameters of the modified Weibull distribution Eq. (1) of fiber strength, obtained by tensile tests of elementary fibers at several gage lengths, are reported in [20]. Based on these data and Eq. (13), the shape parameter was evaluated as α_σ=6.1. The parameters of Eq. (9) were estimated at m=3.2, and $\overline{\sigma}_0=1483$ MPa by Eq. (14), enabling calculation of $\langle\beta_\sigma\rangle$ by Eq. (10). Finally, IFSS for each matrix and loading rate combination considered was determined by solving numerically Eq. (12) for τ and the results are presented in Table 2. The theoretical fiber aspect ratio distribution according to Eq. (11), plotted by dashed lines in Figs. 2 and 3, is close to that of Eq. (5), differing in a somewhat smaller predicted scatter in aspect ratios. This is apparently the result of neglecting the scatter in the scale parameter β_σ values among fibers, described by Eq. (9).

Table 2
IFSS of flax/VE and flax/Tribest.

Matrix	VE		Tribest	
Loading rate (%/min)	8	1.33	8	1.33
IFSS determined using Eq. (6) (MPa)	16.0	15.7	17.1	14.2
IFSS determined using Eq. (12) (MPa)	15.1	14.8	16.1	13.3

It is seen in Table 2 that the IFSS estimates, obtained based on either fiber fragmentation data or fiber tensile tests, differ by less than 7%. The apparent IFSS at the higher loading rate consistently exceeds that at the lower rate. The rate effect on IFSS for flax/VE interface is negligible, ca. 2%, whereas for flax/Tribest is somewhat higher, $\sim$8%. Note that the tensile strength is hardly affected by the relatively modest variation of the strain rate considered in this study, the apparent increase in average strength at the higher rate of 5% for flax/VE and 6% for flax/Tribest being comparable to the data scatter band.

The flax/VE data compare well with those obtained by the same method for a different VE matrix in [19], and are close to the lower bound of IFSS evaluated by fiber fragmentation tests (cf. $\tau = 28 \pm 11$ MPa [6]). The latter may stem from the heterogeneity of flax reinforcement, which contains both elementary and technical fibers (i.e. naturally adhering bundles of elementary flax fibers). Hence during fracture of the composite the elementary fibers can be pulled out not only from the matrix but also from the reinforcing technical fibers. The mutual adhesion of retted, scutched, and hackled flax fibers was found to be rather low, with IFSS estimated at 2.9 MPa [39]. FinFlax employed enzymatic retting of flax, hence the mutual adhesion of fibers is unlikely to be higher than that of traditionally retted fibers. The IFSS derived from fiber pull-out distribution should reflect the effective adhesion of fibers in the composite as affected by imperfect impregnation of the yarns and mutual adhesion of elementary fibers.

5. Conclusions

The model of flax fiber pull-out length distribution has been modified to allow using the modified Weibull distribution of fiber strength. The model has been applied to estimate the IFSS of flax/VE and flax/Tribest composites. The obtained IFSS is lower than that determined by fragmentation tests, which is likely to be related to imperfect impregnation of flax yarns and low mutual adhesion of elementary flax fibers. The obtained IFSS values for flax/Tribest are close to the IFFS values of flax/VE which means that compatibility between flax and both these resins is in the same range. Increasing the loading rate from 1.33%/min to 8%/min lead to an increase by a few percent of the apparent IFSS and a roughly commensurate change in the average tensile strength of the composite materials.

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