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Tensile strength of pine needles and their feasibility as reinforcement in composite materials

Chensong Dong · Daniel Parsons · Ian J. Davies

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Abstract A feasibility study concerning the use of pine needles from Maritime Pine (*Pinus pinaster*) trees as reinforcement in composite materials has been presented in this paper with the tensile strength being investigated for a total of 150 specimens at three gauge lengths, namely 50, 75 and 100 mm. In order to calculate the tensile strength for each specimen, a correlation was obtained between the cross-sectional area and external dimensions of the individual pine needles. The mean value of the tensile strength was noted to vary only slightly between 33.4 MPa for the 50 mm gauge length and 31.4 MPa for the 100 mm case with a minimum and maximum of 15 and 65 MPa, respectively. Analysis of the data using the standard Weibull model indicated the Weibull strength to vary between 33.5 and 36.0 MPa whereas the Weibull modulus varied between approximately 3.5 and 4.5. Further analysis using the Weibull model indicated the presence of a bimodal strength distribution at each gauge length that was consistent with the presence of two distinct flaw populations operating within the pine needles. Overall, it was concluded that the strength of the pine needles was sufficient for inclusion in polymer matrix composites subject to low stress or non-load bearing applications such as fibreboard and thermal or acoustic insulation.

C. Dong · D. Parsons · I. J. Davies (✉)
Department of Mechanical Engineering, Curtin University, GPO Box U1987, Perth, WA 6845, Australia
e-mail: i.davies@curtin.edu.au

C. Dong
e-mail: c.dong@curtin.edu.au

D. Parsons
e-mail: 13667171@student.curtin.edu.au

Introduction

Interest in natural fibre-reinforced biocomposites has grown rapidly during recent years, driven by increased environmental and health concerns, more sustainable methods of manufacture and reduced energy consumption [1]. Whilst it has been suggested that natural fibres can replace high strength glass fibres in composite materials [2], recent research has tended to focus on relatively low stress applications such as a replacement for wood in building materials [3] and within the automotive industry [4]. Common natural fibres used as reinforcement in biocomposites include flax, hemp, jute and kenaf [5] although other materials such as cellulose [6] and straw [7] have also been investigated in addition to powdered reinforcement such as pine needles [8] and macadamia nutshell [9]. The tensile strength of various natural fibres compared to E-glass is presented in Table 1 [4] with the maximum tensile strength of flax fibres (1500 MPa) being noted to approach the lower limit of E-glass fibre (2000–3500 MPa) although the tensile strength of most natural fibres ranged approximately 400 MPa.

The sourcing of suitable natural materials available in third world countries is an important issue since the procurement of conventional composite materials is prohibitively expensive whilst the availability of conventional building materials may be limited. Pine needles are already known to possess a variety of useful properties including their ability to remove dye from water [10], antibacterial ability [11], act against DNA damage [12] and cancer tumours [13] and indicate trends in air pollution [14]. Researchers have been investigating the possibility of utilising pine needles as reinforcement for cement since the late 1980s [15]. However, it has only been relatively recently that the possibility has been raised of utilising pine

Table 1 Tensile strength of typical natural fibres in comparison with E-glass [4]

Fibre	Tensile strength (MPa)
Flax	345–1500
Hemp	550–900
Jute	393–800
Kenaf	350–930
Ramie	400–938
Sisal	468–700
Curaua	500–1100
Abaca	430–813
E-glass	2000–3500

needle litter as a particle or fibre reinforcement in polymer biocomposites based on matrices such as polypropylene [16], phenol–formaldehyde [17–19], urea–formaldehyde [20–24], isocyanate [25–27] and resorcinol–formaldehyde [28] with the main aims being to quantify their mechanical, thermal, thermoacoustic, flammability and biological resistance properties.

Despite the recent work on pine needle-reinforced composites, there appears to be little or no data available on the mechanical properties of the pine needles themselves. Therefore, in this work the researchers have conducted a preliminary investigation on the tensile strength of dried pine needles from the Maritime Pine (*Pinus pinaster*) tree.

Pinus pinaster, also known as the Maritime or Cluster Pine, is native to the Western Mediterranean region which spans from France in the north to Portugal in the west, Italy in the east and Morocco in the south. Through human intervention *P. pinaster* can now be found in areas as widespread as Australia and South Africa where it is considered a serious pest. The *P. pinaster* tree is of similar appearance to most pine species and commonly grows to a height of 20–35 m with a diameter of up to 1.2 m. Each leaf of the *P. pinaster* is comprised of two ‘needles’ which are commonly referred to as ‘pine needles’ and are typically 1.5 mm in diameter and 200 mm in length. Although relatively soft, current uses of *P. pinaster* wood are varied and range from general construction and house framing to paper pulp and medium density fibreboard. *Pinus pinaster* is commonly planted and harvested for the above uses but is not yet used as prolifically as Radiata Pine (*Pinus radiata*).

Experimental procedure

In this study, brown pine needle litter was collected from beneath *P. pinaster* trees and subjected to tensile testing. Individual pine needles were selected randomly from the litter in order to capture a population of pine needles in various stages of their life cycle and with various geometries. The strength of fibrous materials is known to be sensitive to the volume or surface area of the specimens due to a consequence of the so-called Weibull effect [29] where the strength of the specimen population decreases as the volume or surface area increases due to an associated increase in the average number of strength limiting flaws [30]. Therefore, the authors investigated the influence of needle length on the tensile strength by cutting the pine needles according to three different gauge lengths, i.e., 50, 75, and 100 mm. Specimens for each gauge length were cut approximately 20 mm longer than the gauge length to allow room for adherence to the fixtures required for integration into the testing apparatus. The specimens were placed on a plastic sheet with a threaded eyelet at each end and the joint area coated with multipurpose epoxy

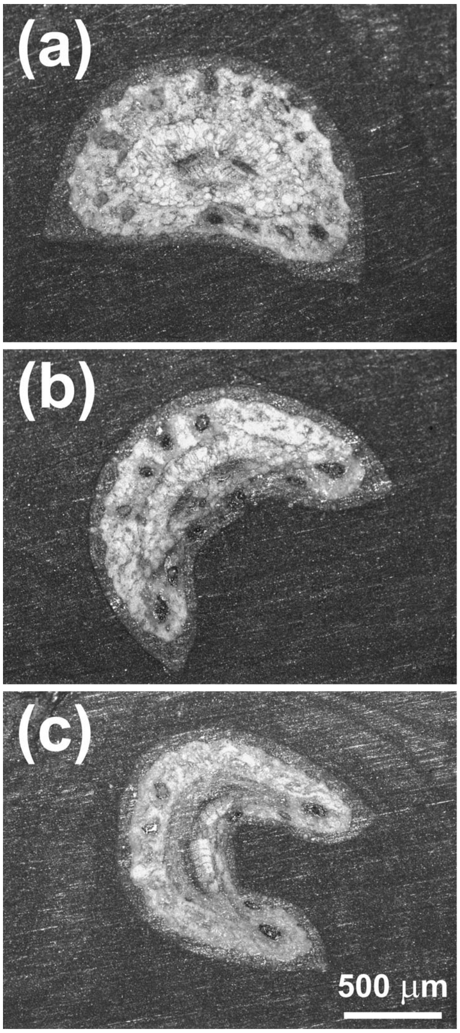

Fig. 1 Optical micrographs showing the three main categories of cross-sectional geometry noted in this work: **a** “half-moon”, **b** “cashew”, and **c** “U shape”

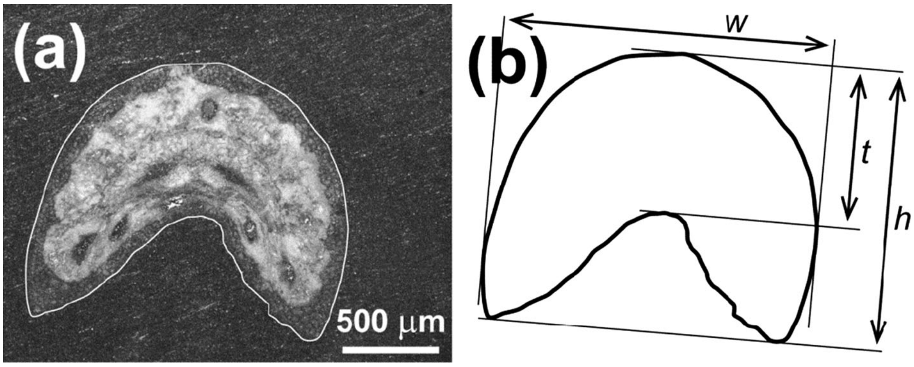

Fig. 2 Typical cross-sectional geometry of a pine needle: **a** optical micrograph and **b** external dimensions that could be readily measured using a vernier caliper

adhesive. Upon drying of the adhesive, the eyelets at either end of the specimen were fixed into the testing apparatus. A tensile load was applied gradually until the point of fracture with the failure load being noted. A total of 50 specimens were tested for each of the gauge lengths, i.e., 150 specimens overall.

Results and discussion

Measurement of the cross-sectional area

An important issue in this work was the requirement to calculate or estimate the cross-sectional area for each pine needle in order to determine the tensile strength. Preliminary investigations from the optical microscopy of pine needles, that had been embedded in epoxy resin, sectioned and then polished, indicated the cross-sectional geometry to vary greatly between individual pine needles. Figure 1 shows typical optical micrographs from the three main categories of cross section that were noted during this work, namely “half moon” (Fig. 1a), “cashew” (Fig. 1b) and “U shape” (Fig. 1c). Given the wide range of cross-sectional geometries present and the arduous requirement of having to calculate the cross-sectional area of each fractured specimen individually, the authors attempted to find a correlation between the cross-sectional area and external dimensions of the pine needle (as shown in Fig. 2) that could be measured with relative ease for each specimen using a vernier caliper.

The specimen width, w, full thickness, h, centre thickness, t, and cross-sectional area, A, for 125 randomly selected specimens that had been embedded in epoxy, sectioned and polished were measured using freely available image analysis software (ImageJ) with the aim of obtaining a general correlation between w, h, t and A. Following an extensive search for correlations between w, h, t (or a combination thereof) and A, the authors concluded that the optimum correlation could be obtained from the product of w, h and t and the cross-sectional area, A, as shown in Fig. 3.

A large number of arbitrary equations were fitted to the data shown in Fig. 3 with the best correlation being found using the following equation:

$$A = \frac{3.111hwt + 0.2292}{hwt + 2.204} \qquad (1)$$

with this relationship between used to estimate the cross-sectional area of failed specimens, and hence the tensile strength, in the following sections.

Tensile strength

Results showing the minimum, mean and maximum tensile strengths of pine needles tested at three different gauge lengths are presented in Table 2. Overall it can be seen that the mean strength decreased slightly (albeit statistically insignificantly) from 33.40 (±1.20) MPa for the 50 mm gauge length to 31.41 (±1.08) MPa for the 100 mm gauge length with the mean strength overall for all gauge lengths being 32.68 (±0.75) MPa. Secondly, the overall tensile strength was found to range between a minimum of 15 MPa and a maximum of 65 MPa. When compared to the tensile strength of typical natural fibres shown in

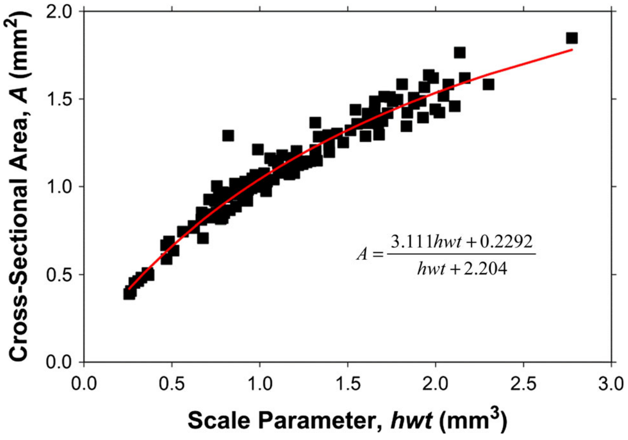

Fig. 3 Correlation between the product of the external dimensions, h, w, and t and cross-sectional area, A, for 125 randomly chosen pine needles

Table 2 Influence of gauge length on the minimum, mean and maximum tensile strength of pine needles

Gauge length (mm)	Tensile strength (MPa)		
	Minimum	Mean (±SD)	Maximum
50	16.70	33.40 ± 1.20	56.33
75	15.05	33.24 ± 1.54	65.03
100	16.96	31.41 ± 1.08	56.47
All specimens	15.05	32.68 ± 0.75	65.03

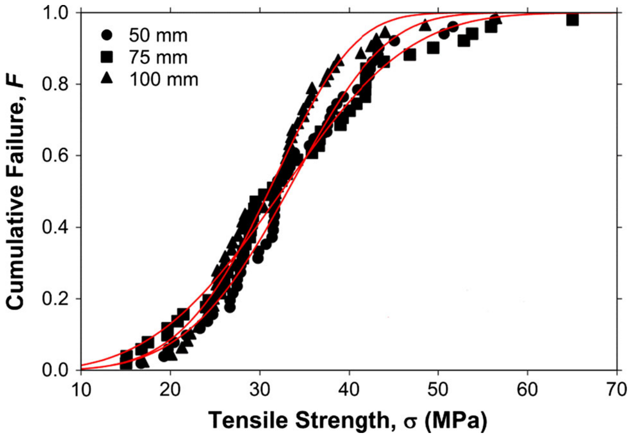

Fig. 4 Cumulative failure plots illustrating the tensile strength of pine needles at three gauge lengths

Table 1, it is clear that the mean strength of the pine needles (32.68 MPa) was approximately an order of magnitude lower than that of commonly used fibres such as jute, sisal and hemp (approximately 400 MPa). This low tensile strength for pine needles has been reflected in the low tensile and flexural strengths of the resulting composites—the tensile strength of pine needle-reinforced polymer matrix composites is known to reach a maximum of approximately 30 MPa [18, 19] but often less than half that value [23, 25, 28, 31], depending on the matrix type and pine needle surface treatment, whereas the flexural strength generally ranges from less than 20 up 100 MPa [23, 25, 27, 28] although surprisingly high values of up to approximately 350 MPa have been reported when utilising phenol–formaldehyde as the matrix [18, 19].

Weibull parameters

In the previous section, the tensile strength data were described in terms of the minimum, mean and maximum as a function of gauge length. However, as mentioned earlier it is well known that the statistical strength of fibres may be influenced by factors such as their gauge length and variation in diameter [32, 33]. In this case the authors used the following Weibull equation [29] to analyse the data as a function of gauge length:

$$F = 1 - \exp\left[-\left(\frac{\sigma}{\sigma_0}\right)^m\right] \qquad (2)$$

where F is the cumulative probability of failure, σ is the tensile strength of the individual specimens, σ_o is a scaling parameter known as the Weibull strength and m is the Weibull modulus which describes the variability in the strength data. Brittle materials such as ceramics and glasses often possess a Weibull modulus value below ten whereas ductile materials may have Weibull modulus values in excess of 50.

In order to fit the experimental data to Eq. 2, it was first necessary to rank the tensile strength data for each gauge length from smallest to largest. The value of F for each specimen was assigned according to the following relationship:

$$F = \frac{i}{N+1} \qquad (3)$$

where i is the rank of the specimen and N is the total number of specimens. The data of σ versus F for each gauge length are presented in Fig. 4 with the values of σ_o and m obtained by fitting Eq. 2 to the data in Fig. 4 being presented in Table 3. Whereas the values of σ_o for the 50 and 75 mm gauge lengths were similar, the value of σ_o for the 100 mm case was significantly lower which was in partial agreement with the Weibull theory which suggests that the tensile strength should become lower as the gauge length increases. Furthermore, the Weibull modulus values exhibited no distinct trend with gauge length and were in the range of 3.3–4.7 which is similar to that of ceramic fibres [34] and confirms the brittle nature of the pine needles.

In order to ascertain any further useful information from the experimental data, Eq. 2 was linearised by taking the natural logarithms of both sides in order to produce the following relationship:

$$\ln\left(\ln\left(\frac{1}{1-F}\right)\right) = m\ln\sigma - m\ln\sigma_0 \qquad (4)$$

Data that follow the Weibull relationship given in Eq. 2 would be expected to produce a straight line when transformed into Eq. 4. However, it is clear that the

Table 3 Weibull parameters obtained from fitting the tensile strength of pine needles as a function of gauge length

Scale length (mm)	σ_0 (MPa) (±SD)	m (±SD)
50	35.99 ± 0.17	4.34 ± 0.13
75	36.23 ± 0.20	3.32 ± 0.09
100	33.53 ± 0.14	4.66 ± 0.14

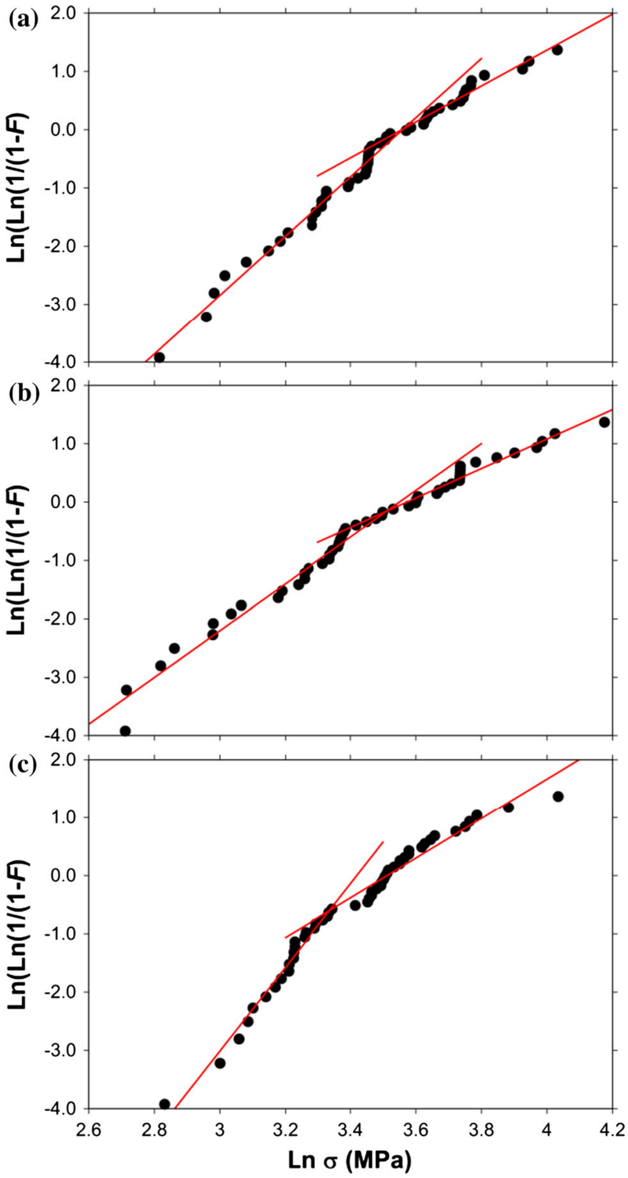

Fig. 5 Linearised cumulative failure plots illustrating the bimodal nature of tensile strength for pine needles at different gauge lengths: **a** 50 mm **b** 75 mm and **c** 100 mm

Table 4 Weibull parameters obtained from fitting a bimodal Weibull distribution to the tensile strength of pine needles as a function of gauge length

Scale length (mm)	Weibull modulus, m_1 (±SD)	Threshold stress (MPa)	Weibull modulus, m_2 (±SD)
50	5.08 ± 0.14	35.30	3.11 ± 0.13
75	4.01 ± 0.17	33.48	2.55 ± 0.08
100	7.17 ± 0.24	28.10	3.52 ± 0.21

transformation of the data into the form shown in Eq. 4 did not exhibit a linear relationship as presented in Fig. 5. Instead, it is clear from the data that the tensile strength of the pine needles exhibited a bimodal relationship, i.e., two distinct linear regions, at each gauge length, with the Weibull modulus being significantly different either side of a threshold stress as shown in Table 4. Such a bimodal relationship is indicative of two distinct defect populations within the pine needles and suggests that lower strength pine needles failed due to one type of flaw whereas the high strength needles failed due to a secondary type of flaw. In all cases, the Weibull modulus at lower strength, m_1, was relatively high and in the approximate range of 4–7 whereas the Weibull modulus at higher strengths, m_2, was significantly lower and in the approximate range 2.5–3.5; the threshold stress above which the Weibull modulus changed was approximately 35.3 MPa for the 50 mm gauge length and decreased to 28.1 MPa for the 100 mm gauge length. The significant difference between m_1 and m_2 for each of the gauge lengths confirmed that two distinct defect populations existed within the pine needles.

Overall, the population of tensile strengths for the pine needles was not found to vary significantly between the three gauge lengths tested with an approximate mean strength of 32.7 MPa and a range of strengths between 15 and 65 MPa. Whilst these tensile strength values were significantly lower when compared to existing natural fibres such as flax and hemp, this work confirms that the strength would be sufficient for low stress and non-load bearing applications currently envisaged for pine needle-reinforced polymer matrix composites.

Conclusions

A feasibility study concerning the use of pine needles from Maritime Pine (*P. pinaster*) trees as reinforcement in composite materials has been presented in this work. A total of 150 specimens for three gauge lengths, i.e., 50, 75, and 100 mm, were subjected to tensile testing. The strength values were noted to range between approximately 15 and 65 MPa with mean values decreasing only slightly from 33.40 MPa for the 50 mm gauge length down to 31.41 MPa for the 100 mm case—this decrease in mean strength with increasing gauge length being consistent with the Weibull effect. These strength values were noted to be considerably lower when compared to existing natural fibres such as flax and hemp.

Analysis of the tensile strength data using the standard Weibull model showed the Weibull strength to be approximately 36.0 MPa for the 50 and 75 mm cases and decreasing to 33.5 MPa for the 100 mm case with the Weibull modulus varying in the approximate range of 3.5 and 4.5 and thus indicative of a brittle materials. Further application of the Weibull model to the data indicated the presence of a bimodal strength distribution for each of the gauge lengths which suggested the presence of two distinct defect populations operating within the pine needles; the weaker specimens tending to exhibit a higher Weibull modulus in comparison to the stronger specimens.

It was concluded that whilst not displaying sufficient strength for the requirements of high strength polymer matrix composites, the strength of the *P. pinaster* pine needles should be sufficient for low stress or non-load bearing applications such as fibreboard and thermal or acoustic insulation.

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