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Mechanical Characterization and Application of Weibull Statistics to the Strength of Softwood Lignin-Based Carbon Fibers

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ABSTRACT: Mechanical characterization of the first generation of softwood kraft lignin-based carbon fibers (CF) was carried out. The single-fiber tensile tests of filaments with different diameters and length were performed to evaluate stiffness and strength of carbon fibers. The average mechanical properties were measured as follows: tensile strength of approximately 300 MPa, the elastic modulus of 30 GPa and a strain at failure within interval of 0.7–1.2%. The fiber strength data was evaluated by the two-parameter Weibull statistics and parameters of this distribution were obtained. Although strength of the produced fibers is still significantly lower than that of commercially available, the experimental results and predictions based on Weibull statistics show a fairly good fit. © 2013 Wiley Periodicals, Inc. J. Appl. Polym. Sci. 130: 3689–3697, 2013

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INTRODUCTION

An increasing demand for high-performance materials, such as carbon fiber reinforced plastics (CFRP) has resulted in a rapid expansion of the carbon fiber (CF) production capacity worldwide.[1] But until recently these materials were used mostly in applications where cost is not the key factor (e.g., various military applications, aerospace, professional sport equipment, etc.). However, with higher demands for the weight-to-performance ratio and durability of materials in everyday applications, advanced composites have attracted attention of other industries. The car manufacturers are considering CFRP for the production also of conventional cars and not only luxury automobiles. Another example is blades of wind turbines which cannot be built anymore only from glass fiber composites due to higher requirements for sustained loads because of their large dimensions. If not for the comparably high price of CF other industries also could make good use of these materials, for example replacement and upgrade of outdated construction materials remains unexplored.[1] Today's nonrenewable main CF precursors Polyacrylonitrile (PAN) and tar- or petroleum pitch represent approximately 50% of the CF cost.[2,3] Moreover, because they are petroleum based the precursor cost will continue to increase with increasing crude oil prices.[1] Meanwhile, low cost CF alternatives are being developed by replacing the currently used precursors. As it was mentioned above, one major area of interest for large-scale implementation is the automotive industry, which is constantly searching ways to lower vehicle weight to reduce fuel consumption.[1,4] However, CFs for car production would not require the same performance grade as those used in aerospace and earlier estimations state that a tensile strength of 1720 MPa, elastic modulus of 172 GPa and ultimate strain above 1% would satisfy the requirements.[2,3]

A potential precursor material should possess properties that allow fiber production by some of the conventional spinning methods,[2] such as wet- or melt spinning, that is, have thermoplastic properties for melt spinning[5] or a liquid crystal behavior for dry or wet spinning.[6]

Other raw materials have been used for carbon fiber production with various results, such as regenerated cellulose[7] and textile PAN.[1] One of the most promising alternatives is lignin,[1] a renewable biproduct from the pulping industry, with high carbon content (above 60 atomic %).[8] Replacing the nonrenewable precursors by lignin could potentially decrease the CF cost by up to 35%.[1] The production scheme for lignin-based carbon fibers have been the same as for conventional CFs,[6,9] starting with the spinning of a precursor yarn that is subsequently stabilized and further carbonized into the final CFs.[10–12] Lignin from different pulping processes have been used in renewable low-cost CF research, such as steam-explosion lignin,[10,13] organosolv lignin,[11] and kraft lignin.[1,11,14,15]

Out of many available pulping processes applied today, the kraft pulping process is by far the most widely used for paper pulp

production. Large amounts of kraft lignin are dissolved in the black liquor from where it can be readily available.[16] Kraft lignin was for long time considered unsuitable for CF production, but methods have been developed that efficiently reduce the amounts of impurities and improve the thermal processability of kraft lignin.[1,17]

It has however been shown that the origin of lignin macromolecules has a great impact on the processability. In contrast to hardwood lignin, softwood lignin was not melt spinnable into fibers and charred upon heating, because of insufficient thermoplastic characteristics.[11,15]

However, it has recently been shown that the spinning difficulties of softwood kraft lignin (SKL) can be overcome by addition of hardwood kraft lignin permeate (HKLP) as a softening agent, and further processed into smooth and homogeneous CFs.[12]

So far, none of the produced lignin-based CFs have reached the target for the mechanical properties mentioned previously. Up until now, CFs from organically purified hardwood kraft lignin are the strongest ones produced, with tensile strength of 1100 MPa and a modulus of 69–83 GPa.[18] Even though these values are quite low compared with the data for conventional CFs,[6] they are encouraging, as one crucial production step for conventional fibers is still lacking here. The precursor fibers were not stretched enough during processing to induce high molecular orientation, a treatment which may lead to significant increase of mechanical properties.[1]

The manufacturing of lignin based fibers is still in an early development stage and there are many parameters that have to be explored and optimized, for example, composition of the lignin feed material, extrusion temperature and rate, stretching ratio. To optimize these parameters simple yet reliable methods for characterization of the CF is needed to get quick feedback. The most direct approach would be to perform tensile tests on single fibers. This method is especially suited in this case because often only small amounts of fibers are available and other experimental techniques cannot be employed (for example fiber bundle or composite tests). CFs are brittle and failure of brittle materials is controlled by the presence of strength-limiting defects. In case of CF such defects are pores, inclusions or irregularities in the crystal structure.[19] The properties of such materials are usually discussed using a "weakest-link" approach. According to this approach, the tensile strength of CFs shows a large scatter and strong size dependence, as a large sample volume probably will contain more defects.[20] One common way of analyzing the strength distribution in brittle material is by using a Weibull probabilistic model, which was firstly developed in the 1950s.[21] As then, Weibull statistics have routinely been used for strength characterization of CFs.[19,22,23] It has been shown that fiber strength increases with decreasing gauge lengths, that is, smaller sample volumes.[22,24] This can simply be explained by the higher probability of finding severe defects with increasing fiber length.

To obtain statistically significant results, a large number of fibers has to be tested. The so far reported mechanical data are however based on at most 20 fibers of one gauge length[3,11,14] which is far too low for a proper statistical analysis. In addition, because of the size dependence of fiber strength, multiple gauge lengths should preferably be analyzed.

In this article, the strength distribution for CFs produced from softwood kraft lignin was studied employing a two-parameter Weibull model using different gauge lengths and fiber sizes. In order to determine the properties of the lignin-based CFs, and to evaluate the impact of the manufacturing parameters, such as die sizes and winding speeds, fibers with three different diameters (≈30, 60, and 90 μm) were made and tested.

Three fiber lengths (10, 20, and 40 mm) for each diameter were tested and strength data statistically approximated in order to obtain parameters of the Weibull strength distribution. This is the first time softwood kraft lignin CFs have been thoroughly characterized using standard mechanical testing methods. Although strength of the produced fibers is still lower than that of commercially available carbon fibers, the fiber properties could be described using common statistical methods, which strongly indicate the potential of this material.

Theoretical Background

It is generally accepted that strength of brittle materials can be described by Weibull statistical distribution, two-parameter distribution is often used:

$$P=1-e^{-\frac{V}{V_0}\left(\frac{\sigma}{\beta}\right)^{\alpha}} \tag{1}$$

where P is probability of failure, σ is stress, V is volume of the sample and V_0 is reference volume, α and β are the shape and scale parameters respectively. This distribution is also valid for brittle fibers such as CFs and glass fibers.

Assuming constant cross-section area (or diameter for circular fibers) within the sample, the volume in (1) can be replaced by fiber length:

$$P=1-e^{-\frac{l}{l_0}\left(\frac{\sigma}{\beta}\right)^{\alpha}} \tag{2}$$

where l is the fiber length and l_0 is the reference length chosen arbitrary (usually it is convenient to use unit length for l_0).

Furthermore, the average strength ($\langle\sigma\rangle$) and standard deviation (s) can be determined for any fiber length l using the following expressions:

$$\langle\sigma\rangle=\beta\left(\frac{l}{l_0}\right)^{-\left(\frac{1}{\alpha}\right)}\Gamma\left(1+\frac{1}{\alpha}\right) \tag{3}$$

$$s=\beta\left(\frac{l}{l_0}\right)^{-\left(\frac{1}{\alpha}\right)}\sqrt{\Gamma\left(1+\frac{2}{\alpha}\right)-\Gamma^2\left(1+\frac{1}{\alpha}\right)} \tag{4}$$

where Γ is a gamma function.

The same quantities described in (2)–(4) can be obtained experimentally for the given fiber length. The average strength and standard deviation are directly calculated from the experimental data, whereas probability of failure is given by:

$$p=\frac{i-0.3}{n+0.4} \tag{5}$$

where i is the sample number in an ascendingly arranged strength data, and n is the total number of samples. Then the probability of failure can be plotted in Weibull coordinates

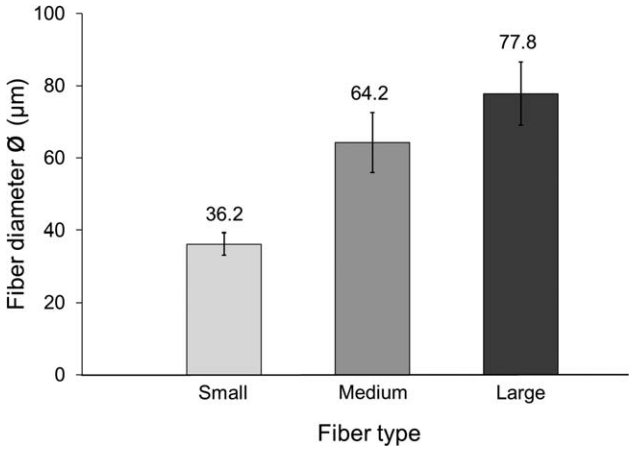

Figure 1. Average diameters with standard deviation for different batches. Small $n = 120$, Medium $n = 200$, Large $n = 168$.

$\ln(-\ln(1 - P)) = f(\ln(\sigma))$ and approximated by the line equation ($y = Ax + B$). The eq. (2) also can be rewritten in a similar way:

$$\ln(-\ln(1-P)) = \alpha \ln(\sigma) + \left(\ln\left(\frac{l}{l_0}\right) - \alpha \ln(\beta)\right) \quad (6)$$

Then if coefficients of line equation (A and B) are obtained from the linear fit of experimental data the Weibull parameters are given as:

$$\alpha = A, \beta = e^{\frac{\ln\left(\frac{l}{l_0}\right) - B}{\alpha}} \quad (7)$$

The shape parameter (also known as the Weibull modulus) of commercial carbon fibers is within a rather broad interval of 4–10.[23,25] The difference in shape parameters reflects the flaw distribution in the material. In case of a homogeneous flaw distribution throughout the material the shape parameter will have high values. But, if shape parameter is low it indicates a much more significant scatter of strength which is caused by the presence of larger defects that are unevenly distributed.

EXPERIMENTAL

Carbon Fiber Production

The manufacturing of CFs consisted of several steps; spinning of a lignin precursor and thermal treatments (stabilization and carbonization).

Lignin. SKL was isolated from industrial black liquor according to the LignoBoost-concept, in which lignin is precipitated using carbon dioxide and re-slurried prior to further washing with acid.[17] HKLP was produced by ultrafiltration of the respective black liquor through a 15 kD ceramic membrane (Groupe Novasep, St Maurice de Beynost, France) prior to isolation.[8] The yields of isolation for unfractionated and fractionated lignin were approximately 80 and 40%, respectively. The lignins were dried in air at room temperature to a dry content above 90 wt %.

Precursor. A 7 g sample of SKL with addition of 10 wt % HKLP was premixed prior to manually feeding the powder into a counter-rotating twin screw laboratory compounder with conical screws (HAAKE MiniLab II CTW5, Thermo Fischer Scientific, Waltham, Germany). The lignin was recirculated at 25 rpm in the extruder for 10 min prior to extrusion at 200°C. Dies of 0.2 and 0.5 mm were used to achieve three sets of single filament fiber diameters (Small—"S,", Medium—"M," and Large—"L"); S fibers by spinning at moderate speed using the small die, and at high and moderate speed using the larger die to produce M and L fibers respectively. The lignin fibers were collected on a fiber collector (TUS, Dynisco, Franklin) at a winding speed of 36 or 76 m min^{-1}. More detailed information about the fiber manufacturing is published previously.[12]

Thermal Treatment. The lignin fibers were oxidatively stabilized in a conventional gas chromatography oven (HP 5890, Hewlett Packard, Palo Alto). The fibers were heated at 0.2°C min^{-1} up to 250°C and held for 1 h before cooling to room temperature.[26]

The stabilized fibers were subsequently carbonized in a tube furnace (VTF 50/15-L, Entech, Ängelholm, Sweden) in nitrogen atmosphere at a flow rate of 10–20 mL min^{-1}. The temperature was increased by 1°C min^{-1} to 600°C and 3°C min^{-1} to 1000°C before cooling to room temperature.[27]

Fiber Characterization

Microscopy. In order to study the fiber structure and to determine the fiber diameters, scanning electron microscopy (SEM) and optical microscopy were performed.

The structure of CFs was characterized using SEM (JSM-6460, Jeol, Tokyo, Japan) at 15 kV acceleration voltage. The fibers were gold-plated prior to analysis to avoid charging of the sample.

The fiber diameter was determined for each sample prior to mechanical testing by optical microscopy (BH2-RFCA, Olympus). Average fiber diameter was obtained from multiple measurements along the fiber length (3, 5, and 7 measurements for the gauge lengths 10, 20, and 40 mm, respectively). These values were used to calculate the fiber cross-section area.

Mechanical Testing. Single fiber tensile tests were performed according to the ASTM standard D3379-75 using an Instron tensile testing device (Instron 4411, Instron, Norwood) with a 5 N load cell and pneumatic grips. In total, 40 samples of each gauge length (10, 20 and 40 mm) were made, except for the longest L fibers, where 24 samples were made due to lack of material. Samples were prepared by mounting a fiber into a paper frame with a slot of the same length as the gauge lengths.[28] The samples are hereafter denoted as S1, S2, S4, M1, M2, M4, and L1, L2, L4, depending on diameter/batch and gauge length.

Tests were performed at strain rates set to 10% min^{-1}, which corresponds to the displacement rate of 1, 2, or 4 mm min^{-1}, depending on fiber length. Load and displacement were recorded during the test for further processing to construct stress–strain curves: stress was calculated from load and cross-section area of fiber, whereas strain was obtained from the initial fiber length and elongation (displacement).

RESULTS AND DISCUSSION

Fiber Morphology

The diameters of the CFs were measured from optical microscopy for each set of fibers. The average values for the different batches (S, M, and L) were determined by pooling together all

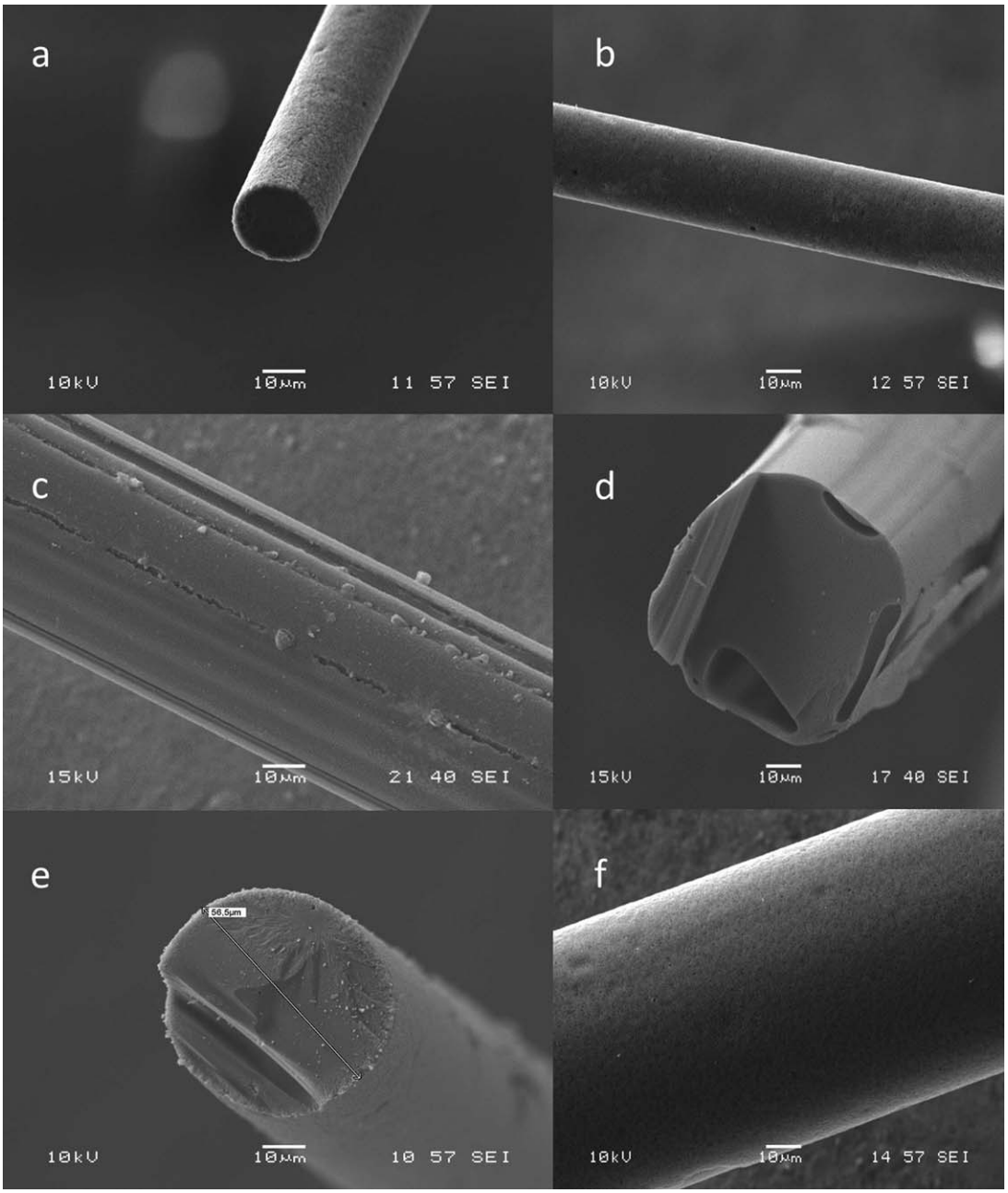

Figure 2. SEM images of the surface and cross sections of S (a-b), M (c-d) and L (e-f) fibers.

measurements made for each individual fiber (Small $n = 120$, Medium $n = 200$, Large $n = 168$). The average results for all fiber batches are shown in Figure 1.

Three sets of diameters could be fairly well distinguished if only average values are compared but there is an overlapping between the M and L fibers if standard deviation is also considered. This is most likely because the material output could not be controlled in the spinning equipment used, which may contribute to the variation in the produced precursor diameters. Moreover, the control of the winding speed appears to be insufficient to obtain satisfactory precision of fiber diameters by the 0.5 mm die used for the M and L fibers. Whereas the S batch has much lower and less scattered values of diameters, since a smaller die of 0.2 mm was used to produce these fibers. The standard deviation in percent was 8.5% for S fibers compared to 13 and 11.3% for M and L fibers respectively. It should be noted that the different drawing speeds might affect not only diameter of filaments but also the mechanical properties of the carbonized fibers because of different degrees of molecular orientation in the precursor.

Characteristic SEM images presented in Figure 2 show that the S (a–b) and L (e–f) fibers had smooth surfaces and circular cross-section without pores or inclusions, whereas the M (c–d) fibers seem to be of poor quality, with large pores and a

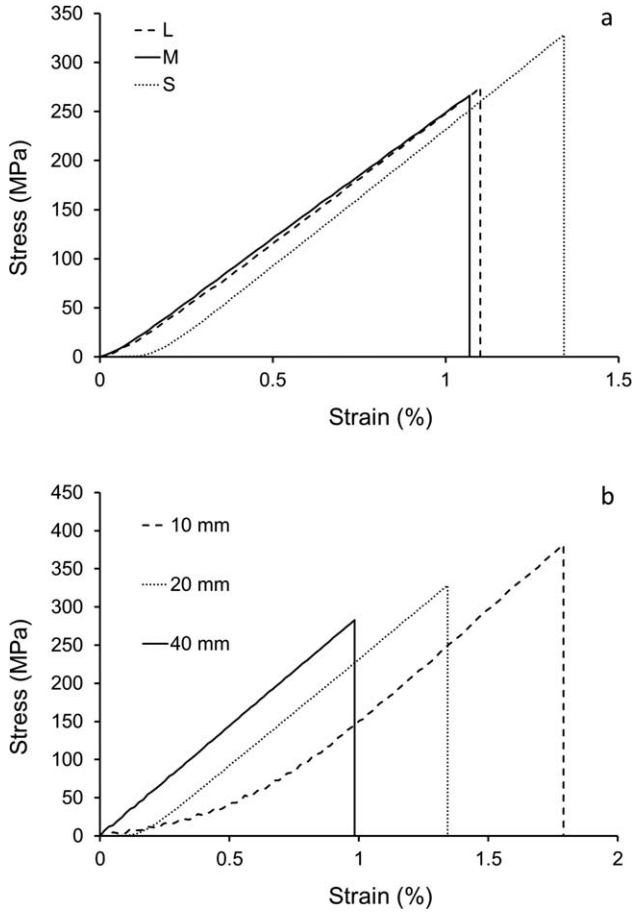

Figure 3. Stress-strain curves for (a) S, M, and L fibers of 20 mm gauge length, and (b) for S fibers of 10, 20, and 40 mm gauge length.

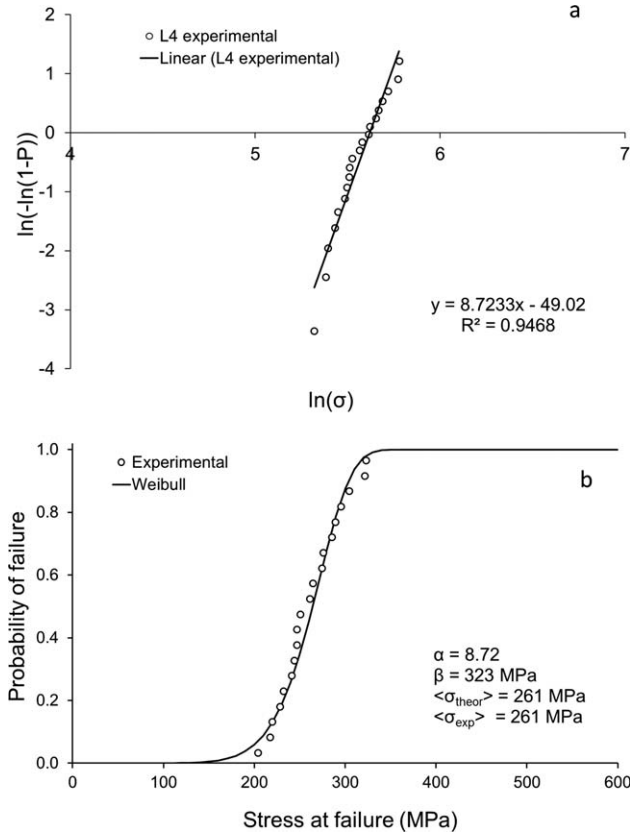

Figure 4. (a) Determination of Weibull parameters; α, β and theoretical average strength (σ_{theor}) for the L4 fibers, by fitting experimental data to the Weibull model. (b) shows experimental and theoretical probability of failure.

noncircular cross-section, the latter is a reminiscence from precursor manufacturing. As it was mentioned above, the different types of defects, their size and location should affect the overall fiber strength distribution.

Stress–Strain Curves. The typical stress–strain curves obtained from tensile tests are shown in Figure 3. These curves are constructed without accounting for the compliance of the test setup and they are shown here only in order to demonstrate their shape (linearity), therefore the strain values should be ignored here. Figure 3(a) compares different fiber types (S, M and L) of 20 mm gauge length and Figure 3(b) shows different gauge lengths of the S fiber. There is a certain lag in the beginning of the stress–strain curves. This is most likely due to some slack in the fiber, although some of it might correspond to unidentified mechanisms in fiber behavior. To assure that only the elastic deformation was included, the calculations of fiber stiffness were performed in a stress interval of 50–100 MPa as the stress–strain curve was linear for all samples in that stress region.

Table I. Summary of Results for All Fiber Batches of Different Length (l); Number of Tested Fibers (n), Average Fiber Diameters (Ø), Experimental (σ_{exp}) and Theoretical (σ_{theor}) Tensile Strength, and Weibull Parameters (α and β)

	l [mm]	n	Ø [μm] (*St. Dev.*)	σ_{exp} [MPa] (*St. Dev.*)	σ_{theor} [MPa] (*St. Dev.*)	α	β [MPa]
S1	10	16	36 (3.07)	377 (40)	375 (44)	10.3	394
S2	20	25		330 (59)	330 (65)	5.92	400
S4	40	19		269 (52)	272 (57)	5.49	376
M1	10	20	64 (8.31)	281 (73)	281 (76)	4.19	309
M2	20	28		274 (71)	274 (76)	4.07	358
M4	40	29		233 (64)	235 (69)	3.80	374
L1	10	15	78 (8.79)	295 (57)	294 (59)	5.75	318
L2	20	37		274 (54)	275 (61)	5.21	342
L4	40	18		261 (34)	261 (36)	8.72	323

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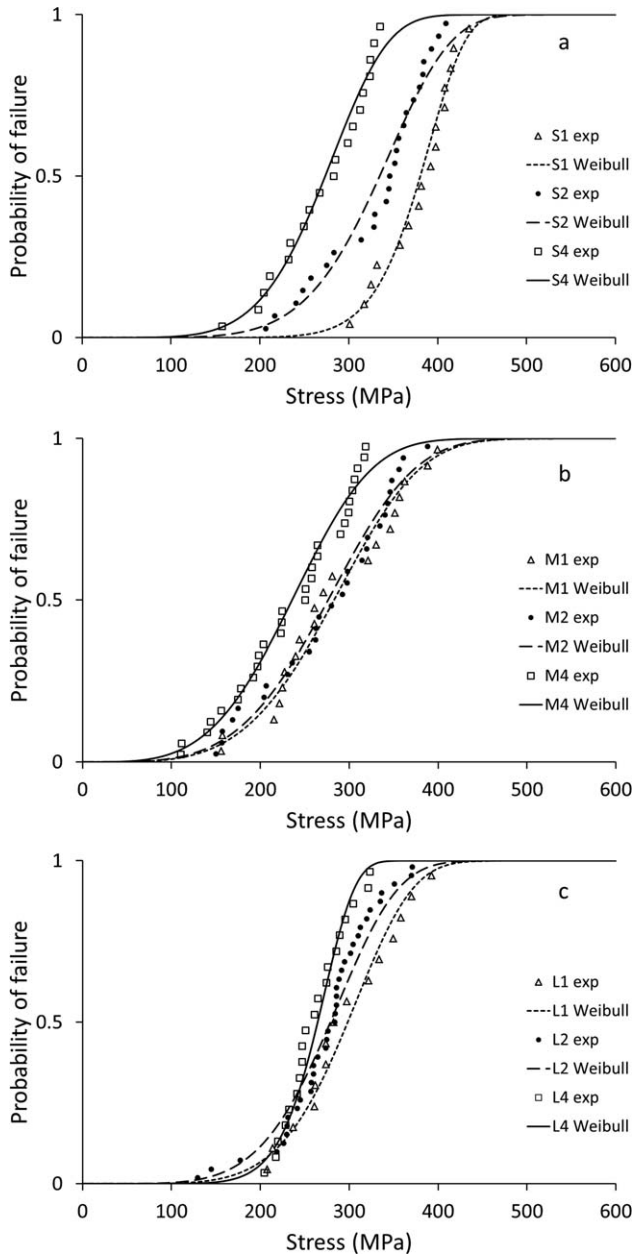

Figure 5. Comparison of Weibull probability of failure and experimental data for S, M and L fibers, tested at different gauge lengths (a: Small fibers, b: Medium fibers and c: Large fibers).

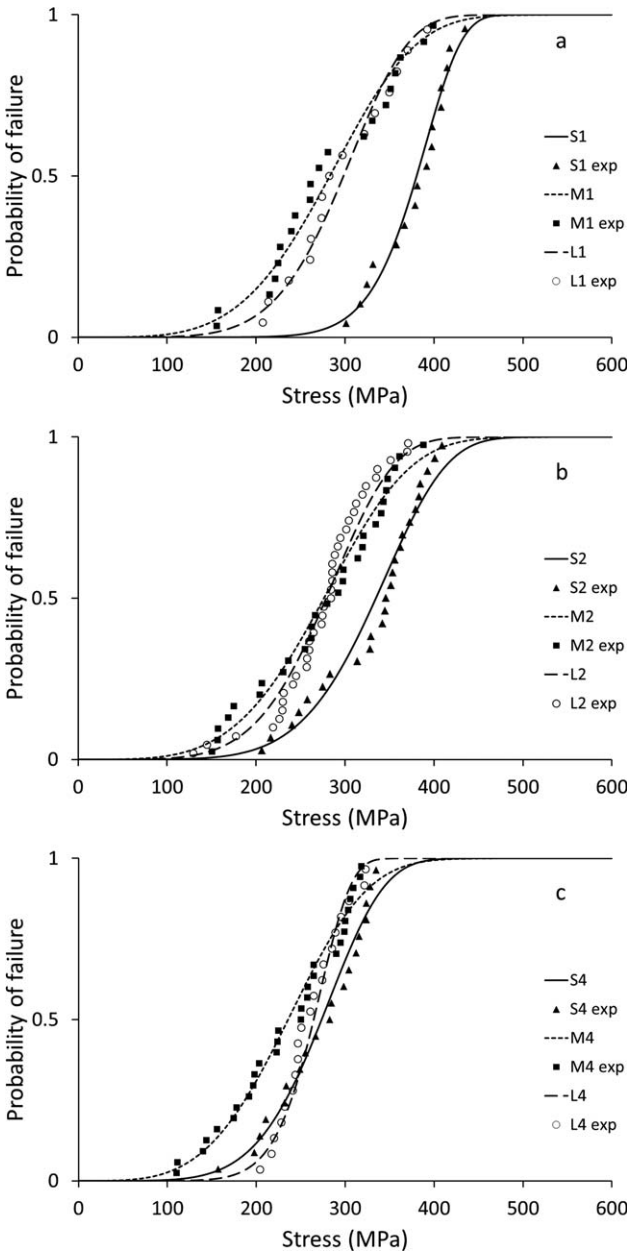

Figure 6. Comparison of Weibull probability of failure and experimental data for 10, 20, and 40 mm fibers obtained for different fiber diameters (a: 10 mm, b: 20 mm, c: 40 mm).

Strength and Parameters of Weibull Distribution. The probability of failure was calculated according to eq. (5) and plotted in Weibull coordinates as shown in Figure 4(a). The experimental data were then approximated by a linear equation as exemplified in Figure 4(a) and Weibull parameters were obtained by using eqs. (6) and (7), see Theoretical Background. The reference length (l_0) was set to 10 mm for all analysis.

The comparison between prediction of strength distribution by using eq. (2) and experimental data is presented in Figure 4(b). Considering that the amount of available data is fairly limited from the statistical analysis point of view, a good fit between experiment and prediction is obtained. The predicted average strength (σ_{theor}) according to eq. (3), of L4 fibers was 261 MPa which is exactly the same as the experimentally obtained value (σ_{exp}).

The produced CF can be assumed to be isotropic, as no stretching is applied during the thermal treatments. Additional stretching of the lignin precursor during the process may increase the properties of the CFs and close a gap between the properties of commercial and lignin based CFs.

Another factor that might significantly influence properties of CF is the heat treatment temperature (HTT). The treatment temperature for commercial CF is ranging within

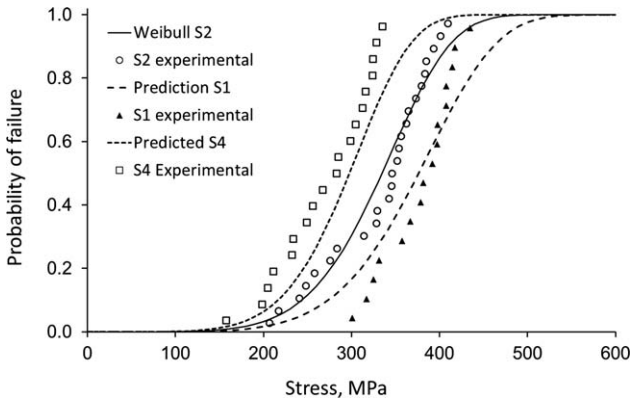

Figure 7. Prediction of the probability of failure for S1 and S4 fibers, based on the Weibull parameters obtained for S2 fibers.

1000–2500°C. The higher HTT will result in higher strength and stiffness of fibers due to changes of crystal order of CF.

The Weibull parameters and the theoretical tensile strength (σ_{theor}) were determined for all fiber batches and different gauge lengths. The results are summarized in Table I. It was not possible to test all prepared specimens since some of them failed prior to loading. Assuming that those were the weakest fibers, the presented strength data can be assumed to be slightly overestimated.

Based on the theory of brittle materials, the average strength should be increasing with decreasing volume. However, this is not the case if batch M and L is compared: the M-fibers have lower (or equal) average strength compare to the L-fibers, even though average diameter (and volume if the same length is considered) of L-fibers is higher than that for M-fibers. One can argue that the results for these fibers are similar because the diameters of the fibers are not that much different. However, the actual reason most likely is the poor fiber quality of the M-fibers, observed from SEM images. Additionally, the M fibers strength data were more scattered, as well as had low α values, as compared to those of the S and L fibers. The additional stretching of the M fibers during spinning compared to L fibers, which should lead to higher molecular orientation and resulting increase of strength, did not compensate for the poor fiber quality or was too negligible to make any difference.

Despite this heterogeneity of M fibers, the strengths followed the theory of brittle materials within each fiber type if fiber length is considered (assuming the same diameter for all fibers): that is the smaller the volume (length), the higher the strength. All three fiber types, S, M, and L fibers, had increasing average strength at decreasing gauge lengths, reaching the maximum strength for the 10 mm samples. This observation was most significant for the S fibers, for which the average strength increased by 40% when the length was reduced from 40 to 10 mm. For the M and L fiber the increase of strength with decrease of fiber length from 40 to 10 mm was 20 and 12% respectively. This trend indicates that with increasing fiber diameter the effect of fiber length on the strength is reduced.

In general, it can be stated that Weibull parameters significantly vary depending on the studied fiber type, that is, diameter, as well as gauge length. Furthermore, strength of the fibers is inferior compared to commercial CFs. This was expected, as the fibers originate from a laboratory process of singe-filament spinning in equipment mainly aimed for granule production. Also, it should be noted once again that there was no substantial stretching of the lignin precursor during the processing and molecular orientation was not induced. Nevertheless, values of the Weibull modulus (between 4 and 10) are within the interval found for the commercial CFs.[23,25] The theoretical average strength (σ_{theor}), calculated according to eq. (3), is very close to the experimental data (almost identical). The theoretical standard deviation, calculated according to eq. (4), showed the same trend as in the experiment, although values were consequently slightly higher than the experimentally obtained.

The experimental and theoretical distributions of fiber strength are compared in Figure 5 and 6, by plotting the probability of failure calculated from (5) and (2) with Weibull parameters corresponding to each fiber type respectively (Table I). In order to see the influence of fiber length, data for the same fiber batch and different gauge lengths are presented in Figure 5. The dependence of strength distribution on fiber diameter can be seen in Figure 6, where data for the same fiber length and different fiber batches (diameters) are plotted together.

The plots in Figures 5 and 6 show that the experimental strength distribution can be approximated fairly well for all fibers using Weibull distribution. It is of course also somewhat expected, since experimental strength results are approximated by the Weibull distribution with parameters also obtained from the experimental results. But according to theory, if parameters of Weibull distribution are known, eq. (2) can be used to predict the strength distribution of fibers of any gauge length. As an example, predictions for S1 and S4 fibers were performed using the Weibull parameters obtained for S2 fibers (Figure 7).

Table II. The Elastic Modulus (E) and Strain at Failure (ε) for All Fiber Types

	Small		Medium		Large	
l [mm]	*E* [GPa](*St. Dev*)	ε [%](*St. Dev*)	E [GPa](*St. Dev*)	ε [%] (*St. Dev*)	*E* [GPa] (*St. Dev*)	ε [%] (*St. Dev*)
10	33.0 (8.1)	1.20 (0.26)	28.1 (5.2)	1.04 (0.29)	25.3 (2.9)	1.17 (0.20)
20	32.9 (3.7)	1.01 (0.18)	30.7 (4.5)	0.90 (0.23)	31.4 (3.0)	0.87 (0.17)
40	30.1 (1.5)	0.90 (0.19)	30.1 (2.0)	0.78 (0.22)	30.3 (1.6)	0.86 (0.11)

The standard deviation is given in parentheses.

The accuracy of predictions is acceptable, although probability is slightly overestimated for S1 fibers at low stress and underestimated for S4 fibers, whereas for higher stresses the prediction for S1 fibers shows lower values. It is likely that in order to improve accuracy, one should account for the fiber-to-fiber strength parameter variation in a batch of fibers (each of which has the Weibull two-parameter strength distribution (1)) by using the modified three parameter Weibull distribution.[28]

The dependence of strength on the fiber size was demonstrated with respect to changes of length. However, it should be noted that the variation of properties of fibers with diameter is also reported in the literature.[24,29] This issue is not fully addressed in the current paper and only length effect on fiber strength is analyzed, by using eqs. (2–7). The variation of fiber diameters within the same "diameter batch" is thus neglected and all values of the strength are pooled together for the analysis within each particular batch.

Although statistical significance of the results may be restricted due to the limited number of experiments performed, the results presented in this study are valid for analysis of average fiber strength and for comparison between different fiber batches. One of the main objectives of this paper was to present experimental values of softwood kraft lignin based CFs, and a comprehensive statistical analysis with more detailed parametric study is currently conducted.

Elastic Modulus and Strain at Failure. The elastic modulus (E) was determined by taking compliance of the tensile machine into account as described by the standard (ASTM D3379-75). The average values of elastic modulus and strain at failure together with the standard deviation are given in the Table II. It should be noted that the procedure described in the standard should be closely followed as the elastic moduli before correction were up to 40% lower than the ones presented in Table II. The strain at failure (ε) was not obtained from the stress–strain curves directly, but calculated from the strength and elastic modulus for each fiber assuming the linear behavior of fiber until the failure (as seen in Figure 3). Therefore, the strain values presented here should only be used as an indication.

The scatter for the elastic modulus is larger for the short fibers (10 mm) as compared to the 20 and 40 mm fibers. This can be explained by the fact that shorter fibers will be more sensitive to misalignment and stress perturbations induced by the clamps. Nevertheless, for most of the fiber types, except S1, M1, and M2, the scatter is within 5–12%. Strain at failure is within 0.7–1.2%, which is rather typical for the CFs.[30,31]

The obtained elastic modulus is pretty similar for all fiber batches, varying within 25–33 GPa. These values are much lower (by an order of magnitude) than those reported for high performance CF. However, they are very close to the values of 33 GPa reported for the isotropic pitch CF[31] or 41 GPa for so-called "high ductility pitch-based" CF.[25] As the lignin precursor was not significantly stretched during processing, the CFs studied here can also be considered as isotropic fibers. However, the strength of the lignin CF is still lower than that of isotropic pitch based CFs (700–800 MPa)[31] or "high ductility pitch-based" CFs (1100 MPa).[25]

Conclusions

Systematic mechanical characterization of lignin based CFs was carried out by performing the single fiber tensile tests of fibers with different diameters and length. The stiffness and strength of carbon fibers were evaluated. The fiber strength was treated by use of Weibull statistical distribution. The two-parameter Weibull distribution was employed and parameters of this distribution were obtained. The experimental results and predictions based on Weibull statistics show a very good fit if distribution parameters for the same fiber length are used. However, if predictions are done for fibers with different length but by use of one set of parameters the accuracy is not as satisfactory, although still fairly good. It is likely that accuracy of predictions can be improved by use of the modified three parameter Weibull distribution. Another factor to consider in order to improve statistical significance of the results would be to increase the number of experimental results. This issue is addressed in the on-going investigation with more comprehensive statistical analysis of the results.

Although strength of the produced fibers (~300 MPa) is still significantly lower than that of commercially available carbon fibers, this is the first evaluation done for softwood kraft lignin-based carbon fibers. It also should be noted that stiffness of the tested CFs (~30 GPa) is comparable with values reported for commercially available isotropic CFs. The estimated strain at failure ranged within 0.7–1.2%, which is rather typical for CFs.

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