

Review Article

# Review on Fundamental Considerations During Lignocellulosic Fiber Characterization in Light Micromechanical Analysis of Their Composites

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## Abstract

Lignocellulose fibers (Cellulosic fibers) are among the major agricultural resources from plant whose potentials are not exploited in some cases and/or underexploited in many cases. If their potentials for industrial application could be exploited, selling the fibers for manufacturing uses would be a win-win situation for both the industries and the farmers, and provide the latter with a much needed source of additional income since composite material reinforced with lignocellulose fibers can be used for diverse application including the production of parts in automotive industry. For this to be successful, it is mandatory to make fiber level characterization. In this process, there are various determinants that affects the characteristics of lignocellulose fiber including agro-ecological zone, plant age from which the fiber is extracted, lignocellulose structure, fiber extraction method and subsequent treatment to enhance properties. This review, therefore, presents the basics of lignocellulose fiber potential and insight into selected deliberation related to fiber level characterization in light of micromechanical analysis for new biocomposite under development. Included in this review, there are considerations to be potted during characterization at fiber level. For fiber diameter measurement and estimation, the following considerations are reported in this paper: measurement method validation, proper cross-section and fiber geometry assumption, lignocellulose structure and internal holes; enough sample consideration, incorporation of analytical method for cross checking. Likewise consideration during estimating fiber density, single fiber tensile strength and stiffness are review and discussed in this review.

## Keywords

Lignocellulose, Industrial Application, Mechanical Characterization, Fiber, Biocomposite

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## 1. Introduction

Due to the increased consciousness of environmental aspect importance and ecological advantages of using renewable resources, there has been a renewed interest in lignocellulose fibers [1-4]. Likewise, the need to replace petroleum based energy systems used in the production of synthetic materials by eco-friendly alternatives is a strong motivation in favor of natural materials [2, 5-8]. Then again, Cellulosic fibers are not abrasive to the processing equipment, have neutral emission of CO<sub>2</sub> and are an important source of income for the people (farmers and others) living in rural areas if their potential is properly exploited [5, 9]. Since they are naturally available in fiber form and don't need further fiber making manufacturing process, they have significant cost advantages in addition to ecological, environmental and industrial application related advantages [4, 8, 10].

One of the major benefits of lignocellulose fibers is their potential to be used as reinforcement in polymer matrix to produce to biocomposite with various advantages [11-13]. Biocomposites are, generally, low-cost with high specific properties, low density and eco-friendly material that can be used for wide range of applications [2, 11, 14-16]. Development of biocomposite will be an alternative way to develop the material which can be particularly used for daily needs of common people whether it is house hold furniture, house and light weight car components and other equipment [5, 7, 17]. This effort to develop biocomposite materials with improved performance for global applications is an ongoing process and the expected future trends also confirm the same. Hence, it is important to study the lignocellulosic structure of lignocellulose fiber as well as their characteristics at macro-mechanical and micromechanical level to possible exploit their industrial potential [7, 18].

## 2. Lignocellulose Structure of Fibers

The lignocellulose structure of a lignocellulose fiber produces substantially affects the characteristic of the fibers [8, 19-21]. It has direct impact on the physical and mechanical characteristics of the fiber and their composite [22-25]. Some fibers contain high cellulose and hemicellulose content and low lignin while others have slightly different composition all the three [8, 19, 26, 27]. For instance, Enset (Ethiopian indigenous plant with scientific name 'Enset Ventricosum'), also called false banana, is characterized by high amount of cellulose above 69.5% and low lignin content of about 5.7% compared to other wood and many non-wood fiber sources [8, 24]. Some have higher amount of cellulose while other contains relatively smaller; the same holds true for complementary parts called hemicellulose and lignin content of the plant fiber. There might, also, be slight value difference between the lignocellulose fiber extracted from different ecological zone and soil condition while age of the plant from which the fiber is extracted has also influence on the difference [28-30].

Again, lignocellulose fiber have a structure that contains shape irregularities, damages, holes, cavity and/or lumen in their structure [1, 27]. For instance, Enset (false banana) fibers have an average length of about 1.66mm, width of 28.5  $\mu$ m, and have wider lumen (25.9  $\mu$ m) and thinner cell wall (2.9  $\mu$ m) [8, 31]. This characteristic varies from one lignocellulose fiber to the other based on various reasons. And, lumen affects, the accuracy of diameter measurement and interpretation which in turn alters density estimation result based on the size of the fiber considered during measurement [1, 27, 31]. So, considering different size of fiber to portray the resulting characteristics of the fiber including density would be crucial [32-34]. For this to be possible there are fundamental considerations, required that are assessed from other previous findings. The main characteristics under focus in this review are the consideration to be made estimating of measuring the diameter, density, tensile strength and stiffness at fiber level.

## 3. Single Fiber Characteristics

### 3.1. Lignocellulose Fiber Diameter

One of the descriptive characteristics of lignocellulose fiber is its tensile strength. And, the tensile strength value of lignocellulose fiber can majority be used as material selection criteria and comparison parameter; and it is one of the most importantly as an input to micromechanical analysis of lignocellulose fibers and their resulting composite [35, 36]. However, the accuracy of tensile strength measurement mainly depends on the accuracy of diameter measurement since the load applied on the fiber axially can easily be known from the universal tensile testing machine display system. Hence, basic considerations to accurately measure the diameter can entail the resulting accuracy and precision of tensile strength of the fiber and micromechanical analysis thereof using this tensile strength [7, 10, 37, 38].

Because of the small size of fibers cross-section, irregularity in cross-section as well as non-uniformity across the length, accurately measuring the fiber diameter is not a trivial task [1, 7, 38, 39]. Several approaches have been reported in the literature to measure diameter under such determinant factors. One of this is to assume the fiber diameter based on manufacturer-specified values with no direct measurements made in their experimental work. It has been found, though, that the diameters of certain types of fibers do deviate from these assumed values and this can have significant effects on the calculation of the tensile properties of a single fiber [23, 39, 40]. High-resolution scanning electron microscope (SEM) images were used to more accurately measure the diameter of fibers [24, 41].

However, it is difficult to conclude the diameter correctness based on the high-resolution scanning electron microscope because of the lignocellulosic structure. Because of their

structure, the measurement can only tell the external diameter. Since lignocellulose fiber incorporates internal hole and/or lumen, the effective diameter that carries the tensile load would not be accurately found using microscopy alone [7, 10, 37]. On the other hand, the accuracy of the measurement has to be verified for it to, even, be accepted as external diameter. This can be done measuring diameter of synthetic fiber with known diameter and provided on datasheet from manufacturer. And, still, the diameter is assumed to be cylindrical while using microscopy though that is not the case. Hence, a synthetic fiber with known diameter should be used and the result is expected to be compared with the data sheet. Simultaneously, multiple spot across the length need to be measured so that a representative cross-section size is found.

The other possible method to estimate the diameter is to measure the mass and length of the fiber and use the density value (if already studied) and implement the following relationship on equation 1 below. Assuming the fiber geometry to cylindrical geometry with circular cross-section, cross-sectional area of the assumed cylindrical geometry of fiber can calculate this relationship; and the result should be compared with data sheet is synthetic fiber is used to validate the process before doing the same analysis for lignocellulose fiber. Density can be calculated using equation 1 below.

$$\rho = \frac{m}{V}, V = AL = L \frac{\pi d^2}{4}, d = \sqrt{\frac{4m}{4\rho l}} \quad (1)$$

Where;  $m$ ,  $\rho$  and  $l$  are mass, density and length, respectively.

Consequently, lignocellulose fibers diameter measurement should consider the following for result accuracy and precision [1, 23, 39, 40, 42].

- 1) Validation of the microscopic measurement using a synthetic fiber with known diameter, given on datasheet, before using the process for lignocellulose fiber. Measuring the synthetic fiber and comparing the two diameters (Measured and the on data sheet). Based on the acceptable difference (>90% agreement), the process can be used afterward.
- 2) Proper assumption about the geometry of the natural fiber (usually considered circular cross-section) and measuring on as many spot as possible across its length. In line, fiber cross-section and associated morphology study is required for more certainty.
- 3) Proper consideration of lignocellulosic structure of the fiber under study and associated content to identify effective diameter of the fiber (cellulose part of the lignocellulose). This is important when the subsequent usage of the fiber is for fiber tensile test and related analysis.
- 4) Proper consideration of the internal hole and lumen, especially when the reason for measuring the diameter is to use it for single fiber tensile characterization, especially strain measurement (Using digital image cor-

relation or direct).

- 5) Due to the variation of the size of different fiber from the same plant, adequate number of samples testing is highly recommended. The fiber diameters not just vary from fiber to fiber but also along the length of the same fiber.
- 6) Comparison with the result found using the relationship between mass, volume and density whenever the density of the material is known. Sometime, measuring the density of the fiber using Gas Pycnometry is required.

### 3.2. Lignocellulose Fiber Density

The density of lignocellulose fiber is one of the most important parameter giving highlight about how it fits to practical applications. In fact, fiber density lignocellulose fiber is essential property which can be used to estimate composite weight before compounding it with the matrix of a known density [33, 34, 43, 44]. It is, also, used to evaluate fiber content for property predictions in fiber reinforced polymer matrix composites [32, 33, 45, 46]. There are two possibilities in the density estimation process of lignocellulose fibers; not known plant density value or the varying density value. The density is not known whenever the lignocellulose fiber under consideration is new and is at early stage of investigation while the variation may emanate from different reason including agro-ecological zone from where the lignocellulose fiber is extracted, the soil type where the plant and characteristics thereof, the plant age from which the fiber is extracted; lignocellulosic structure of the lignocellulose fiber, the condition under which the measurement is take and the measurement method used [9, 14, 32, 47].

Therefore, to boost the density estimation result accuracy and precision appropriate methods should be selected and proper consideration need to be taken into account. And, there are various methods to measure density of lignocellulose fiber but with pros and cons. Some of the possible methods include Archimedes by Weight, Diametric and linear density, Gradient column, Liquid Pycnometry and Gas Pycnometry [32-34, 48-52]. Out of this gas Pycnometry is proven to be the relatively best method for lignocellulose fiber and suggested by number of researchers due to the hydrophilicity of lignocellulose fiber, physical structure and associated challenges; gas Pycnometry (mainly helium gas Pycnometry) being the commonly recommended one [32-34, 53].

Therefore, lignocellulose fibers density estimation should consider the following for accuracy and precision of the result [32, 34, 43, 53-55].

- 1) Appropriate diameter measurement value using the consideration mentioned in section 3.1 above is required for comparison and validation using equation 1 above.
- 2) Setting the upper and lower possible limit taking into account the lignocellulos structure and calculation based on diameter, length and mass measurement with

employing the relationship on equation 1; and with right assumptions.

- 3) Proper drying and desiccation of the lignocellulose fiber is required before every test since lignocellulose fibers are hydrophilic.
- 4) Conducting number of tests is recommended to find representative result since there is difference in characteristics between different fibers from the same plant.
- 5) Comparison with the result found using the relationship between mass, volume and density is advised to enhance the accuracy and precision of the result found.

### 3.3. Single Fiber Tensile Strength

Fiber tensile tests are a way to determine the fiber's mechanical properties before using them as a reinforcement in composite material [7, 10, 37]. It is the preferred method when limited material is available, at material development stage [1, 7, 18]. This would privilege researchers save fiber materials and even matrixes since it allows investigation of the fiber and its composite before compounding. The fiber's tensile properties are needed to perform micromechanical analyses and mechanical modeling of these materials and their composites using limited material [1, 7, 18]. For synthetic fibers, the single fiber tensile properties are often provided by the manufacturers. However, when testing new materials, and especially from natural resources including lignocellulose fiber, datasheets are often not available and need to characterize lignocellulose fibers' tensile characteristics is inevitable [19, 23, 33].

The need for consistent single fiber properties is increasing due the aforementioned reasons. Hence, fiber manufacturers, or cultivators in the case of lignocellulose fibers, should be able to rely on fiber properties to measure the quality of the produced material [10, 18, 38, 42]. Unfortunately, the literature reveals a large range of the measured lignocellulose fiber properties and number of reason for this variation can be mentioned. A part of this variation can be attributed to the inherent variability in plant materials, but this is unlikely to be a complete explanation. There is a need for a consistent fiber testing method where the scatter is only a function of the material variability and not of the testing setup [18].

Therefore, proper method to make single fiber tensile characteristics is needed; and digital image correlation is one of the preferred methods for such application [1, 7, 18]. There are some challenges when testing lignocellulose fibers' tensile characteristics, that can lead to a wide range of results [1, 7]. The guiding principle during such measurement is that the tensile strength depends on the applied load and the cross-sectional area of the fiber under consideration.

Hence, the following deliberation needs to be taken while doing single fiber tensile strength test using digital image correlation [7, 10, 12, 36, 56].

- 1) Due to the irregularity of single lignocellulose fiber cross-section, sound cross-section assumption and

consideration need to be made when using microscopy. And, deriving the diameter from the density estimation value using equation 1 above and comparing it would enhance the accuracy.

- 2) Since the fibers are small in size and mounted on some other paper material; proper care need to be made either to avoid slippage or consider whenever it happens inevitably.
- 3) Consideration of the preloads applied to straighten the fiber mounted on the universal tensile testing machine is also important.
- 4) The model used during the digital image correlation using VIC-2D software package has to also be the proper one.
- 5) Sometime, back-calculation of the characteristics of the composite of the fiber under study in a unidirectional orientation might be required to validate the result at single fiber level.

### 3.4. Single Fiber Stiffness

The other important parameter to compare materials and select it for specific use is to identify its stiffness at fiber level. Such characterization is mandatory to exploit potential of lignocellulose fibers since most of them don't have available datasheets [1, 7, 10, 18]. Because of diameter disparity, shape irregularity, lumen and their structure, the strength values of single lignocellulose fibers fluctuate substantially in wide ranges. However, stiffness is relatively stable. Consequently, it can be used to describe lignocellulose fibers better and to make profound comparison among materials [1]. Hence, appropriate stiffness estimation is needed and result accuracy and precision depends on various parameters that allow calculating in a deterministic way.

Digital image correlation technique mentioned in section 3.3 above as a powerful tool to measure single fiber tensile properties is still one of the preferred methods to estimate single lignocellulose fiber stiffness; similar procedure used on the single fiber test could be implemented [1, 7]. Based on the same token about single fiber tensile strength, there are strategies to be used to enhance stiffness estimation accuracy and precision employing direct and indirect measurement methods [1].

Hence, the following considerations are recommended when estimating lignocellulose fibers stiffness [1, 10, 15, 56].

The accuracy of stiffness estimation result depends on the accuracy of diameter, length, density, and fiber mass measurement. Thus, the accuracy and precision enhancement recommended on these parameters need to be given due consideration following the recommendations mentioned above.

The fiber tensile tests as a way to determine the fiber's mechanical properties is required and the stiffness can be either can be calculated directly or estimated using the technique called digital image correlation.

Comparison of the direct method with the method that



employs digital image correlation is recommended for validation of the result.

## 4. Conclusion

With the majority of the world's fiber use relying on wood, cotton and petrochemicals, the use of alternative fiber from natural sources in manufacturing is rapidly gaining importance in reaching the Sustainable Development Goal's on innovation and responsible production. Lignocellulose fibers are among the major natural resource in this regard. However, the uses of this natural resource demand their appropriate characterization before the exploitation of their potential. For this to be possible, at least, the following parameters should be investigated for specific fiber under consideration; appropriate diameter measurement, density estimation, single fibers tensile strength and its stiffness thereof. And, there are various factors affect the characteristics of lignocellulose fiber including agro-ecological, plant age, extraction mechanism and subsequent treatment should be taken in to account while estimating all the four parameters including diameter, density, tensile strength and stiffness.

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## Conflicts of Interest

The authors declare no conflicts of interest.

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