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Effects of Lignocellulose Structure on Single Fiber Tensile Characteristics Portrayal: A case of Enset fiber

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Abstract

Fiber-level tensile characteristics are vital for micromechanical analysis and mechanical modelling of materials and their composites. This portrayal depends on diameter estimation accuracy, as the applied load is determined from the testing machine. Inline, natural fibers possess an internal cavity; the diameter found using microscopy denotes the external diameter which is larger than the diameter pertaining to actual load-carrying cross-section. This study presents a new approach that estimates diameter considering lignocellulose structure and hydrophilicity, thereby enabling the portrayal of more accurate tensile strength values. First, the fibers' diameter is measured using a laser microscope, on various spots axially and the internal cavity was then considered to determine the actual diameter. The density of milled fibers is measured using Pycnometry. The diameter which relates to a solid load-carrying cross-section is identified using the relationship between density, mass and volume. The experiment design was framed and analyzed using Python and JMP Pro 13. The measured density of Enset is 1.38 g/cm3. The average overestimation of microscopy result is significant; it is 27.7µm which is about 21.8%. This underrates the actual tensile strength of Enset fiber by about 37.5%. That is σext=0.627σact. This, in turn, would affect micromechanical analyses and mechanical modelling. Thus, the need to consider lignocellulose structure for testing the tensile strength of Enset fiber is inevitable and the method utilized in this study can be used for other natural fibers of the same nature customizing the context.

1. Introduction

The properties of natural fiber-reinforced composites depend upon the characteristics of the fibers considered [1]-[3]. The tensile tests performed on fibers play a key role in establishing the mechanical properties of the fibers, thereby aiding the assessment of their viability for utilization as reinforcement materials [4]-[6]. This particular approach is the preferred option when faced with limited resources in the material development process [6]-[8]. And, portrayal of the tensile properties of fibers is essential for conducting micromechanical analyses and mechanical modelling of fibers and their composites [7], [9], [10]. Manufacturers provide fiber properties for synthetic fibers; however, for natural fibers, datasheets are often not available. The absence of data for natural fibers necessitates the characterization of plant fibers' tensile characteristics when evaluating new materials [11]-[13].

Single fiber tensile strength is affected by both the applied load and the cross-sectional area of the fiber [11], [14], [15]. The exerted load is derived from the universal testing machine display system. Yet, the accuracy of diameter estimation affects the cross-sectional area of the fiber. Some researchers used microscopy with certain assumptions to measure external diameter but they didn't consider the inherent lignocellulose structure [5], [8], [16]. Since natural fibers have internal cavities, it is difficult to conclude the diameter measured using microscopy to be the effective load-carrying cross-section's diameter [17], [18]. Literature reports different diameters for the same natural fibers. There are also studies which use image analysis software to establish load-carrying cross-sections, but are limited to account for the non-uniformity of the cross-section along the length [19], [20]. Thus, it is crucial to determine the appropriate diameter corresponding to a solid cross-section carrying the load. The diameter of a fiber is also contingent upon the density, and other various factors [5], [8]. The lignocellulose structure affects natural fiber's diameter [18]. Besides, Enset has a 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 [18], [21]. There is a slight variation in the cellulose and lignin content found in Enset fibers extracted from different zones and plants of varying ages [22]–[24]. So, the fiber length, width and internal holes are vital during density measurement and subsequent result interpretation. Enset fibers have an average length of about 1.66 m, width of 28.5 µm, and have wider lumen (25.9 µm) and thinner cell wall (2.9 µm) [18], [25]. These values certainly affect the estimation of the effective diameter and in turn, affect the tensile characteristics of a single fiber.

Therefore, the need for a method to analyze the effective diameter that considers the lignocellulose structure of the natural fiber is required. It is also important to consider other factors related to the testing machine, digital image correlation facilities and software thereof [6]–[8].

Considering every important determinant, in this regard, it is possible to estimate the representative diameter which can result in the appropriate tensile characterization of a single fiber [4], [5], [26], [27]. The result can be used as an input for micromechanical characterization and mechanical modelling of their composite. As a result, this investigation focused on improved methods for carrying out diameter estimation and analyzes its effect on single fiber tensile analysis. A better approach has then been suggested.

2. Materials and methods

2.1. Chemicals

Enset Fiber/False banana: Fibers (Fig.1) were sourced from Ethiopian (Ensete Ventricosumn) from Kokosa, Oromia, Ethiopia, found at 2627 m altitude with minimum and maximum annual average temperature of 12 and 180C, respectively [8], [28], [29]. Fibers obtained from the Enset plant were manually extracted using an in-house developed technique described in Figure 1 (C) below. These fibers were obtained from 3 distinct stages of the plant's growth, specifically 1, 2, and 3 years after the pulp underwent the initial [6].

Fig. 1. A) Enset Plant, B) Enset Plant, C) Manual Extraction and D) Extracted Fiber.

Steel fibers: Cold drawn stainless steel (*type 316L*) fibers with a stiffness (Young's modulus) of 193 GPa and a diameter of 30µm supplied by *NV Bekaert SA* is used to validate the method using a material with known diameter and tensile properties comparing against the measured value using digital image correlation.

The first step involves the extraction and preparation of fibers before they are brought to the lab (Fig 2,). These fibers are conditioned in a room kept at a RH of 50% and 21oC for 3 weeks. Afterward, the diameters of Enset fibers were measured using Laser Microscope (Keyence,VHX-x1 series) on 25 spots across the 10 cm length. This measurement is limited to finding the external diameter, and natural fibers contain an internal cavity. The fiber's lumen distribution is not uniform throughout the length. Hence, the solid cross-section equivalent diameter needs to be estimated; and the following relationship on Equation (1) below can be used.

$$\text{Density } \dot{\rho} = \frac{m}{v} \quad \text{But, } V = AL = \frac{\pi d^2}{4}\text{L} \quad \text{thus, } \quad d = \sqrt{\frac{4m}{4\dot{\rho}l}} \qquad (1)$$

where; m, $\dot{\rho}$ and l are mass, density and length, respectively.

Density is measured using Helium Gas Pycnometry. The fibers were cut to a length of 10 cm, dried for 24h at 60 0C and later cooled using a desiccator without absorbing moisture. In the latter state, the mass of the fibers was measured using an analytical balance with an accuracy to 10-5g; Mettler AT 261 Delta Range. And, fibers are cut to different lengths (powder, 1, 3, 5 and 10 mm) to measure the density. Each fiber weight is recorded on the machine using the data input interface. Then, the samples are put into the Pycnometer using the selected volume cell after calibration using the two standard spheres with a volume of 56.56 cm3 and 2.15 cm3. In this process, all three available volume cells are used; their volume is 147.39 cm3, 27.21 cm^3 and 11.43 cm^3 for the large, small and micro respectively. Also, the effects of the uniformity of purging duration have been checked by altering the three-purging duration (5, 10 and 15 min).

The tensile test is done using ASTM C1557-14 standards. A result from steel fibers with known stiffness measured using the same method was used as a benchmark [7]. The fiber was glued onto a paper frame using a double-sided glue roller (Permanent Pritt glue roller, Henkel) and adhesive (SICO MET 8300). Fig 2A below shows the paper frame for a test gauge length of 50mm. The frame facilitates sample mounting and fibre alignment. Tensile tests were performed on an Instron 5943 equipped with a 100N load cell according to the ASTM C1557-14 standard in a conditioned environment at 50% RH and 21 °C. The frame was pneumatically gripped with a gripping force of 200N. A pre-load of a maximum 0.01N was applied to the fiber to straighten it. Fiber straightness is critical when the strain is to be derived from the crosshead displacement, especially for comparison of the result with Digital Image Correlation (DIC). The crosshead displacement rate was chosen according to the ASTM C1557-14 standard, which suggests achieving fracture within 30s of testing. For the investigated fibers, this translates to a crosshead displacement rate of 1mm/min.

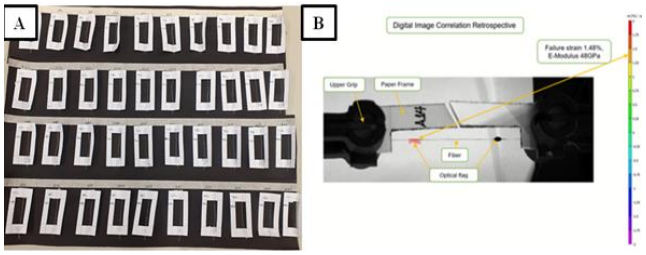

Fig. 2. Specimen preparation for SFTT (A) and digital image correlation (B).

Glueing, optical flag and speckle formation - potent glue is used to fully make the fiber rigid around grip. Double-side glue is used to straighten the fiber at the contact point of fiber and paper and SICO MET 8300 is used. Optical flag is prepared using TIPEX followed by speckle formation and dot making using the tick marker to make visible spot on the fiber.

Fiber straightness and preload - straightness during attaching and gripping is vital; it affects the strain value estimation on both methods since the straightening stage itself is taken as elongation. Hence the fiber has been straightened by using of a preload less than 0.01N.

Digital Image Correlation (DIC) - Images were processed with Vic 2D 2009 (Correlated Solutions, Columbia, USA) correlation software to determine the pixel displacement of both optical flags. During DIC process, images before elongation starts and after failure have been filtered, first. Initial guesses have also been conducted to check whether it is possible to correlate before starting the analysis and assist the correlation process if required. In case the initial guess faces problems, going to the picture where the correlation stops and assisting with the possible options is required.

3. Results and discussion

3.1. Optimal experiment design

The experiments were designed using the DOE platform of JMP 13 pro software. Constraints were imposed to ensure that the experiment closely mimics real-life situations. Following the minimum number of experiments suggested by JMP, extra tests were done to increase the reliability of the results. 24 tests were suggested by JMP for fiber extracted from each age plant; yet a total of 40 tests were considered to enhance the category. It is crucial to note that natural fibers exhibit variations and fluctuations in their properties, not only among different fibers from the same plant but also within the same fiber being examined. Therefore, it is essential to incorporate supplementary tests to enhance the descriptiveness of the results to the scenario. The estimation of the diameter for each fiber in each category has yielded the findings presented in section 3.2.

3.2. Diameter estimation

The sample of image pertaining to the measurement of Enset fiber diameter conducted using a laser microscope is illustrated in Fig. 3 below. The smallest diameter measured using laser microscope is 50.1µm while the average and large fiber diameter are 163.7µm and 298µm respectively. Yet, 97.8% of the fibers fall in the range between 70µm and 240µm; while 85% of fibers diameter falls between 90µm - 210µm. Conversely, the external diameter of Enset does not maintain a perfectly cylindrical shape with a uniform circular cross-section, as illustrated in Figure 4 below. It contains groove-like structure on the surface.

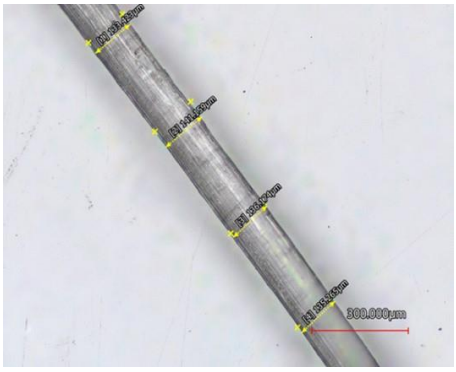

Fig. 3. Microscopic image of Enset fiber diameter.

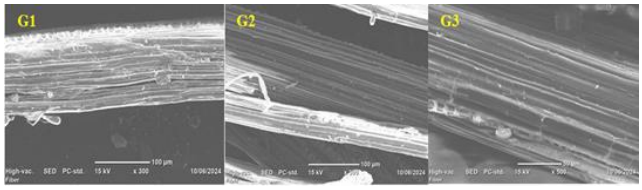

Fig. 4. SEM image of Enset fiber (G1, G2, and G3 ordered in increasing plant age).

This diameter found using laser microscope measurement can only show an external diameter that is greater than the actual load-carrying diameter during a single fiber tensile test. Since diameter and density are inversely related, the overestimation of the diameter underestimates the density. Thus, the density calculated using equation 1 above considering the largest possible diameter found using a laser microscope could be used as the minimum possible density when commencing density measurement using helium gas Pycnometry. The following Table 1 shows a sample of density values found using diameter found using laser microscopy.

Table 1: Density derived from diameter measured by laser microscope.

S.N	S1	S2	S3	S4	S5	S6	S7	S8	S9	S10	Mean
Diameter (µm)	171	185	78	201	69	82	60	50,12	101	141	113,81
Length (mm)	101	99.5	100	100.5	101	101	98.7	100	100	100.8	100.25
Density (g/cm^3)	1.079	1.319	1.184	1.011	1.207	1.236	1.298	1.136	1,273	1,287	1.203

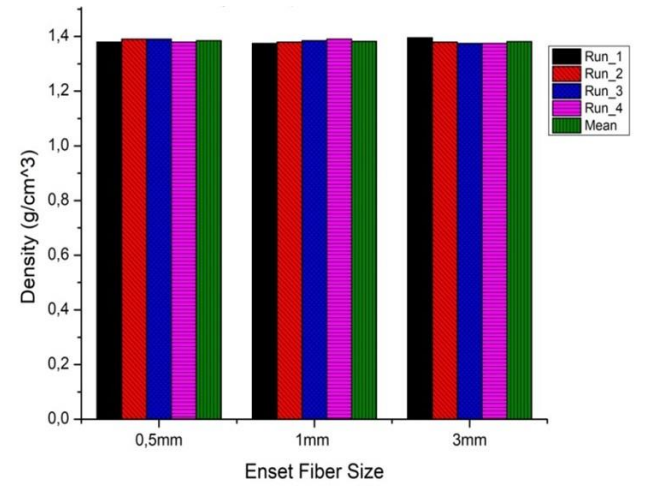

Fig. 4. Enset fiber density estimation results.

Now, considering 1.203g/cm^3 as the lower limit of expected density during measurement using helium Pycnometry would help reduce error and give insight into the expected accurate density. Using this limit and other important factors including moisture content, fiber size, purging pressure and duration, and volume cell consideration the following density values shown in Fig. 4 are found.

Fig. 4 above shows the density of Enset, with an average density being 1.38gcm-3. This particular value is used jointly with Equation 1 for each fiber, where the cross-sectional areas are computed by taking into account the diameter and then recorded on the universal testing machine. Once the diameter for each fiber under consideration is properly found, the preloads applied to straighten the fiber should be given due care and it is elaborated upon in section 3.3.

3.3. Preloads considerations and failure spot

The preload used to straighten the fiber greatly affects the resulting tensile strain since the machine drives it only from the crosshead displacement. When a higher preload is utilized to align the fiber, the tensile test may commence with the fiber already under strain, leading to strength and strain values that do not accurately reflect the material's inherent properties. It should, therefore, be in the acceptable range. The result found from the universal tensile testing setup analog data revealed the result on the graph shown in Fig. 5. The result has been used as an input to the simulated correlation system. Fig. 5:

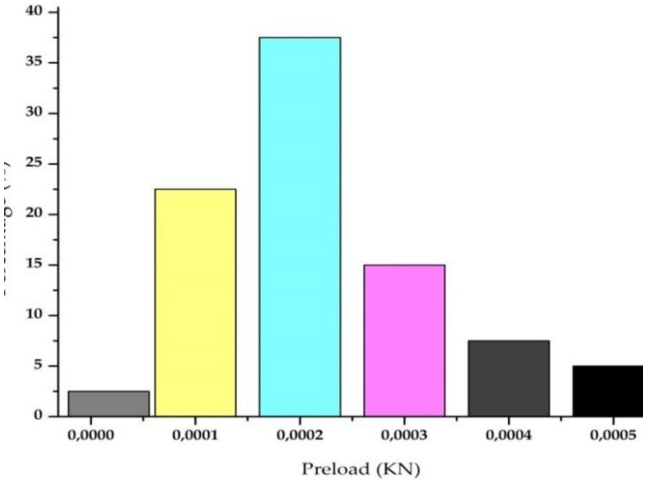

Fig. 5: Ranges of the preloads applied.

From the result presented in Figure 5 above, 72.5% of the testing was conducted with a preload of less than 0.00025KN. Given the minimum strength observed of nearly 200MPa, this accounts for less than 0.05% of the minimum strength achieved. The applied load has a minimal effect on strength, with only a small percentage being insignificant [7], [27]. Preloads of 87.5% are below 0.0003KN, and 95% of preloads are less than 0.0004KN. The use of smaller preloads ensures the reliability of test results. Yet, no tests have been conducted with preloads exceeding 0.0005KN. The preloads fall within the acceptable range, prompting the need to investigate the failure spot since it plays a key role in determining the fiber's attribute during a single fiber tensile test. Failures near the grip may be attributed to either the gripping load or the adhesive applied at the attachment point of the fiber to the paper. The findings related to the failure spot are shown in Fig. 6.

Figure 6 indicates that 82.5% of fiber failures occurred at the midpoint, showing that these failures are primarily due to the applied load rather than grip. Conversely, the remaining 17.5% of failures were located around the grip and are not considered in the subsequent.

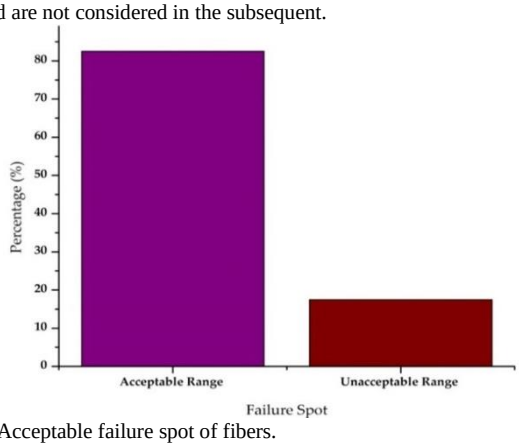

Fig. 6: Acceptable failure spot of fibers.

3.4. Tensile Strength Characteristics

The majority of the failure falls within the acceptable range, as shown in Figure 6 above. Thus, a study reinforced with DIC described in section 3.5 below is carried out to determine the tensile strength of each fiber failing the acceptable range. Besides, evaluation is done to analyze the failures that occurred at the desired spot and under the permissible preloads. Fig. 7 below presents the range of tensile strength of a single Enset fiber.

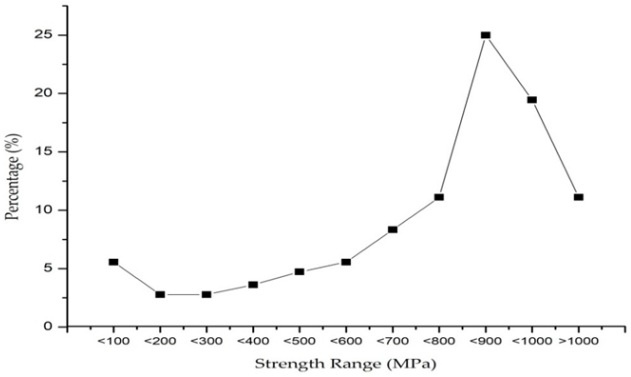

Figure 7: Tensile strength of single Enset fiber.

Initially, it was observed that nearly 5% of the fibers experienced failure before attaining strength of 100 MPa. This value deviates significantly from the average strength. The study reveals that only 14.72% of the fibers possess strength below 500 MPa. On the contrary, a significant share, 69.44%, of the fibers fall within the strength range of 500-1000 MPa. So, the 5% of fibers failing before reaching strength of 100 MPa is not indicative of the overall scenario. It is, thus, suggested to neglect e the early failure since it does not provide an accurate depiction of the fiber properties. The probable cause of this premature failure can be attributed to the damage incurred during the manual extraction process, resulted from the indentation. Also, the storage conditions might also play a role in this untimely failure. This finding shows that a significant proportion of the fibers, specifically 25%, exhibited a strength falling within the range of 800 MPa to 900 MPa. Equally, the majority of the fibers, nearly 70%, displayed a strength surpassing 500 MPa. Also, more than 30% of the fibers showed a strength exceeding 900 MPa, while 11% achieved the highest possible strength of over 1000 MPa. It is vital to note that there were also instances where fibers with the smallest diameters reached a strength of 1000 MPa, albeit infrequently. These results are consistent with the existing literature, as shown on Figure 8 below [7], [11], [19], [21], [30].

All fibers showed in Figure 8, except Buriti fiber, exhibit 1000MPa strength for a diameter less than 60µm. For Jute and coir fibers, this threshold is below 20 µm. This result confirms the possibility to attain strength above 100MPa for natural fibers; the fibers with smallest possible diameters are those resulting in higher strength value.

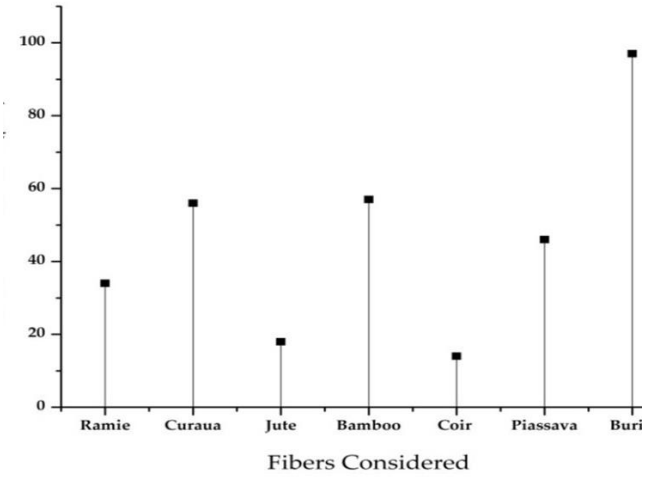

Fig. 8: The diameter of natural fibers that produces strength of 1000MPa.

3.5. Digital image correlation

The correlation established using Vic2D software ensures that the acceptable error range, which is below 5% (0.05), is maintained for all data points and tests. By analyzing the displacement in the images and calculating the corresponding strain, the tensile strength and tensile modulus of Enset fiber is determined. The comprehensive results of the tensile strength for various test categories conducted under tension are presented above in Section 3.4. Based on the DIC result, above 84% fibers strength falls between 41MPa-723MPa for first category and 67 MPa-923 MPa for third category. This range is given without considering the remaining 16%; 5% falling before attaining 100 MPa and 11% attaining above 1000 MPa. This is consistent with literatures reporting about other related natural fibers [6]-[8], [11]. The stiffness is calculated using the strain derived image correlation for the same material.

The observed minimum strength limits in the data presented may be ascribed to a range of factors, such as the manual fiber extraction technique. This method has the capacity to induce fiber damage, consequently leading to premature breakage under loading conditions and yielding reduced strength values. Another subset of fibers exhibiting low strength failed either at the clamp or near the clamp, potentially causing damage that affects the Poisson ratio. Thus, enhancing the extraction means to preserve the fiber during extraction will minimize breakage and increase the strength as a result.

3.6. Effects of lignocellulose structure on tensile characteristics

The correlation between diameter and strength can be described as an inverse and quadratic. The strength is inversely proportional to the square of the cross-sectional area, resulting in a hyperbolic association. This is derived from the relationship in the following Equation (2) below.

$$\sigma(d) = P \Big/ \left(\frac{\pi d^2}{4}\right) \qquad (2)$$

The correlation between the diameter measured using Equation (2) and the corresponding tensile strength of single Enset fiber has been established through software analysis. The equation derived from analysis is expressing the relationship in Equation (3)

$\sigma(d) = 0.02243553d^2 - 8.6359d + 1379.96$

And, Enset can have a Lumen exceeding 29 µm. This affects the tensile strength greatly; actual tensile strength of Enset fiber is underrated by about 37.5%. That is $\sigma_{ext} = 0.627\sigma_{act}$. Hence, it is crucial to consider the lignocellulose structure during characterizing tensile strength of Enset fiber and this can be used to other natural fibers of the similar nature.

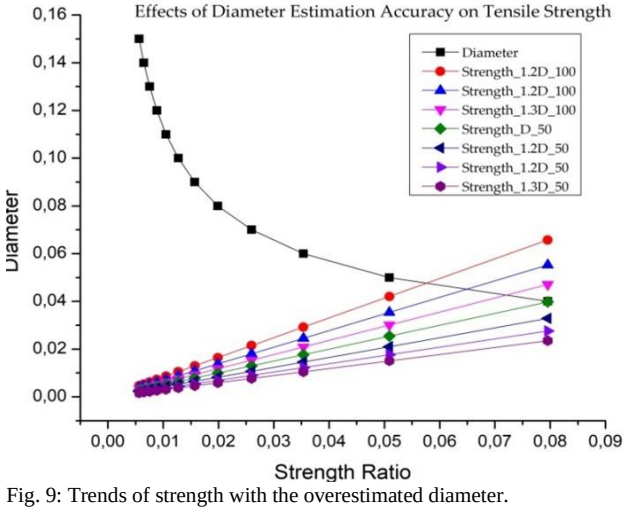

Fig. 9: Trends of strength with the overestimated diameter.

The portrayal on Fig. 9 above is the response of the fiber for the loading of 50 N and 100 N applied to different diameter. The relative increase and decrease in fiber strength due to the underestimation and overestimation of fiber diameter respectively is shown on. The effect of error during diameter estimation is greater when the load applied is larger. And, literatures mention that the lumen size of Enset fiber is 25.9 µm which is relatively larger than some other related natural fiber [18], [25]. Based on the findings of this study, the lumen value could potentially exceed 29µm as indicated by the back-calculation derived from the Pycnometry analysis. Therefore, it is suggested to exercise caution when estimating the effective diameter and subsequent tensile strength of the individual fiber.

4. Conclusions

The mechanical properties of Natural fiber, considering, is characterized in this study. The inverse and quadratic relationship between tensile strength and fiber diameter leads to a significant effect on strength value when a small underestimation or overestimation of the diameter is made. The method utilized in this study, which strengthens the microscopic measurement of diameter with density estimation via helium gas Pycnometry, elevates the precision of the diameter and in turn of the tensile strength of the fiber. Still, in contrast to the direct measurement technique that calculates strain by analyzing crosshead movement, the approach utilized in this investigation (DIC) produces notably better results owing to its enhanced ability to account for slippage effects. The average overestimation of microscopy results revealed to be crucial and it can be as high as 27.7µm which is about 21.8%. This results in the underrated actual tensile strength of Enset fiber by about 37.5%. This, in turn, would affect micromechanical analyses and mechanical modeling. This outcome aligns with the findings reported in the literature regarding the properties of flax fiber, which indicate variations in the levels of underestimation and overestimation. It is, thus, crucial to consider the lignocellulose structure during characterizing the tensile strength of Enset

fiber and the method utilized in this study can be used for other natural fibers of the same nature customizing the context

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