Determinants of Single Natural Fiber Stiffness Estimation Accuracy and Enhancement Possibilities

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Abstract
Characterization is mandatory to exploit potential of natural fibers since most them don't have available datasheets. Coping with diameter disparity, lumen and their nature, the strength values of single natural fibers fluctuates substantially in a wide ranges. But, stiffness is relatively stable that it can be used to describe fibers and to make profound comparison among materials. Hence, appropriate stiffness estimation is needed and result accuracy is depending on various parameters that allow to calculate in a deterministic way. This paper describes a strategy to enhance stiffness estimation accuracy and precision employing direct and indirect measurement methods jointly with digital image correlation techniques. Enset fiber is used to display the efficacy of the methods. Using these methods, materials with known stiffness are considered as a benchmark and above 5% accuracy enhancement has been achieved compared with conventional method. The effect of errors can potentially lead to wrong conclusion about the properties of the material. Plus, the effect of each of the influencing parameter on stiffness result has been characterized. Thus, stiffness can be used to typifies the tensile property of a fiber and its estimation accuracy, which can be enhanced using digital image correlation, plays significant role to compare materials.

Keywords: Natural Fiber, Stiffness, Digital Image Correlation

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 natural fibers (Kumar & Anbumalar, 2015). Also, 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 (Monteiro et al., 2010). Natural fibers are not abrasive to the processing equipment, have neutral emission of CO_2 and are an important source of income for the people living in rural areas (Alves Fidelis et al., 2013). Plus, fiber reinforced composites have been found to be the most promising material available and they are gaining more attention as demands for light-weight materials with high strength for specific applications are growing (Rajak et al., 2019).

Polymer-based composite materials are ideal for applications where high stiffness-to-weight and strength-to-weight ratios are required (Gloria et al., 2011). Rapid growth in manufacturing has led to the need for the improvement of materials in terms of strength, stiffness, and lower cost with improved sustainability (Rajak et al., 2019). Tendency to exploit the potential of natural fiber is increasing and growth of the natural fiber reinforced composite has made a substantial impact on the polymer composite research (Vigneshwaran et al., 2020). Natural fibers abundance and possibility to be used to reinforce polymers to obtain light, stiff and strong materials is appreciable (Maleque et al., 2007). Indeed, far-ranging studies on natural fibers, including sisal, flax, kenaf, and bamboo, demonstrate that natural fibers possess amazing potential as an effective reinforcing phase in composite materials (Salih et al., 2020). The benefits of using lignocellulose fibers in place of synthetic fibers relate to their specific properties, such as light weight, low cost, high specific properties, low density, good thermal properties, eco-friendliness, and biodegradability (Salih et al., 2020). Due to their high specific strength and modulus, natural fiber reinforced polymer composites are receiving widespread attention (Ranga et al., 2014). To choose among wide range of available natural fibers, characterization at fiber level is required. Ethiopian Enset fiber, extracted from Enset plant which serves as a staple food for above 20% (> 20mil) of the population, with desirable potential and abundance is used for this study (Borrell et al., 2020). This fiber has been considered because of the introduction of regulatory norms demanding more environmental-friendly products, the use of natural fibers as composites reinforcements materials increased recently (Arun Kumar et al., 2017).

There are some problems when testing natural fibers, that can lead to a wide range of results (Haag & Müssig, 2016). This can emanate from different sources. Firstly, the literature does not always clearly state whether tests are performed on an elementary fiber or a technical fiber (Depuydt et al., 2017). Secondly, when testing technical fibers at very short gauge lengths, elementary fibers can get gripped from end to end. Besides these problems, some other test related influencing factors are known: the strain rate, the environmental conditions and the gripping method should thus always be reported (Kim et al., 2015). The main variables among them are the method used to isolate the single plant fibers and a suitable gripping system (Yang et al., 2016). The mechanical properties of single fibers may be influenced by many factors, such as moisture content, microfibril angle, plant species, as well as the age and location (Yang et al., 2016).

Still, the accuracy of strength and stiffness estimation result depends on the accuracy of the diameter, density, mass measurement. So, fiber tensile tests as a way to determine the fiber's mechanical properties is required (Depuydt et al., 2017). It is the preferred method when limited material is available and cost reduction is crucial, in material development stage (Abdela et al., 2020). Various determinants including fiber slippage can contribute to the underestimation of the stiffness since this is not considered in the correction procedure (Depuydt et al., 2017). Hence, it has to be addressed accordingly. Besides, the fiber's tensile properties are needed to perform micromechanical analyses and mechanical modelling of these materials and their composites (Depuydt et al., 2017). This paper contains a means to enhance the estimation result accuracy.

2. Material and Methods

2.1. Material

Enset Fibers extracted, using an in house developed technique meant only for extracting the pulp (Edible part Enset), from west Arsi, Ethiopia is used. Enset plant with three different ages, after maturity time (1, 2, and 3 years) with no previous depiction for their mechanical properties are considered. Plus, different material for preparing the specimen including paper frame on which the fiber is attached, attaching glue, speckle formation spray, optical flag formation TIPEX are used.

Figure 1. A-Enset Plant, B-Extracted Enset fiber, C- Fiber gripping and orientation

2.2. Method

Assumption

- The single fiber is assumed to be cylindrical and the cross section circular
- Fiber diameter is assumed to be the average fiber diameter.

Fiber preparation

The density of the fibers was determined using a gas Pycnometer, Beckman model 930, in which helium gas was used as the displacement medium. During material preparation prior to the density measurement, the fibers were cut to different length and vacuum dried for 24hr at 60 ^{0}C (Kim et al., 2015). The density of the powder is used because of assumed circular cross section and solid cylindrical shape. The mass of these fibers to determine the density is then measured using sensitive balance with an accuracy of 10^{-5}g. The measured density was 1.49 ± 0.05 g/cm^3. For the single fiber tensile tests, the fibers were cut to a length of 10cm, dried for 24hr at 60 ^{0}C and subsequently conditioned at 50% relative humidity (RH) and 21 ^{0}C for 24 hr. In the latter condition, the mass of the fibers was measured (Abdela et al., 2020). Then, the fiber is attached to the paper frame using the glue and then optical flags are formed.

Fiber tensile test setup

To check the reliability of the method, result from steel fibers with known Young's modulus measured using the same method was used as a benchmark (Depuydt et al., 2017). The fiber was glued onto paper frame using a double-sided glue roller (Permanent Pritt glue roller, Henkel) and cyanoacrylate adhesive (SICO MET 8300). Fig. 1C shows the paper frame for a test gauge length of 50 mm and the position of the fiber in this frame. The frame facilitates sample mounting and fiber alignment in the grips. 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 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 DIC. The crosshead displacement rate was chosen according to the ASTM C1557-14 standard, which suggests to achieve fracture within 30s of testing. For the investigated fibers, this translates to a crosshead displacement rate of 1mm/min.

Digital Image correlation

The specimens were attached to a paper frame and two optical flags with an approximate diameter of 3 mm were attached to the fiber surface using white correction fluid or a dot made with black marker is applied. The gauge length was fixed to 50 mm to manipulate the fibers easily, Enset fiber from three different ages were successfully tested; for each age, 40 tests were performed. Tests with different gauge length to eliminate slippage of fibers were not considered. In natural fibers, also the stiffness can be affected when the gauge length becomes so small, that at least one elementary fiber is gripped from end to end, this results in an increase in the stiffness (Shah et al., 2016). For the registration of the images during tensile testing, a digital camera (Limess messtechnik & software GmbH, Krefeld, Germany) was used. The distance between sample and camera was 1m. The analog data of the tensile bench was send to the computer and compared with the visual information to find the strain at failure. Finally, the image and analog data are analyzed using Vic2D 2009, corelated solution software. The result is then compared with the crosshead movement based results.

3. Result and discussion

3.1. Strength value wide range

The strength value of the single fibers considered lies in wider range though majority of the values are in a certain allowable ranges, as depicted on the figure below.

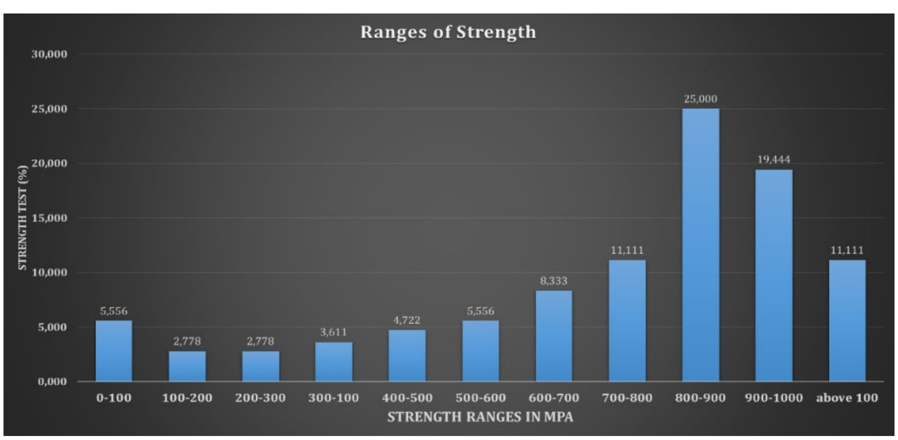

Figure 2: Strength value variation

As it is evident from the above figure, the value of the strength of individual figure varies in different ranges though majority of the fiber strength is in the range of between 500Mpa to 1000Mpa. Because of this the comparison of natural fiber using stiffness play significant role. It is also important to find to Stiffness result enhancement possibilities is crucial in addition to using it as a reliable means to compare it as it is described here below.

3.2. Density estimation effect on stiffness

Diameter of the cylindrical fiber which is important to find the strength and stiffness. Density is required to calculate diameter using the following relationship.

$$\rho = \frac{4m}{\pi d^2 L} \Rightarrow d^2 = \frac{4m}{\pi \rho L}; d = \sqrt{\frac{4m}{\pi \rho L}} \qquad (1)$$

Density estimation accuracy directly affects Modulus with large magnitude. An increase of density by 30% proportionally increases the modulus by 30%, ideally as shown in equation 2 below.

$$E = \frac{\sigma}{\varepsilon} \Rightarrow E = \frac{4F}{\varepsilon \pi d^2}; Ed = 1.69 Ed_{1.3} \qquad (2)$$

Where d is original diameter $d_{1.3}$ is the diameter with 30% error

Some of the main factors affecting density measurement using pycnometer includes the following. Firstly, the standard fiber drying and cooling without moisture absorption need to be secured. Once the fiber is ready, it is placed into cups with known volume which are in three range (micro, small and large). The larger the cup selected, the better the result is. For the same cup, the larger the amount of material inside, the better is the accuracy. Then, the sample is weighed and placed into the system. One of the major factor affecting density result is the purging of 1Psi for 1 minute. 1 minute is appropriate for hydrophobic powder but natural fiber require more time. For Enset, accuracy has been enhanced when purging of 1bar pressure for 10-15 minutes. 15 minutes purging enhanced the result by 0.12 g/cc^3, about 8%, compared with 1 minute purging. This translates into 8% variation on stiffness, about 3GPa, values result. This may vary for different materials and 15 minutes gave the best result for Enset. After purging, four runs for density measurement were performed using ideal gas law principle between reference and cell volume. It is advisable to make similar waiting time for pressure drop for all four runs in case the pressure drop don't get stable due to nature of fiber.

3.3. Mass error effect on Stiffness

For known density (1.49g/cc^3) and length of the fiber (100mm), error during measuring mass also have an significant effect on diameter estimation and in turn on stiffness. Since the weight of a single fiber is very small, small error can take significant percentage of the value. The relation between mass and diameter is found using equation 4 below.

$$\rho = \frac{4m}{\pi d^2 L} \Rightarrow d^2 = \frac{4m}{\pi \rho L} \qquad (3)$$

As it is shown on the Fig 2 below, an increase of mass by 30% increases diameter by 14% and in turn the modulus by 26%. Hence, mass has to be as accurate as possible and accuracy and sensitivity of the balance has to be given a due attention. Sensitivity of the balance used is critical. The treatment condition has to be as per the standard and reported.

$$d_{new} = \sqrt{1 + m_{error}} * d_{old} \qquad (4)$$

The effect of mass error on stiffness follows similar trend as diameter shown on Fig 3 below with coefficient shown on equation 4 above.

3.4. Diameter error effect on Modulus value

Diameter of the cylindrical fiber can be found using equation 3 below.

$$\rho = \frac{4m}{\pi d^2 L} \Rightarrow d^2 = \frac{4m}{\pi \rho L}; d = \sqrt{\frac{4m}{\pi \rho L}} \qquad (5)$$

An increase of diameter by 30% decreases the modulus by 59% and decrease in diameter by 30% increases the modulus by 98% as shown on Fig 3 below. Attention has to be taken into account since its decreasing value almost triples stiffness variation.

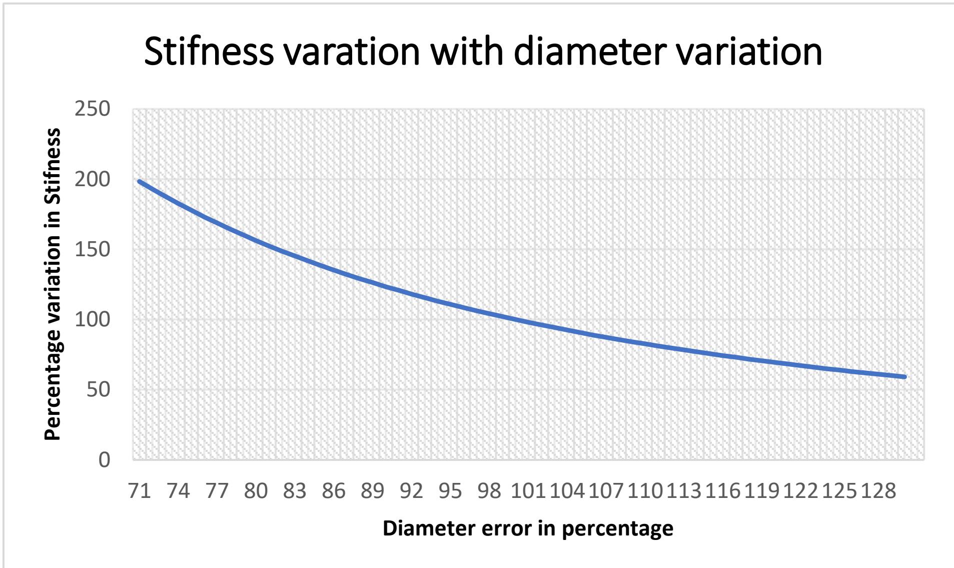

Figure 3: Stiffness variation with diameter value

Some of the main factors that affects error in diameter are density error, weight balance accuracy, balance sensitivity (10^{-5}g), and treatment conditions. Since natural fibers incorporate lumen, it is not advisable to take diameter measured using microscope since it gives external diameter ignoring the lumen which is not capable of load transmission.

3.5. Digital image correlation result

Digital image correlation mitigates the problem with elongation driving from crosshead movement, where the slip is not considered. The figure below describes the estimation of strain using digital image correlation and as a result making possible estimation of stiffness.

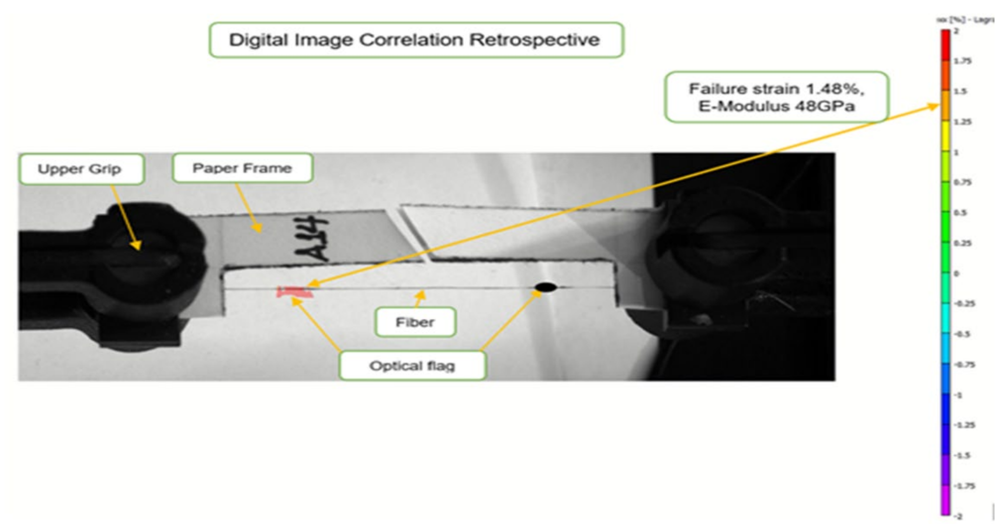

Figure 4: Strain estimation to find stiffness of the material using digital image correlation

On the other hand the error in balancing the elongation may result exaggerated value of stiffness. The comparison of the result direct and indirect method shows that the accuracy of 5-11% with average of 7% is achievable using digital image correlation techniques to find strain at failure and Stiffness as a result.

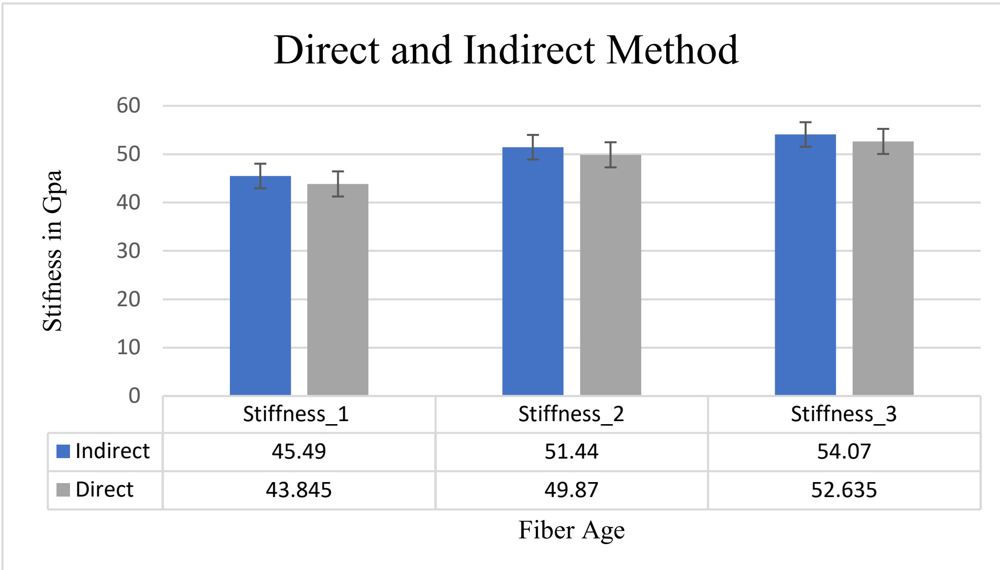

Figure 5: Direct and Indirect method comparison

4. Other factors

4.1. Sample preparation and machine factors

A. *Gluing:-* Unlike abrasive paper frame, the glue type used has to be carefully selected, when normal paper frame is used. The double sided glue allows slippage because normal 80g paper is very smooth. Conversely, use of potent glue, SICO MET 8300 fully can make the fiber rigid around grip and facilitate failure at the grip leading to premature failure. For Enset case, double side glue is used to straighten the fiber at the contact point of fiber and paper and SICO MET 8300 is used around the paper frame end.

B. *Optical Flag and speckle formation:-* Optical flag type affects the correlation process. It is possible to prepare the optical flag by using TIPEX application followed speckle formation and dot making using the tick marker to make visible spot on the fiber. The one with TIPEX is better corelated.

C. *Fiber straightness:-* Fiber straightness during attaching and gripping is critical to come up with an acceptable result. It affects the strain value estimation on both methods because the straightening stage itself is considered as elongation. Hence the fiber has to be straight during preparation and is expected to be straighten by using of a preload less than 0.01N.

D. During digital image correlation process, images before elongation starts and after failure has to be first filtered. It is also advisable to make initial guess to check whether it is possible to correlate before starting the analysis and assist the correlation process if required. In case the initial guess face problems while correlating, going to the picture where the correlation stops and assisting with the possible options available is required.

5. Conclusion

Estimation of natural fiber stiffness, specifically one of the major indicator of its property, can be affected by various factors. Error during density and weight measurement affects the diameter and in turn the stiffness value. One of the major reasons for the density result variation is the purging time and waiting time during pressure drop using the pycnometer. Similarly, digital image correlation reduces error emanated from cross head movement based strain and stiffness estimation.

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Biographies

Abebayehu Abdela obtained his Bachelor Degree in Mechanical Engineering from Jima in 2008 and His master's Degree in production and industrial system Engineering from Ethiopian Institute of Technology-Mekelle in 2011. He is now a PhD Student, under collaborative cooperation of KU Leuven – faculty of Engineering Technology, Belgium and Ethiopian Institute of Technology-Mekelle, Ethiopia. His PhD research considers Ecofriendly sustainable composites materials development from natural fibers and biopolymers. He is also a lecturer at Ethiopian Institute of Technology-Mekelle and is actively engaged in industrial projects where he combines his knowledge, expertise and Mechanical, Materia and industrial engineering experience. He has also led different research projects in the university and actively participate in university-industry engagement via providing different community service projects. He is a founding president of IEOM Ethiopian chapter and 2020 service award receiver from the IEOM society hosted in Zimbabwe. He is also serving as reviewer in IEOM society for couple of years.

Professor Desplentere obtained his master degree in mechanical engineering at the KU Leuven in 1999. Immediately afterwards, he started his professional career as researcher combined with lecturing at the university college KHBO (KU Leuven association) in Ostend. His Phd study was titled: "multiscale modelling of stochastic effects in resin transfer moulding for thermoplastic composites"
Nowadays, he teaches rheology, polymer processing techniques and computer simulations (validated within lab sessions on state of the art equipment) for polymer processing in the master program polymer processing within the faculty of Engineering Technology at the KU Leuven Bruges campus.
Besides his educational tasks, he cooperates in industrial and research projects (national and international), to simulate new products and processes. Next, he also guides different projects in the framework of polymer processing.
Nowadays, he is the head of the research group Propolis (processing of Polymers and Innovative Material Combinations) located at KU Leuven Bruges Campus. Currently, the group exists of 2 Professors, 3 post docs, 2 researchers and 13 phd students. This research group is concentrating on sustainable polymer engineering. To realise this, the interaction between material characterisation, numerical simulations and the validation on industrial relevant equipment is the backbone of the main research projects.

Bart Buffel obtained his master degree in Chemical engineering at the KHBO university college in 2007 and a master degree in polymer processing in 2008 at KU Leuven. Immediately following this, he started his professional career as a researcher combined with lecturing at the university college KHBO. He obtained a PhD at the KU Leuven material science department with the title "Development of tools for the analysis and prediction of the mechanical properties of glass fibre reinforced polyurethane foam sandwich panels"
Nowadays, Bart is active as research manager in the Polymer Processing research group ProPoLiS at the KU Leuven Bruges campus. In this role, Bart is responsible for initiating, preparing and coordinating of different research projects. Additionally he guides multiple colleagues in the elaboration of their projects. These projects are both long term fundamental research projects as well as short and mid-term industrial projects.
Bart teaches Heat transfer and processing of composites in the master program polymer processing within the faculty of Engineering Technology at the KU Leuven Bruges campus.

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