

## Conference Paper

# Looking for Links between Natural Fibres' Structures and Their Physical Properties

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Natural fibres have excited growing attention in the last decade since they offer the potential to act as candidates substituting for man-made fibres as composite reinforcements. Their superiority over synthetic fibres is that they are environmentally friendly and biodegradable. Numerous industrial sectors are interested in such composites, including but to name a few the aeronautical and the automotive fields. However natural fibres tend to suffer from large variability in properties compared to the “traditional” man-made fibres, and the performance of their composites often does not conform to that theoretically predicted from single-fibre tests. This study investigates the properties of the single fibres. The mechanical properties of the fibres were correlated to their microstructure. There are factors that were found to contribute to the reported variability, some of which are inherent in the fibres and some are related to testing parameters.

## 1. Introduction

Fibre reinforced composite materials are gaining popularity in the industry day by day for their superiority over individual materials [1]. Synthetic fibres, such as glass and carbon fibres, are most commonly used in this area. However, there are lots of motivators to shift to renewable sustainable materials, such as rising oil prices and its depletion, in addition to the aim of living in a cleaner environment. Moreover, producing all natural/green composites is becoming more popular as environmental regulations are being initiated in response to the international calls for renewable/recyclable sources for materials. Hence comes the industrial interest in ecofriendly composite materials [2, 3]. Therefore, there is interest in studying the feasibility of using natural fibres in composite materials. Documentation of the structural properties of natural fibres is difficult because of the wide range of variability in the reported properties (Table 1). For engineers, this variation within the mechanical properties of natural fibres is a challenge towards designing reliable

components for industry since they are accustomed to the accurate, precise, and repeatable properties of synthetic fibres. Variability of properties of natural fibres is caused by several factors, some are already inherent in the fibres (due to maturity, age, location, source, fibre extraction technique, and fibre's microstructure), and some are related to the testing/characterization techniques [4–6].

When designing a composite material, it is important to be able to have an estimate of the expected mechanical properties. The rule of mixtures is a key tool for predicting the tensile properties of composites. However, it is not always found to be suitable when using natural fibres as reinforcements although its predictions in some cases are close to the experimental results depending on the constituents [7]. There are different forms of modified rules of mixtures that are being developed to fill the gap of deviation from the ROM [8–10]. This deviation could be attributed to several factors such as the variability within the mechanical properties of each fibre [5, 6] or poor adhesion between the fibre and the matrix that does not allow a homogeneous load transfer from

TABLE 1: Ranges of mechanical properties of natural fibres from the literature compared to man-made fibres.

Fibre type	Tensile strength (MPa)	Young's modulus (GPa)	Elongation to failure %
E-glass [33]	200–2400	68.9	3
S-glass [11, 33]	2400–4500	73–86	2.8–3
Carbon [33]	4000	227–241	1.4–1.8
Coir [11, 20, 33, 34]	130–580	4–6.2	15–40
Flax [6, 11, 20, 33–35]	345–1500	28–100	1.2–3.2
Jute [6, 11, 20, 33, 34]	380–800	10–30	1.16–1.8
Hemp [11, 20, 33]	550–900	68.9–70	1.6–4

one entity to the other for the load to be carried by the stiffer constituent [11, 12].

Despite the difficulties that face research exploring the uses of natural fibres, it is becoming more and more important to invest in this field. One of the pros of using natural fibres to substitute man-made fibres is the fact that they are not only environmentally friendly during their growth but also after using them, since natural fibres are biodegradable [12–18]. However, the setbacks of using natural fibres cannot be overlooked, for example, their poor wettability, being incompatible with some polymers, and high moisture absorption [11, 14]. In addition, from an environmental sustainability point of view, natural fibres reinforced composites are not yet recyclable. This is not a problem that is specific to natural fibre reinforced composites but to all composite materials due to the difficulty of separating the different entities building up the composite [19].

Lots of work is being carried out in order to enhance the mechanical behaviour of composite materials in which natural fibres act as reinforcements whether by using different coupling agents or fibre treatments prior to composite processing. However, not much work is directed towards understanding the root cause behind this unpredictability. This study aims at understanding the fibre-related factors that make accurate predictions of the mechanical properties such as Young's modulus of natural fibre reinforced composites challenging [20, 21].

A natural fibre is itself a composite material by nature of the fact that it consists of cellulose fibrils embedded in a lignin matrix [6]. This complex structure could contribute to the factors affecting the accurate prediction of their mechanical properties. For single fibre testing (SFT), special arrangements are needed to handle and align these ultrafine fibres. Consequently, the testing systems are usually custom made to accommodate the small load and elongation required for the deformation of the ultrafine fibres. Examples of these systems are precision load cells, cantilevers, and atomic force microscope-based nanoindentation system. However, this setup is not an easy one for testing since it requires high skills

for manipulation of individual fibres [22]. The commonly followed standard test methods are ASTM D3822-07 and the BS 3411-1971. There is also the ASTM D3379-79 that was used by Sathishkumar et al. [23].

The results from a single fibre test are affected by several factors such as the clamping length, area measurement, elongation measurement, and modulus calculation. Osorio et al. [12] and Nechwatal et al. [24] reported that the clamping length has an inversely proportional relationship to the strength of the fibre due to the increase in the existing defects that weaken the fibre. The reason behind the inverse relation between the fibre length and its mechanical strength was clearly explained by Defoidt et al. [15]. The probability of breakage is expected to be at the weakest point of the fibre. The longer the fibre, the higher the probability of existence of a weak section that is prone to failure at a certain load. Defroidt et al. [15] also stated that the strain to failure of the fibre is inversely proportional to its length, which they attributed to the same factors.

The area measurement highly affects the stress calculations in SFT, and according to some of the literature it is the main contributor to the variation in the reported properties. It is usually determined either through density measurement and backcalculation of the fibre fineness/diameter [25], or through direct measurement along the gauge length from microscope images, or measuring the diameter at the point of failure [25, 26]. The problem with the first method is that it is not accurate as it depends on the apparent density and that with the latter is that it assumes the fibre to be of circular cross-section, which is rarely the case. Nechwatal et al. [24] reported that the correlation between using the two methods is significantly high. Hu et al. [25] dedicated a full study to the different methodologies of diameter measurements. In agreement with lots of previous research, it was established that increasing the diameter of the fibres leads to a depression in the tensile properties. Thomason et al. [4] compared the optical average diameter method with measuring imaged cross-sections for natural fibre diameter determination. The reported results showed that the calculated average diameter from optical microscopy is always at least double the measured diameter since the former technique ignores the cross-section irregularities and assumes a circular cross-section. This finding shows how much the reported Young's modulus values are influenced since the majority of the published data relies on the calculated average diameter method. However, they did demonstrate that the variation in diameter along the length did not have a major effect on the mechanical properties variation. Tomczak et al. [27] reported that increasing the fibre diameter results in depressing the tensile strength of the fibre. Similar behaviour was reported for the influence of the length on the strength, as they are also inversely proportional.

Another factor affecting the SFT results is the measurement of the elongation, which is an issue bearing in mind the effect of the fibre slippage from the adhesive that cannot be monitored directly [24].

Polarized optical microscope images showed the structure of hemp fibres to contain series of dislocations [25]. Sawpan et al. [28] also observed the kinks in the structure

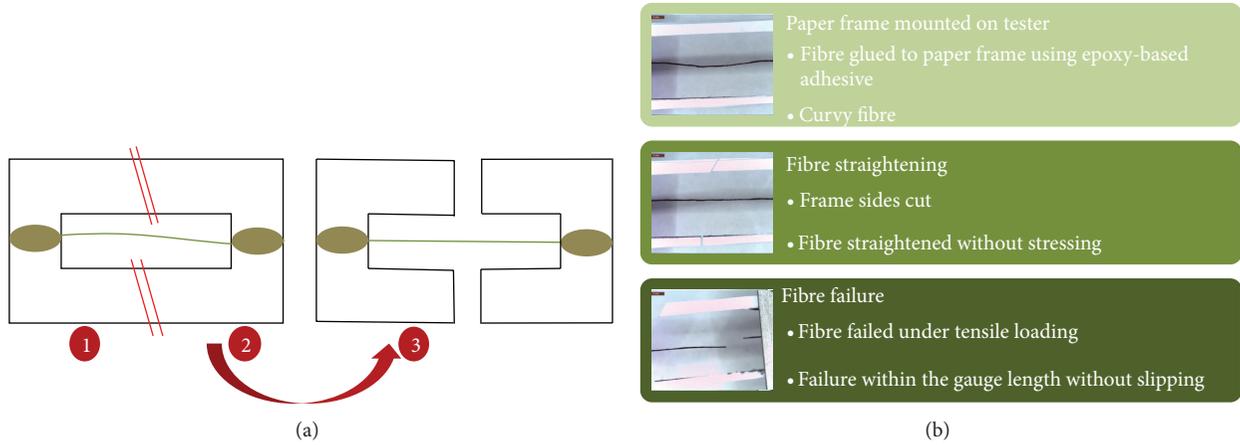


FIGURE 1: Schematic presentation showing preparing the fibre for tensile testing and optical microscope images taken during the process.



FIGURE 2: Optical microscope images of coir fibre under tensile loading slipping out of the adhesive (a), and the adhesive after the fibre has slipped out with its empty space clearly obvious (b).

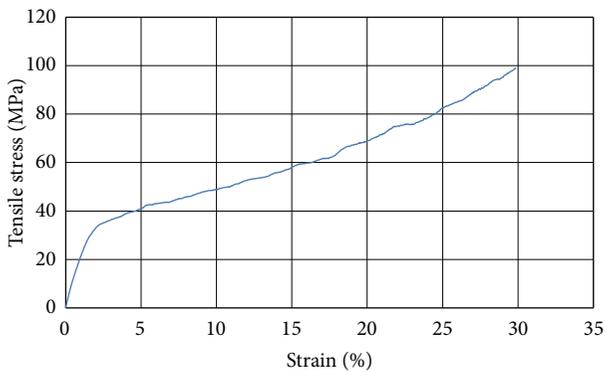


FIGURE 3: Representative stress-strain curve showing the tensile behavior of coir fibres.

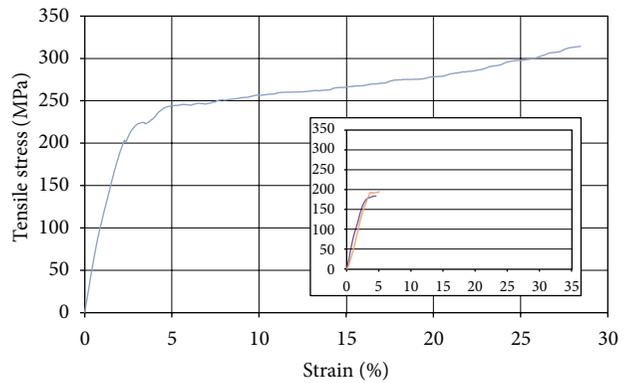


FIGURE 4: Representative curves for tensile behaviour of some of the tested cattle hair fibres with the inset showing samples with early premature failure.

of hemp fibres and attributed the variation in the mechanical properties to their presence. Kompella and Lambros [29] concluded that the strength of the fibre is highly dependent on the amount of surface flaws in the fibre. Studies of the fracture surface of the fibre showed that failure took place through

fracture of fibre cells, delamination within the fibre cells, and delamination between the fibre cells [5].

Materials are built up of smaller elements that are linked together. A material would fail at the weakest point, which is where dislocations, kink bands, or defects exist, or where

diameter variation occurs, or even both simultaneously. Dislocations do not only affect the tensile behaviour of single fibres but also, from a broader perspective, affect the behaviour of the composite material since debonding between the fibre and matrix usually takes place at a dislocation as it is considered a location for stress concentration [30, 31]. This study aims at correlating the structure of the fibres with their mechanical properties and the influence of the abovementioned defects on the tensile behaviour of the fibres.

## 2. Experimental Work

Cattle hair, horsetail hair, hemp, flax, jute, and coir fibres were supplied by ENKEV (UK) Ltd. A Deben microtensile tester using a 200 N load cell was used to test all the single fibres. The testing speed employed was 1.5 mm/min. The gauge length of the fibres was 30 mm. The tension tests were conducted in accordance with the ASTM D3822-07. Each fibre was glued to a paper frame using Araldite adhesive, which is left for 24 hours in the laboratory before testing for the adhesive to cure and the fibres to be conditioned. The frame would then be mounted to the tensile tester clamps. After that the sides of the frames are cut for the load to be fully carried by the fibre. Some of the fibres exhibited a curly structure and so they had to be straightened by the tester without stressing the fibre before running the test. The test procedure is shown in the schematic presentation in Figure 1.

The type of adhesive to use for mounting the fibres was investigated at the start of the experimental work to prevent the fibre from slipping out of the adhesive during testing. Figure 2 shows a coir fibre under tensile loading with evidence of slipping. The epoxy-based adhesives were found to be the most satisfactory for minimizing slippage. Using epoxy-based adhesive did not yield a 0% slippage of fibres, but it was diminished to the minimum and the fibres that slipped during the test were discarded.

Images along the length of the fibre were taken using an optical microscope before testing the fibre. The cross-section of the fibre was assumed to be circular and hence the diameter was directly measured from the images using ImageJ software. To consider the variation along the length of the fibre, the diameter was measured at 20 different locations and an average value was then calculated.

A Phillips XL30 scanning electron microscope SEM was used to characterize the longitudinal and cross-sectional surfaces of the fibres. Moreover, the fracture surfaces of the tensile test specimens were investigated. Since natural fibres are not conductive materials, the fibres were first coated with platinum for successful imaging.

## 3. Results and Discussion

Representative curves for the stress-strain behaviour of coir fibres are demonstrated in Figure 3. The behaviour of coir is seen to start with a nearly linear elastic region that is followed by a second phase showing a more gradual increase in force per % increase in strain. This two-phase phenomenon

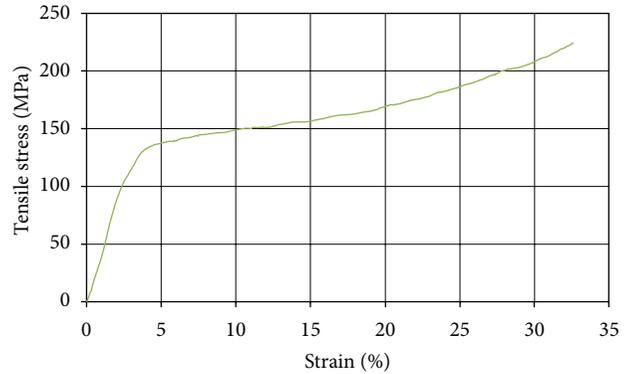


FIGURE 5: Representative curves for tensile behaviour of some of the tested horsetail hair fibres.

led to a relatively high elongation to failure [32], and it could be attributed to stress relaxation taking place during loading the fibres for residual stresses already existing in the fibres or microfibrils self-alignment during deformation. The diameter variation along the gauge length was found to be a factor affecting the measured tensile strength of the fibre. As the variation in diameter (standard deviation of diameter measurements) increases for a particular fibre, the mechanical behaviour, in terms of strength and modulus, is depressed considerably. However, when designing for industry it will not be feasible to check the diameter variation for each single fibre embedded in the composite. But this should be generally considered as a factor that is highly affecting the modulus of elasticity.

The changing slope of the tensile curve was also observed for the cattle and horsetail hair (Figures 4 and 5); the slope of the curve changes at a specified strain value. It is possible to make rough estimates for the strain value after which the slope of the curve changes, and these are 2.5%, 3.2%, and 3.5% for coir, cattle, and horsetail fibres, respectively. Although the mentioned three types of fibres behave in the same manner, white cattle hair is seen to have both the least scatter of data and the highest tensile strength parameters. It is important to note that both hair types tend to be highly ductile as most of the tested specimens have not failed/fractured at the specified strain. For those reaching an elongation equal or greater than 30%, the test was stopped before failure because the tester reached its maximum travel limit. On the other hand, some of the hair samples failed prematurely right after the slope change, that is, the transition from one form of deformation to another. Representative tensile stress-strain curves for the fibres that failed prematurely are shown in the inset in Figure 4. The reason behind this early failure would be better understood by witnessing the difference in morphology of both fibres that behave differently in such a different manner.

Unlike coir, cattle, and horsetail fibres, flax, hemp, and jute fibres stress-strain curves show only one stage, which is almost linearly elastic, in agreement with Müssig et al. [32]. Representative stress-strain curves of hemp, flax, and jute are

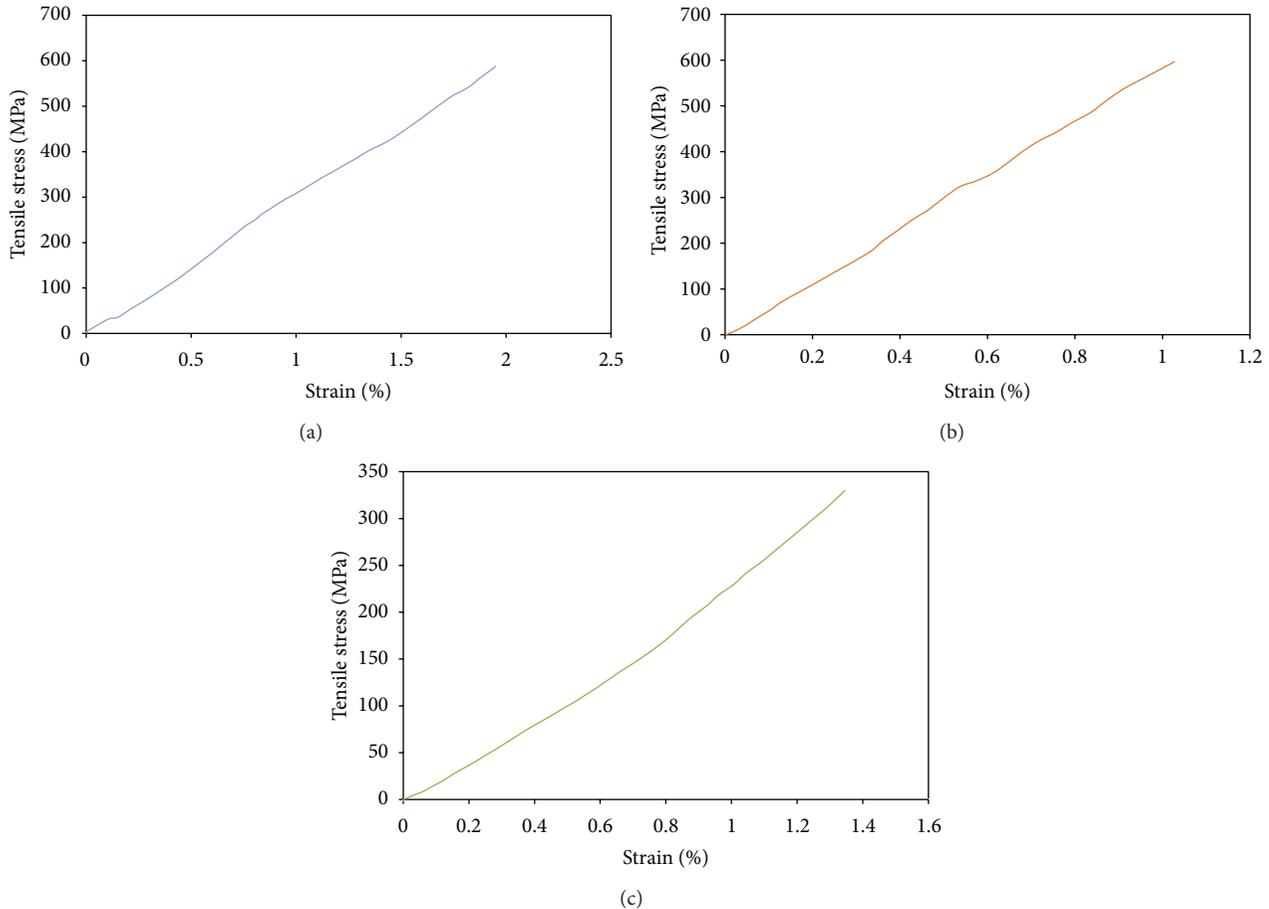


FIGURE 6: Representative curves for tensile behaviour of (a) hemp single fibres, (b) flax single fibres, and (c) Jute single fibres.

demonstrated in Figure 6. The scatter in the tensile behaviour of hemp, flax, and jute fibres is quite large. This is expected for natural fibres as suggested by the literature. Despite the scatter, the overall behaviour of these three fibres is superior to that of coir, cattle hair, and horsetail hair when considering the modulus of elasticity. But it is evident that the ductility of the plant fibres (excluding coir) is much lower than that of hair. The stiffness of both types of fibres is quite comparable though.

A summary of the tensile properties of cattle hair is shown in Figure 7. These graphs show the large scatter within each property. However, a general trend for the behaviour can be determined and the significance of this trend is established through the R-squared values on the plots. The measured Young's modulus shows no statistical trend with measured diameter. However, it can be concluded that the measured stiffness (force per unit elongation) of cattle hair increases with increasing the fibre diameter, whereas its strength to failure shows a slight tendency to decrease with increasing the diameter. The graphs for hemp fibres (Figure 8) show weak correlation between the fibre diameter and the different mechanical properties. It could be observed that the higher population of the fibre's properties tend to be at the lower range for Young's modulus and strength at failure.

Figure 9 shows SEM images of the cross-section of coir fibre. This image points out the dilemma that has been reported to be a factor affecting the mechanical properties of natural fibres through biased calculations, that is, the method used for diameter measurement. Although using optical microscope images to determine the diameter of the fibre has been stated to be an adequate technique, still the assumption that the cross-section of the fibre is circular influences the estimation of the cross-sectional area. For instance, the maximum diameter measurements for the fibre in Figure 9(a) are almost double the minimum diameter. This means that depending on the view observed in the optical microscope image, an error of up to 50% can exist. Nevertheless, this statement cannot be generalized since some of the fibres do have circular cross-sections such as the coir fibre shown in Figure 9(b). Moreover, measuring the diameter using this method and calculating the failure stress of the material are not only ignoring the fact that the fibre is not circular but also assuming a uniform density across the cross-section. This is not the case for lots of the natural fibres as can be seen in SEM images of coir fibres (Figures 9 and 10) since the fibre cross-section shows a large amount of hollowness in the fibre that is not considered in diameter measurement. This means that the strength measurements

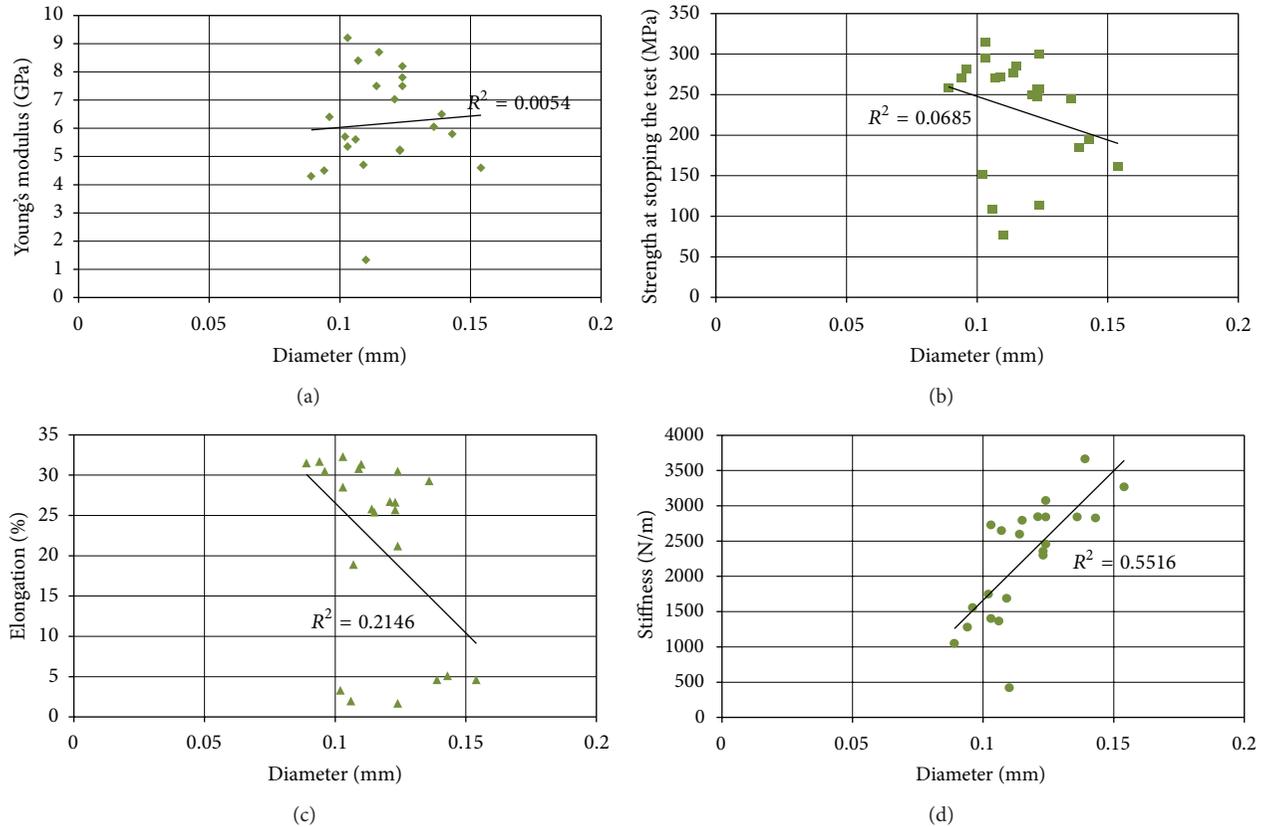


FIGURE 7: Summary of cattle hair tensile properties.

are misled as the area effectively carrying the load is well less than the measured one.

The high magnification SEM image in Figure 8(a) suggests that the microfibrils towards the centre of the fibre tend to have a coiled spring-like structure, with this spring being lined with a thin layer as shown in the inset in the same figure. The scenario of failure could be that this thin layer first is stretched (bearing the load), then it fails, and that would represent the first stage in the stress-strain curve. After that the spring itself is carrying the load and this is the second part of the curve until it fails, which means that the whole fibre failed. This hypothesis was also suggested by the SEM image in Figure 8(b) of the fracture surface of a tensile test coir fibre. Observation of the deformation of the fibre under tensile loading in an SEM could help verify this hypothesis.

A characteristic feature that was noticed in the structure of cattle hair (as shown in Figure 11) is that, unlike other hair types (keratinous), it has a smooth surface. This is clearly demonstrated by comparing the surface of the cattle hair to that of Merino wool. Moreover, some of the observed hair samples contained a hole in the middle and others did not (as shown in Figure 12). This observation needs further investigation and better understanding of the anatomy of the cattle hair. Another feature that was noticed in the cattle hair is the presence of kink bands that could act as structural defects leading to the premature failure that was observed in

tensile stress-strain curves. This kind of defect/feature can be seen in Figure 12 as well.

The structure of jute fibres shown in Figure 13 is similar to that of most plant fibres (flax and hemp in this study); each fibre is a bundle of microfibrils waxed together. The abovementioned statement about the inaccuracy of assuming the cross-section of the fibre to be circular is also supported by the SEM images of jute fibres here.

#### 4. Conclusions

Natural fibres have the potential to be candidates to replace synthetic fibres in lots of applications. There are barriers that are hindering their progress into industry [2]. First and foremost is the variability within the properties of the fibres, which affects the final properties of them when used as reinforcements in composite materials. There are two sorts of variability: the apparent variability that is caused by measurement and testing techniques and the actual property variability of the fibre by nature. The former type of variability is caused by experimental methods such as the diameter measurements as well as variability in microstructure.

It has already been well established by previous work that the use of plant fibres such as hemp, flax, jute, and coir in composite materials is beneficial from an engineering perspective as well as the environmental impact scope. There

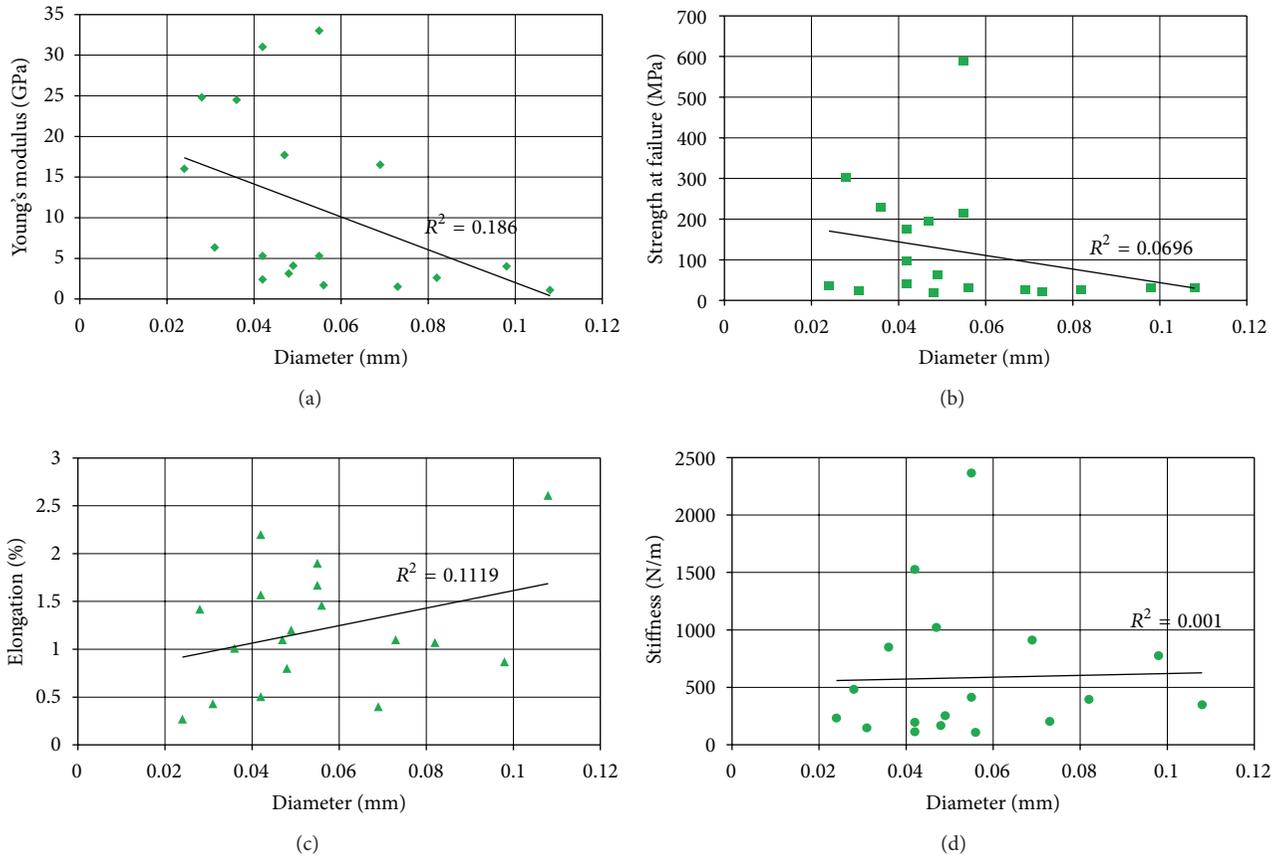


FIGURE 8: Summary of hemp tensile properties.

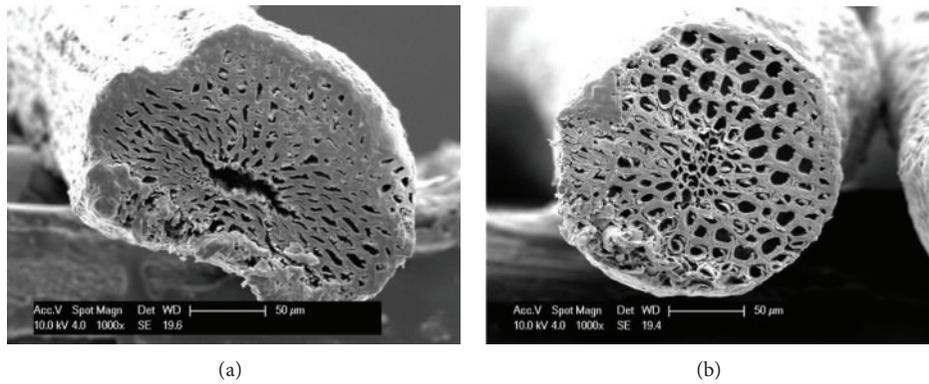


FIGURE 9: SEM images of the cross-sections of coir fibres.

are no relevant published investigations for the properties of cattle hair and horsetail hair. This study has shown the animal hairs to have good mechanical properties, in terms of ductility (in the range of 20% elongation on the average) and strength to failure (in the range of 250–300 MPa on the average), suggesting that they could be utilized in several applications.

Natural fibres usually have the mechanical properties suitable for their role in nature. For instance, coir fibres are

expected to have high impact strength/resistance as their role in nature is to protect the nut from breakage upon falling. Understanding the role of each fibre in nature could help utilizing them in the suitable applications. In addition, the structural defects in the fibres cannot be overlooked as they seem to be playing a vital role in the deformation of the fibres. Further investigation is required in order to monitor the microstructural evolution of the fibres while deformation is under tensile loading.

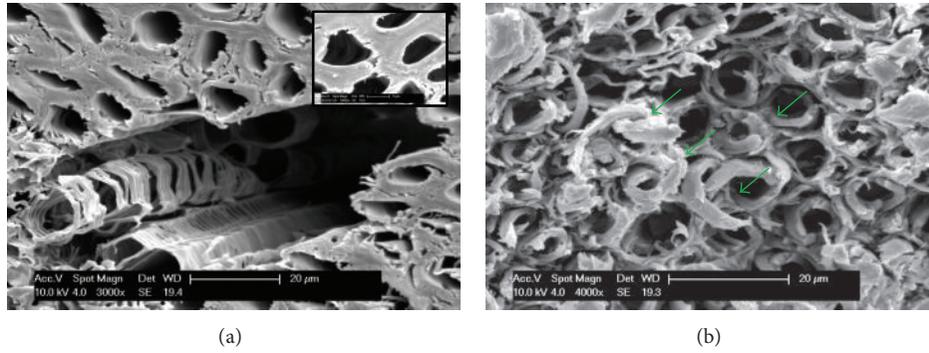


FIGURE 10: SEM images showing (a) coir microfibrils at higher magnification with the spring-like structure and the interconnecting layer and (b) fracture surface of a coir fibre that failed under tensile loading (the arrows point towards features demonstrating spirality of the microfibrils).

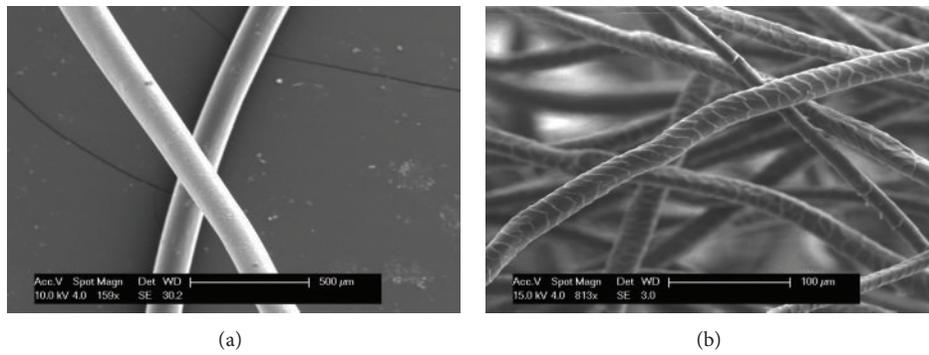


FIGURE 11: SEM images showing the smooth surface of cattle hair (a) versus keratinous structure of wool (b).

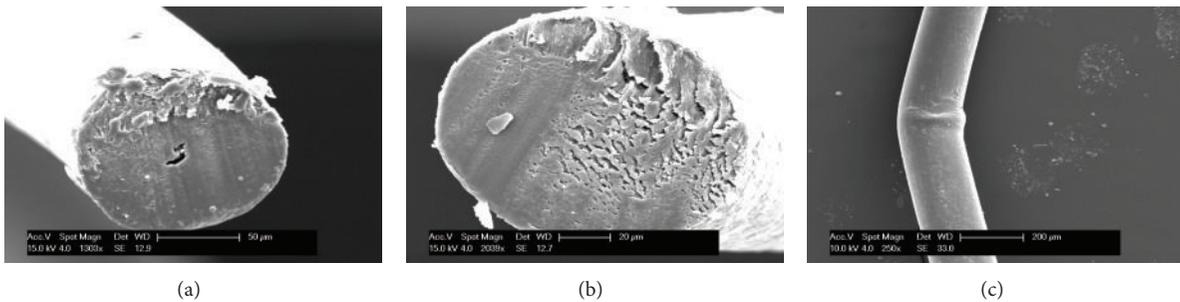


FIGURE 12: SEM images showing different features of cattle hair (with and without a hole in the middle, kink bands).

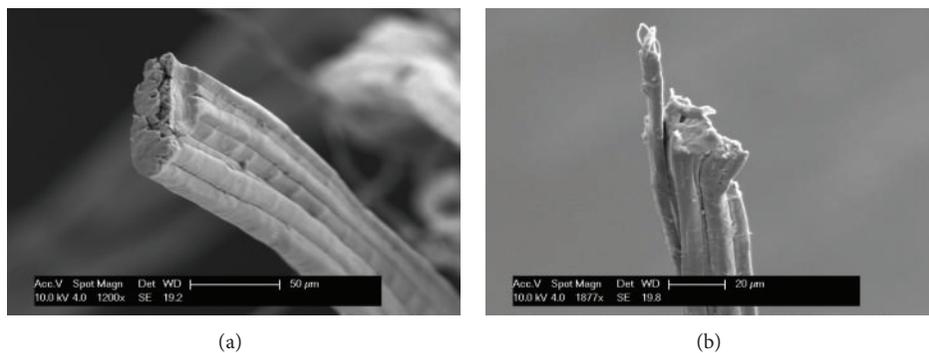


FIGURE 13: Jute fibres shown to consist of several elementary fibres waxed together.

## Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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