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MECHANICAL ENGINEERING

On the characterisation of structure and properties of sorghum stalks

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Abstract This work investigates the thermal, mechanical and chemical characteristics of sorghum stalks rind. Sorghum stalks have been divided into three equal regions: bottom, middle, and top. The thermal degradation behaviour has been studied using static and dynamic thermogravimetric analysis. The results of this study show that it is not recommended to subject the fibres to elevated temperatures (> 200 °C) for time periods exceeding 10 min. The highest tensile strength of 280 MPa was found at the middle region of the sorghum stalk. However, based on practical and economic aspects, the use of the complete stalk is recommended, since variations are minor. Microscopic investigation has been done for the sorghum stalk using optical microscope to relate mechanical properties to structural appearance.

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1. Introduction

Due to their versatility in type and cultivation conditions researchers are often concerned with investigating and characterising new types of natural fibres targeting their use as engineering materials. A common objective that is herein being explored since the late 1990s is the use of various fibres of plant origin as reinforcements for polymer composite applications. This is due to their optimum characteristics regarding, costs, availability from natural resources, environmental friendliness, CO_2 neutrality, low density, in addition to their acceptable mechanical properties.

One important renewable natural fibre resource, that has not been well investigated yet is the sorghum stalks. Sorghum takes the fifth place among the cereal crops produced worldwide with 56,098,260 tonnes annually [1]. It is essentially a plant of hot and warm countries due to its drought resistance [2]. The oven dry weight of the sorghum agro residues constitutes 74% by weight of the crop [3]. The United States of America is the first country producing sorghum with 11,998,040 MT per year, followed by Nigeria with 9,318,000 MT, and further by India producing 7,925,900 MT [1]. Egypt occupies the fourteenth place with 866,948 MT [1]. In Egypt, sorghum stalks are either open-field burned (most of the

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stalks), or underutilised in applications, such as thatching and fencing.

The agro residues of the first four cereal crops, maize, wheat, rice, and barley, were subjected to several characterization research attempts [4–9], whereas the agro residues from sorghum have not been well studied for higher added-value applications. Generally, natural fibres have gained great attention within the last decade, aiming at their use as technical textiles or as reinforcement materials in composite systems, especially with polymer matrices. The existence of a huge amount of annual residues, the immense variation in plant types and the wide dispersion of their properties makes them attractive for use in the field of composites. Beside this vast diversity in raw materials, natural fibres are also a low-cost, light-weight, and eco-friendly alternative for some synthetic reinforcements, showing acceptable specific properties.

Next to the wide dispersion in mechanical properties of natural fibres, due to biological differences among plants, the climatic conditions, and the practices of cultivation, the geographic area, and the applied fibre extraction technology [10], the influence of the sample location also plays a vital role. This effect was studied on several plants, such as flax, hemp, date palm midrib, and bamboo. In case of flax, fibres from the middle region of the plant were reported to possess higher mechanical properties, compared to fibres from the top or bottom regions [11]. Hemp fibres, however, seem to provide only slight variations in properties over the bast length [12]. Amada and Untao studied thin sliced specimens from various locations along the culm's radius and along the stem length of bamboo. Tensile testing showed variations in mechanical properties [13]. A study of the variation of the tensile strength across the cross section of date palm leaves' midrib shows that the tensile strength is maximum at the outer layer and continuously decreases in the direction of the centre [14].

Sorghum is a monocotyledon plant, which belongs to the grass family (Gramineae) [15]. It can reach up to 6 m in height [2], and the stalks are usually erect, consisting of nodes, each with a leaf [2] (Fig. 1a). In the radial direction the stalks consist of pith and rind (Fig. 1b). Similar to sugar cane and maize, sorghum has a solid stem.

The objective of this work is to characterise sorghum stalks, which is one of the most important renewable resources available in Egypt. The characterisation includes structure and its chemistry, thermal properties and tensile behaviour, which represent the key issue for the preparation of polymer matrix composites.

The sorghum stalks under investigation are by-products of the sorghum plant, cultivated at the beginning of June and harvested after 95 days. Sorghum stalks morphological, mechanical, and thermal properties, as well as the chemical analysis, are studied for three equally divided regions: bottom, middle, and top regions of the rind, which is easily separated from the pith. The rind is a very dense and fibrous tissue, thus promising higher strength and mechanical properties than the pith, where the fibre bundles are few in number, and scattered within the pith tissue. Chemical analysis has been performed to support the choice of rind in our study. Whereas the higher mechanical properties of the rind suggest their application as composite reinforcements, the pith can further be utilised for low-strength applications, such as low density particleboards, as suggested by Wang [16]. A modified method in sample preparation has been used to overcome the difficulty in fixation of the samples due to the curved nature of the rind.

Figure 1a Sorghum plant.

Figure 1b Sorghum stalk cross section.

2. Materials and methods

2.1. Materials

Sorghum stalks were collected from local farmers of the Assuit governorate in Egypt, directly after crop harvest. Each stalk was divided into three regions of equal length; bottom (B), middle (M), and top (T). Leaves and nodes were removed and stalks were further gently scrubbed in tab water. The rind was manually peeled and any residual pith on the inner rind surface was removed using a knife.

2.2. Structure of the sorghum stalk

The measurements of the diameter, internodes length (distance between two successive nodes), and rind thickness was done using vernier calliper, measuring tape, and micrometre, respectively.

Ten random stalks were investigated and results were averaged.

2.3. Microscopic investigation

Young sorghum stalk was examined. Thin slices of 12 μm thickness were cut using a Spencer Lens Co., Buffalo, NY, USA microtome. The slices were further microscopically examined using a LABOMED optical microscope. A transverse section was inspected showing pith and rind.

2.4. Chemical analysis

The rind of each region was ground using a blender. Chemical composition of the rind was further analysed, where the cellulose, hemicelluloses, lignin, crude fibre contents were determined for each region. Analysis was performed using an ANKOM^{2000} Fiber Analyzer, ANKOM Technology, US, in accordance to the methods reported by [17,18].

Three replicates were used for each test. In addition, the pith of the top region was ground, and its fibre ratio was determined to compare rind and pith results.

2.5. Dynamic thermogravimetric analysis

Dynamic thermogravimetric analysis (DTGA) aims to analyse the thermal stability at continuously increasing temperatures for each of the three regions of the sorghum stalk rind. The tests were performed on ground samples using a TGA Q5000 V3.13 Build 261 at a heating rate of 10 °C/min in nitrogen atmosphere up to a temperature of 700 °C. Three replicates were used for each test. The critical degradation temperature was determined, at which rapid weight loss occurs.

2.6. Static thermogravimetric analysis

Samples of the three regions were respectively subjected to constant temperatures of 200 °C, 220 °C, and 230 °C for 15 min, and the weight loss was recorded in a 5 min interval. The temperature was set slightly below the critical temperature, determined by the DTGA analysis. Again, three replicates were used for each condition.

2.7. Tensile testing

Tensile specimens were longitudinally cut from the rind of the sorghum stalk. Dumble-shaped samples were prepared with dimensions as indicated in Fig. 2a. Smooth fillets at the shoulders were prepared by abrasive machining, by gluing emery cloth on a 6 mm cylinder and further rotating this cylinder by means of an electric motor.

To avoid sample failure when gripped, due to its curved cross section (Fig. 2a), tabs of epoxy putty were bonded to the gripping length. The assembly (Fig. 2b) was left to cure over night prior to tensile testing. These tabs successfully prevent sample crushing, as they are known to smoothly transfer lateral compressive loads from machine grips to the specimen. Moreover, the tabs also prevent sample slipping between the grips.

Tensile tests were conducted at room temperature on an LRXPLUS universal testing machine using a 2.5 kN load cell at a cross head speed of 3 mm/min up to rupture. Ten specimens were randomly taken from each region of different stalks.

3. Results and discussion

3.1. Structure of the sorghum stalk

Measurements of stalk diameter, internodes distance and rind thickness are presented in Figs. 3–5 respectively. Node count from the bottom to the top has resulted in an average number of 16 nodes per stalk. The bottom (B), middle (M) and top (T) regions are of equal length, (but this results in different number of nodes in each region). The stalk diameter is observed to gradually decrease within the middle region in the upward direction, whereas the bottom and top regions show more or less uniform diameters. Although this is an observation of a natural occurrence, it lies in logic conjunction with the design requirements, where the bottom is of higher diameter to provide stability and withstand higher bending moments. Towards the ends, the diameter becomes smaller.

Generally, the average length of a sorghum stalk was found to lie around 3.35 m. The internodes distance, as illustrated in Fig. 4, gradually increases towards the top within the bottom region, where it reaches maximum and further decreases until reaching a constant value at the top region. Such variation ranging from about 100 to 275 mm again provides best stability to the stalk avoiding fractures in internodes positions due to severe wind actions, for example.

In contrast, the rind thickness continues to decrease gradually from the bottom (1.2 mm) towards the top (0.4 mm), as can be depicted in Fig. 5.

3.2. Microscopic investigation

Microscopic picture of the cross-section, as seen in Fig. 6 reveals the microstructure of both, pith and rind, respectively.

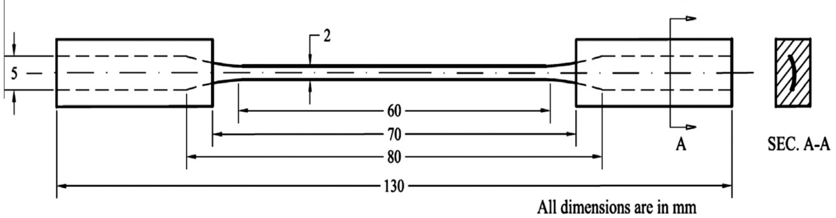

Figure 2a Tensile test specimen: drawing with dimensions.

Figure 2b Tensile test specimen: real image of the specimen.

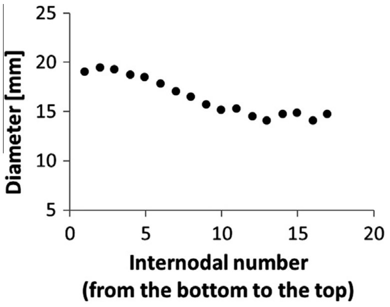

Figure 3 Relation between internode number and internode diameter.

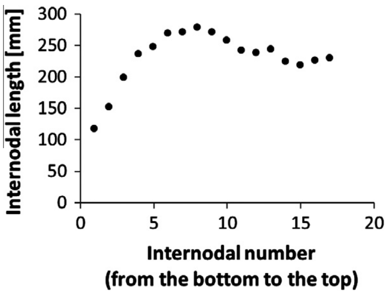

Figure 4 Relation between internode number and Internode length.

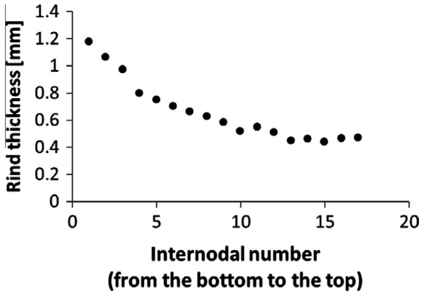

Figure 5 Relation between internode number and rind thickness.

It can be observed that the pith consists of vascular bundles surrounded by weak tissue of large and thin-walled parenchyma cells, whereas the rind is found to be composed of compacted fibre bundles. By comparison it can also be observed, that the rind reveals higher density of fibre bundles compared to the pith. In fact, it is this higher fibre ratio that provides for higher tensile strength of the rind.

3.3. Chemical analysis

Table 1 summarises the results of the chemical composition of the three stalk regions. It can be observed that the main strength providing constituent, i.e. the cellulose microfibrils, vary only marginally over the entire length of the stalk. While at the bottom a cellulose content of 41% is present, its ratio slightly increases to 45% in the middle and top regions.

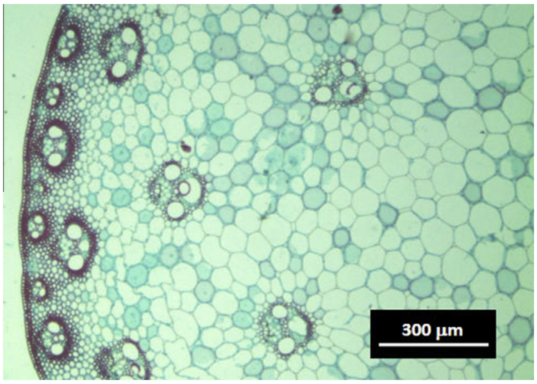

Figure 6 Transverse section of sorghum stalk.

Table 1 Chemical composition of the rind in all three regions, given as weight percent of dry matter.

	Cellulose	Hemicellulose	Lignin	Fibre ratio
B	$41.47^{\pm 0.22}$	$13.34^{\pm 0.69}$	$13.47^{\pm 0.39}$	$45.71^{\pm 0.44}$
C	$45.15^{\pm 0.76}$	$12.99^{\pm 1.27}$	$12.40^{\pm 0.25}$	$49.2^{\pm 0.48}$
T	$45.69^{\pm 0.37}$	$15.29^{\pm 0.31}$	$11.66^{\pm 0.20}$	$47.35^{\pm 0.46}$

Similarly, the hemicellulose content slightly varies between 13% and 15%. Contents of further constituents are indicated in Table 1, and generally show a variation range of around 1–3%.

Further chemical analysis of the pith resulted in a fibre content of only $32.5^{\pm 0.32}$%, compared to a fibre ratio of around 45–47% for the rind. This great difference supports the selection of the rind material for reinforcing purposes, while using the pith for other low-strength applications.

3.4. Thermal analysis

Targeting composite materials, thermal analysis of natural fibres is essential for the proper selection of adequate matrix materials. Relatively low degradation temperatures limit the matrix selection to polymeric matrices. Next to thermoset polymers, thermoplastics are usually limited to materials such as polyethylene, polypropylene, polyvinyl chloride and polystyrene, where processing temperatures are lower than those at which thermal degradation of fibres would take place [19].

The beginning of the DTGA curve, presented in Fig. 7, shows the evaporation of about 5% moisture at 100 °C, followed by slight weight loss, due to the degradation of lignin at around 150 °C [20–22]. Due to this slow degradation rate, lignin acts as a thermal barrier, protecting other constituents, such as pectin, hemicellulose and most importantly cellulose [20–22].

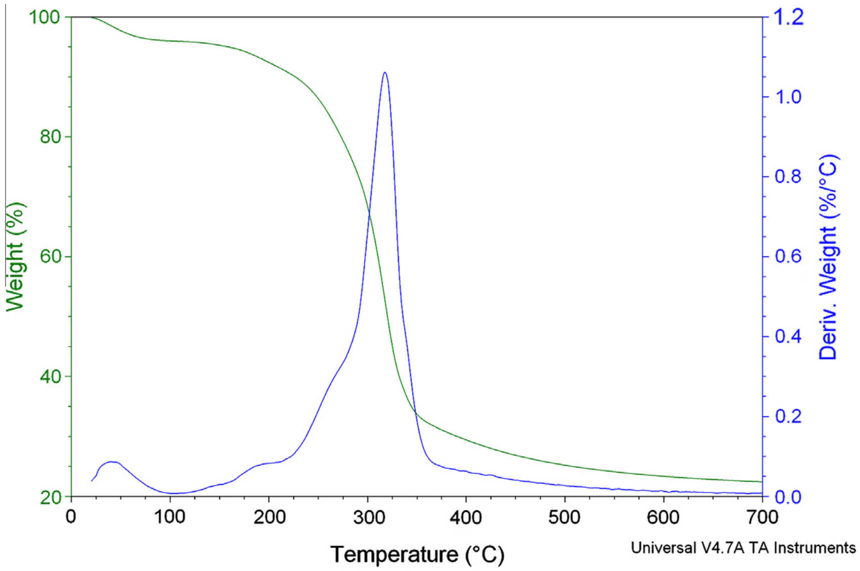

Figure 7 Thermogravimetric analysis for the sorghum stalk rind.

Table 2 Critical temperatures and the corresponding weight percent of the three regions.

	B	M	T
Temp (°C)	217.5 ± 2.18	223.3 ± 2.89	221.67 ± 3.06
W (%)	89.5 ± 0.5	89.3 ± 0.58	92 ± 1.73

Continued temperature increase results in a subsequent sharp weight reduction at a critical temperature T_c. Marginal differences in the onset of severe degradation (T_c ranging between 218 °C and 223 °C, as stated in Table 2) are observed among the three regions of the sorghum stalk. As indicated by Fig. 7, the maximum degradation rate is observed at temperatures exceeding 300 °C. Similar T_c values are reported for corn stalk, corncob, and wheat straw [4]. These slight variations can be attributed to the compositional changes discussed above. The slightly higher resistance to degradation of the top and middle regions are attributed to their higher cellulose content, where cellulose is reported to have degradation temperatures lying around 305 °C [20–22].

A statistical hypothesis test has been used to determine whether or not it is worthwhile to deal with the three regions independently, or if deviations are so marginal, that it is more efficient to consider the stalk as a whole. It has been proven, that statistically there is no significant difference between these values at a confidence level of 98%, giving good reason for considering the whole stalk in practical applications.

Based on the above results, ground fibre from the complete rind was subjected to isothermal heat for 15 min, at temperatures around the T_c. Three temperatures were considered: 200 °C, 220 °C and 230 °C. Weight loss along time is graphically represented in Fig. 8.

As time and/or temperature increases fibres were observed to undergo discolouration. After 10 min, a weight loss of around 5% indicates the evaporation of moisture, implying no significant fibre degradation.

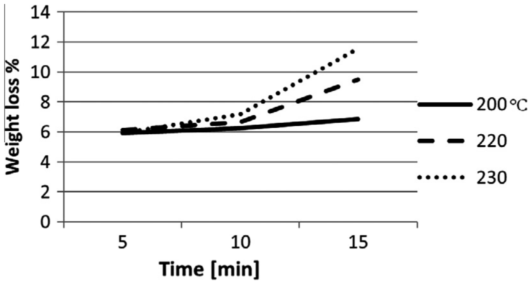

Figure 8 Static thermogravimetric for the sorghum stalk rind.

As can be seen from Fig. 8, fibres start degradation at all temperatures under investigation. As expected, degradation rate increases with increased temperature. However, thermal stability for duration of 10 min is acceptable when processing sorghum rind with most polyolefins with common extrusion or injection techniques.

3.5. Tensile testing

Fig. 9 shows a typical stress–strain curve of a sorghum stalk rind from the central region. Generally, all regions follow the same tensile behaviour. Specimens show a brittle fracture mode with abrupt load drop at the breaking point.

Tensile tests of the rind at the three regions resulted in variation of the tensile strength (TS) and modulus of elasticity (MOE), whereas the elongation only showed minor differences. The highest tensile strength of 280 MPa was found at the central region, followed by the upper region 245 MPa and further the bottom region with 208 MPa (Table 3). Applying statistical techniques, the variations in tensile strength and modulus were found to be significant, which is in agreement with the results reported by Charlet et al. for flax fibres [11]. The changes in the mechanical properties among the three regions may be attributed to the change in the chemical composition [23]. From chemical analysis results, it was shown that

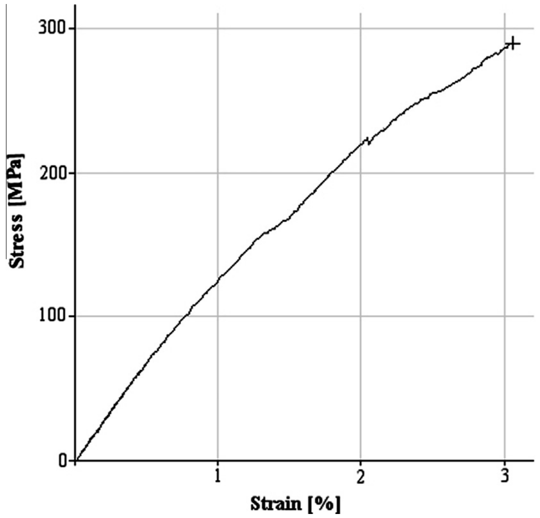

Figure 9 Stress strain curve for a rind from the central region.

Table 3 Mechanical properties of the rind for the three regions.

	B	M	T
TS (MPa)	208 ± 20	280 ± 46	245 ± 27
MOE (GPa)	11.9 ± 2.1	17.4 ± 3.7	16.6 ± 3.1
Elongation (%)	2.47 ± 0.71	2.22 ± 0.47	2.17 ± 0.55

the fibre ratio, as well as the cellulose content is maximum at the middle region.

Moreover, Charlet et al. [11] also reported that the environmental and growing conditions greatly affect the plant. The bottom of the stalk is the oldest region, which was most affected by all weathering conditions, causing a deterioration in properties. On the other hand, the growth of top fibres has been precociously interrupted, leading to immature stalk properties at the top region. Hence, the middle region presents the optimum properties making a compromise between immaturities and weathering deterioration.

Although the differences in failure strains are marginal, the highest elongation is that of the bottom region, which may be attributed to the highest lignin content in that region. Tomczak et al. [24] observed the highest elongation of 59.87% for coir fibres, which are reported to have a lignin content reaching up to 45% [20–22].

4. Conclusions

From the present work, the following conclusions can be drawn:

1. Microscopic investigation supports the chemical analysis in proving the higher fibre density of the rind.
2. Thermal analyses indicates that the fibres cannot withstand elevated temperatures higher than 200 °C for time periods exceeding 10 min, where degradation processes start to take over.
3. Tensile test indicates that the middle region of the rind possess the highest tensile strength and modulus, followed by the top regions.

The data endorse the use of sorghum stalk rind in common polymer composite processing techniques, provided that the fibres are not subjected to elevated temperatures exceeding 200 °C. Although that the middle region of the rind is superior for reinforcing purposes, due to its highest tensile strength and modulus, however, the use of the complete stalk rind is recommended based on economic and practical aspects.

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