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Peer-reviewed version available at Materials 2017, 10, , 479; doi:10.3390/ma10050479

Article

In-depth Analysis of the Structure and Properties of Two-variety Natural Luffa Sponge Fibers

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Abstract: The advancement in science and technology has led to luffa sponge (LS) being widely used as a natural material in industrial application as its polyporous structure and light texture. In order to enhance the utility of LS fibers as the reinforcement of lightweight composite materials, this study investigate its water absorption, mechanical properties, anatomical characteristic and thermal performance. Hence, moisture regain, tensile properties of LS fiber bundles were measured in accordance with standards and the structural characteristics were investigated via microscopic observation. Scanning electron microscopy (SEM) was used to observe the surface morphology and fracture surface of fiber bundles. Test results shows that the special structure where the phloem tissues degenerate to cavities had a significant influence on the mechanical properties of LS fiber bundles. Additionally, the transverse sectional area occupied by fibers in a fiber bundle (S_F), wall thickness and ratio of wall to lumen of fiber cell, and crystallinity of cellulose had an impact on the mechanical properties of LS fiber bundles. Furthermore, the fiber bundles density of LS varies range of 385.46-468.70 kg/m^3, much less than that of jute (1360.40 kg/m^3) and Arenga engleri (950.20 kg/m^3) while LS fiber bundles has superior specific modulus.

Keywords: luffa sponge fiber bundles; mechanical properties; anatomical characteristic; moisture regain; thermal performance

1. Introduction

The luffa sponge, commonly referred to as vegetable sponge or luffa cloth, is obtained from matured dried fruit of luffa cylindrica. It consists of crisscrossed fibers and presents a three-dimensional reticular structure. The luffa cylindrica and luffa sponge are shown Figureure. 1.The luffa sponge is a rich resource for plants and is cultivated in the tropical countries of Asia and Africa and some sub-tropical areas. The plantation areas in China include Jiangxi, Henan, Sichuan, Guangdong, Jiangsu, Anhui and other places [1-3]. The luffa sponge is a low-density, eco-friendly resource which is non-toxic, biodegradable and has stable physical and chemical properties [4, 5]. There are two common varieties of luffa sponge, namely, luffa sponge of low density and high density, among whom the luffa sponge of high density is the improved variety [6]. The luffa sponges have a long history of being studied in China. The Chinese classics "Compendium of Materia Medica" recorded about the luffa performance 400 years ago and specified that “luffa sponge, with sweet and clod nature, has the effect of clearing heat, cooling blood, detoxifying, promoting circulation of blood and dredging collaterals”. The luffa sponge has been used as cleaning equipment (for instance, as a dish cloth), insole materials, and pillow filling materials. The advancement in science and technology has led to luffa sponges being widely used in several fields such as pharmaceutical engineering [7, 8], environmental engineering [9-11], biotechnology [12-14], and industrial products [15-17]. Since, luffa sponges are porous material with a high degree of lignification [18-20], they have a great potential for application in composite materials and fabric fibers.

Of particular interest when studying the luffa sponge fibers and its application in composite materials are the water absorption and mechanical properties of sponge gourd-polyester composites, luffa sponge cellulose and nanometer composite material, and the structure and mechanical strength of luffa sponge fiber bundles. Boynard et al. investigated the water absorption and mechanical properties of sponge gourd-polyester composites. The results have demonstrated that in comparison with general plant fiber reinforced composites, luffa sponge reinforced composite material has greater moisture absorption and lesser mechanical strength. However, these properties can be enhanced if a barrier layer avoids the close contact between the fiber and the

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external environment [21]. Hence, it is expected that water absorption in luffa sponge fiber has a great impact on the behavior of sponge gourd–polyester composites. However, till now, the water absorption properties of luffa sponge fibers haven't been given a significant attention. In 2010, Siqueira et al. investigated the behavior of luffa sponge nanocomposites and compared the mechanical properties of luffa cylindrical micro fibrillated cellulose (MFC) and whiskers films. The results showed that the degree of crystallinity had a significant impact on mechanical properties of luffa sponge nanocomposites [4]. Also, in 2012, Shen et al. studied the structural strength of luffa sponge column and indicated that structural strength has a close relationship with density of luffa sponge column [15]. Chen et al. performed a multi-scale study to understand the relationship between the structural and mechanical properties at different levels of its hierarchical organization (the fiber of inner layer, foam-like blocks of lateral position and different forms columns) and different orientations including, longitudinal direction, tangential direction and radial direction. The results showed that the inner layer fibers of hoop wall made significant contributions to longitudinal properties of luffa sponge column followed by the tangential properties, while the core part had lesser mechanical strength [22]. One of the possible explanations of the results could be the variable porosity distribution in the luffa sponge with lower porosity in the hoop wall and higher porosity in the core part. However, the mechanical strength of different parts and the effect of density on mechanical strength haven't been investigated yet.

Moreover, a significant number of studies have shown that, as the reinforcing phase of polymer composites, mechanical strength of fibers has a significant impact on the mechanical properties of polymer composites [23, 24]. Zhai et al. have indicated that the structural characteristics of diameter of fiber bundles, transverse sectional area occupied by fibers in fiber bundles, transverse sectional area occupied by vessels and phloem tissue in fiber bundles, diameter and wall thickness of fiber cell, microfibril angle and the degree of crystallinity have a great influence on the mechanical properties of windmill palm fiber bundles. In summary, it is essential to conduct an in-depth study on the moisture absorption, mechanical properties and structural characteristics for enhanced understanding of luffa sponge reinforced composite materials. In the current work, the moisture absorption, mechanical properties, structural characteristics, and the thermal performance of fiber bundles in four layers of luffa sponge including high density (31Kg/m³-65Kg/m³) and low density (15Kg/m³-30Kg/m³) have been studied.

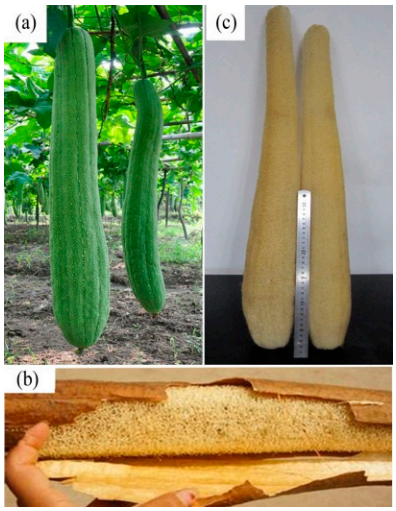

Figure 1. (a) luffa cylindrical, (b) luffa sponge with cortex, (c) luffa sponge.

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Figure 2. The luffa sponge of high density and low density (left: low density luffa sponge, right: high density luffa sponge)

2. Materials and methods

2.1. Sample preparation

In order to account for aging contraction, the two ends of luffa sponge were amputated and the middle section of luffa sponge columns (Figure 4a) which are similar to cylindrical ones were selected. Before collection, luffa sponge columns were rinsed several times in rinsing water and thereafter, dried. Subsequently, the luffa sponge columns were placed in humidity chamber at 65% relative humidity (RH) at 21°C for 24h. The humidity chamber was set up using distilled water and held at room temperature. The density of luffa sponge columns was calculated using Eq. (1) (2):

$$\rho = \frac{m}{v} \quad (1)$$

$$v = \frac{(s_1+s_2)\times 2}{H} \quad (2)$$

where ρ is density of luffa sponge columns in kg/m^3, M is the mass of luffa sponge column in Kg and v is the volume of luffa sponge columns in m^3. S_1 and S_2 are the areas of the top and bottom transverse section from luffa sponge column (excluding the porous structure on the transverse section), respectively. The transverse section of luffa sponge column is showed in Figure. 3. The area is expressed in m^2, while, H, the height of luffa sponge column, is expressed in m.

Luffa sponge can be classified into two categories on the basis of density-namely-low density (L.D) and high density (H.D). The density range of 15 to 30 kg/m^2 is considered as low density, while the density range of 31 to 65kg/ m^2 is considered as high density.

As shown in Figure 4, each luffa sponge column is composed of an outer, inter, middle and inner layer. The eight groups of single fiber bundles (length~ 30mm) were obtained from four layers in the luffa sponges of two densities for tensile strength test, moisture regain determination, and surface morphology observation. The single fiber bundles of the same length from jute and Arenga engleri were obtained for contrast test. In addition, the eight groups of short fiber bundles (length~2mm) were obtained from luffa sponge to prepare freezing microtome sections.

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The fiber bundles of four layers from luffa sponge of two densities, jute and Arenga engleri were dried in an air oven at 100 °C for 24 h, and thereafter, eight kinds of powder specimens were obtained by ball mailing for X-ray diffraction test (XRD) and thermogravimetric analysis test (TGA). The speed of ball mailing used was 170 turns/min, and the time of ball mailing was 5 minutes.

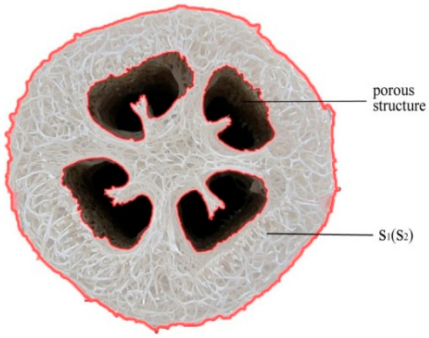

Figure 3. The transverse section of luffa sponge column

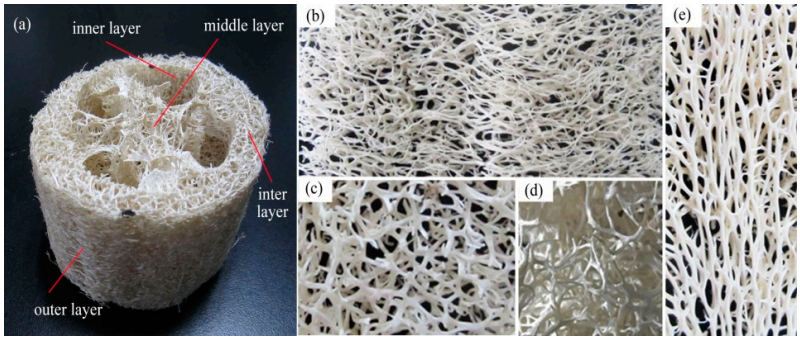

Figure 4. The geometrical features of luffa sponge column (a) Different regions, (b) Orientation of outer layer, (c) Orientation of inter layer, (d) Orientation of middle layer, (e) Orientation of inner layer.

2.2. Microscopic observation and imaging quantification

Subsequent to section collection, diameters of fiber bundles from luffa sponge, jute and Arenga engleri were measured via digital optical microscope (Nikon Corporation, Nikon Eclipse E100，Japan).

In order to observe the morphological characteristics of fiber cells in fiber bundles and measure the length of fiber cells in luffa sponge, five to ten fiber bundles were bound tightly together by cotton threads, cut into lengths of 20mm. Subsequently, they were segregated in solution of 1:1 glacial acetic acid and hydrogen peroxide for duration of 8h. Thereafter, fiber cells were rinsed several times until reaching a neutral PH. The digital optical microscope was used to observe morphological characteristics of fiber cells. The length measurements were carried out using MIAS image analysis software. In order to gather significant statistical data, 90 fiber cells from each group were tested.

To observe the micromorphology characteristics of transverse sections of fiber bundles, eight groups of fiber bundles (length~2mm) from luffa sponge were embedded in tissue freezing medium. The temperature of frozen embedding ranged from 23 to 13°C below zero. The tissue freezing medium was offered by AMOS SCIENTIFIC PHY.LTD. Australia. Thereafter, freezing microtome sections of 10um thickness were cut from embedded specimens using a freezing microtome (Hestion Co. Ltd., CM3800, China). A few of the transverse sections were stained with safranin in order to clearly observe the lignified tissue via digital optical microscope. The diameters of fiber cell, and the thickness and diameter of fiber cell wall were measured via Motic Images, an image analysis software.

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The other sections were observed under polarized-light microscopes (Olympus Corporation, BX51, Japan) in order to obtain data on fiber bundles including the area of transverse section (S), the amount of transverse sectional area occupied by fiber in a fiber bundles (S_F), and the amount of transverse sectional area occupied by non-fibers including cavity, vessels, and phloem tissue (S_N). The microscopes equipped with LCD OLYMPUS video camera (model DP70) were used to resolve images and Motic Images image analysis software was used to conduct measurements.

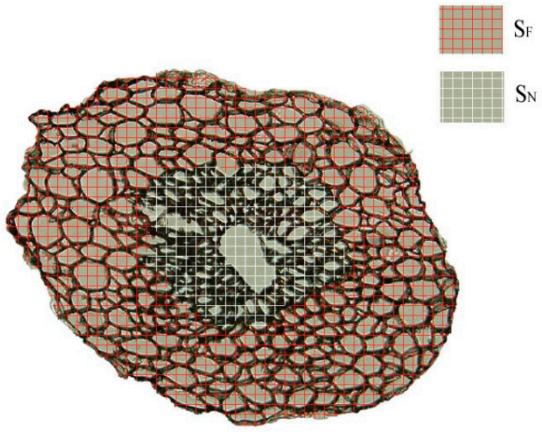

Figure 5. Areas of S_F and S_N in a fiber bundle of Luffa sponge. S_F: transverse sectional area occupied by fibers in a fiber bundle, S_N: transverse sectional area occupied by non-fibers (including cavity, vessels and phloem tissue) in a fiber bundle.

2.3. Tensile measurements

For the tensile strength measurements, the fiber bundles of luffa sponge, jute and Arenga engleri were air-dried till their moisture content ranged from 8 to 10% by weight (wt%) and thereafter, cut into sections of 30mm in length. All the tensile tests were conducted on an universal testing machine (Shimadzu Corporation, Shimadzu AG-X Plus, Japan.) with a 1KN load cell and the crosshead speed of 0.5 mm/min. The testing room conditions stayed around 25±2°C with a relative humidity (RH) of 70% . In order to immobilize fiber bundles, in accordance with ASTM D 3379-75 standard [25-28], a 15mm gauge length paper frame using two part epoxy adhesives was used in our study (Figure 6a). Prior to tensile measurements, the middle part of the supporting paper frames was cut as shown in Figure 6b. In order to obtain effective statistical data, 50 fibers from each group were tested. Furthermore, the mean diameter of each fiber bundle was measured using digital optical microscope (magnification =200x) at ten random points along the gauge length. As discussed by Munawar et al. [25, 27], a mean diameter was used to calculate fiber's transverse sectional area, which is a vital parameter for determining fiber tensile strength. Finally, the fractured fiber bundles were collected and fractured surfaces were observed via scanning electron microscope (Hitachi Co. Ltd., S-4800, Japan).

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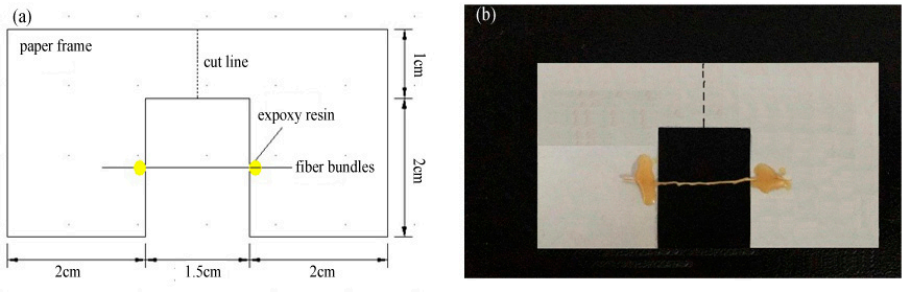

Figure 6. (a) Paper frame support used for tensile strength tests, (b) A fiber bundle is fixed on the paper frame by means of epoxy adhesive. The paper is then cut in two along the dotted line, and the paper supports are pulled apart.

2.4. Moisture regain

In accordance with standard ASTM D 2654-89a, bundles of single fibers bound together were weighed and dried in an air oven at 105 °C for 4 h. The samples were weighed using a balance with accuracy to four decimal places (±mg) and recorded as M_d. The samples were thereafter placed in humidity chamber at 65% relative humidity (RH) at 21 °C for 24 h [29, 30]. Subsequently, the fibers were weighed again after exposure to humidity and recorded as M_h. Finally, moisture regain (MR) was determined using Eq. (3):

$$MR = \frac{M_h - M_d}{M_d} \times 100\% \quad (3)$$

where M_h= mass of the sample after exposing it to humidity and M_d=mass of the dried sample.

2.5. *X-Ray Diffraction analysis*

The crystallinity index of the tested fibers was calculated from X-Ray Diffraction patterns recorded on an XRD-3 X-ray diffractometer (Beijing Purkinje General Instrument Co., Ltd., XRD-3, China). Diffractograms were obtained by varying angle (2θ) from 10° to 40°. The crystallinity index (C_rI) of the fiber was calculated using the Eq. (4) [31, 32]:

$$C_rI = \frac{I_{002} - I_{am}}{I_{002}} \times 100\% \quad (4)$$

where C_rI is relative crystallinity index, I_{002} is the maximum intensity of diffraction of the (002) lattice peak at a 2θ angle between 22° and 23°, and I_{am} is the intensity of diffraction of the amorphous material, which is taken at a 2θ angle between 18° and 19° when the intensity is minimal.

2.6. *Thermo-gravimetric analysis (TGA)*

In order to determine thermal stability of luffa sponge fibers, thermogravimetric analysis (TGA) was carried using TG209 thermogravimetric analyzer (Netzsch Corporation, TG209, Germany). The samples of weights between 5 and 10mg were placed in an alumina pan and heated from 30 to 700 ◦C. To prevent oxidation, TGA analysis was performed under nitrogen atmosphere at a flow rate of 20 ml/min. The heating rate was maintained at 10 °C/min during heating between 30 and 700 °C, kept the temperature at 100°C for 10 min [33].

3 Results and discussion

3.1. Structure of fiber bundles

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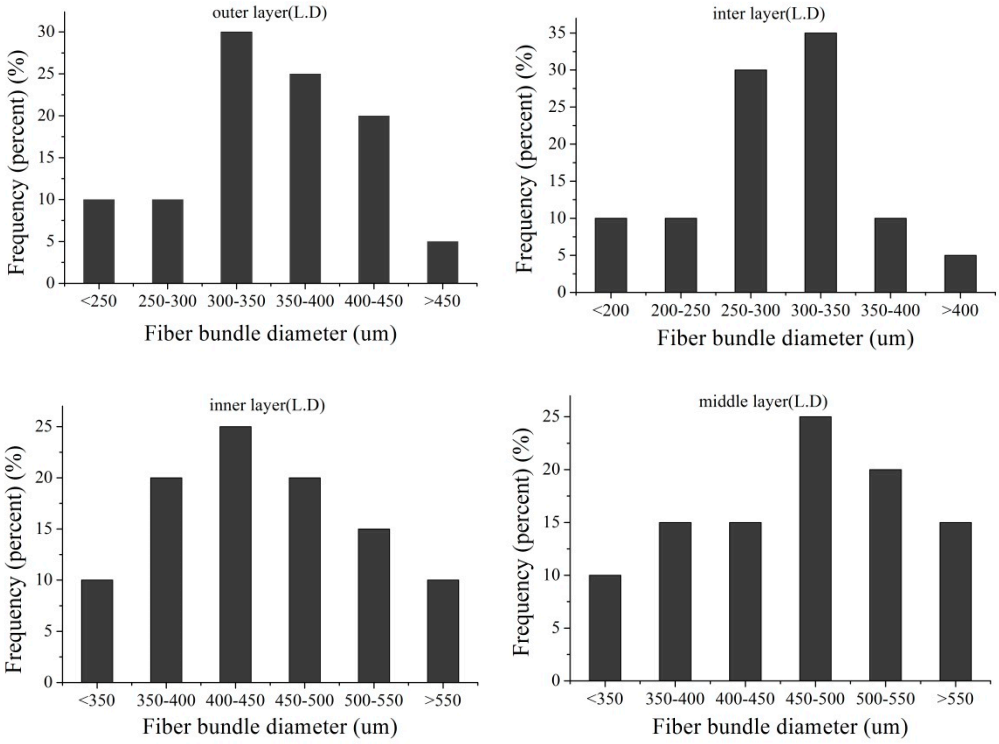
The difference in the diameters of fiber bundles of low density luffa sponge, high density luffa sponge, jute and Arenga engleri were investigated (Figure 7, Figure 8). Moreover, the diameters of four layers from density in same range are different. In the case of luffa sponge of L.D, the fiber bundles in middle layer and inner layer have a larger diameter followed by outer layer and inter layer. The diameter of fiber bundles in the outer, inter, inner and middle layer was 353.3um, 288.7um, 451.3um and 481.3um in turn. However, in the case of luffa sponge of H.D, the diameter of fiber bundles was the largest in the inner layer, followed by outer layer and middle layer, and the smallest is inter layer. The diameters of outer, inter, inner and middle layer were 550.3um, 457.0um, 634.3um, 535.3um, respectively. It was observed that there was a slight difference in diameters of middle layer fiber bundles between luffa sponge of L.D and H. D; on the contrary, there was a significant difference in diameters of fiber bundles in the hoop wall (including outer layer, inter layer and inner layer). The mean diameter of fiber bundles in the hoop wall of luffa sponge of L.D is about 200 um larger than that of luffa sponge of H.D. The structural division of the sponge is shown in Figure 4. Each layer of the sponge has its unique feature. The outer layer is composed of thin fibers with the orientation roughly surround the sponge (Figure 4b). The fiber bundles of inner layer running along the vertical axis were straight (Figure. 4e). The inter layer consisted of a complex branched fiber network (Figure. 4c). In comparison with the hoop wall, fibers of the core part were loosely interconnected with each other, and a thick single fiber ran along the central line of the complete luffa sponge (Figure 4d) [4].

Also, the diameters of fiber bundles from Arenga engleri were distributed in the range of 170 to 320um and those of jute had a relatively more concentrated distribution in the range of 40 to 120um. Zhai et al. indicated that mean diameters of outer layer from sheath of windmill palm is 345um, while those of middle layer and inner layer were 418um and 202um, respectively. The diameters of Sisal aggregates (Agava sisalana) have been reported to be in the range of 100–400 um in the literature while the typical diameter of coir fiber bundles from the coconut palm (Cocos nucifera) has been reported to be around 200 um [34, 35]. Hence, the fiber bundles of luffa sponge have larger diameters than common plant fibers.



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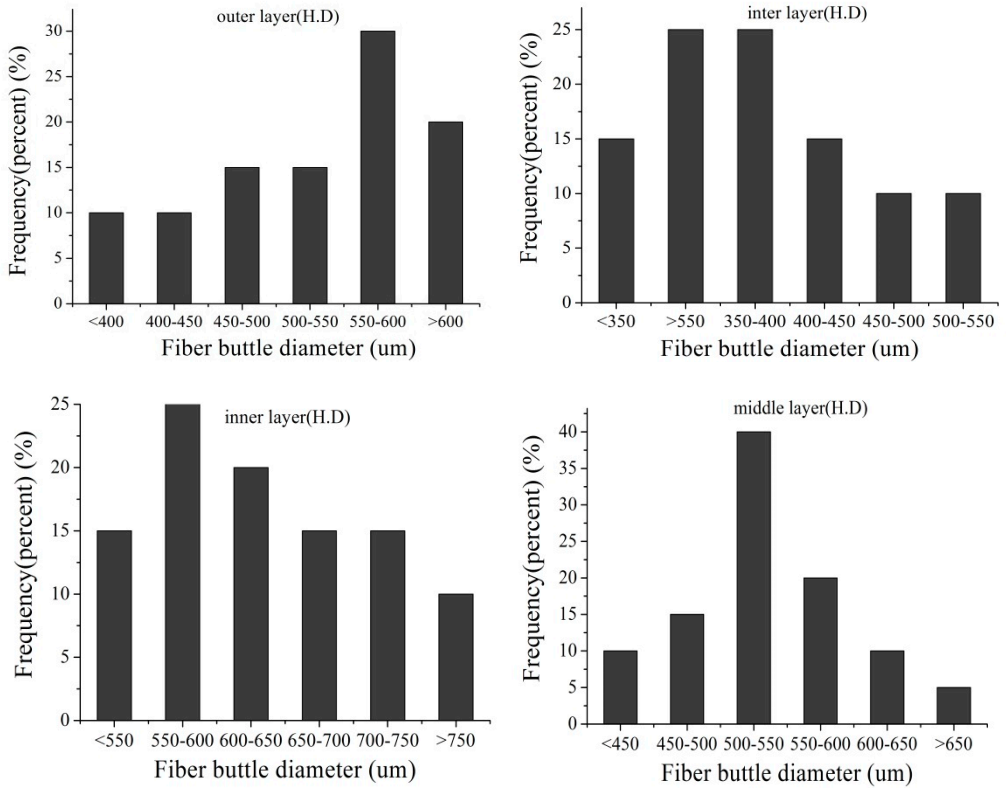
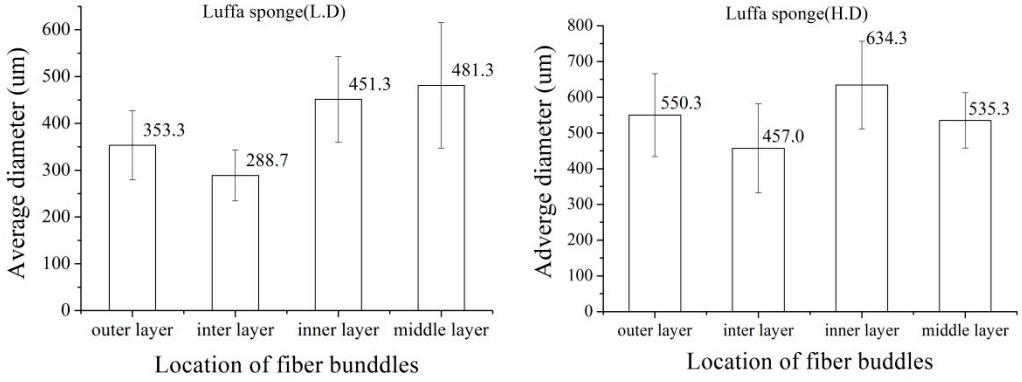
(a)



(b)

Figure 7. The diameter distributions of fiber bundles (a) taken from luffa sponge of low density, (b) taken from luffa sponge of high density.



(a)

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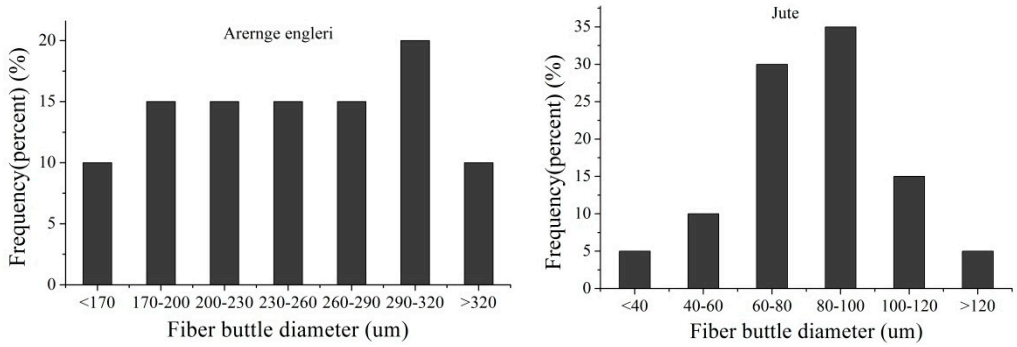

(b)

Figure 8. (a) The average diameters of fiber bundles taken from luffa sponge, (b) The diameter distribution of fiber bundles taken from Arernge engleri and jute.

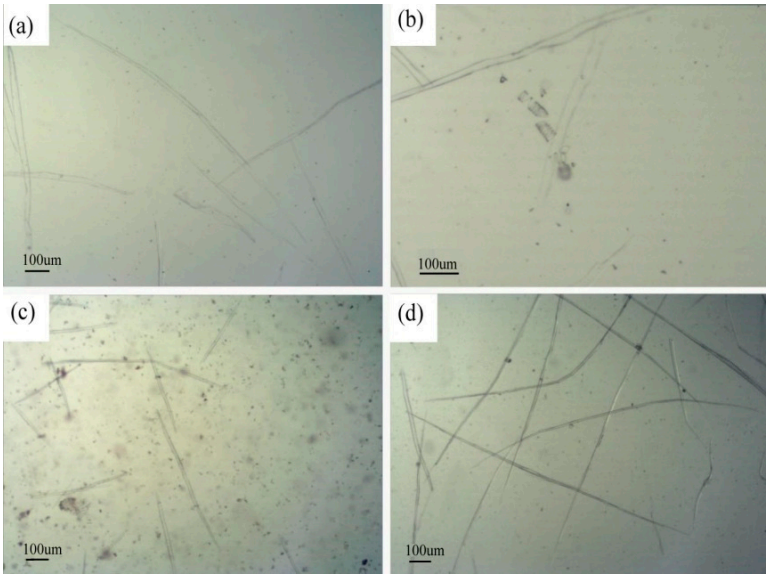

Figure 9. Morphology of fiber cells (a) Fiber cells of luffa sponge, (b) Threaded pipe of luffa sponge, (c) Fiber cells of Arenga engleri, (d) Fiber cells of Jute.

Table 1. Fiber cell characteristics of luffa sponge, Arernge engleri and jute.

		Fiber cell of luffa sponge(L.D)				Fiber cell of luffa sponge(H.D)				Arenga engleri	Jute
		outer layer	inter layer	inner layer	middle layer	outer layer	inter layer	inner layer	middle layer		
Length(um)	Mean	1359	1708	1610	1331	1594	1075	1488	1072	717	2331
	SD	430	450	482	398	503	349	602	458	14.98	36.14
Diameter(um)	Mean	25.25	22.29	20.78	24.35	22.37	17.32	26.53	22.65	14.14	19.6
	SD	6.57	6.48	5.19	7.23	6.34	3.5	7.08	5.71	0.33	0.13
Lumen	Mean	21.23	23.82	17.45	20.53	17.16	12.52	19.04	16.55	8.9	6.11
diameter(um)	SD	6.32	6.09	4.99	7.12	5.09	3.44	6.15	5.05	0.24	0.11
Wall	Mean	4.02	4.41	3.51	3.82	5.21	3.85	7.48	6.1	5.24	7.41
thickness(um)	SD	0.83	0.82	0.72	0.74	2.16	1.02	1.96	1.74	0.08	0.07

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ratio of wall	Mean	0.19	0.18	0.2	0.18	0.3	0.29	0.39	0.36	0.59	1.21
to lumen	SD	0.06	0.05	0.07	0.07	0.12	0.10	0.15	0.17	0.01	0.05

The Morphology and characteristics of fiber cells from luffa sponge, Arenga engleri and jute are present in Figure 9. and Table 1. The fiber cell of luffa sponge is shuttle-like in shape and is easy to bend. It has a characteristic length in the range of 1050-1070um, diameter in range of 17-28um, lumen diameter in range of 12-24um, and wall thickness in range of 3-8um. There is a sight difference in length and diameter between luffa sponge of L.D and H.D. Nevertheless, the wall thickness and ratio of wall to lumen of fiber cell from luffa sponge of H.D is larger than that of L.D (excluding the inter layer).

Secondly, the fiber cell of luffa sponge is longer than that of Arenga engleri, but shorter than that of the jute. The ratios of wall to lumen for luffa sponge are significantly smaller than that of Arenga engleri and jute. Thus, it can be concluded that in comparison with other common plant fiber cells, the fiber cell of luffa sponge contains less material substance, which is the primary reason for lightness of luffa sponge. Furthermore, the ratio of wall to lumen of fiber cells in inner layer of luffa sponge of L.D and H.D is 0.20 and 0.39, respectively, and is slightly larger than that of the other layers.

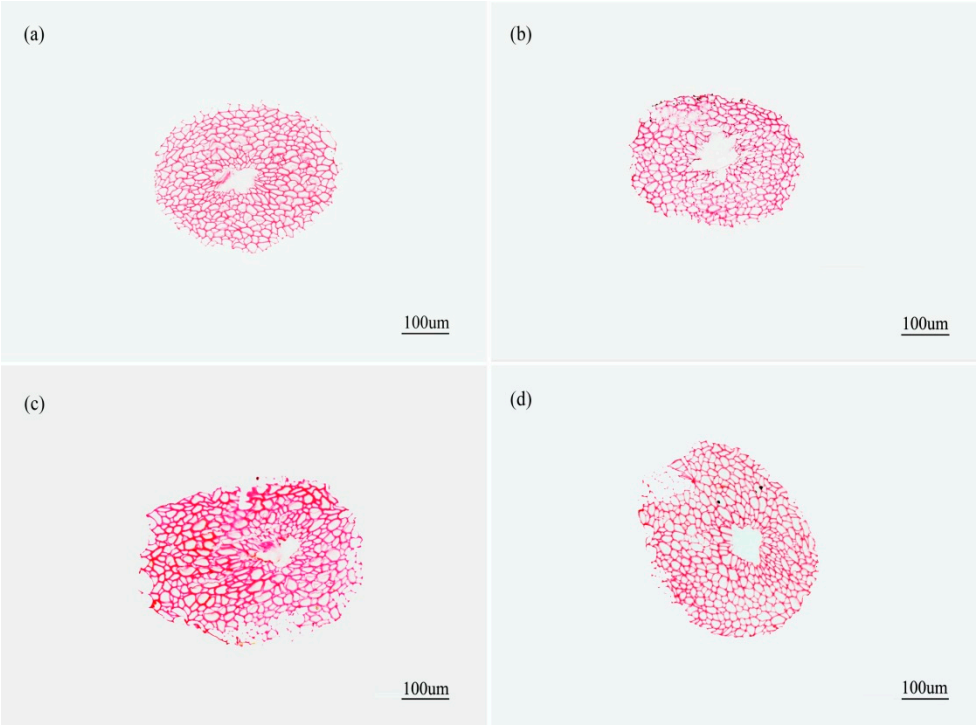

Figure 10. Transverse sections of fiber bundles taken from luffa sponge of low density (a) outer layer, (b) inter layer, (c) inner layer, (d) middle layer.

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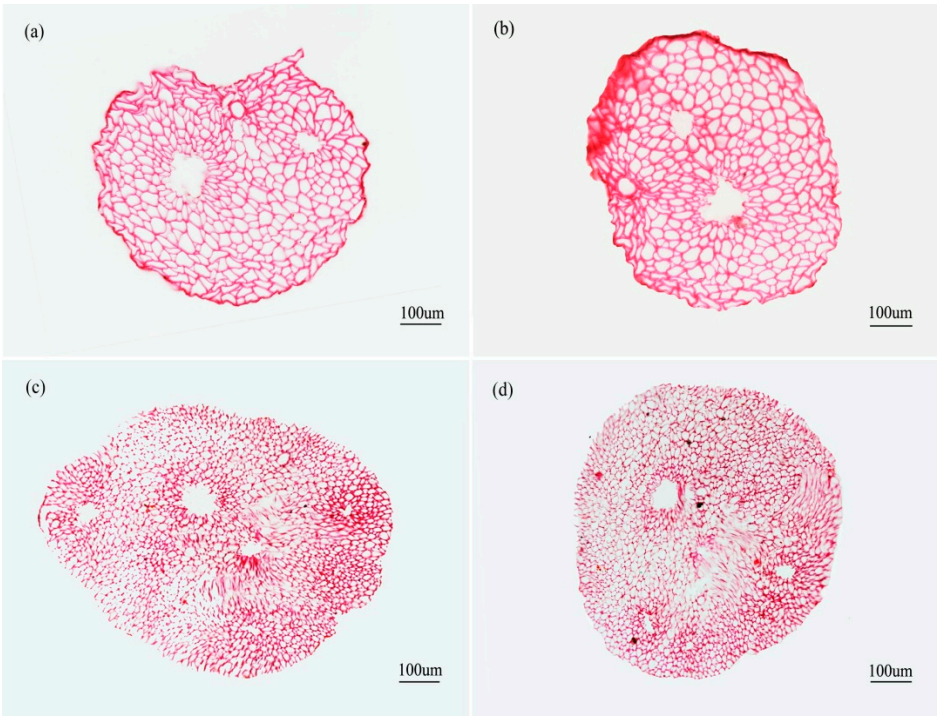

Figure 11. The transverse sections of fiber bundles taken from luffa sponge of high density, namely (a) outer layer, (b) inter layer, (c) inner layer, and (d)middle layer.

Figure 12. The transverse section of fiber bundles taken from Arenga engleri.

The transverse sections of the safranin-stained fiber bundles clearly revealed lignified tissue. According to Figure10, Figure 11, Figure 12 the diameters of fiber bundles from luffa sponge of H.D is significantly larger than that of luffa sponge of L.D. Besides, the fiber bundles from Arenga engleri have smaller diameters than those of luffa sponge. It can be observed that fiber bundles of luffa sponge primarily consist of fiber cells. According to Figure. 10 and Figure. 11, there are cavities, with a small number of vessels and phloem tissue nearby, around the center of fiber bundles [36]. However, as shown in Figure 12, similar phenomenon could not be observed in the fiber bundles of Arenga engleri. Moreover, it was also discovered that there are two or more cavities with uneven distribution in the fiber bundles of luffa sponge of H.D, while there is only one cavity around the center of fiber bundles in luffa sponge of L.D.

Figure. 13 shows transverse sectional images of the fiber bundles of luffa sponge as observed in the dark field by polarized-light microscope. The dark regions in the transverse sectional of each individual fiber bundle were clearly observed. In combination with Figure 13, two or more dark regions existed in the fiber bundles of

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luffa sponge of H.D, while only one dark region existed near the center of the fiber bundles from luffa sponge of L.D. The dark regions were proved to be the cavities, vessels and phloem tissue.

Hence, S, S_F and S_N of a fiber bundle, were measured for each of the four layers of luffa sponge. Statistical data is shown in Table 2, where S_F% is the ratio of S_F to S, and S_N% is the ratio of S_V to S. The S_N% of luffa sponge is in the range of 13-28%. The S_N% of fiber bundles of corresponding layer in luffa sponge of H.D is larger than that of L.D. The value of S_N% represents the immaterial substance in the fiber bundles; in other words, the lower is the value of S_N%, the higher is the amount of substantial fibers in fiber bundles. Zhai et al. have indicated that the amount of substantial fibers have an important influence on the mechanical properties of fiber bundles [25].

Table 2. Fiber bundles characteristic of luffa sponge

	Fiber bundles of luffa sponge(L.D)				Fiber bundles of luffa sponge(H.D)			
	outer layer	inter layer	inner layer	middle layer	outer layer	inter layer	inner layer	middle layer
S (100 um^2)	1100	940	1277	1381	1819	1481	2000	1766
S_F (100 um^2)	878	705	1109	1050	1307	1096	1568	1413
S_N (100 um^2)	222	235	168	331	512	385	432	353
S_F%	80%	75%	87%	76%	72%	74%	78%	80%
S_N%	20%	25%	13%	24%	28%	26%	22%	20%

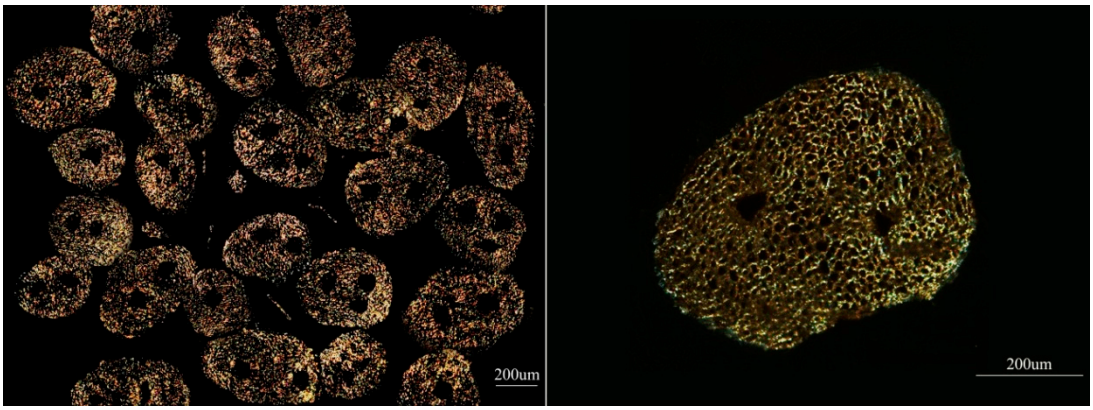

Figure 13. Transverse section image of fiber bundles taken from high density luffa sponge observed by polarized-light (The noticeable dark region is the area occupied by non-fiber including cavity, vessels and phloem tissue).

3.2. Mechanical properties

The typical stress-strain curves for fiber bundles from Arenga engleri, jute and four layers in luffa sponge were obtained (Figure 14). The curves showed a yielding, followed by plastic deformation until breakage from 3 to 10% strain for fiber bundles of luffa sponge. Table 3 presents the mechanical properties of fiber bundles taken from Arenga engleri, jute, and four layers in luffa sponge. In comparison to fiber bundles in each layer from luffa sponge of H.D, the fiber bundles in corresponding layer from luffa sponge of L.D demonstrated higher tensile strength, Young's modulus (excluding the middle layer), and elongation. In other words, the mechanical properties of single fiber bundles in luffa sponge of L.D were superior to luffa sponge of H.D. The phenomenon may be attributed to S_N%, since the S_N% of the fiber bundles in each layer (excluding middle layer) of luffa sponge of H.D are higher than the corresponding layer of luffa sponge of L.D. The presence of

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fibers predominantly contributes to the mechanical strength of the fiber bundles while the presence of cavities vessels and phloem tissue tends to reduce the mechanical strength [25]; In other words, higher the value of S_N% in fiber bundles, lower is the amount of substantial fibers in fiber bundles, resulting in worse mechanical properties. In the case of same kind of luffa sponge, mechanical property of fiber bundles in each layer also decreased with the increase of S_N% in fiber bundles. Moreover, the mechanical property of fiber bundles from luffa sponge of H.D decreased dramatically with a slight increase in S_N%. There are several factors responsible for these results, one of which is the presence and contribution of cavities in the transverse section of luffa sponge. The simplified models for transverse section of fiber bundles taken from two-density luffa sponge are shown in the Figure. 15. As mentioned above, only one cavity was observed near the center in fiber bundles from luffa sponge of L.D, resulting in a circular distribution of fiber cells, while two or more cavities contributed at random in fiber bundles from luffa sponge of H.D, making the fiber cells unable to gather. The mechanical properties of the fiber bundles are a combination of the mechanical strength of all the fiber cells and the adhesion among fiber cells [37, 38].The aggregation degree of fiber has an important influence on the mechanical properties of fiber bundle. For instance, higher aggregation degree of fiber cells results in better mechanical property of fiber bundles. This is one of the reasons for lower mechanical strength of fiber bundles from luffa sponge of H.D as compared to the ones from luffa sponge of L.D.

Also, it is interesting to note that the density of fiber bundles taken from luffa sponge is in the range of 385.46-468.70kg/m^3 and is far lower than that of fiber bundles from Arenga engleri (950.20kg/m^3) and jute (1360.40kg/m^3). It could be that the low density for luffa sponge fiber bundles due to the hierarchical polyporous structure for fiber bundles and fiber cells as shown in Figure. 15. Hence, luffa sponge fiber bundles are a low-density and light-weight natural fiber material. Although, the tensile strengths of luffa sponge fiber bundles are lower than those of fiber bundle from Arenga engleri and jute, the specific modulus of luffa sponge fiber bundles were in a normal range. Specifically, specific modulus of luffa sponge of L.D is higher than that of Arenga engleri fiber bundles. The natural plant fiber, as the reinforcement of the composite material, with higher mechanical strength and specific modulus, can manufacture the polymer matrix composite material with advantages of portability and good mechanical properties [39, 40].

Table 3. Mechanical properties of single fiber bundles in different layers taken from two-density luffa sponge and single fiber bundles of Arernga engleri and jute.

		Density (kg/m^3)		Young's modulus (MPa)		Specific modulus (MPa*m^3/Kg)		Tensile strength[a] (MPa)		Elongation (%)	
		mean	SD	mean	SD	mean	SD	mean	SD	mean	SD
Luffa sponge (L.D)	outer layer	458.31	2.37	1208	145.9	2.64	1.54	73.46	34.25	4.98	3.94
	inter layer	431.85	1.68	958	236.5	2.22	1.23	53.82	17.42	5.16	3.95
	inner layer	468.70	0.60	1897	320.1	4.05	1.24	91.63	14.32	9.81	3.19
	middle layer	443.80	3.69	918	245.9	2.07	0.78	61.80	24.22	6.27	2.43
Luffa sponge (H.D)	outer layer	391.50	0.93	635	82.09	1.62	0.56	24.03	3.12	2.54	0.39
	inter layer	385.46	1.22	555	193.1	1.44	0.43	24.31	8.8	3.2	1.62
	inner layer	411.73	1.91	1133	299.8	2.75	1.03	39.91	11.94	3.73	1.19
	middle layer	421.90	1.69	880	577.2	2.09	0.85	42.55	8.18	6.67	3.70
Arenga engleri		950.20	0.90	1735	187.5	1.83	0.58	102.34	13.18	15.76	3.58
Jute		1360.40	0.40	25119	530.9	18.46	3.21	378.59	113.14	0.83	0.25

[a] Excluding the S_N area and recalculating tensile strength using the effective area (S_F).

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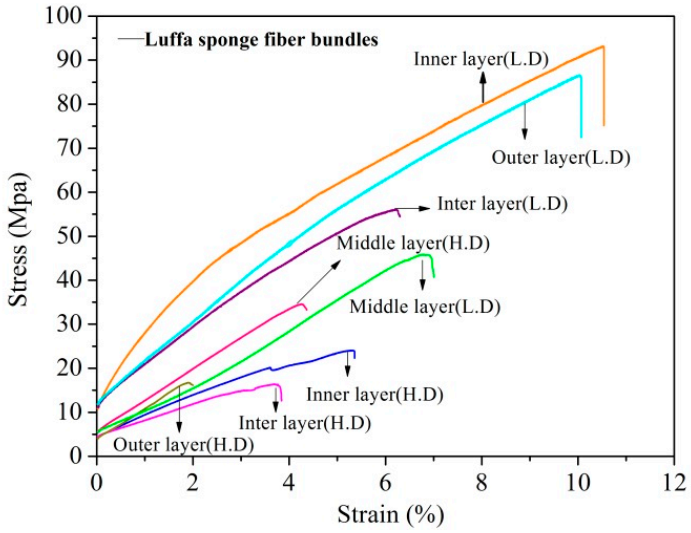

Figure 14. Typical stress–strain curves of fiber bundles in different layers taken from two-density luffa sponge.

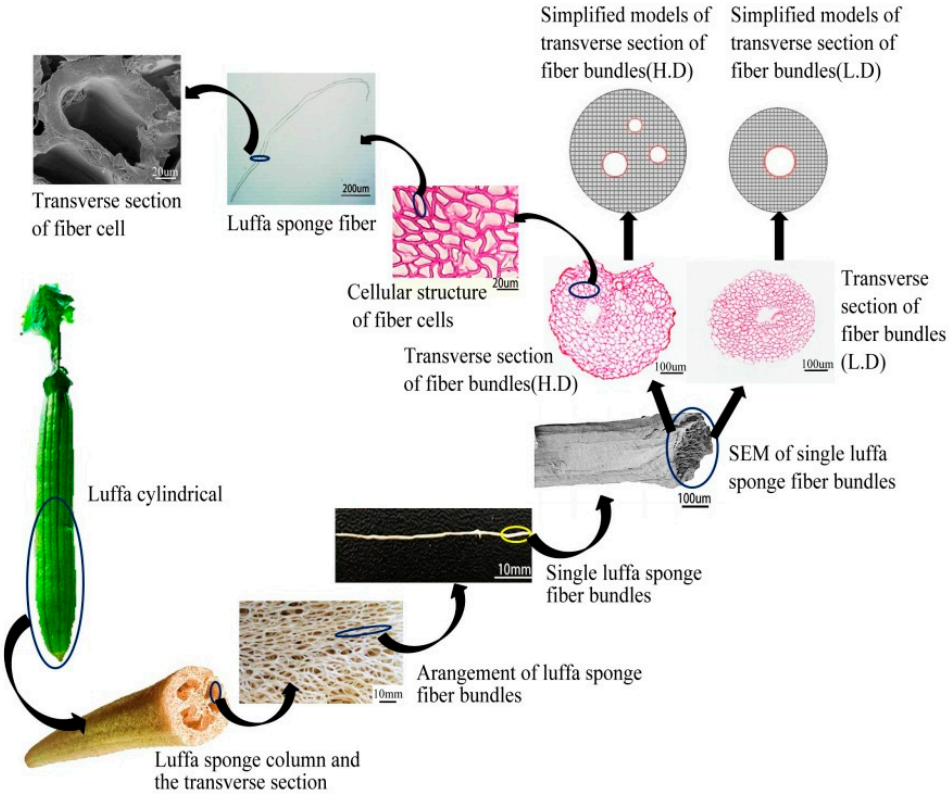

Figure 15. The hierarchical polyporous structure of luffa sponge (including luffa sponge column, luffa sponge fiber bundles and luffa sponge fiber cells)

3.3. SEM analysis

In order to further understand the fracture mechanism, the fracture surface of broken fiber bundles was observed under SEM. As shown in Figure. 16a and Figure. 16c, fiber cell of luffa sponge of L.D has an obvious

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slip phenomenon at broken, while the fracture surface of fiber cell of luffa sponge of H.D is flat. Since each fiber cell was connected with the surrounding material, external force had to overcome the strains of fiber cells and the adhesion stress among fiber cells, when being stretched. If the strength of fiber cells was superior to adhesion stress, the fiber cells would survive, otherwise the fiber cells would break. In this regard, the strength of the fiber cells of luffa sponge is relatively high, and is the primary reason behind superior mechanical strength of fiber bundles from luffa sponge of L.D in comparison with luffa sponge of H.D.

Furthermore, as shown in Figure. 16e and Figure. 16f, abundant spring-like spiral vessels were observed in the fiber bundles. It was observed that the behavior of pull-out spiral vessels were similar to that of the spring lost elasticity.

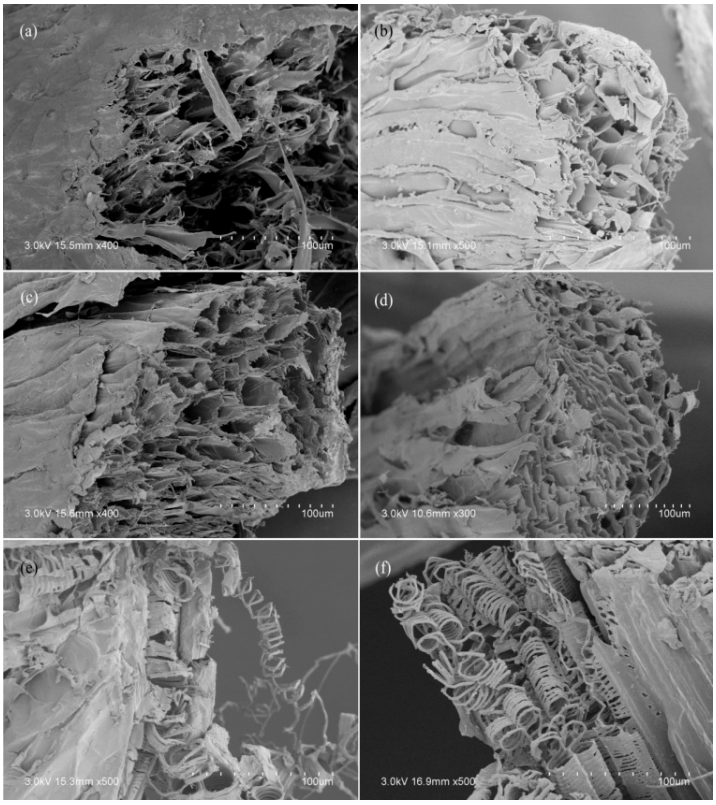

Figure 16. Fracture surface of fiber bundles taken from two-density luffa sponge (a) (c) (e) are from luffa sponge of low density, (b) (d) (f) are from luffa sponge of high density.

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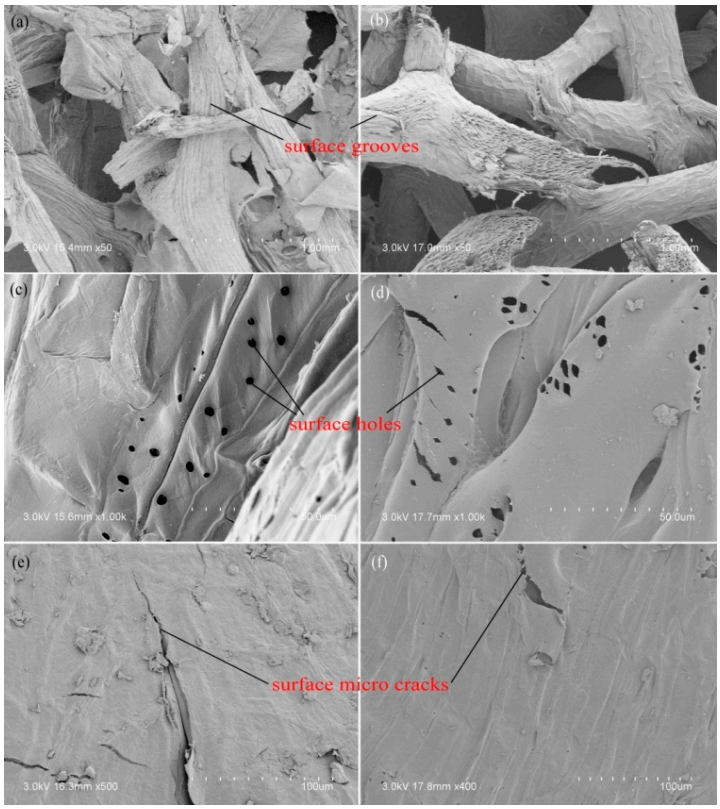

Figure 17. The surface topography of fiber bundles taken from two-density luffa sponge (a) (c) (e) are from luffa sponge of low density, (b) (d) (f) are from luffa sponge of high density.

In Figure. 17, representative surface topography of luffa sponge fiber bundles are presented. It was observed that the grooves, holes and micro cracks on the surface of the fiber bundles from luffa sponge of L.D were more abundant than those on luffa sponge of H.D.

3.4. Moisture regain

The moisture regain of fiber bundles from luffa sponge, Arenga engleri and jute is presented in Table 4. It was observed that the luffa sponge fiber exhibited a higher moisture regain than Arenga engleri and jute. The higher moisture regain could probably be attributed to the presence of the porous structure, surface groove, and micro cracks in luffa sponge fiber bundles. Tan et al. have found that slender hole and surface exists in the bamboo fiber and indicated that these were important for fine moisture regain and moisture dispersion [41]. In comparison with the luffa sponge of H.D, luffa sponge fiber of L.D has higher moisture regain in the range of 10.2-10.9%, as compared to 7.1-9.3% for luffa sponge of H.D. There were slight differences in moisture regain among four layers in fiber from luffa sponge fiber of L.D., while those in the corresponding layers in luffa sponge fiber of H.D were significant. In the case of luffa sponge fiber of H.D, the middle layer demonstrated highest moisture regain of 9.3%, while, the inner layer demonstrated the lowest moisture regain of 7.1%. Additionally, as shown in Figure. 17, the grooves, holes and micro cracks on the surface of the fiber bundles from luffa sponge of L.D were more abundant than those on luffa sponge of H.D. These results could be a possible explanation for higher moisture in luffa sponge fiber of L.D as compared to luffa sponge fiber of H.D. The moisture regain is an essential parameter as it affects the dimensional and physical properties of the fibers. The moisture regain influences the dimensional stability, electrical resistivity, tensile strength, porosity and swelling behavior of natural fiber reinforced composites [42, 43]. In this regard, low moisture content of luffa

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sponge fiber of H.D is beneficial for fabricating luffa sponge fiber reinforced polymer composites due to their lesser ability to hold up water molecules.

Table 4. The moisture regain of luffa sponge fibers.

	Luffa sponge(L.D)				Luffa sponge(H.D)				Arenga engleri	Jute
	outer layer	inter layer	inner layer	middle layer	outer layer	inter layer	inner layer	middle layer		
Moisture regain	10.4%	10.2%	10.2%	10.9%	8.9%	7.1%	9.3%	8.8%	8.0%	7.3%

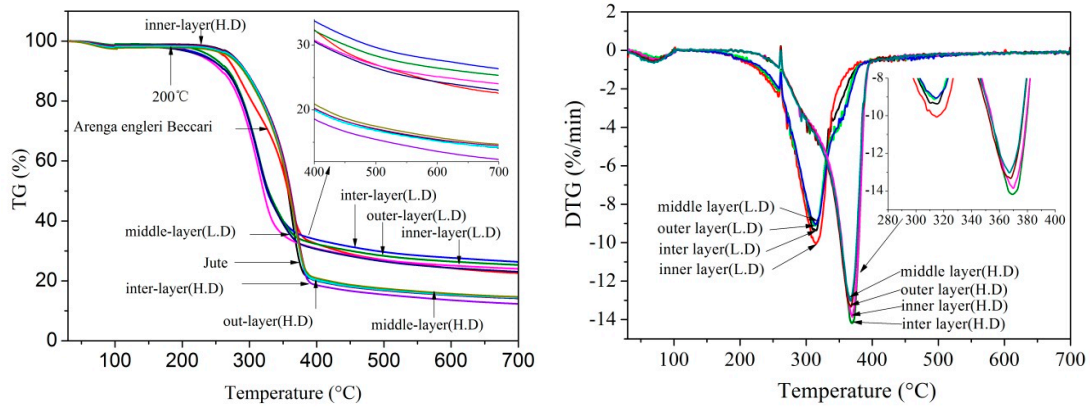
3.5. X-Ray Diffraction analysis

Table 5 shows the relative crystallinity of luffa sponge, Arenga engleri and jute. The relative crystallinity of each layer of luffa sponge of L.D is higher than that of corresponding layer in luffa sponge of H.D, leading to better mechanical strength of luffa sponge of L.D. Moreover, the trend of variation in relative crystallinity is in accordance with the variation trend of mechanical strength of luffa sponge, indicating that relative crystallinity had some influence on mechanical properties of luffa sponge. However, in comparison with Arenga engleri and jute, luffa sponge fiber bundles have higher relative crystallinity but lower mechanical properties. The possible reason could be the existence of cavities and smaller ratio of wall to lumen for luffa sponge. Furthermore, in the case of luffa sponge of H.D, the fiber bundles in middle layer have lower relative crystallinity but demonstrated better mechanical properties. The luffa sponge fibers, as the natural fibers, have complex chemical components with the complex crosslinking of lignin and hemicellulose. All the above factors certainly affect the mechanical properties of luffa sponge fiber bundles.

Table 5. The relative crystallinity of luffa sponge, Arenga engleri and jute.

	Luffa sponge(L.D)				Luffa sponge(H.D)				Arenga engleri	Jute
	outer layer	inter layer	inner layer	middle layer	outer layer	inter layer	inner layer	middle layer		
Relative crystallinity (%)	42.3	37.7	47.1	30.1	39.0	33.1	39.6	24.4	29.0	31.8

3.6. TGA



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(a) (b)

Figure 18. (a) TGA curves of luffa sponge, Arenga engleri and jute, (b) TGA–DTG curves of different layer from luffa sponge of low density and high density.

Table 6. Thermogravimetric Analysis of fibers taken from luffa sponge, Arenga engleri and jute.

		initial degradation temperature /°C	final degradation temperature/°C	peak degradation/°C	Final residue/%
Luffa sponge (L.D)	outer layer	269.6	309.8	276.1	32.7
	inter layer	270.2	346.1	314.1	35.1
	inner layer	270.3	344.6	307.8	31.9
	middle layer	271.3	350.8	314.3	34.0
Luffa sponge (H.D)	outer layer	321.4	384.8	368.3	19.2
	inter layer	321.9	387.4	366.3	18.7
	inner layer	324.6	386.5	369.9	19.2
	middle layer	319.7	385.7	367.0	21.0
Arenga engleri		300.9	383.0	357.4	31.0
Jute		325.6	379.3	365.7	19.6

Figure. 18 and Table 6 show the curves and TG and DTG data of luffa sponge, Arenga engleri, and jute. As shown in Figure. 18, the TG curves of each layer of luffa sponge, Arenga engleri and jute are similar. The biomass combustion included four main phases: water evaporation stage, the devolatilization and combustion stage, the fixed carbon combustion stage, and the burnout stage [44]. As shown in Figure. 18, the degradation temperatures of each layer from luffa sponge of L.D are higher than those of corresponding layer from luffa sponge of H.D In the case of luffa sponge of H.D, initial degradation temperature is in the range of 319.7-324.6°C, final degradation temperature is in range of 384.8-387.4°C and the peak degradation is in range of 366.3-369.9°C, while those of luffa sponge of L.D is 269.6-271.3°C, 309.8-350.8°C and 276.1-314.3°C, respectively. It is generally agreed that the higher the relative crystallinity, the better is the stability, including thermal stability of fibers [45]. However, our results have shown that fiber from luffa sponge of L.D has higher relative crystallinity, but they demonstrate worse thermal stability. The difference in content of cellulose, hemicellulose and lignin in both kinds of luffa sponge should be taken into consideration. Under normal circumstances, firstly, the hemicellulose degrades, followed by cellulose and lignin. Hence, the lignin content in luffa sponge of H.D could be higher. Furthermore, the thermal stability of luffa sponge of H.D is similar to jute and superior to Arenga engleri. In the case of jute, initial degradation temperature is 325.6°C, final degradation temperature is 379.3°C and the peak degradation is 365.7C, while those of Arenga engleri are 300.9°C, 383.0°C and 357.4°C, respectively. In 2004, Tang et al. reported that, for the bamboo fibers, the initial degradation temperature is 342.6°C, final degradation temperature is 380.4C and the peak degradation is 368.8°C [41]. In 2016, Ridzuan et al. reported that the peak degradation of Pennisetum purpureum is 364.7°C. Hence, it is evident that the fiber of luffa sponge of H.D is the preferred natural fiber material in regards to thermal stability.

4. Conclusions

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This study investigated the relationship between their structural characteristics and mechanical properties for luffa sponge fiber bundles and its moisture regain, thermal performance as the reinforcement of lightweight composite. From above discussion, the following conclusions can be drawn:

1. Luffa sponge fiber bundles are the natural fiber materials of light texture with a density range of 385.46-468.70kg/m^3 which is significantly lower than that of Arenga engleri (950.20kg/m^3) and jute (1360.40kg/m^3). Furthermore, luffa sponge fiber bundles have excellent specific modulus. Specifically, specific modulus of luffa sponge fiber bundles of L.D (2.07-4.05MPa*m^3/Kg) is even higher than Arenga engleri, considering the fact that luffa sponge fiber bundles have lower tensile strength.

2. Special structural characteristics of luffa sponge fiber bundles have a significant impact on the mechanical properties. The phloem tissues of vascular bundle degenerate to cavities leading to a reduction in the mechanical strength of luffa sponge fiber bundles. Interestingly, only one cavity was observed in transverse section fiber bundles from luffa sponge of L.D, while two or more cavities were observed in fiber bundles from luffa sponge of H.D. The tensile tests proved that luffa sponge fiber bundles of H.D possess lower mechanical strength than luffa sponge fiber bundles of L.D. From the point of structural features, the mechanical properties of the fiber bundles were affected by the ratio of SN, the number and contribution of cavities in the transverse section of luffa sponge fiber bundles. In other words, luffa sponge fiber bundles would have excellent mechanical property if they have high ratio of SN, even-distribution and high aggregation degree of fiber cell.

3. The smaller wall thickness of fiber cell and the cavity in the transverse section contribute to lower density of luffa sponge fiber bundles than jute and Arenga engleri fiber bundles. The luffa sponge fiber cells had higher ratio of wall thickness to lumen; in other words, the luffa sponge fiber cells had more substantive material. Hence, in this regard, fiber bundles from luffa sponge of H.D would ideally have possessed better mechanical strength. However, it is in contrast with our results that fiber bundles from luffa sponge of L.D have superior tensile strength. The probable reason for the discrepancy could be the higher crystallinity of cellulose in luffa sponge of L.D.

4. The luffa sponge fiber bundles had relatively higher moisture regain in comparison with the common fibers. The possible explanation for the results could be the porous structure, the surface grooves and the surface micro cracks in the luffa sponge fiber bundles. The moisture regain of luffa sponge fiber bundles of H.D (7.1-9.3%) was lower than that of luffa sponge fiber bundles of L.D (10.2-10.9%). Moreover, each layer fibers of luffa sponge had significantly different moisture regain in case of luffa sponge of H.D while the corresponding difference was negligible in luffa sponge of L.D.

5. The biomass combustion of luffa sponge fiber consisted of four main phases: the water evaporation stage, the devolatilization and combustion stage, the fixed carbon combustion stage, and the burnout stage. There were remarkable differences in thermal degradation temperatures between luffa sponge fiber of L.D and H.D. The luffa sponge fibers of H.D demonstrated better thermal performance than luffa sponge fiber of L.D due to lower crystallinity of cellulose. This result is in contradiction with the widely accepted notion that higher the crystallinity of cellulose, the better is the thermal stability of fiber. The difference in the content of cellulose, hemicellulose and lignin in two kinds of luffa sponge is a considerable factor for thermal performance. The luffa sponge fiber of H.D has relatively more lignin.

Acknowledgments: The current research work was supported by the Doctoral Fund of Ministry of Education of China under Grant Numbers 20133418120008, Anhui Provincial National Science Foundation under Grant Numbers 1308085QH133, and China Postdoctoral Science Foundation under Grant Numbers 2013M531499. The authors also thank Dagang Li, Zhongkai Wang, Liang Zhou, Yamei Liu, and Yunian Wang for their assistances in conducting numerous analyses.

Author Contributions: Yuxia Chen and Na Su participated in the design of this study, they both performed the statistical analysis and drafted the manuscript. Yong Guo carried out the study, collected important background information, carried out literature search, manuscript editing and performed manuscript review. Kaiting Zhang, Liushi Zhu and Lei Zhao carried out the concepts, definition of intellectual content, literature search, data acquisition and manuscript preparation. Linyan Ren provided assistance for data acquisition. Fei Fang provided assistance for manuscript editing. All authors read and approved the final manuscript.

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Conflicts of Interest: The authors declare no conflict of interest.

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