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This paper presents an approach to examine the microstructural properties and mechanical behavior of coconut husks, banana pseudo-stem, pineapple leaf, and sugarcane bagasse fibers by scanning electron microscope and mini-tensile tester, respectively. Single fiber bundles were examined by using scanning electron microscope. Tensile tests were performed at different diameters (0.15–0.55 mm) and gauge lengths (10, 15, 20, and 30 mm/min) to assess the effects of diameter and gauge length on tensile properties. It was found that fibers consisted of different types of regularly arranged cells. The tensile strength (310 MPa) and Young's modulus (7.4 GPa) of pineapple leaf fiber bundles showed the highest value compared to the other fibers. The tensile strength and Young's modulus decreased with the increase of diameter and gauge length of fiber bundles. Scanning electron microscopic fractography analysis showed comparatively heterogeneous ruptures associated with more participants of microfibrils for pineapple leaf and banana pseudo-stem fibers compared to coconut husk and sugarcane bagasse fibers. These fractographic observations were discussed in the light of current knowledge of the microstructure of each fiber and the corresponding mechanical properties.

Keywords: agricultural residue, fiber diameter, fractography, gauge length, mechanical properties, Young's modulus

本文提出了一种通过扫描电子显微镜和微拉伸测试仪对椰子壳,香蕉假茎，菠萝叶和甘蔗渣纤维测试的研究微观结构特性方法。单纤维束通过扫描电子显微镜检查。拉伸试验是在不同的直径（0.15-0.55毫米）上进行，并测量长度（10，15，20，和30毫米/分钟），以评估标距长度和直径对拉伸性能的影响。结果发现，纤维由不同类型的规则排列的单元组成。相比其他纤维，拉伸强度（310 MPa）,杨氏模量（7.4 GPa），菠萝叶纤维束显示最高值。在纤维束的直径

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和标距长度的增加时，拉伸强度和杨氏模量降低。扫描电镜断口分析表明，与更多的参与者的微纤维. 菠萝叶，香蕉假茎纤维相比，椰子壳，甘蔗渣的纤维较为异构破裂。用显微镜观察，用在每一纤维的微观结构的现有知识，讨论了与相应的机械性能。

关键词：农业残余物，纤维直径，显微镜观察，标距长度，力学性能，杨氏模量

INTRODUCTION

A growing tendency in the use of natural fibers is taking place in recent years for many engineering applications. The generalized pollution due to the nondegradable synthetic materials and climate changes attributed to CO_x emission during their industrial production are the major events of global concern (Gore 2006). The need to replace petroleum-based energy systems by environmentally friendly alternatives is also a strong motivation in favor of natural materials (Crocker 2008). Natural fibers, mainly those lignocellulosic obtained from agricultural residue, constitute important examples of renewable and sustainable materials. The interest on these agricultural residue fibers has grown rapidly due to its abundance, biodegradability, low density, nontoxic nature, less abrasiveness to plastic processing equipments, useful mechanical properties, and low cost (17–40% of glass fiber) (Mathew et al. 2006, Bledzki and Gassan 1999) which make them a suitable replacement for man-made fibers. However, agricultural residue fibers have yet many challenges to overcome like lower mechanical properties compared to glass fibers (Wambua et al. 2003), in order to become largely used as reliable engineering materials for structural elements (d'Almeida et al. 2006). The profound knowledge on the basic properties of these fibers would help to conquer these challenges and would ensure an enhanced utilization with superior properties for many structural and general applications.

In composites, plant fibers are used in the form of fiber bundles as reinforcement therefore, a close investigation on strengthening and failure mechanisms of fiber bundle itself is needed. Plant fibers are like microscopic tubes with cell walls surrounding the lumen. The fiber consists of several cell wall layers. These cell wall layers are formed from oriented reinforcing semi-crystalline cellulose microfibril embedded in a hemicelluloses–lignin matrix of varying composition (Thomas et al. 2011). Table 1 and 2 listed the physical and chemical composition of different agricultural fibers that shows high variability in properties even for the same type of fibers. This situation is well understood as the properties of natural fibers depend not only on the type of plant, locality in which it is grown, age of the plant, and the extraction method employed as well as on the fiber structure,

TABLE 1
Agricultural fibers characteristics

Types of fiber	*Technical length (mm)*	*Diameter (μm)*	*Cell length (mm)*	*Diameter (μm)*	*Lumen width (μm)*	*Cell wall thickness (μm)*	*References*
COIR	20–150	10–50	0.7–0.9	18.9–19.3	12.5	3.19–3.41	Van Dam et al.,2006 Satyanarayana et al., 2011
BPS	300–900	12–30	0.9–4.0	80–250	–	–	Reddy and Yang, 2005 Satyanarayana et al., 2011
PALF	900–1500	–	3–9	20–80	–	–	Reddy and Yang, 2005 Satyanarayana et al., 2011
SCB	10–300	10–34	1.59	20.9	9.72	5.64	Hemmasi et al., 2011 Satyanarayana et al., 2011

TABLE 2
Chemical composition of different agricultural fibers

Types of fiber	*Cellulose (%)*	*Hemicellulose (%)*	*Lignin (%)*	*Ash (%)*	*References*
COIR	43.4	4.0	48.3	3.5	Barbosa Jr et al., 2010
	21.2	12.6	46.5	1.05	Bilba et al., 2007
BPS	69.9	19.6	5.7	–	Abraham et al., 2011
	31.2	14.9	15.0	8.6	Bilba et al., 2007
PALF	75.3	13.3	9.8	–	Abraham et al., 2011
	69.5	–	4.4	0.9	Banik et al., 2011
SCB	32–38	–	19–24	1.5−5	Rowell et al., 2000
	55.7	–	20.5	1.8	Hemmasi et al., 2011

microfibrillar angle, fiber (cell) dimensions, and chemical composition (Osorio et al. 2010). The lignocellulosic nature of plant fibers also makes it prone to thermal degradation. Structural constituents of the fiber (cellulose, hemicelluloses, lignin, etc.) are sensitive to the different range of temperatures. It was reported that, lignin starts degrading at a temperature around 200°C, and hemicelluloses and cellulosic constituents degrade at higher temperatures (Kabir et al., 2011).

The major roles of fibers within a plant and their commercial application are largely based on their mechanical properties. Baley (2002) stated that tensile tests would be a well-adopted way to investigate the mechanical properties of elementary and bundle of plant fibers. Properties of different agricultural residue fibers like coir (Buana et al. 2013, Priadi et al. 2013, Tomczak et al. 2007), kenaf (Lavadia and Fronk 2013, Nitta et al. 2013, Ochi 2008), pineapple (He et al. 2013, Wan et al. 2012), sisal (Belaadi et al. 2013, Mwaikambo and Ansell 2006), banana pseudo stem (BPS) (Jayaprabha et al. 2011, Mukhopadhyay et al. 2009), sugarcane bagasse (Hemmasi et al. 2011), piassava (d'Almeida et al. 2006), and oil palm fibers (Abdul Khalil et al. 2008) was studied by many researchers over the past few years. Most of the studies were focused on the importance of microfibrillar angles, cellulose ratio, or their location in the stem on mechanical properties of plant fibers. The performance of these natural fibers in reinforced thermoplastic composite depends on the intrinsic properties and structure of the fibers used as well as the sizes of the fibers after processing. The toughness of a composite material depends on the fiber stress–strain behavior, i.e., strong fibers with high failure strain impart high work of fracture on the composites.

Thus, the characterization of those fibers is an important aspect of fiber material research and plant breeding (Gorshkova et al. 2012). Unfortunately, a very limited number of studies have been reported on the systematic evaluation of microstructure, tensile strength, and fracture analysis of tropical agricultural residue fibers such as coconut husks (COIR), BPS, pineapple leaf (PALF), and sugarcane bagasse (SCB) fibers. Thus, the objective of this paper was to study the microstructure and to evaluate the tensile properties of COIR, BPS, PALF, and SCB fibers as a function of fiber diameter and gauge length. Study the fractured fiber was also an objective of this study.

MATERIALS AND METHODS

Materials

Four different types of agricultural residues were used in this study, viz., banana (*Musa sapientum*) fibers obtained from the pseudo stem of the plant, sugarcane (*Saccharum officinarum*) bagasse fibers, coconut (*Cocos Nucifera*) fibers, and pineapple (*Ananas comosus*) leaf fiber. All the residues were collected from the commercial grower of these plants in Kedah, Malaysia. All the samples were soaked in cold water for 3 days to separate the fiber bundle. The fibers were then washed and dried in air for a week before keeping it in a closed container for further analysis.

Methods

Microstructure and fracture analysis

Analysis of microstructure of the isolated fiber bundles and fractured fibers after tensile test was conducted through scanning electron microscope (Leo Supra, 50 VP, Carl Ziess, Germany) after suitably preparing the samples. The analysis of scanning electron microscope was performed on gold sputtered samples using secondary electrons with a beam voltage of 15–20 kV.

The diameter of fiber was measured to calculate the cross-sectional area for determining the tensile strength. At least fifty fiber bundles were randomly selected from each type of fiber for measuring the diameter. The isolated single fiber bundle was examined by the optical microscope (Olympus BX50, Japan) to determine the outer diameter. The cross-sectional area of the fiber bundle was calculated by measuring the bundle diameter assuming that fibers were perfectly cylindrical and uniform throughout their length. The effective cross-sectional area was calculated by using the Equation 1:

$$A = \frac{\pi d^2}{4} \tag{1}$$

where, A is cross-sectional area, $\pi = 3.142$, and d is the diameter of fiber bundle.

Tensile test

Tensile properties (Young's modulus, tensile strength, and elongation at break) of the fiber bundles were tested according to ASTM D3379 standard (ASTM 1978) by using a miniature tensile tester (MTT 175, Dia-Stron, UK) equipped with a 20 N load capacity at room temperature (23°C) with four different gauge lengths (10, 15, 20, and 30 mm). Every fiber bundle was mounted with two polyvinyl chloride lined brass tube mounts. The schematic representation of fiber bundle specimen is shown in Figure 1. The fiber was loaded at a constant crosshead displacement rate of 20 mm/min until rupture. A total of 60 samples for each fiber type were tested.

Data analysis

Young's modulus, tensile strength, and elongation at four different gauge lengths were determined. Analyses of variance (ANOVA) were performed with linear models in a completely randomized design using the following equation. The correlation between fiber bundle diameter

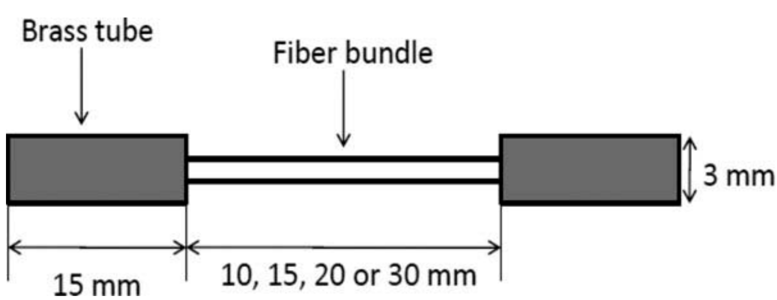

FIGURE 1 Schematic drawing of tensile testing for fiber bundle.

and Young's modulus, tensile strength and elongation were also analyzed. All statistical tests were performed using SAS at 95% confidence level.

$$Yij = \mu + \tau i + \varepsilon ij \tag{2}$$

where, Y_{ij} is the 50^{th} observation for treatment (i), j = 1–50, μ is the overall mean, τ_i is the effect of fiber diameter, and ε_{ij} is the random error.

RESULTS AND DISCUSSION

Fiber Bundle Surface Morphology

Figure 2 shows the surface of fiber bundles used in this study. It was found that the structure was formed by several bundles of elementary or ultimate cells overlapped and bonded together by lignin along the bundle length. It was observed that nodes were at irregular distances which divided the fiber into individual cells. This observation was apparent for BPS. According to Vincent (2000), fibers in plant were rarely found as individual cells, but were assembled into bundles depending on their position in the plant, the chemistry, and morphology of the fiber wall. It was seen that fiber bundles were externally covered by some impurities which caused variable roughness and nonuniform surfaces of the fibers especially for SCB. These observations were supported by other studies done for several agricultural residue fibers (Monteiro et al. 2011, De Rosa et al. 2010). This rough and uneven surface provides good adhesion to polymer matrix of composites by providing good fiber-resin mechanical interlocking (Thomas et al. 2011). On the other hand, waxy substances on the outer surface of fiber cover the reactive functional groups of the fiber and act as a barrier to interlock with the matrix resulting poor surface wetting. However, surface modifications by different chemical treatments, reactive additives and coupling agents would optimize the interfacial bonding between fiber and matrix.

Fiber Diameter Distribution

For each type of fiber, histograms corresponding to the frequency of the diameter distribution are shown in Figure 3. The figure shows that the minimum number of fiber bundles is associated with the thickest and the thinnest fibers except for PALF. According to the histogram, PALF showed smaller bundle diameter distribution compared to other fibers, while COIR showed the largest bundle diameter. The variation in fiber bundle diameter distribution for PALF and BPS were in the same range as were reported by Mohamed et al. (2010) and Mukhopadhyay et al. (2009), respectively. However, fiber bundle diameter distribution of COIR and SCB were higher than the results reported by Reddy and Yang (2005). This variation was due to the species, nature of growth, age of plant, the environmental conditions (Mylsamy and Rajendran 2010) and the fiber extraction method employed (Osorio et al. 2010). Investigation on greater number of fiber bundles could possibly extend the range of histograms by finding even thinner and thicker fiber bundles.

Stress-Strain Curve

Typical stress-strain curves obtained from tensile tests performed at room temperature for different agricultural residue fibers are presented in Figure 4. Initially, all the measured stress–strain curves were evaluated based on their curve shapes. The shape of stress–strain curve varied between different fibers. Both linear and nonlinear curve was observed for the studied fibers. The linear curve appeared as a straight line up to maximum load and was truly elastic observed for BPS and PALF

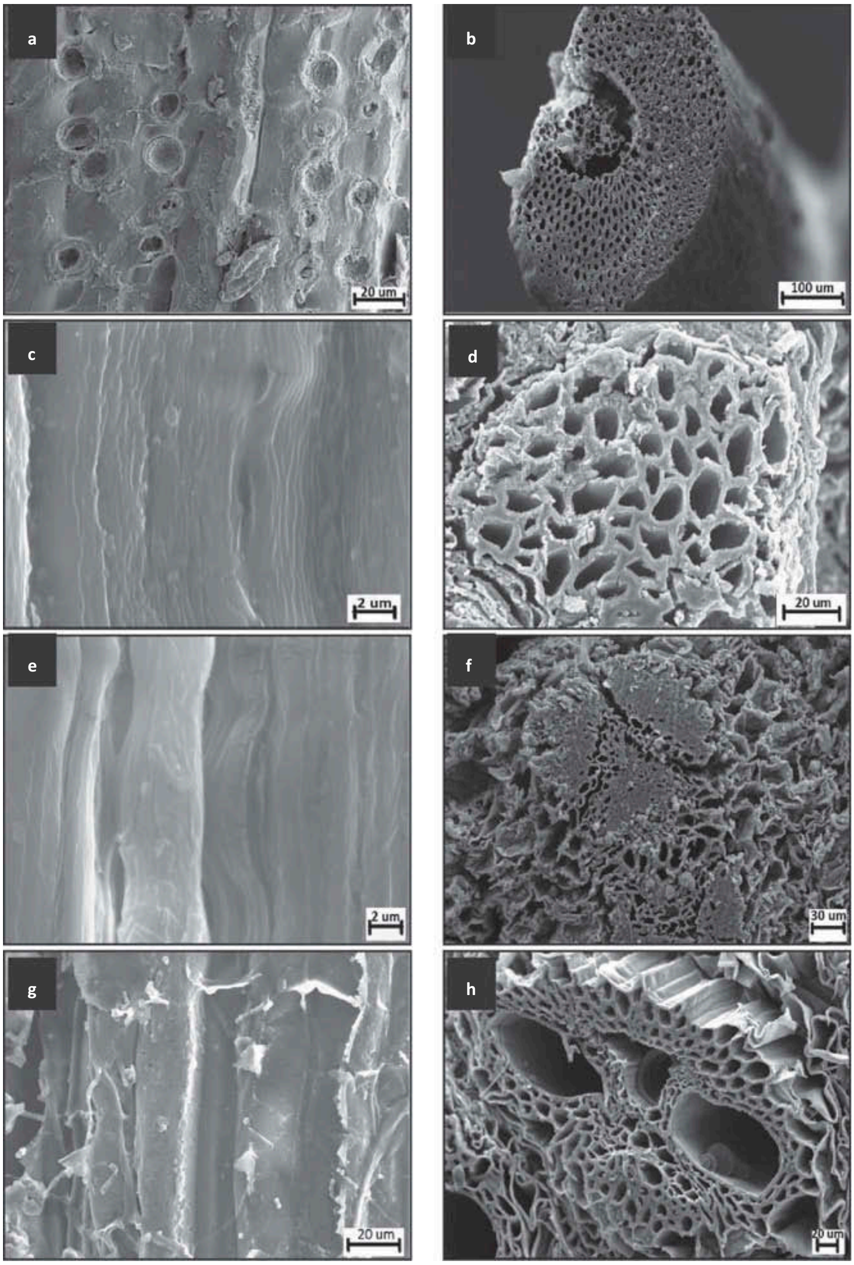

FIGURE 2 SEM micrograph of longitudinal surface of (a) COIR 654×, (c) BPS 4670×, (e) PALF 4650×, and (g) SCB 726×, and cross-sectional view of (b) COIR 170×, (d) BPS 2050×, (f) PALF 297×, and (h) SCB 282×.

fibers. COIR and SCB fibers showed nonlinear curve which included the plastic flow. This nonlinear stress-strain behavior for COIR and SCB showed that during tensile loading, the fibers deformed more before breaking. The linear stress–strain curves showed a higher tensile strength, a higher Young's modulus, and a lower strain to failure than the nonlinear curves. Similar phenomenon was

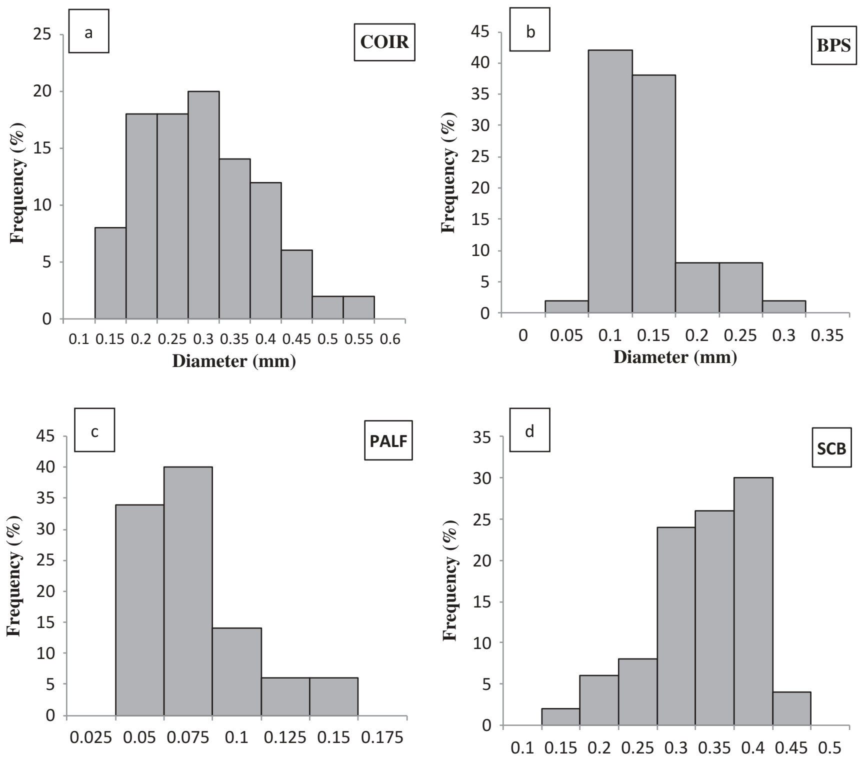

FIGURE 3 Fiber diameter distribution of COIR, BPS, PALF, and SCB fiber (based on 50 readings of each fiber along the length).

also reported by Aslan et al. (2011) and also reported that both linear and non-linear curves were correlated to the defects of the fibers.

Tensile Properties

Average tensile strength, Young's modulus, and elongation at break are presented in Table 3. PALF fiber showed the highest tensile strength and Young's Modulus, while, SCB showed the lowest value for both the properties. The tensile strength for COIR and BPS fibers were in the same range with data reported by Tomczak et al. (2007) and Mukhopadhyay et al. (2009). However, PALF tensile results were significantly lower compared to the results reported by Munawar et al. (2007). Meanwhile, tensile strength for SCB fibers were higher than the results obtained by Muensri et al. (2011) which was 29MPa. PALF and BPS fibers have higher cellulose content and lower microfibril angle which help them to support higher weight of the fruits and are less perishable (Mukhopadhyay et al. 2009, Mishra et al. 2004). According to Reddy and Yang (2005) and Mwaikambo and Ansell (2006), fibers having higher cellulose content and lower microfibrillar angle generally showed higher mechanical properties, although cellulose content was not exactly correlated with the measured strength of natural fibers. This would help to explain the lower strength of SCB fibers associated with lower cellulose content and reasonably higher microfibril angle (Satyanarayana et al. 2011). The measured elongation at breaks of the agricultural residue fibers were in the range of 2–37%.

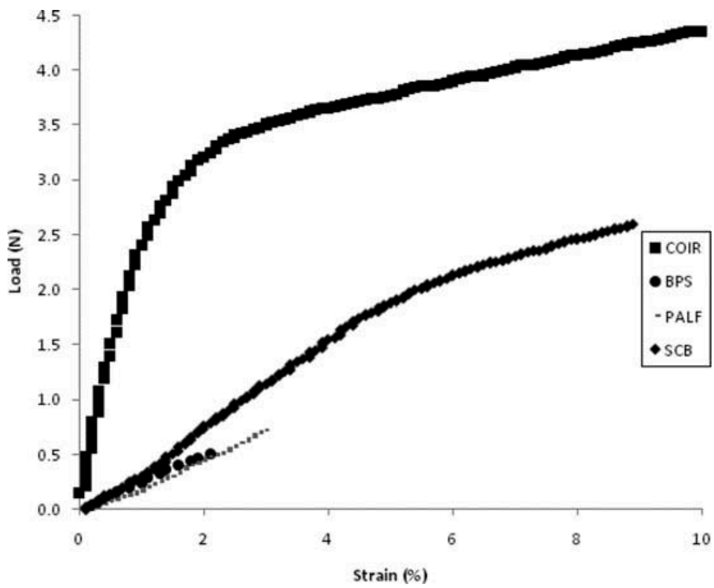

FIGURE 4 Typical load-strain curves of COIR, BPS, PALF, and SCB fiber obtained at a crosshead displacement rate of 20 mm/min.

TABLE 3
Mechanical properties of different agricultural residue fibers at different gauge length

Types of fiber	*Gauge length (mm)*	*Tensile strength (MPa)*	*E-Modulus (GPa)*	*Elongation at break (%)*
COIR	10	120.8 (41.2)	3.4 (1.0)	37.2 (1.8)
	15	104.4 (52.1)	3.1 (2.1)	37.1 (0.9)
	20	92.6 (41.5)	1.8 (0.8)	28.5 (1.5)
	30	83.4 (46.1)	1.7 (0.5)	26.5 (1.2)
BPS	10	295.1 (50.4)	7.1 (1.0)	3.0 (1.5)
	15	284.8 (30.7)	6.1 (2.6)	2.6 (1.3)
	20	269.3 (43.5)	6.1 (2.3)	2.5 (1.0)
	30	265.8 (40.1)	5.7 (1.2)	2.4 (1.3)
PALF	10	309.7 (50.0)	7.4 (0.8)	2.2 (0.6)
	15	290.1 (43.5)	5.4 (2.3)	1.9 (0.6)
	20	275.8 (50.5)	5.1 (2.7)	1.9 (0.7)
	30	273.7 (50.9)	5.6 (1.3)	1.8 (0.8)
SCB	10	60.4 (19.0)	1.3 (0.7)	7.9 (1.8)
	15	52.8 (14.2)	1.3 (0.5)	6.4 (2.4)
	20	29.1 (8.9)	0.8 (0.7)	6.7 (2.7)
	30	26.5 (7.2)	0.8 (0.8)	5.3 (2.0)

Data in parenthesis are the standard deviation. Based on 60 readings for each type of fibers.

COIR fiber showed the highest elongation at break (37%) because of its lower cellulose content and higher microfibrillar angle (Silva et al. 2000) when compared to other natural fibers. Coiled microstructure of COIR fibers might be the cause of this higher microfibril angle (Kulkarni et al. 1981).

Effect of Fiber Bundle Diameter on Tensile Properties

There was negative correlation between tensile strength and fiber diameter, i.e., tensile strength decreased with the increase of fiber bundle diameter (Figure 5). The relationship was statistically significant for most of the cases (Table 4). Tomczak et al. (2007) reported similar negative relationship between diameter and tensile strength of fiber when they studied the Brazilian coconut fiber. This trend might be because of the lumen size which increased with the increase of fiber diameter.

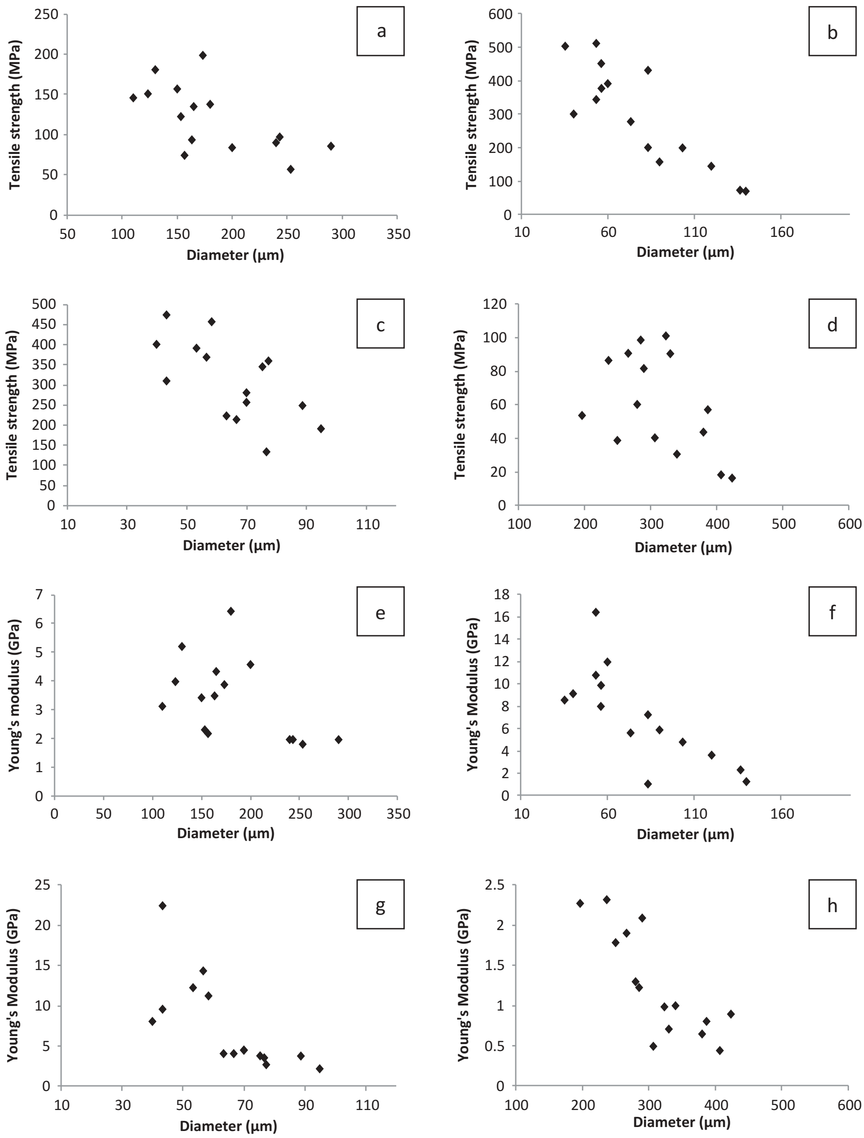

FIGURE 5 Tensile strength and Young's modulus as a function of fiber diameter for COIR (a and e), BPS (b and f), PALF (c and g), and SCB (d and h) at 10 mm gauge length.

TABLE 4
Analysis of Variance (ANOVA) and correlation for different types of agricultural residue fibers

ANOVA for Gauge length and fiber types				
	Source	*df*	*F value*	*Prob. >F*
Tensile strength	Types of fibers	3	82.72	0.0001*
	Gauge length	3	1.24	0.2947
	Types of fibers × gauge length	9	0.01	1.0000
Young's Modulus	Types of fibers	3	51.33	0.0001*
	Gauge length	3	3.06	0.0292*
	Types of fibers × gauge length	9	0.43	0.9183
Elongation at break	Types of fibers	3	453.15	0.0001*
	Gauge length	3	6.25	0.0004*
	Types of fibers × gauge length	9	3.84	0.0002*

Correlation between diameter and properties			
Fibers	*Properties*	*R*	*Prob. > F*
SCB	Tensile strength	0.35384	0.0055
	Young's modulus	0.56344	0.0001
	Elongation at break	0.00174	0.9895
PALF	Tensile strength	0.61989	0.0001
	Young's modulus	0.54518	0.0001
	Elongation at break	0.26301	0.0423
BPS	Tensile strength	0.71181	0.0001
	Young's modulus	0.62370	0.0001
	Elongation at break	0.18921	0.1477
COIR	Tensile strength	0.62771	0.0001
	Young's modulus	0.16271	0.2142
	Elongation at break	0.32653	0.0109

*Significant at 95% confidence level.

Lumen is not generally considered for calculating the cross-sectional area and fiber bundle is considered as perfect cylinder due to its difficulties in determination which might also add some errors for calculating the tensile properties (Bourmaud et al. 2010). Besides, thicker fiber has also higher probability to have more flaws and defects compared to the thinner ones (Zhang et al. 2002) which might contribute to this trend. The result was also supported by Munawar et al. (2007) where they investigated physical and mechanical properties of fibers from several nonwoody plants. The internal structure and properties (chemical composition and mechanical properties) of lignocellulosic fibers depend on their origin, maturity, species, and extraction methods. In addition, the strength properties of the fibers also depend on test conditions, microfibril angle, and density of weak links or flaws.

Effect of Gauge Length

Results of the effect of gauge length on tensile strength and Young's modulus of the studied fibers with varied gauge length are shown in Figure 6. It was found that tensile strength, Young's modulus, and elongation of the fiber decreased when the gauge length increased. The decrease in tensile strength, Young's modulus, and elongation of the fiber with increasing the gauge length could be understood as defects/weak-links and nonhomogeneity of the fibers increase with their increasing

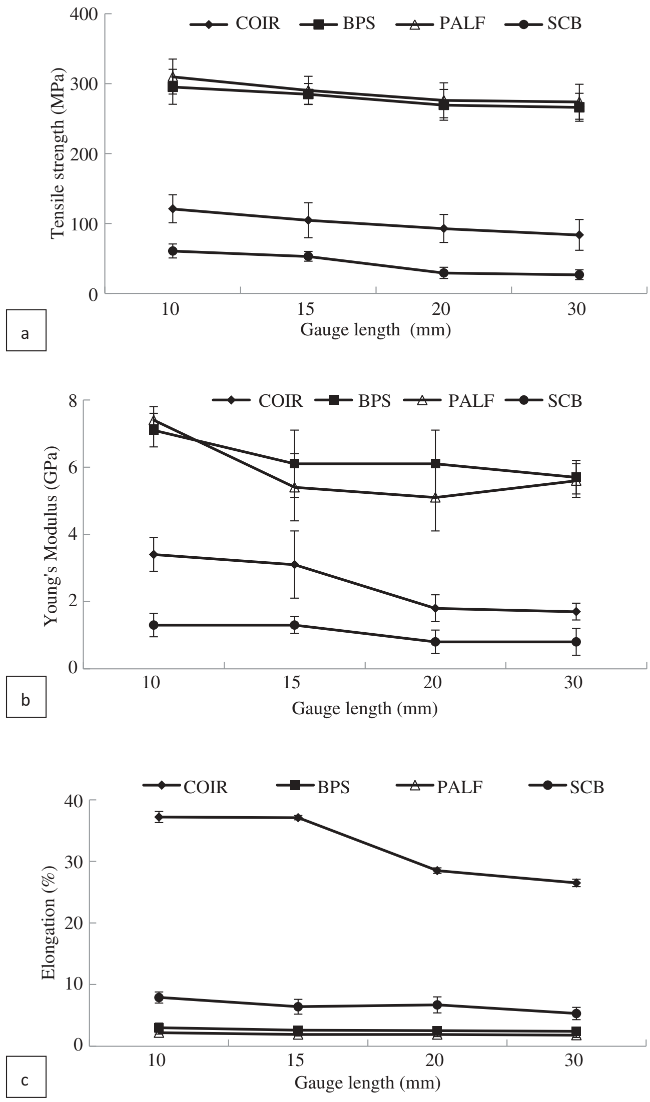

FIGURE 6 Tensile properties, i.e., (a) tensile strength, (b) Young's modulus, and (c) elongation, as a function of gauge length for COIR, BPS, PALF, and SCB fiber.

length (De Rosa et al. 2010). This result was consistent with the results reported for other natural fibers (Xia et al. 2009, Silva and Chawla 2008). Zafeiropoulos and Baillie (2007) reported that gauge length of tensile tests endorsed variation for mechanical test results. Longer the fiber's length, the higher would be its lignin content, and hence, the higher would be its resistance to applied stress (higher stiffness or modulus) which in turn resulted in lower elongation (Tomczak et al. 2007).

Fracture Analysis

Scanning electron microscopic (SEM) fractography analysis of tensile-ruptured fibers is shown in Figure 7. The fracture mechanism of PALF and BPS showed fiber pullout failure mode with comparatively heterogeneous rupture associated with more microfibrils. It is worth emphasizing that

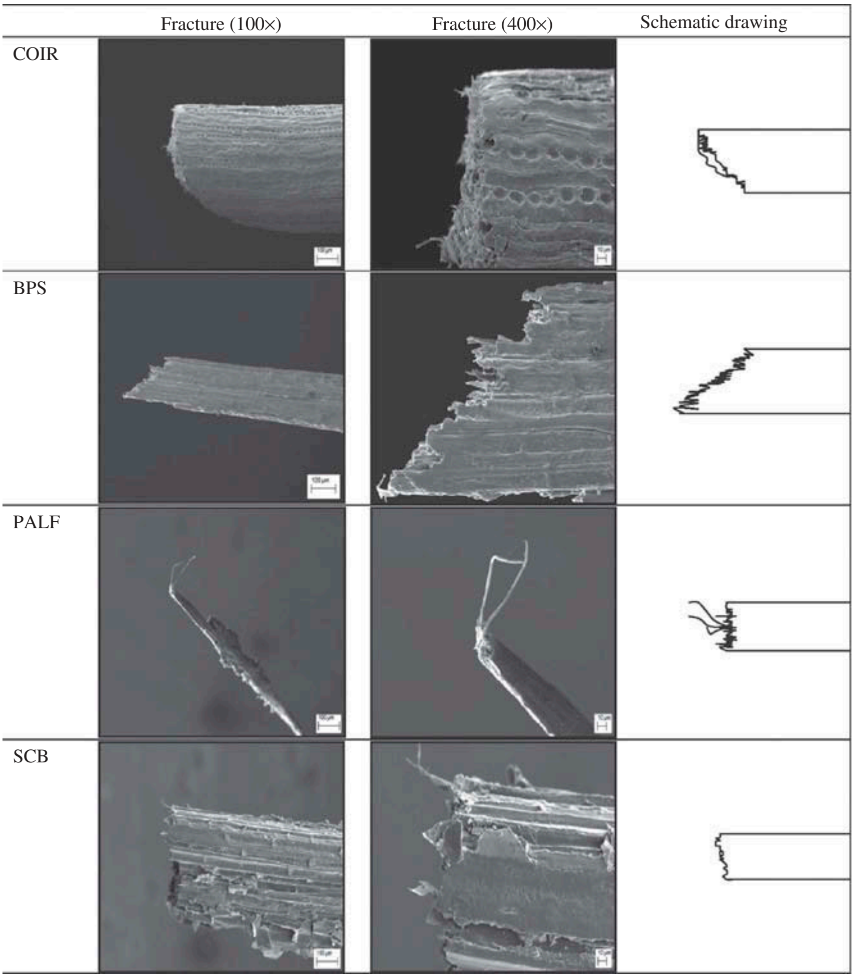

FIGURE 7 Scanning electron micrographs of different types of agricultural residue fibers on fracture surface and their corresponding drawing.

microfibrils are based on cellulose chains and constitute the strongest part of the fiber bundle. Thus, the difference in strength between fibers depends on the relative amount of microfibrils (cellulose composition) and lignin. It also depends on the spiral angle of microfibrils around the fiber axis. COIR and SCB fiber bundle showed more homogenous microstructure with fewer participants of fibrils as there was only a few fibrous debonding on the fracture surface for both fibers. According to Monteiro et al. (2011), a thicker fiber with more microfibrils had larger distribution of both weaker and stronger microfibrils. Thus, the weaker microfibril in the thicker fiber could break at a lower stress than the weaker microfibril in the thinner fiber. Once the weakest microfibril is broken, it causes a flaw in the fiber structure. The flaw may act as a microcrack, which swiftly propagates in a brittle mode until total rupture. Moreover, different fiber cells do not fracture at the same stress level, possibly due to the presence of cell wall defects along the fiber length which creates stress intensities leading to ultimate failure (De Rosa et al. 2010).

CONCLUSIONS

Microstructure and tensile properties of four different types of agricultural residue fibers were analyzed and evaluated for potential application as reinforcement in polymeric composites. Fiber bundles showed a cylindrical shape consisting of a bundle of ultimate fibers bonded together in a matrix of lignin and hemicelluloses. The diameter range of COIR, BPS, PALF and SCB varied between 0.15–0.55, 0.05–0.30, 0.05–0.15, and 0.15–0.45 mm, respectively. BPS and PALF showed linear curve whereas COIR and SCB showed nonlinear stress-strain curve. PALF showed the highest tensile strength and Young's modulus while the highest elongation was observed for COIR. Mechanical properties of fibers showed increasing trends with the decrease of diameter and gauge length. SEM fractography analysis demonstrated the brittle behavior of fibers as well as fiber pulled out with microfibril debonding on the surface of fiber fracture. The overall mechanical properties of the fibers were higher than the previous studies which might be related to the various factors like location, environment, and maturity of plant. Thus, this systematic evaluation of different agricultural residue fibers would help to find out suitable fibers for different composite applications by optimizing the production process and choosing the suitable end use.

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