STRUCTURAL CHARACTERISTICS AND PROPERTIES OF WINDMILL PALM LEAF SHEATH FIBER

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Abstract. A study was carried out on a special kind of fibrous material—windmill palm leaf sheath fiber (palm fiber)—with the aim of full utilization of the bioresource. Morphological feature and fine structure of palm fiber were investigated using light microscopy, scanning electron microscopy, and transmission electron microscopy (TEM). The results indicate that palm fiber is subcylindrical with a rough surface and large diameter (359.15 μm). Ultrastructure from TEM confirmed that cell wall layers of palm fiber have a structure similar to that of wood cell wall. Individual fibers in the palm fiber are elongated cells (length–diameter ratio is about 100) with lumen, tapering ends, and thick cell walls (about 1 μm). In addition, crystallinity, tensile properties, and moisture regain of palm fiber were studied and compared with flax, ramie, and bamboo fiber. Palm fiber has relatively lower crystallinity and tensile strength compared with the fibers, but it has extremely higher elongation.

Keywords: Windmill palm leaf sheath fiber (palm fiber), individual fiber, morphology, structure, properties.

INTRODUCTION
Windmill palm is one of the most widely distributed palms in the world, especially in East Asia. The windmill palm leaf sheaths originate from the stem and surround it layer by layer. The palm fibers interweave in a network and form the leaf sheath fiber layers. As an abundant natural resource, the development and utilization of the windmill palm leaf sheath fiber (palm fiber) is being increasingly explored. Due to its excellent performance such as high toughness, low density, erosion resistance, and environmental friendliness, palm fibers have been used for thatch, marine ropes, mattresses, brushes, and traditional raincoats in ancient China (Essig and Dong 1987; Zhang et al 2010; Zhai et al 2012). Use of palm fiber in composites, filters, elastic materials, etc, is being explored by some researchers (Al-Sulaiman 2002; Sreekala et al 2002; Sreekala and Thomas 2003; Kaddami et al 2006; Zeng and Zhu 2006; Alawar et al 2009).

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A large body of literature exists on research of palm fibers on the view of biology, anatomy, and wood science (Munawar et al 2007; Sahari et al 2012; Zhai et al 2012), but a basic understanding about the correlation between microstructure and fundamental properties of palm fiber is still lacking. Therefore, further explorations on the palm fiber’s characteristics are necessary for evaluating its potential application.

Morphology and intrinsic structure properties are important factors for fiber processing technology and end-use performance characteristics. Much of the literature focuses on morphology and microstructure of natural fibers, such as ramie, hemp, and flax (Wang and Wang 2005), bamboo fiber (Zou et al 2009), lotus fibers (Pan et al 2011), and cellulose fibers from cornstalks, cotton stalks, and hop stems (Reddy and Yang 2005, 2009a, 2009b). Most of them have interpreted the relationship between the structure and properties of those fibers. Meanwhile, palm fibers were studied from a botanical perspective (Zhai et al 2012). Palm fiber-reinforced composites have been also widely investigated by scientists and technologists (Al-Sulaiman 2002; Abu-Sharkh et al 2004; Alawar et al 2009), but there is an increasing need for further study on intrinsic structure properties, such as microstructure and cell wall layer structure of palm fibers, arrangements of individual fibers, etc.

To completely evaluate the potential applications of palm fibers, a comprehensive study on the fine structure and the fundamental properties of the palm fiber has been carried out in this article. Morphological features and fine structure of palm fiber were investigated using light microscopy, scanning electron microscopy (SEM), and transmission electron microscopy (TEM). In addition, individual fibers were obtained by a degumming technique. The diameter and length of the individual fibers were measured by a light microscope. The longitudinal and transverse connections among palm fibers were analyzed. Some properties of palm fiber were compared with those of flax, ramie, and bamboo. The results of the research should lead to a better understanding of morphology, microstructure, cell wall structure, and some properties of palm fibers. These fundamental studies can be used by material scientists, wood technologists, and polymer chemists for further applications.

**MATERIALS AND METHODS**

**Material**

The palm leaf sheath (Fig 1a-b) was peeled with knives directly from mature palm trees from a local plantation in Honghe country, Yunnan province of China. They were manually washed and rubbed with water to remove dirt and then dried at 50°C in an oven for 2 h. The palm fiber samples were pulled out from the leaf sheath (Fig 1c); then they were cut off at a length of 5-30 cm for different tests.

**Individual Fiber Extraction**

Palm individual fibers were obtained by a chemical method according to the extraction of bamboo fiber (Chen et al 2011). The main chemicals are sulfuric acid (H2SO4), sodium hydroxide (NaOH), nitric acid (HNO3), potassium chlorate (KClO3), and hydrogen peroxide. The process is shown in Fig 2.

**Microstructure and Cell Wall Structure**

The morphology and microstructure of palm fibers were observed by a light microscope (AXIOSKOP40; Carl Zeiss, Oberkochen, Germany), scanning electron microscope (S-4800; Hitachi, Chiyoda, Tokyo, Japan; FEI QUANTA 200 FEG, Eindhoven, The Netherlands), and transmission electron microscope (H-7500; Hitachi). The transverse sections were prepared by the brittle fracture method: the fiber was broken off after being immersed in liquid nitrogen for 5 min. Internal longitudinal sections were obtained by splitting the fiber along the axial direction. The samples for cell wall structure observation were fixed in 50% ethanol for 4 days. Fixed samples were dehydrated in an ethanol series and embedded in epoxy resin. Ultrathin sections
(0.1 μm) were cut by an ultramicrotome (Leica UC6, Vienna, Austria).

Properties of Palm Fiber

The crystallinity of palm fiber was evaluated from X-ray diffraction obtained by a MSAL-XD3 X-ray diffractometer system with CuKα (λ = 1.5406 Å). The X-ray generator system was operated at 36 KV and 30 mA. The two θ angles ranged from 5 to 36°. The samples were cut up with scissors.

Fiber samples for tensile tests were made using the Munawar method (Munawar et al 2007) in a natural dry condition. The tensile tests of the fibers were performed on an electronic multi-filament strength tester-YG012 with a gauge length of 20 mm. Fifty samples were mounted for testing conducted at the cross-girder rate of 0.5 mm/min.

Moisture regain of palm fiber was determined according to ASTM 2654 (ASTM, 1989) standard method. Both moisture absorption and liberation were tested in the standard reference atmospheric condition (21°C temperature and 65% RH). About 5-g palm fiber samples were used in the moisture regain test.
RESULTS AND DISCUSSION

Morphological Structure of Palm Fiber

Surface morphological features of palm fiber. Figure 3 shows the surface morphology of palm fiber at different magnifications. It can be seen that palm fiber is subcylindrical with a rough surface. As observed at higher magnification in Fig 3b, areas of the palm fiber surface are covered by arrays of protrusions. These protrusions are shown detailed in Fig 3c and thought to be Si particles, which result in the excellent natural anticorrosion of palm fiber. Palm fibers have potential mechanical characteristics as reinforcement in composites because the rough surface of the fibers could enhance bonding of the matrices.

Transverse section of palm fiber. The transverse sections of palm fiber are shown in Fig 4. In Fig 4a, we can see that palm fiber is a multicellular fiber with a central portion called a “lacuna” (mark 1) and other parts mainly are individual fibers (mark 2). The lacuna shown in Fig 4b is made up of phloem, xylem, and parenchymatous tissues. The inner wall of xylem is spiral with wave-like wrinkles, and this kind of vessels is defined as spiral vessels (Zhao 2009). Figure 4c shows the individual fibers that have lumens and cell walls. Average diameter of the lumens is about 5-8 μm, and cell wall (marked 3) thickness is about 1-3 μm. The cellular structure of palm fiber may make them potential candidates for filter and adsorption materials.

Longitudinal structure of palm fiber. Figure 5 shows the longitudinal sections of palm fiber. Figure 5a reveals that the vessels are broken and some membranes (marked 4) are left. Obviously, all the individual fibers (marked 5) are elongated cells aligned in the longitudinal direction. Figure 5b shows a separated individual fiber (marked 6) which indicates that the individual
fibers are independent in the fiber assembly with a unique wall of its own.

**Palm fiber diameter.** Figure 6 shows the diameter distribution histogram of palm fiber. The palm fiber diameters varied from 150 µm to 600 µm, and the average value is about 359.15 µm. The diameter also varies at different measurement spots along the longitudinal direction of fibers. Generally, the diameter of the fiber decreases gradually along the growth direction. The diameter of palm fiber is much larger than that of jute (25-200 µm), flax (10-40 µm), and sisal (50-200 µm). It is a kind of large-diameter fiber, and it is difficult to be used in producing garments and textiles now even though palm fiber is also a kind of promising bioresource in the material science and wood science, etc.

Microstructure of Palm Individual Fiber

Partly separated palm fibers are shown in Fig 7. Individual fibers are bonded tightly to each other in Fig 7a, because the fiber is not easy to separate in the individual fiber extraction process. The ends of individual fibers (marked 7) are randomly distributed in the palm fiber. Obviously, the individual fibers are much shorter than the palm fiber bundle and they are randomly distributed along the longitudinal direction. From Fig 7b, individual fibers separated from the palm fiber assembly can be found distinctly. This type of fiber assembly is analogous to that of bast fibers (Baley 2002). Figure 8 shows the microstructure of completely separated individual fibers. In Fig 8a, individual fibers have tapering ends along their length direction. The fine structure of individual fiber in Fig 8b reveals that the surface is rough. This surface feature helps enhance the cohesive forces between individual fibers and improve the strength of palm fiber.

Length and diameter distribution of palm individual fiber is shown in Fig 9. In our study, the diameter in the middle part of an individual fiber is larger than those in the near end parts (3 µm from ends). The very ends have a diameter of
zero. Individual fibers in the middle part have an arithmetic mean diameter and standard deviation with a value of $11.57 \pm 1.47 \mu m$ (Fig 9a), while those of the near end part are $6.10 \pm 1.23 \mu m$ (Fig 9b). The length distribution of the individual fibers is shown in Fig 9c. The average value of individual fiber length is 0.78 mm, and it ranges from 0.19 to 1.36 mm. The relationship between fiber diameters and their length is shown in Fig 9d. Obviously, diameters, both in the middle part and the near end parts, are randomly distributed with the increase of individual fiber length. So, there is no marked essential relation between the fiber diameter and its length.

**Cell Wall Structures**

A transmission electron microscope provides a conventional way to study fiber cell wall structure. The cell structure of palm fiber can be seen clearly and comprehensively using TEM. In Fig 10, various wall layers (primer and secondary) can be seen clearly from the ultrathin sections of the fiber. The TEM graphs confirmed that the cell wall structure of palm fibers consisted of a primer layer (P) and secondary layers (S1, S2, and S3). This structure is similar to that of the wood cell wall structure reported by researchers (Harada and Cote 1967). Individual fiber is almost round in shape from Fig 10a. The S1, S2, and S3 layers are bonded together and the microfibril angles of S1 and S3 are almost parallel with the S2 layer. The special structure gives additional strength to the fiber. This is because the layered structure will give high hoop strength to the S1 and S3 layers which will prevent the buckling process of the S2 layer when stress is applied at a low level (Booker and
Sell 1998). The S2 layer and S1 layer can be clearly seen and differentiated. The thickness of S2 and S1 layers are about 1.3 μm and 0.6 μm, respectively. S2 layers mainly influence the thickness of the overall cell wall. The S3 layer is much thinner than S1 and S2, and it is hardly differentiated. There are some phenols besides the S3 layer which contribute to good antibacterial

Figure 9. Diameter and length distribution of palm individual fiber: (a) diameter of individual fiber ends; (b) diameter of individual fiber middle parts; (c) length of individual fibers; (d) relationship between individual fiber length and diameter.

Figure 10. Transmission electron micrographs of palm fibers: (a) ×12000; (b) ×20000 (ML, middle lamella; P, primer layer; S1, S2, and S3, secondary layers; Ph, phenols).
Properties of Palm Fiber

Table 1 summarizes the properties of palm fibers compared with lignocellulosic fibers such as flax (Baley 2002), ramie (Goda et al 2006), and bamboo (Murali et al 2010). Experimental results show that crystallinity of palm fiber is 37.92%, which is lower than that of flax (65-70%), ramie (70-74%), and bamboo (78.14%). The lower crystallinity means a greater number of amorphous regions and more disorderly molecular arrangement in the fiber (Reddy and Yang 2005). Therefore, palm fibers tend to be more permeable to water and chemicals. Figure 11 depicts the stress–strain curves for palm, flax, ramie, and bamboo. From both Table 1 and Fig 11, it can be seen that the strength of palm fibers ranges from 89 to 222 MPa, which is much lower than that of flax (1339 ± 486 MPa), ramie (560 MPa), and bamboo (341 MPa). The Young’s modulus of flax, ramie, and bamboo is over 10 times higher than that of palm fiber (0.44-1.09 GPa). However, palm fiber shows much higher breaking elongation (14-23%) than the other three fibers. It is well known that the strength and elongation of multicellular fibers depend on the crystallinity, microfibril orientation, and cellulose crystals in the fibers. The lower tensile strength and Young’s modulus of palm fibers can also be attributed to its lower crystallinity. Lower crystallinity leads to easier molecular chain movement. So, palm fiber exhibits higher elongation. In addition, the number, length, and diameter of individual fibers affect the strength of the fiber (Reddy and Yang 2005). Compared with flax, individual palm fibers are much shorter and thinner because there are more individual fibers in the palm fiber bundle than in the same size of the flax fiber bundle. The larger number of individual fibers means a greater number of binding spots, which could have more weak links in the fiber. A larger amount of weak links may lead to easier slipping between individual fibers during stretching. Therefore, compared with flax, this feature contributes to the lower tensile strength and higher elongation.

The moisture regain of palm fiber is about 16.65%, which is similar to that of bamboo (14-16%) and ramie (15-17%), but higher than that of flax (12%). According to the fiber moisture absorption mechanism (Yu and Chu 2009), fiber macromolecules arranged orderly and closely in the crystalline regions. Hydrophilic active groups formed crosslinks among the molecules and the water molecules cannot get into the crystalline regions easily. As a result, the moisture regain mainly happened in the uncrystalline parts. The
higher moisture absorption property of palm fibers may be attributed to lower crystallinity as well as the rough morphology structure and abundant hollow sieve tubes.

CONCLUSIONS

The palm fiber is subcylindrical with a rough surface, and it has a large diameter. Cell wall structure of palm fiber is similar to that of wood. Individual fibers in the palm fiber are elongated cells with lumen, tapering ends, and thick cell walls (about 1 μm). Diameters and lengths of individual palm fiber range from 1.1 μm to 19 μm and 0.1 to 1.4 mm, respectively.

Palm fiber has relatively lower crystallinity, tensile strength, and Young’s modulus, but a higher breaking elongation than do flax, ramie, and bamboo. Moisture regain of palm fiber is about 16.7%.

The special structural characteristics and properties endow palm fiber with distinctive characteristics for potential applications. Palm fiber and individual fibers could be explored and utilized in many fields such as material science and wood science.

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