

Studies on the physical characteristics of natural fibers from the wine palm and roselle

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Abstract

Fiber strands from Hibiscus sapdariffa (Roselle) and Wine Palm (Caryota urens) plants were isolated and described in this study. In order to compare the extracted fiber strands with their dried equivalent, they underwent a water treatment. The micro structure, morphological characteristics, and chemical compositions were ascertained using methods including energy dispersive X-ray spectroscopy (EDX), Fourier transform infrared spectroscopy (FTIR), and scanning electron microscopy (SEM)-FTIR. The Universal Testing Machine (UTM) was utilized to investigate mechanical qualities. With water treatment, the fiber strands' crystallinity increased, according to XRD data, and FTIR spectroscopy shows that the functional groups in these fibers varied. SEM examination shows that after water treatment, the surface becomes smoother. For CU and HS fibers, the corresponding fineness of these fiber strands was determined to be 372.18 denier and 13.63 denier. Tensile strength for both types of fibers increases after water treatment, according to UTM data. Comparing these fibers with other natural fibers reveals their competitive advantages.

Introduction

Many civilizations have made extensive use of natural fibers. According to certain reports, hemp was first used more than

6000 years ago (Beckermann 2007; Peter 1992; Roulac 1997). Growing global waste issues, rising environmental consciousness, and steadily rising crude oil prices. Globally, prices have prompted governments to step up legislative pressure. Natural fiber and other alternative energy sources, like new industrial products, are made from renewable resources found in agricultural and forestry products. Green relevance lies in the use of natural fibers to create affordable, environmentally beneficial composite materials. Since the price of crude oil keeps rising, the cost of conventional plastics also keeps rising. Furthermore, there is a global conversation about sustainability, climate change, and reducing CO₂ emissions. Since novel non-oil-related materials are in demand, numerous businesses in the packaging, automotive, and construction sectors are searching for them. The amount of energy needed to produce hemp and jute fibers is only 10% and 12%, respectively, less than that needed to produce glass fiber (Carus and Gahle 2008).

The species Caryota urens (CU), known as "Baine" in Kannada, and Hibiscus sabdariffa (HS), known as "Pundi" in Kannada, are abundantly and readily available in the Western Ghats. The wine palm plant has a long history of usage in medicine, having been used to alleviate

rheumatic swelling and hemicranias. According to Re et al. (1999), CU's fruit skin and immature fruit have antibacterial action against the investigated pathogens. The fleshy red calyces are the most well-known because they are frequently used to make non-alcoholic drinks in the form of beverages or juice. A variety of highly valued food and medicinal items are generated from HS components, including seeds, leaves, fruits, and roots (Adebayo-Tayo and Samuel 2009). These plants, which are depicted in Figures 1(a) and (b), are employed in daily life to make things like bags, mats, ropes, and other items, as well as for agriculture. Preparing a nose rope or Cavesson to control domestic animals during tilling is one of the significant uses. Unlike synthetic polymers, the ropes composed of these fibers do not cause damage to their skin. The wine palm and Roselle natural fibers that are readily available in our area, as well as their strength in both wet and dry situations, will be scientifically characterized by this study. These natural fibers' low cost, low density, and good mechanical qualities have made them attractive for use as reinforcement in polymer composites in a variety of technical applications (Alamri and Low 2012).



Figure 1. (a) wine palm and (b) Roselle plants.

Experimental Extraction of fibers

The extracted fibers are shown in Figure 2. These fibers are extracted by retting, the process of extracting fibers from the long lasting life stem or bast of the bast fiber plants. CU fiber extraction is done using mechanical retting. Through this method, the plant's outer layer of stem is cut off at a young stage of growth.



Figure 2. Extracted fibers

The fine fibers can then be extracted by pounding these bast portions of the stem. Yet, water retting is used to remove the

fiber strands of HS. For two to three weeks, HS stems are submerged in water (ponds, rivers, or tanks) and are periodically checked. However, if the fiber is of higher quality, the retting procedure might take less time. Water retting is a labor- and capital-intensive technique that yields higher-quality, more uniform fibers. Following the fiber extraction process, the samples are submerged for 24 hours in two different 1-liter beakers, as depicted in Figure 3, to evaluate their physical characteristics in both a wet and dry state.

Characterization

X-ray diffraction (XRD) was recorded using a graphite monochromator and $\text{CuK}\alpha$ radiation with a wavelength of 1.5406\AA using a Rigaku Smart lab. For a range of 6° – 60° , the sample was scanned at a speed of 5° per minute with a step size of 0.02° . The scanning range for the FT-IR spectroscopy was 4000 cm^{-1} to 400 cm^{-1} . The Hitachi S-3400N scanning electron microscope (SEM) with tungsten filament, which allows for accelerating voltages up to 15 kV and a spot size of 50, was used to perform SEM-EDX. This setup is equipped with a Thermo Fisher Scientific Noran System 7 Energy-dispersive X-ray detector (EDX) for chemical analysis. A 5 kN load cell from Lloyd Instruments UK's Universal Testing Machine (UTM) was used to record the mechanical properties of the fibers. Every fiber underwent testing at a 50 mm gauge length, 5 mm/min cross head speed, and room temperature. Utilizing UTM, the Tensile Test was carried out. Five samples were evaluated in each case, and the average value was calculated. Samples were cut in accordance with ASTM D638 test protocol for the tensile test. Tests were conducted on the samples at room temperature until tensile failure was seen.



Figure 3. Fibers immersed in the water.

Results and discussions

XRD analysis

Figure 4 displays the XRD pattern options for CU and HS fibers, both wet and dry. The size and strain of these fibers' crystallites have been determined through analyses. Cellulose, hemicelluloses, and lignin make up the majority of natural fiber (Joonobi et al. 2010; Tserki et al. 2005; El-Sakhawy and Mohammad 2007; Dominique et al. 1999). Amorphous hemicelluloses envelop cellulose stalline microfibrils, and a lignin matrix encases the hole. According to Avella et al. (2009), Koronis, Arlindo, and Mihail (2013), Dittenber and Ganga Rao (2012), Thakur, Thakur, and Gupta (2013), the XRD patterns exhibit the usual crystalline nature of natural cellulose. The locations of the Bragg peaks are $2\theta = 16^\circ$ and $2\theta = 23^\circ$. The primary crystalline peak of cellulose is seen by the strong peak at $2\theta = 23^\circ$. The XRD pattern has large peaks at higher 2θ values, which are indicative of the amorphous nature of these fibers. The locations of the peaks in wet fibers remain unchanged as the water content rises, but the X-ray intensity does. This is probably certainly the result of the enhanced mobility allowing disordered chains to slot into the proper crystal orientation,

resulting in a steadily rising crystallinity with water content. This suggests that the crystallites have changed in shape and have a larger surface area (Kondo and Chie 1998). The values that were determined using the Williamson Hall plot (W-H plot) equation and relate to the crystallite size and lattice strain associated with the fiber samples are shown in Table 1. We can infer from the results that when the water content rises, the fiber's crystallite size increases

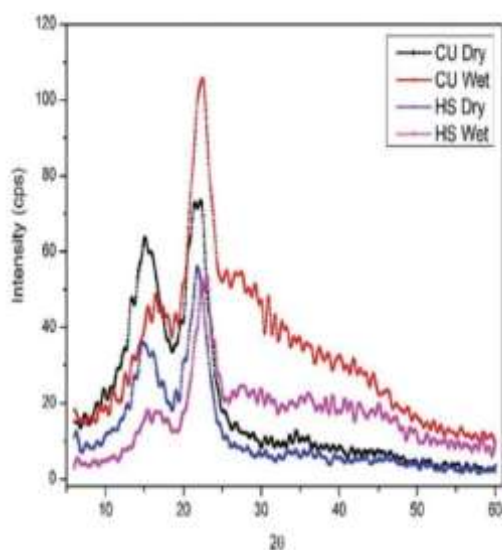


Figure 4. XRD of CU and HS both dry and wet.

Fourier transform infrared spectroscopy (FTIR) analysis

In general, ligno-cellulosic substrates with a heterogeneous structure make up natural plant fibers. The broad absorption band at 3340–3355 cm^{-1} in Figure 5 displays the FTIR spectra of CU and HS for both wet and dry fibers. These spectra are characterized by an O–H stretching and H bonded bond structure, which primarily contains major functional groups of phenols, alcohols, and water from cellulose, hemicelluloses, and lignin (Reddy, Guduri, and Varada 2009). Wet fibers exhibit an increase in the region's broad absorption band and O-H stretching. due to the cellulose's insertion of hydrogen bonds. The wet fiber strand's spectrum and the dry fiber strand's spectrum are comparable. For HS wet, a significant absorption is noted. The infrared transitions of each sample are displayed in Tables 2 and 3.

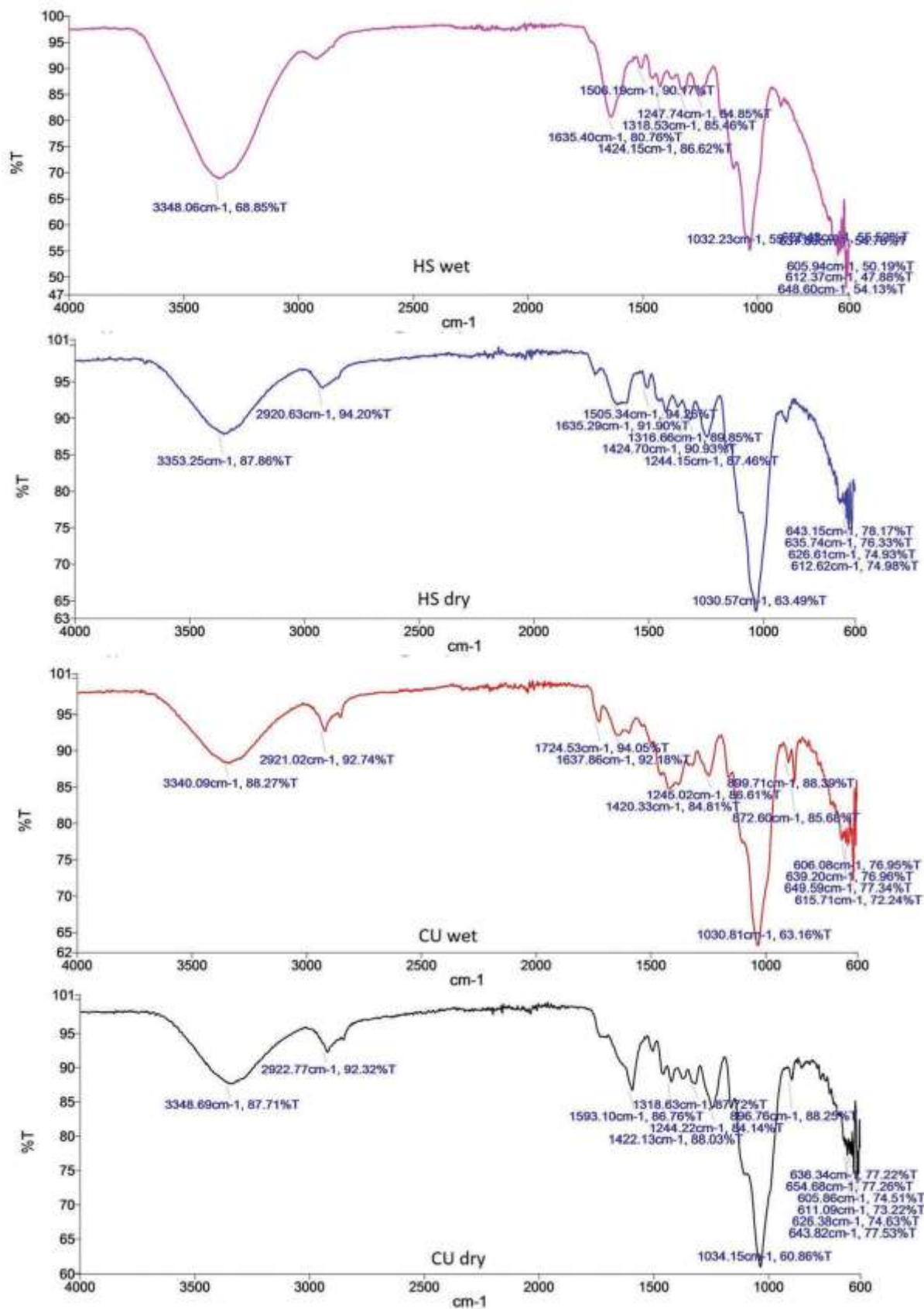


Figure 5. FTIR of fibers

Table 2. Infrared transition for CU dry and wet fibers.

CU dry fibers (wavenumber/cm)	CU wet fibers (wavenumber/cm)	Vibration	Source
3348.69	3340.09	OH stretching H-bonded alcohol, water, phenils	Cellulose, hemicellulose, lignin
2922.77	2921.02	C-H symmetrical stretching	Cellulose, hemicellulose
1593.1		C=C aromatics stretching	Lignin
1422.13	1637.86	OH bending of absorbed water	Water
1318.63	1420.33	HCH & OCH in plane bending	Cellulose
1244.22		CH2 rocking vibration	Cellulose
1034.15	1245.02	C=O & G ring stretching	Lignin
896.76	1030.81	C-O(primary alcohol)	Cellulose, hemicellulose, lignin
	899.71	COC,CCO and CCH deformation & Stretching	Cellulose
	872.6		
654.68	649.59	C-OH out of plane bending	Cellulose
643.82			

Table 3. Infrared transition for HS dry and wet fibers.

HS dry fibers (wavenumber/cm)	HS Wet Fibers (wavenumber/cm)	Vibration	Source
3353.25	3348.06	OH stretching H-bonded alcohol, water, phenils	Cellulose, hemicellulose, lignin
2920.63		C-H symmetrical stretching	Cellulose, hemicellulose
1635.29	1635.4	OH bending of absorbed water	Water
1505.34	1506.19	C=C aromatic stretching	Lignin
1424.7	1424.15	HCH & OCH in plane bending	Cellulose
1316.66	1318.53	CH2 rocking vibration	Cellulose
1244.15	1247.74	C=O & G ring stretching	Lignin
1030.57	1032.23	C-O (primary alcohol)	Cellulose, hemicellulose, lignin
643.74	648.60	C-OH out of plane bending	Cellulose
635.74	637.59		

SEM analysis

The micrographs obtained for both wet and dry fibers are displayed in Figures 6(a, b). Using "ImageJ," an image analysis program, the diameter of the CU and HS fiber strands was determined to be 450 μm and 46 μm, respectively, from the SEM pictures.

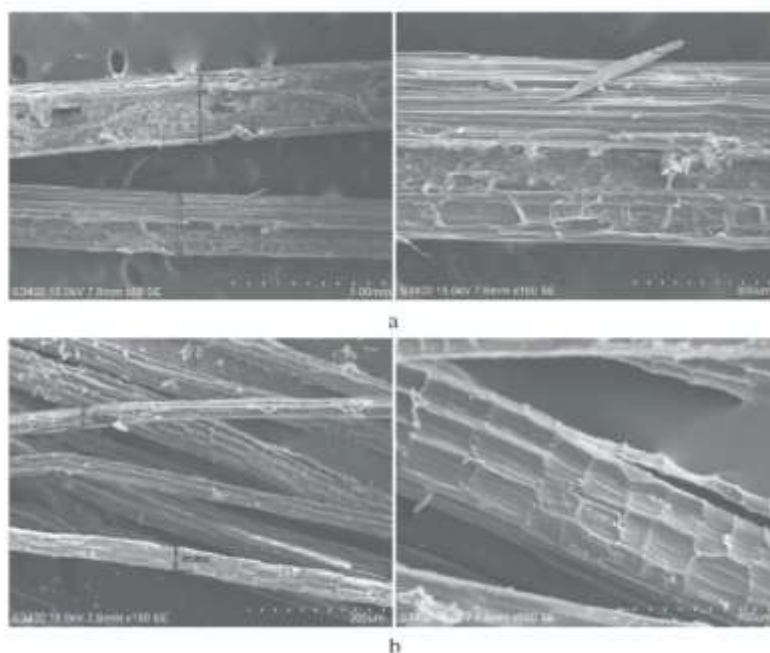


Figure 6. SEM figures of fibers

For CU and HS, the fiber fineness determined by the gravimetric technique is 372.18 denier and 13.6 denier, respectively. The graphs show demonstrate that the fiber's surface roughness and smoothness differ in damp and dry environments. When considering the elasticity of the fibers in relation to the outcomes of mechanical testing, wet fibers have a smoother surface than dried ones.

UTM

Elastic modulus, tensile strength, and percentage of elongation at break are metrics that are connected to the chemical structure and mechanical characteristics of the sample. Stress-strain curves for the dry and wet fibers of both samples are displayed in Figures 7 and 8. Because hemicelluloses and surface contaminants are removed after water treatment, the tensile strength of CU wet fiber is clearly better than that of all the available fibers given in the table, according to the results obtained for these samples. Additionally, the fibrils improved their ability to compactly rearrange themselves, which resulted in a closer packing of the cellulose chain and an increase in the tensile and strength of the fibers. However, CU dry fiber is not as strong as Vakka fiber. It suggests that wet fiber has a higher elastic strength than dry fiber. The materials become more stiff after water treatment since it is found that there is more elongation at the break point in the case of CU wet.

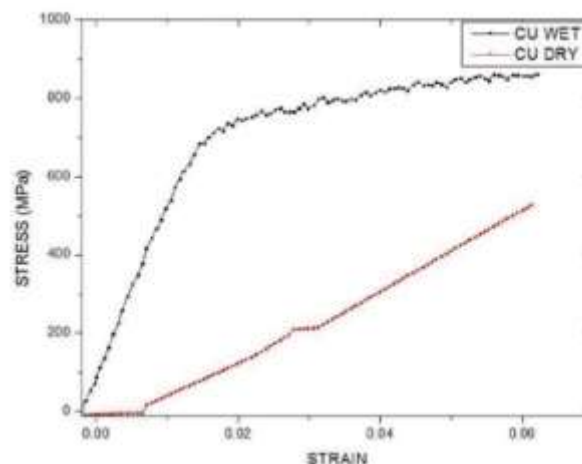


Figure 7. Variation of stress with strain for CU dry and wet fibers.

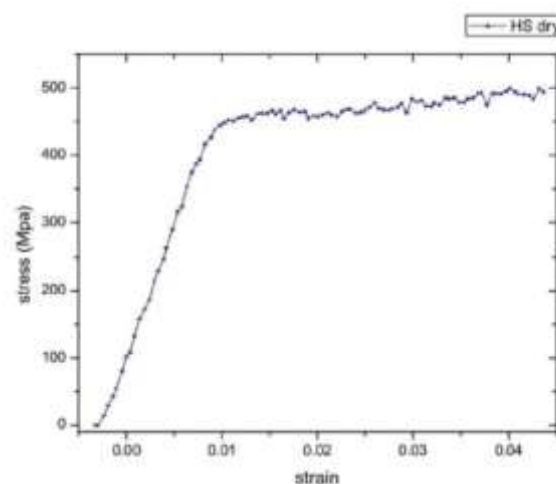


Figure 8. Variation of stress with strain for HS dry fibers.

Conclusions

We have defined Wine palm and Roselle, two naturally occurring fibers, as wet and dry samples for the first time. According to XRD research, in both dry and wet forms, CU has a more ordered area than HS. The functional groups and their features are identified by the FTIR result. It is evident from the XRD and FTIR results that they are cellulose fibers. SEM images show that when the water is treated, the fibers' surface smoothness rises. According to the UTM study, CU is more useful than other natural fibers. This study promotes the use

of these fibers in place of hazardous and non-renewable reinforcing materials in the manufacturing sectors, particularly in the packaging, rope-making, automotive, agricultural, and furniture industries.

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