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Morphological Characterization of Soapbark Fibers

Sivasubramanian Palanisamy[1,2*], Mayandi Kalimuthu[2], Murugesan Palaniappan[3], Azeez Alavudeen[2], Nagarajan Rajini[2] and Carlo Santulli[4]

[1]Department of Mechanical Engineering, Dilkap Research Institute of Engineering and Management Studies At Village Mamdapur, Post Neral, Tal- Karjat, Dist Raigad – 410101. Maharashtra, India.
[2]Department of Mechanical Engineering, Kalasalingam Academy of Research and Education, Anand Nagar Krishnankovil, Srivilliputhur, Tamil Nadu 626128, India.
[3]Department of Mechanical Engineering, College of Engineering, Imam Mohammed Ibn Saud Islamic University, Riyadh 11432, Kingdom of Saudi Arabia.
[4]School of Science and Technology, Università di Camerino, 62032 Camerino, Italy.

Authors' contributions

This work was carried out in collaboration among all authors. Authors SP, MK, MP and CS designed the study and discussed its structure. Authors SP, AA, MP and NR performed the experimental study and examined the results Authors SP and CS wrote the initial draft. Author CS corrected the draft to submission. All authors read and approved the final manuscript.

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ABSTRACT

The local availability of biowaste, which can be used as possible source of fibers, is an important trigger for research into new lignocellulosic materials for potential introduction into biocomposites: to evaluate this possibility, characterization is needed. In this work, soapbark (*Acacia Caesia*) fibers are obtained by peeling the bark fibrous structure out of these climbers, which are diffuse in Kerala, a state of southern India, particularly in the Western Ghats. Acacia Caesia bark is widely available and is used for ayurvedic medicine purposes to reduce the skin issues, and therefore in the wider context of cosmetics. The fibers extracted from the bark have not previously been researched for their potential use in materials, though them being lightweight, renewable, cheap, entirely or partially recyclable, and biodegradable. In particular, Acacia Caesia fibers' properties, once extracted from the bark, have been investigated to determine their appropriate applications in

** Corresponding author: Email: sivaresearch948@gmail.com;*

the future, starting from soapbark fiber morphology. The fibers were therefore extracted, and their fibers' chemical composition, density and morphological features, such as diameter, regularity, compactness, presence of porosities, were determined in particular by using scanning electron microscopy (SEM). In practice, soapbark fibers do appear quite similar to coir, with the added difficulty of cumbersome extraction process.

Keywords: Soapbark fiber; morphology; scanning electron microscopy; Acacia Caesia; chemical properties.

1. INTRODUCTION

In recent years, a large number of researchers have been focusing their studies on environmentally friendly materials to develop the environmental sustainability of products [1,2]. More specifically, the search for new or scarcely used natural fibers constitutes an important topic for research, providing additional data on potential applications, deduced from the characteristics of new natural fibers [3]. Insofar, technologies for processing such materials are either adapted from other sectors or purposely developed: it can also be anticipated that the future request for new natural fibers will increase. These developments can lead to improved environmental results for biodegradable products. As sustainable and biologically degradable materials, natural fibers can be extracted from the spikes, stem, leaf, seed, plant roots, and bark. They are preferred for different domestic and industrial applications due to their wide availability, potential for carbon dioxide sequestration, improved energy recovery, low production cost, reduced weight, biodegradability, high strength, and specific modules lower health risk [4]. Some of these plants can also derive from agro-waste or as a by-product from other applications: this can be for example, the case for roselle, Sunn hemp, okra, banana, pineapple, Napier grass, etc. [5]. Their possible use into "green composites" would bring the no-value agro-waste to be a constituent of valuable materials, notable for their potential high-specific strength, other than for cost-effectiveness. For this aim, new plants with reasonably straightforward extraction methods that would easily and cheaply not impair the fiber properties should be found. In various applications, such as aerospace, construction, sports equipment, food packaging, and the automotive industry, natural fiber composite materials are used already [6]. Due to their low cost, low density, environmental friendliness, ready availability, non-abrasion of processing equipment, and non-irritability, natural fibers are reported to be likely to replace, at least partially, synthetic fibers [7].

The interest on bark fibers is reflected by their easy availability as by-product or forest waste during pruning operations. Various studies have been carried out on other natural bark fibers and their composites, which are possible competitors of soapbark fibers, such as cotton stalks [8], eucalyptus globules [9], pine bark [10], *Ceiba pentandra* [11], western red cedar [12], etc. The applications are the most different, such as active carbons, food dyes or horticultural uses, yet not specifically for fillers in composites, although in principle this would also appear to be possible, namely for obtaining sufficiently long stretches of aligned fibers.

In particular, studies on modes and facility of extraction together with information of physico-chemical characteristics and more specifically cellulose yield have recently been triggered in the Indian context. A work on *Thespesia populnea* bark fibers highlighted the excellent potential of them, despite the irregularity and the presence of porous segments between the fibrils, which offered a tensile strength of over 550 MPa, owed to a cellulose content exceeding 70% and a crystallinity above 48% [13]. It is recognized though that most bark fibers would require some form of treatment, such as with alkalis like sodium hydroxide (NaOH) or potassium hydroxide (KOH), and to prepare for it, a thorough characterization study of the fibers would be necessary [14]. Among these fibers, a number are obtained from plants belonging to the Mimosaceae family: for example, *Acacia Concinna* offered an effective thermal stability up to 225 °C, which suggested the possibility of their probable application as the filler into biocomposites [15].

One species of the Mimosaceae, *Acacia Caesia* is an armed woody straggling shrub, known in India as 'Cabool', and extracts from the plant have recognized ethnographic medicine properties, for skin, sexual problems, lesions, stomach, and tooth problems, which allowed it to be used for a long time [16]. Many Acacia Caesia herbal products, like baboon paste, ayur shampoo, Nyle shampoos, etc., are sold on markets in pure or mixed forms. Acacia Caesia offers strong active substances, with antioxidant and antibacterial properties, in Indian medicinal systems. Acacia Caesia bark (ACB) (L.) Willd. fiber was not until now investigated for use in material despite their presence worldwide and abundance in tropical, subtropical, and warm temperate areas. In particular, ACB extracted from the foothills of Western Ghats were generally selected, from a large number of districts, including Wayanad, Palakkad, Kottayam, Kollam, Kasaragod, Idukki, Pathanamthitta, Thrissur, Kozhikkode, Kannur, in Kerala, India.

Although the information available on soapbark is substantial, it is surprising that not a single report is available to the best of our knowledge on ACB fiber characterization. This study has been carried out based on this lack of literature information, to explore a possible alternative application to this bark fiber than the ethnography medicine and cosmetic one. A possible suggestion is that Acacia Caesia fibers could be used as filler in composites or rubber. However, to assess this possibility, characterization studies will be needed, starting from the morphological one, which is the objective of this work.

2. MATERIALS AND METHODS

2.1 Materials

The raw material used was soapbark, which has been removed from the plant stem by mechanical stripping. This process allowed obtaining large portions of the bark, with larger dimensions of up to 60-70 cm, such the one depicted in Fig. 1, which included aligned fibers to be further extracted.

2.2 Fiber extraction

Acacia Caesia bark fibers from the forest of Malayatoor, located in Thrissur's district in Kerala, India, have been gathered. The barks of AC bark stems have been soaked to allow for the microbial degradation in water during 15 to 20 days, after which the AC bark fibers (ACBF) have been removed by water recovery process [17,18]. The fibers were washed by freshwater and placed under the sun for three days after the water washed off. After this, fibers were stripped off from the stem using a knife. The obtained fiber bundles to be used for analysis are depicted in Fig. 2. Some situations are evidenced, namely the difficulty to obtain sufficiently straight fibers and also the variability of diameters and the presence of parts with more flattened, or strip-like, section. Sixty fiber that were initially extracted were measured at five different sites along their length and the average value was reported.

2.3 Thermal and chemical analysis

Standard test methods measured cellulose, hemicellulose, lignin, moisture and ash content to determine the composition of ACBF. The ACBF density was evaluated using a Mettler Toledo xsz05 balance [19], while ash content in ACBF was analyzed using the ASTM E-830 standard [20], by using a muffle oven and heating up to 600 °C with a heating rate of 10°C/minute, and a method described elsewhere was applied to determine the moisture content (ASTM E-871) [21]. Cellulose, hemicellulose and Klason lignin were measured with the dry weight method [22]. In practice, Klason lignin is the insoluble organic residue from acid hydrolysis with 72% sulfuric acid (H_2SO_4), then diluted and refluxed with dilute acid. Hemicellulose was determined by summing up arabinose, xylose, mannose and galactose, while cellulose by the total amount of glucose [23]. Sulfuric acid 98 vol. % was purchased by Bhagwati Industries, Chhatral, Gandhinagar, Gandhi Nagar, Gujarat, India.

2.4 Scanning Electron Microscopy (SEM)

After some initial observation of the surface of ACBF using an optical microscope with 40x magnification, for their improved visualization, a scanning electron microscope (FEI ESEM model Quanta 200) has been used, with a speeded voltage of 20 kV and an achieved vacuum level of 1.5×10^{-3} Pa. The specimens were covered in a thin gold layer in order to prevent the accumulation of electric charges during the examination.

3. RESULTS AND DISCUSSION

3.1 Chemical properties

ACBFs consist of cellulose (37 wt. %), made up of helically coiled microfiber cellulose-binding to an amorphous matrix of lignin. The main compatibilizer between cellulose and lignin was hemicellulose (20 wt. %). The lignin (18 wt. %) in ACBFs protects against biological fiber attack. ACBFs also contains a significant amount of moisture (11.71 wt. %), as measured at 105 °C, and has a density of 1200 kg/m^3.

Fig. 1. Soapbark after decortication from plant stem

Fig. 2. Acacia Caesia fibers after extraction

The residual weight determined the quantity of ashes at 600 °C, which were measured as equal to 4.32 wt. %. The chemical compositions of ACBFs have been compared to those of several other natural fibers presented and indicated that the closest similarity has been observed with the composition of ficus leaf, as it had been reported in [24]. The fibers deemed particularly comparable with ACBFs have been discussed in [24-29] and relevant values are exposed in Table 1. In practice, this indicates for Acacia Caesia an intermediate amount of cellulose among ligneous fibers for mats, rugs, etc., such as coir, and typical textile fibers, such as flax, yet closer to the former than the latter.

3.2 Surface morphological analysis

A few short stretches of ACBF, constituted by aligned smaller fibers, not yet ready for tensile properties, since possibly subject to further splitting over time, with variable diameters, as reported in Figure 3, are obtained by stripping. These were observed under the optical microscope, and reported in Figure 4, clarified that straight portions of the fibers are obtainable, even with no treatment and with this very basic extraction method, though not exceeding around 10 mm length. These would have quite constant diameter, despite presenting a rough surface, sometimes with defects at the edges and some loose fibrils being gradually separated from the bulk of the material.

The similarity to other natural bark fibers is clearly indicated in SEM images of ACBFs: fiber bundles with diameter as low as around 150 µm have been further extracted as "technical" fibers for further developments, such as introduction as filler in composites. In particular, Fig. 5 shows the relative ductility, as opposed to brittleness, by oblique and complex crack propagation at failure, which can make it a candidate as a filler for polymers. In addition, surface cellular porosities tend to be concentrated, therefore tending to coalesce: consequently, most of the sclerenchyma bark does not appear to have significant porosity, as it has been seen to be the case, even for the sawdust of other Acacia species [30,31]. The above characteristics make ACBFs similar to other natural bark fibers.

More specifically, the fibers tend to be constituted by aligned bundles kept together by more viscous and glue-like material. At higher magnification, as depicted in Fig. 6, fibrils arrangement can be also observed with a multi-cellular structure on the quite irregular ACBF surface [32]. This suggests also the possible partial removal of parenchyma tissue, yet on the other side it raises concerns about the control of surface roughness, another factor that is crucial for technical application of natural fibers [33]. Regarding the possible use of Acacia Caesia fibers, the references for an initial evaluation are fibers in which cellulose, hemicellulose and lignin are present in quite comparable amounts and with the idea of having aspect ratios not very high, possibly not exceeding 100 as an order of magnitude, such as coir. This will be caused by the observed difficulty to separate single fibers from bark sclerenchyma, therefore applying fiber bundles for the purpose, which is likely to be eased by applying a chemical treatment [34,35].

Limitations of this preliminary study would concern the need for study of water absorption and of mechanical properties, in particular tensile strength of single fibers. To perform this, the separation of fibers from bundles needs to be optimized, so to lead to significant aspect ratio (length/diameter) fibers. This will occur, considering the inherent length limitations of the bark portions obtained after decortication, once single technical fibers are consistently separated, with diameter not exceeding 100-150 microns.

Table 1. Comparison of the chemical composition of ACBF with different fibers

Fibers	Cellul. (%)	Hemicell. (%)	Lignin (%)	Moisture (%)	Ash (%)	Density (kg/m^3)	Ref.
Acacia Caesia	37	20	18	12	5	1200	Here
Ficus leaf	38.1	30.5	23.4	-	4.5	-	[24]
Coir	32.69	22.56	42.1	10 [25]	-	860	[26]
Sah. aloe vera	67.4	8.2	13.7	5.8	-	1325	[27]
Ficus religiosa	55.58	13.86	10.13	9.33	3.96	1246	[28]
Cal. Gigantea	63.56	19.29	10.38	-	5.78	1324	[29]

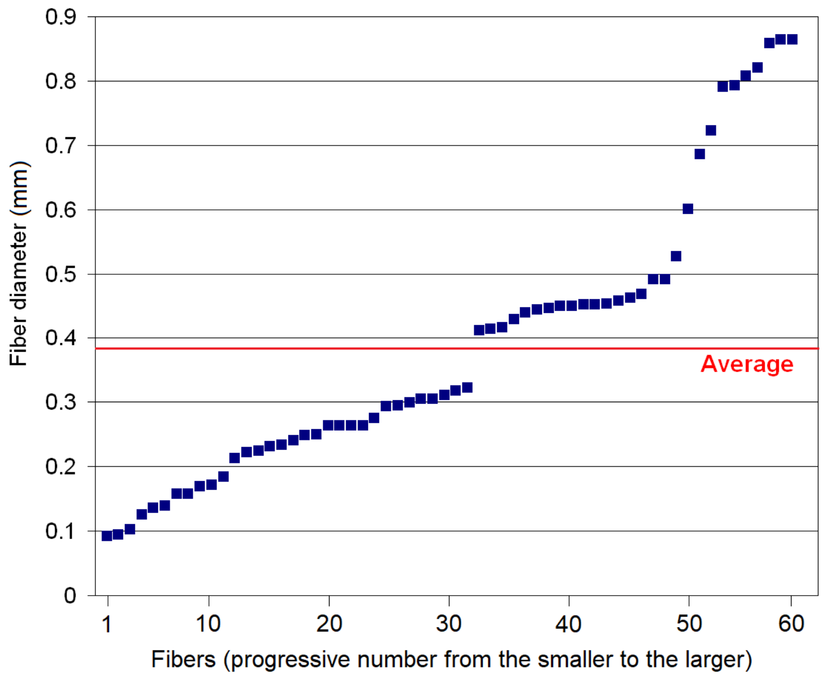

Fig. 3. Average diameter of each fiber along its length

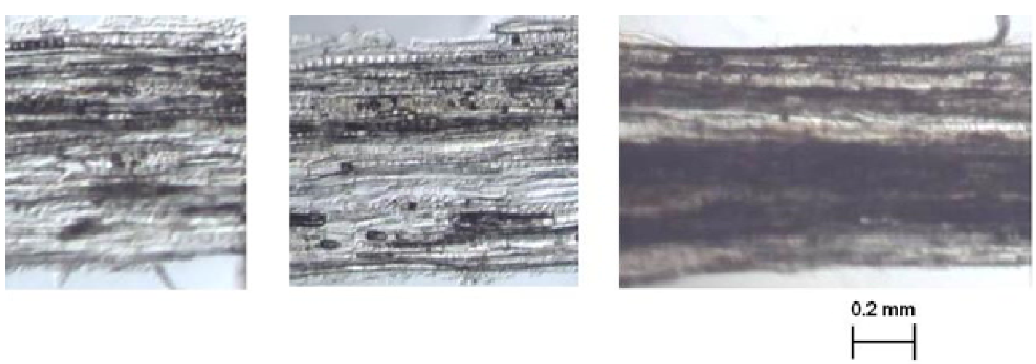

Fig. 4. Different portions of Acacia Caesia fibers (magnification 40x): the scale to the right refers to all the sections

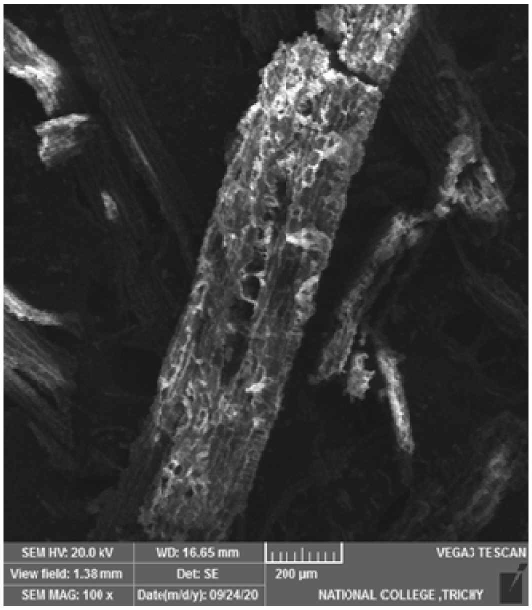

Fig. 5 Technical (minimum separable dimension) Acacia Caesia bark fiber (100x magnification)

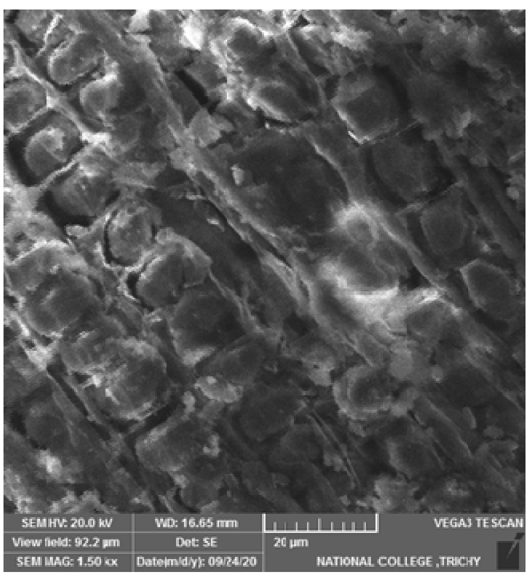

Fig. 6 Detail of the technical fiber (1.5kx magnification)

4. CONCLUSION

The outcome of this preliminary work suggests the possibility that quasi-aligned bundles of Acacia Caesia fibers are used as the reinforcement of composites, in the understanding that the extraction of the single fiber appears cumbersome and would reduce the availability of material. This is particularly important because these fibers are a by-product, or secondary raw material, from other processes.

In particular, the scanning electron microscopy investigation on the possible application of Acacia Caesia bark fibers indicated a considerable potential in terms of ductility of the structure, which would possibly reduce crack propagation in polymers and limited, though concentrated, porosity. The main limitation appears on the other side of the difficulty in extracting long as reasonably straight portions of fibers. This would imply needing to use whole fiber bundles and therefore require keeping (length/diameter) aspect ratios low and possibly using a chemical treatment to improve the obtained results.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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