

Article

Effect of Alkali Treatment on the Properties of Acacia Caesia Bark Fibres

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Abstract: As possible substitutes for non-biodegradable synthetic fibre, ligno-cellulosic fibres have attracted much interest for their eco-friendliness; a large number of them are already used for the production of green polymer composites. The search for further green candidates brings into focus other fibres not previously considered, yet part of other production systems, therefore available as by-products or refuse. The purpose of this study is to explore the potential of alkali treatment with 5% sodium hydroxide (NaOH) to enhance the properties of bark-extracted Acacia Caesia Bark (ACB) fibres. The microscopic structure of the treated fibres was elucidated using scanning electron microscopy (SEM). Moreover, the fibres were characterised in terms of chemical composition and density and subjected to single-fibre tensile tests (SFTT). Following their physico-chemical characterisation, fibre samples underwent thermal characterisation by thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC), and their crystallinity was assessed using X-ray diffraction (XRD). This level of alkali treatment only marginally modified the structure of the fibres and offered some improvement in their tensile strength. This suggested that they compare well with other bark fibres and that their thermal profile showed some increase of degradation onset temperature with respect to untreated ACB fibres. Their crystallinity would allow their application in the form of fibres with an average length of approximately 150 mm, even in thermoplastic biocomposites.

Keywords: *Acacia caesia*; alkali treatment; SEM; single-fibre tensile tests; thermal characterisation



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1. Introduction

Today, the growth of worldwide environmental awareness is more significant than ever before in the development of recyclable and environmentally sustainable composites, such as those based on natural fibres [1,2]. These fibres can be extracted, according to the single species, from different parts of the plant, such as stem or bast (e.g., hemp, flax, etc.), leaves (e.g., pineapple), fruit hair (e.g., coir fibres) or seed hair (e.g., cotton). This leads to a variable amount of lignin and cellulose present in the fibres and therefore to a wide range of morphologies and mechanical properties [3]. Despite the availability of data on a number of natural fibres, research is continuous to elicit the potential of other possible lignocellulosic reinforcements for composites extracted from plants.

In particular, natural fibres may be of interest whenever they belong to a more complex productive system, becoming by-products or waste. Recently, bark fibres of different species have been often used with the idea of obtaining reinforcements for polymer matrices [4–6]. Concerns were raised about the possible brittleness of composites obtained.

The subsequent need to optimise the extraction of technical fibres was an important requirement, for example dealing with difficult cases where the irregularity of the extracted material is significant. Examples of conspicuous cases, which led nonetheless to acceptable yielding, were aspen bark [7] or water hyacinth fibres [8]. The difficulties, which are also more frequent and characteristic of other more common ligno-cellulosic fibres, and which influence the behaviour of bark fibres, are the presence of kink bands and nodes along the fibre length [9]. As well as extraction, the treatment of fibres, most usually achieved through chemicals, is normally required: this removes loose matter, regularises the diameter of the fibres and makes them harder [10]. Treatment does not normally alter much the proportion between lignin, cellulose and hemicellulose, though some amount of the latter is normally removed [11].

Once extracted, bark stretches can be used in the form of non-organised fabric [12]. An alternative option can be to separate the single filaments and cut them to avoid having split ends, therefore obtaining a more regular shape, which enables the fibre to withstand higher tensile loads. This option is necessary in cases such as the introduction of rubber composites, which have been investigated on isora fibres [13].

This study aims to elucidate the physico-chemical, mechanical and thermal bark properties of *Acacia Caesia* bark (ACB) fibres, contextualising and comparing them with previously investigated natural bark fibres. The bark of this plant is used in the Indian tradition as a scrubber, while leaf extracts are used in cosmetics and herbal products. The stem of *Acacia Caesia* (L.) Willd. also contains strong active substances with antioxidant and antibacterial properties [14]. Initial studies on untreated ACB fibres suggested the potential for further investigation, revealing the relative ease of extracting from the bark technical fibres of diameter 100–150 microns, with the best fibres measuring over 100 mm in length [15,16]. These ACB fibres are characterised to collect information and we propose that they could be used as novel reinforcements for composites, therefore in a typical alkali-treated state.

2. Materials and Methods

2.1. Materials and Treatment

Acacia Caesia (AC) (L.) Willd, whose different parts are depicted in Figure 1, is a growing shrub belonging to the family of Mimosaceae. AC is generally available in different parts of southern India, especially in Kerala, in the Thrissur district and Malayatoor forest. After its maturity is reached, such as in Figure 1a, as opposed to Figure 1b, the bark starts fracturing and can be easily removed from the stem; the inside of the bark is used for bathing scrubs and ayurvedic purposes. When cutting the shrub to obtain the stem, the bark is detached and left to rot in fields, although it biodegrades very slowly. It is therefore a ligneous waste which could still be available for possible use in materials.

Once the bark is available in the form of fibrous almost-flat portions, as the one reported in Figure 2a, a water-retting process was adopted to remove fibres from the bark by allowing microbial degradation in the water [17]. The freshly matured stems were cut from the plant and the AC bark was soaked in water for 15–20 days, after which it was possible to obtain fibres that could be split, as in Figure 2b. After a further three days of soaking, the single fibres were arranged into bundles with different lengths, between 100 and 150 mm, and left to dry under the sun for around 6 h: these are shown in Figure 2c.

The separated technical fibres which, after removing split ends, measured around 150 mm in length, were divided into two sets. Of the two sets, one was tested in the untreated state, whilst the other was immersed in a 5 vol.% sodium hydroxide (NaOH) solution at ambient temperature, and then left to dry overnight at 20 °C. All subsequent measurements were carried out on alkali-treated fibres, where it is not explicitly said otherwise.

A comprehensive scheme of the experimental activities carried out in this work is shown in Figure 3.

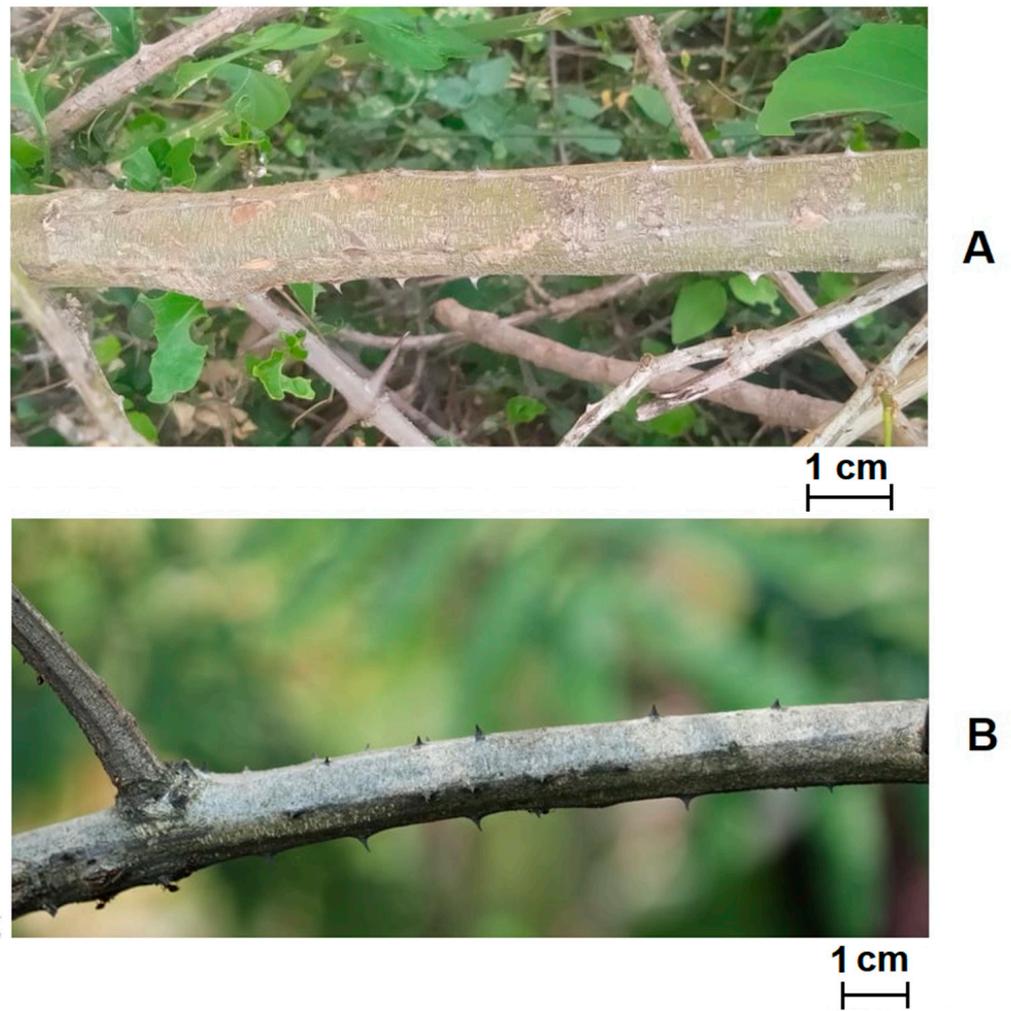


Figure 1. Acacia Caesia stems with bark. (A) Mature stem; (B) immature stem.



Figure 2. Acacia Caesia extraction. (A) Manually extracted bark stretches; (B) splitting into fibres after water soaking; and (C) fibres arranged into bundles according to their length.

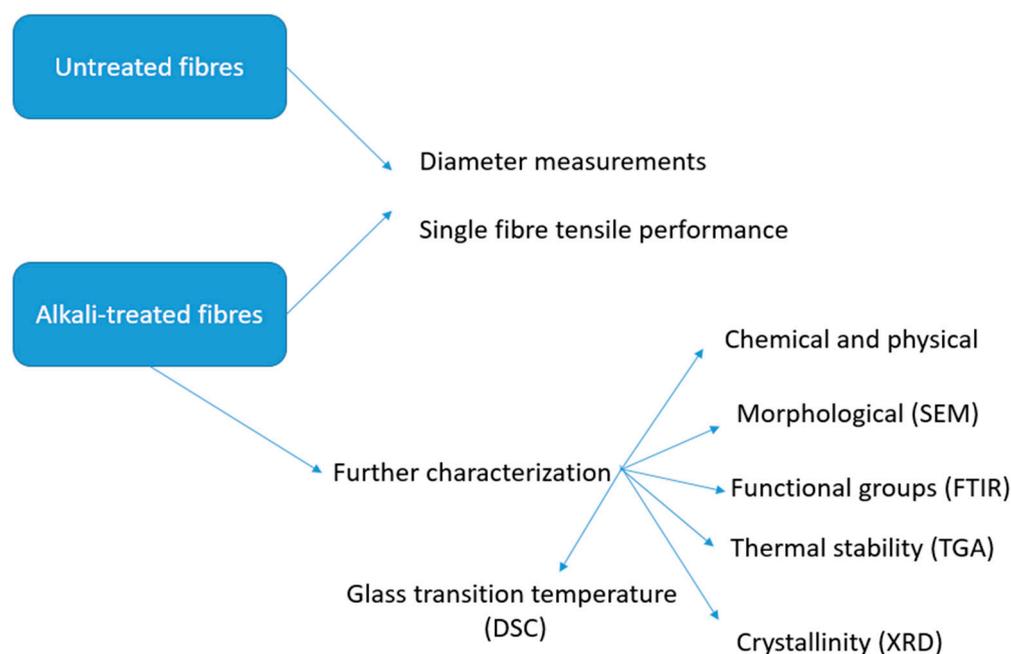


Figure 3. Scheme of experimental activities performed.

2.2. Scanning Electron Microscopy (SEM)

Scanning electron microscope (SEM) model Quanta 200 (FEI, Hillsboro, Oregon, United States), with a speeded voltage of 20 kV and a nominal vacuum level of 1.5×10^{-3} Pa, was employed. The fibre specimens, three untreated and three treated, were covered in a thin gold layer to prevent the accumulation of electrical charges during the examination. The purpose of the study with the SEM was to observe the surface morphology and evaluate whether the treatment produced any modification of the aspects of the fibres.

2.3. Single Fibre Tensile Testing (SFTT)

The single fibres were secured to a small paper frame according to ISO 11566, then aligned and fixed in the clamping mechanism of the testing machine. For this purpose, a ProLine tensile machine (Zwick Roell GmbH & Co., Ulm, Germany) equipped with a tensile head for a maximum force of 5 kN, was used. The tests were carried out with a gauge length of 50 mm on fibres 125 ± 5 mm long in displacement control mode, with a crosshead speed of 5 mm/minute. A total number of 70 fibres were tested, 35 before treatment and 35 after treatment, both for diameter measurements and for mechanical testing.

2.4. Chemical and Physical Characterization

Standard test methods for the determination of ACB fibres composition were adopted to determine in particular the content of cellulose, hemicellulose, lignin, moisture and ash content in both the untreated and treated fibres. The ACBF density was assessed using a Mettler Toledo (Columbus, OH, USA) xsz05 balance, while the ACBF ash content was analysed using the ASTM E-830 standard; the moisture content determination method according to ASTM E-871 was followed. The dry weight method was used to measure cellulose, hemicellulose and Klason lignin, following what was suggested in [18].

2.5. Fourier Transform Infrared (FTIR) Spectroscopy

In order to determine the presence of free functional groups on the ACBF, the FTIR Spectrometer (OPUS 7.5.18, from Bruker GmbH, Ettlingen, Germany) was used. In a range of 4000 to 400 cm^{-1} , spectral outputs were obtained using 32 scans and recorded as a function of the wave number in transmitting mode.

2.6. Thermogravimetric Analysis (TGA)

Thermogravimetric analysis (TGA) was used for the measurement of thermal stability and the temperature degradation interval of biofibres using a temperature scanning procedure. A STA 449 F3 Jupiter (STA 449 F3) (NETZSCH GmbH & Co. Holding KG, Selb, Germany) was used for analysis, which could accurately record the sample weight between room temperature and 1550 °C. In particular, in the course of this study, 5 mg of ACBF were heated in aluminium, their temperature increasing by 10 °C per minute from 30 to 550 °C. Nitrogen (N₂) was delivered to the oven at a flow rate of 30 mL/min to preserve the inert environment.

2.7. Differential Scanning Calorimetry (DSC) Analysis

Differential scanning calorimetry (DSC) was used to measure the glass transition temperature (T_g) and other transition phenomena in polymers, such as cellulose and lignin. The instrument used, DSC214 (NETZSCH GmbH & Co. Holding KG, Selb, Germany) simultaneously recorded heat exchange, due to thermal reactions occurring during fibre heating and cooling, and operated at a temperature rate of up to 500 K/minute. For this scanning, a temperature rate of 5 K/minute was applied with an acquisition rate of 300 points per minute.

2.8. X-ray Diffractometer (XRD) Analysis

XRD analysis, to measure the crystallinity of the cellulose fraction in the fibres, was carried out through a powder X-ray diffractometer by Malvern Panalytical (Malvern, UK). The model was a PANalytical X-Pert3 PRO with copper (Cu) K α 1 energy, therefore equal to 8.04 keV and 0.039° resolution. A voltage of 40 kV was used, while the intensity of current was 15 mA. The treated fibres were pressed to ca. 1 cm diameter pellets prior to perform the X-ray measurements.

3. Results

The objective of this work is to assess the suitability of alkali-treated Acacia Caesia bark (ACB) fibres and their use as the fillers in polymer composites, through the acquisition of physico-chemical data. This was done in comparison with the limited evidence obtained so far from the untreated ACB fibres. In particular, the measured average density of the fibres was 1150 kg/m³, slightly inferior to the value of 1200 kg/m³ obtained on the untreated fibres [16]. This value appeared similar to what was observed on fibres containing a substantial amount of lignin, for example curauà or coir fibres. On the other hand, typical fibres for textile uses, such as hemp, sisal, flax and cotton, had higher densities, which have been related to the presence of a larger quantity of crystalline cellulose. It is typical of studies on vegetable fibres to indicate considerable variability in physical properties such as density, due to the cultivars used and the mode of extraction, etc. However, data obtained from some works, reported in Table 1, suggested a clear collocation of the density values for ACB fibres amongst high lignin ones.

Table 1. Density of different fibres.

Fibres	Density (kg/m ³)	Reference
ACB	1150–1200	[16] and this work
Curauà	1200	[19]
Coir	1150–1250	[20,21]
Hemp	1393	[22]
Sisal	1450	[23]
Pineapple	1440	[23]
Flax	1540	[24]
Cotton	1520	[25]

With regard to the chemical composition, the application of alkali treatment does appear, as indicated in Table 2, to slightly reduce the amount of hemicellulose and in general of loose matter, reported as extractives. In particular, it is noticeable that the amount of cellulose obtained is similar to that which is obtained from coir fibres in [26,27], though generally coir fibres contain a very limited amount of hemicellulose in comparison to what we found in ACB fibres. In addition, the removal of hemicellulose appeared much less effective than in other bark fibres, such as *Hardwickia Binata*, where using the same amount of sodium hydroxide (5 vol %) for an even shorter time than in the present work (only 2 h) caused hemicellulose to decrease from 22.24 to 7.01% [28]. This suggests that ACB fibres are particularly resistant to alkali treatment, so that, in view of their introduction into composites, alkali treatment with more concentrated solutions of NaOH might also be suitable.

Table 2. Chemical composition.

Properties	Untreated ACB Fibres [15]	Alkali-Treated ACB Fibres (This Work)
Cellulose	37	40.1
Hemicellulose	20	17.3
Lignin	18	23.5
Ash	4.3	5.4
Moisture	11.7	11.5
Wax	0.4	0.2
Extractives	8.6	2

The extraction process of the fibres via mechanical stripping resulted in obtaining partially separated bundles rather than recognisable single fibres. The partial linking of the fibres into bundles was due to the presence of loose matter, therefore the fibres needed further separation by hand before possibly performing the single-fibre tensile tests (SFTT). This was also due to the considerable scattering of their diameter values. The diameters of these initial stretches and then of the further separated fibres, before and after treatment, were measured under a light microscope. The results of these diameter measurements are summarised for better clarity in Table 3. The relative accuracy of these measurements in only one region along the fibres, neglecting their non-circular section, needs to be emphasised. It is suggested that the slight reduction in diameter which occurred with alkali treatment might be ascribed to the removal of some loose material. On the other hand, some split edges remained, which were not considered in the regions that were measured. In every fibre, a number of measurements (at least five) were taken perpendicularly to the fibre edges, such as those shown in Figure 4, performed on further separated fibres.

Table 3. Results of diameter measurements.

Extraction Method	Average Diameter (μm)	Standard Deviation (μm)
Untreated mechanically stripped	423	240
Treated mechanically stripped	372	211
Untreated further separated	127	25
Treated further separated	123	22

Fibres were then cut at a length of 125 mm to prepare them for mechanical testing. Typical curves obtained from untreated and alkali-treated fibres are reported in Figure 5. Considering the tensile strength results, which are reported in Table 4, the values obtained were lower than those measured in textile fibres, such as flax, where values of around 1200 MPa were encountered [29], while the elongation at break is always around a few percent of the initial length. This treatment results in some improvement of the ultimate load and of the elongation in a somewhat reduced scattering of the results. For ACB fibres, neglecting in the first instance the variability of the diameters, and considering the average

diameter of the fibres and the average ultimate load, an ultimate stress equal to 852 MPa for untreated fibres and 937 MPa for treated fibres was observed, respectively. Data on elongation appear quite promising, considering that a number of investigations on the tensile performance of bark fibres did not indicate an elongation to break exceeding 2%, such as in the case of *Calotropis Gigantea* (L.) Dryand ex W.T. Aiton [30]. Alternatively, the variability of the ultimate stress for similar bark fibres showed variances largely superior to 100, i.e., a standard deviation exceeding 10% of the average value, as shown in *Acacia Leucophloea* Roxb. Willd. [31].

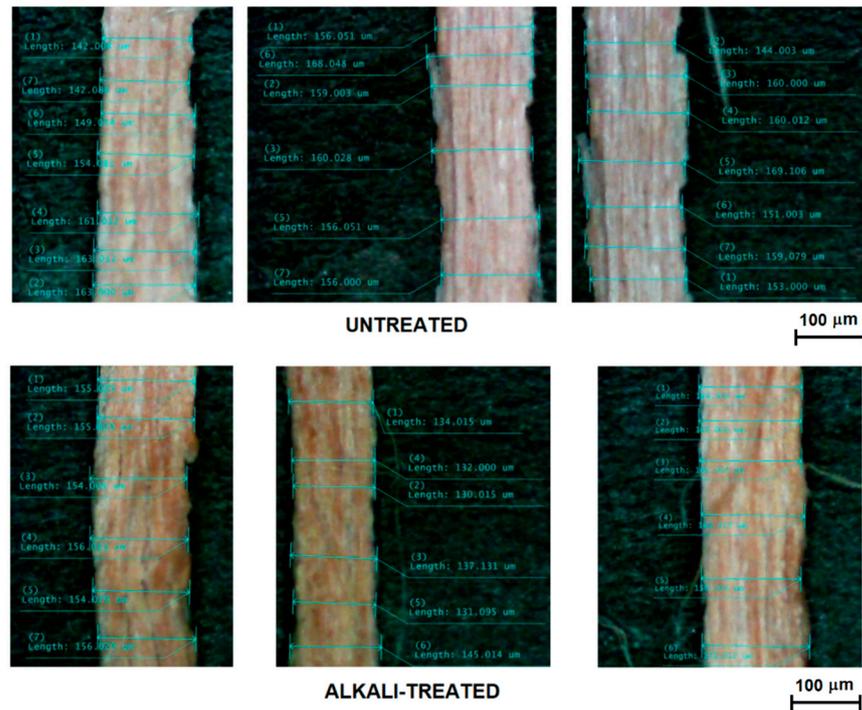


Figure 4. Examples of diameter measurements along several untreated and alkali-treated fibres (the scales refer to all three images in a row).

Table 4. Single-Fibre Tensile Tests (SFTT) results.

		Avg. Value	Std. Deviation	Variance
Untreated fibres	Ultimate load (N)	10.8	5.21	48.31
	Elongation (%)	3.4	1.0	30.75
5% NaOH treated fibres	Ultimate load (N)	11.14	4.56	20.82
	Elongation (%)	4.25	0.80	0.64

In Figure 6, the SEM micrographs of the fibre surface at different magnifications are represented. The ordered alignment of the fibres, and conversely the discontinuities in the bundles, were visible at lower magnification, in particular considering that only some parts of the bark were adapted to obtaining oriented fibre structures (Figure 6a,b). In contrast, higher magnifications allowed for the identification of other features, such as the detachment of the hexagonal cells protecting the internal cellulose structure, most likely due to alkali treatment (Figure 6c,d) (e.g., in [32]). In Figure 7, the typical appearance of the surface of a further separated fibre, therefore adapted to the application of some tensile load, is depicted. The clear presence of striations, indicating boundaries between fibrils which are therefore likely to split during loading, was observable.

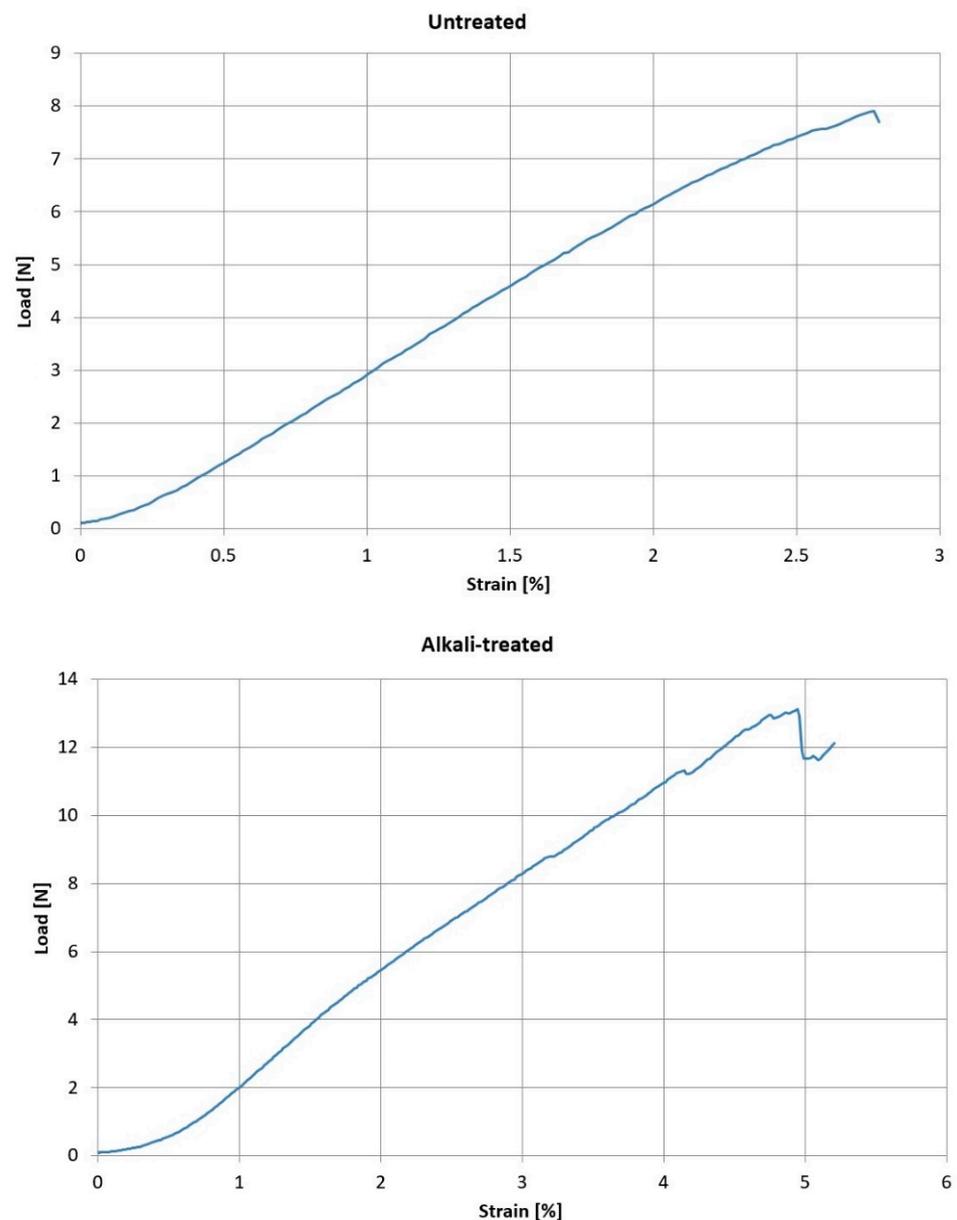


Figure 5. Typical tensile curves for untreated and alkali-treated fibres.

Moving on to thermal characterisation, DSC curves, reported in Figure 8, showed two endothermic peaks. A first peak culminates at 60 °C indicating the loss of absorbed water which occurs at quite a low temperature with respect to studies carried out on other bark fibres, such as *Ficus Natalensis* [33]. A considerably smaller peak was also observed, which culminated at 149.7 °C, and was attributed to the rupture of inter- and intra-molecular linkages and the loss of volatiles; some hemicellulose was possibly among these. Moreover, from the TGA analysis performed in Figure 9, some mass loss, starting at around 60 °C and reaching no more than 7% at 250 °C, was initially observed. Following this, a single degradation curve was revealed with an onset at 326 °C by measuring the intersection of the tangents at the two different parts of the curve, as suggested in [34]. This is very close to results reported in [35] for pure cellulose extracted from oil palm empty fruit bunches (OPEFB). The degradation temperature was also higher than the one observed in [16], which was equal to 308 °C.

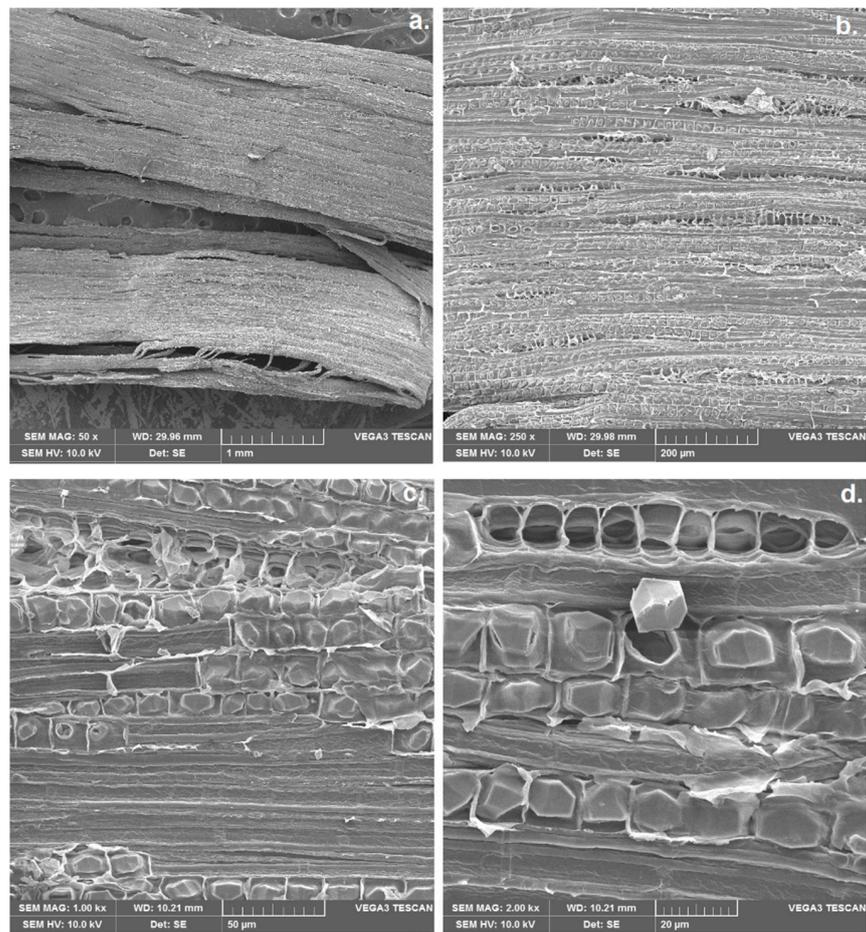


Figure 6. SEM micrographs of the alkali treated mechanically stripped fibre surface at different magnifications: (a) 50×; (b) 250×; (c) 1 k×; and (d) 2 k×.

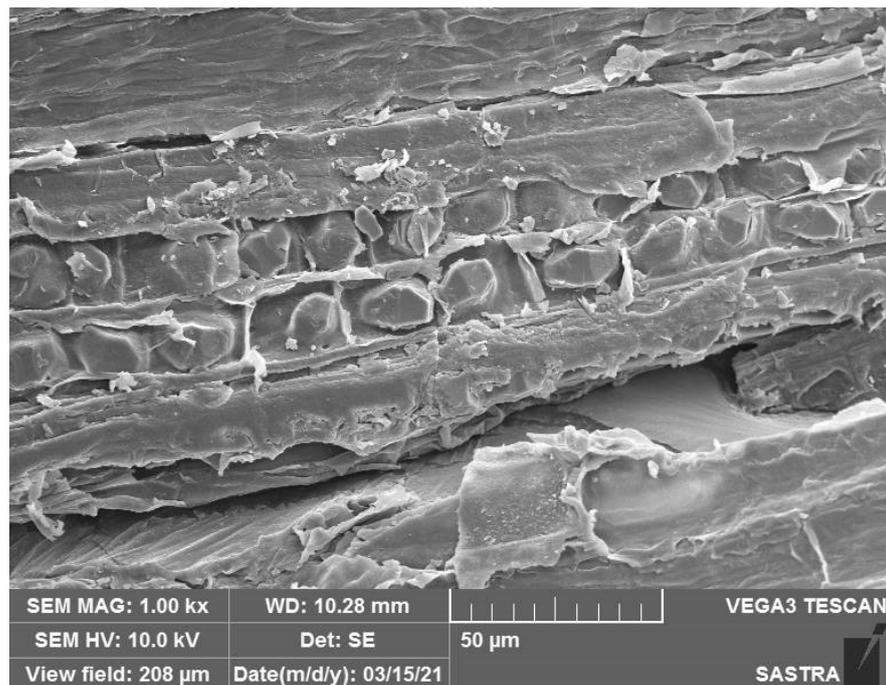


Figure 7. Typical appearance of the surface of a further separated fibre.

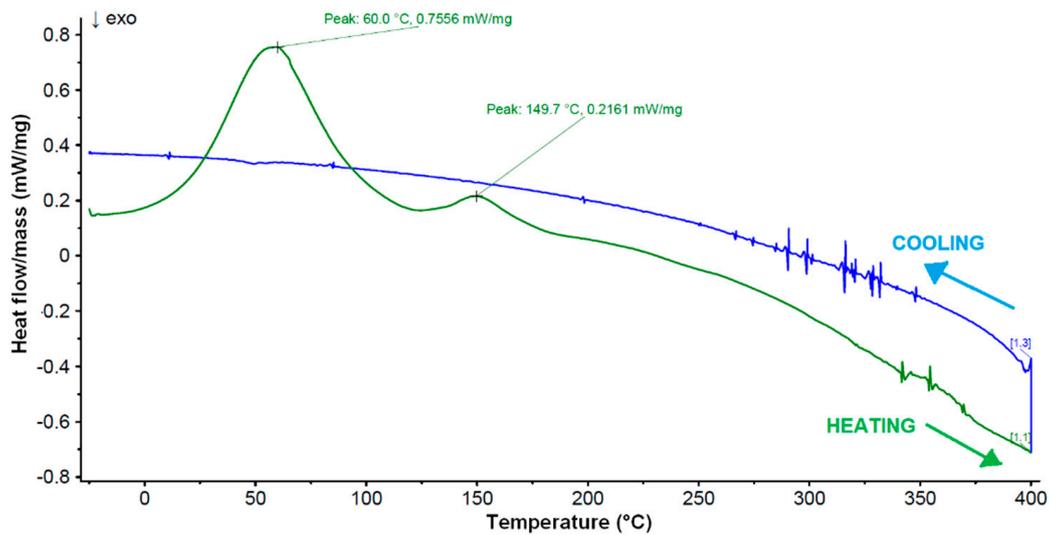


Figure 8. DSC scanning of a fibre sample (heating and cooling) over the temperature interval from -25 to 400 °C.

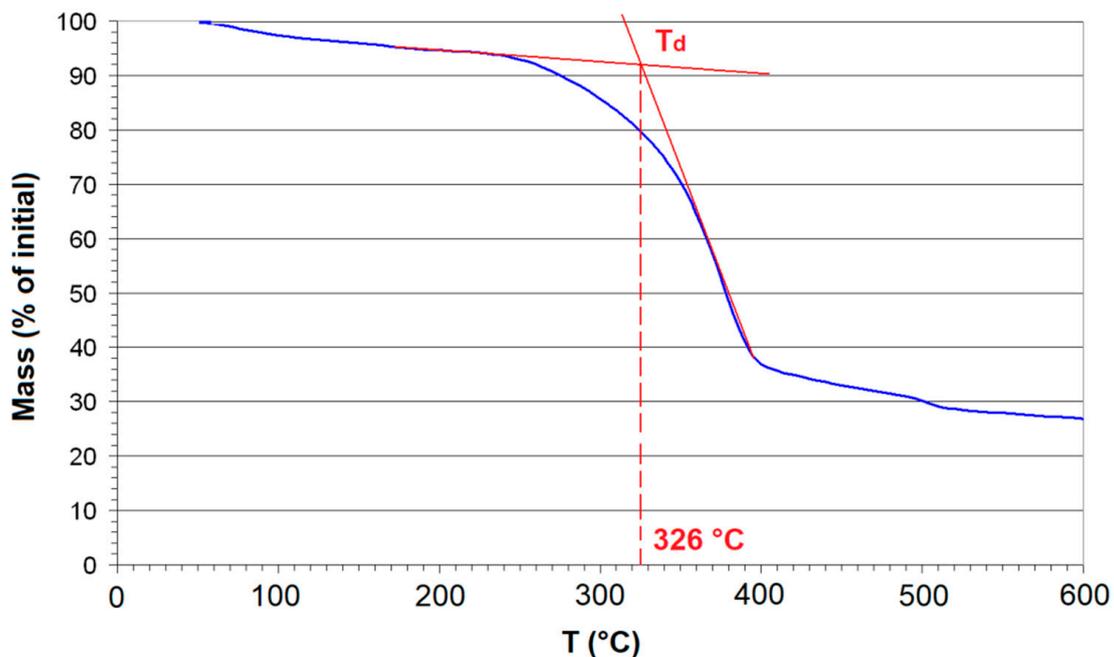


Figure 9. TGA curve during heating from 30 to 600 °C.

Regarding the identification of different components with further chemical characterisation, FTIR spectroscopy (Figure 10) indicated a number of regions and peaks of absorption. In particular, absorption bands at 3344 – 3231 cm^{-1} represented the stretching vibration of O-H, due to moisture content where lignin, hemicelluloses and cellulose presented the hydroxyl groups. The C-H stretching vibration in cellulose, hemicelluloses and lignin was identified in the spectrum at 2994 – 2932 cm^{-1} [36]. The very evident peak at 1604 cm^{-1} can be attributed to aromatic skeletal vibrations in lignin [37], possibly enhanced due to alkali treatment. The peak at 1311 cm^{-1} could be ascribed to the C-H bending of cellulose [38], whereas the sharp peak at 1016 cm^{-1} was elsewhere reported as characteristic of a C-O-C glucopyranose ring [39]. No peaks that corresponded to the presence of wax were identified.

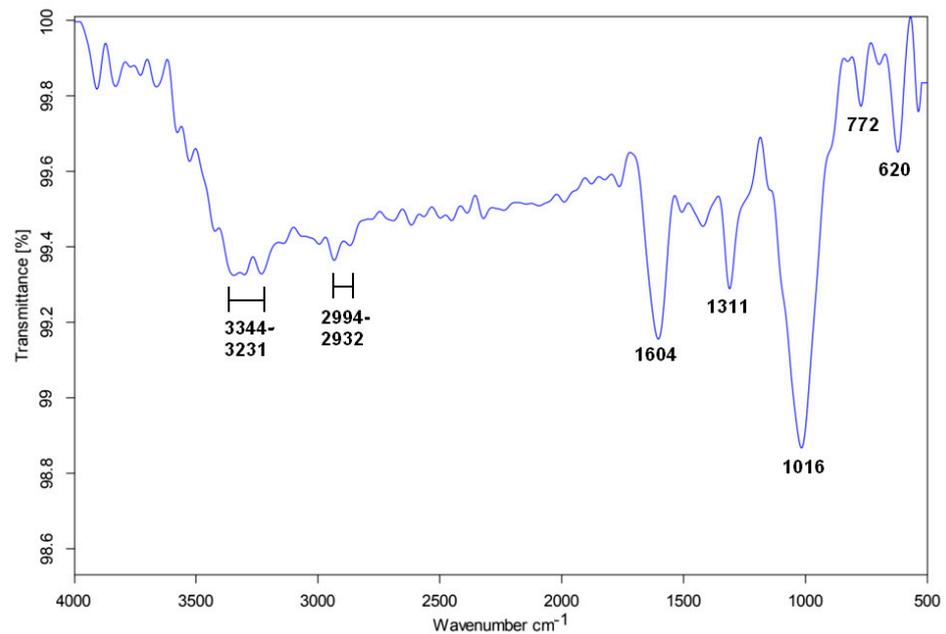


Figure 10. FTIR spectrum in the interval between 4000 and 500 cm^{-1} .

The alkali treatment of lignocellulosic fibres usually leads to the transformation from cellulose I to cellulose II [40]. From the XRD diagram in Figure 11, it is possible to see that the 002 crystalline cellulose peak is visible at around 22.7° and the amorphous cellulose peak (1-0-1 plane) at around 16° . According to Segal's law [41], the crystallinity of the cellulose is obtained by the ratio offered by two respective intensities, $I_{(22.7-I_{16})}/I_{22.7} = \sim 40\%$. This value was found to be just a little lower than what was found on other bark fibres recently investigated for applications in composites, whose outcomes are summarised in [42]. The two further and smaller peaks at around 30° and 38° were also observed, yet not commented on, in a study on *Prosopis juliflora* (Sw.) DC. [43]. Indications provided in [44] suggest that the presence of these peaks may refer to an increased level of order, as they were detected in the XRD spectra of cellulose microfibrils, with respect to cellulosic banana plant waste.

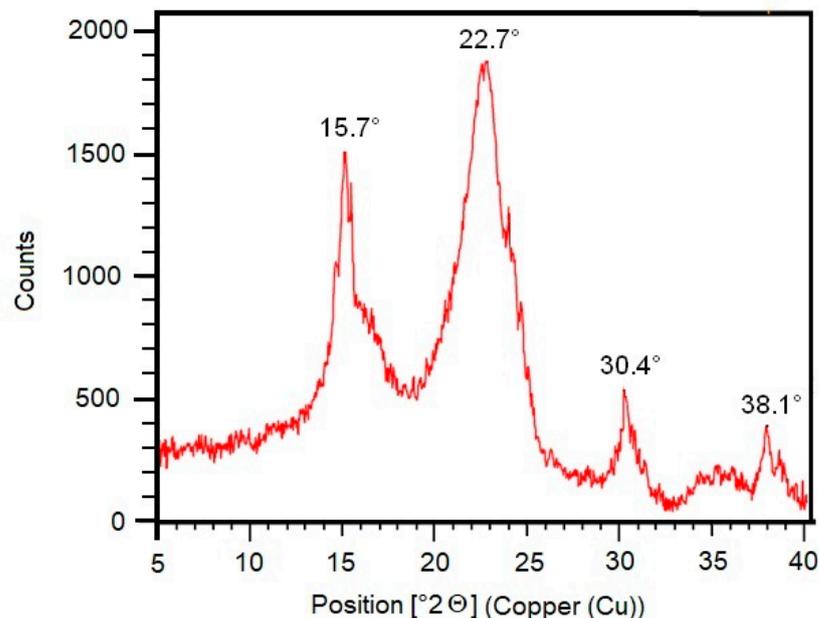


Figure 11. XRD analysis in the range 5– 40° .

4. Discussion

This part of the work aims to comment on the results obtained in a wider sense, trying to elucidate the operation of investigating *Acacia Caesia* bark fibres for possible applications. The rationale of this study was exploring the possibility of using this particular type of bark fibre as a local substitute for other ligno-cellulosic fibres with lower densities (around 1200 kg/m³) and as a composition including a similar amount of cellulose and lignin, such as coir. In principle, this makes sense as the bark material is widely available and can be introduced in biodegradable matrices, such as cellulose-based ones, e.g., cellulose acetate-butyrate (CAB), with the concept of “bark biorefinery” [45].

Some obvious difficulties appear to be present nonetheless, prior to the idea of application in a composite. First and foremost is the extraction to the level of a “technical fibre”, which is able to provide some tensile resistance with no excessive splitting. The latter is unfortunately an inherent characteristic of bark fibres, due to the limited regions that present a recognizable linear orientation in the fibres [46]. This poses the idea of treatment to improve the characteristics of the fibres, starting with the removal of loose and non-structural matter, then improving tensile performance. In general, sustainable treatments, such as ultraviolet treatment, would preferably be used, starting with an exposure to sunlight [47]. However, substantial improvements are in reality only obtained from procedures mostly applied to ligno-cellulosic fibers, such as flax, hemp and sisal, etc., where alkali treatment is performed on bark fibres; an example is given in [48].

It is promising that some improvement is observed in very mild alkali treatment, in terms of tensile strength and the degradation temperature of the fibres, which would facilitate their tentative introduction in a composite. However, there is a complex balance between the suitability to increase the bark fibre content in the polymer matrix and the need for further, more concentrated treatment, alkaline or otherwise [49]. To better control the properties of the fibres pre- and post-treatment, more information on the crystallinity of the cellulose fraction of the bark materials is needed [50], where results in the case of *Acacia Caesia* would require further investigation.

5. Conclusions

Preliminary studies on alkali-treated *Acacia Caesia* bark (ACB) fibres offered some evidence regarding the possibility of extracting fibres, with diameters not exceeding 150 microns, from the bark, fibres which may offer some potential for application as the reinforcement of polymer composites. However, the presence of loose matter and the limited regularity of the fibres suggested the need for the chemical treatment of ACB fibres. The present study indicated that alkali treatment at 5% NaOH for one hour provided some limited improvements in terms of tensile strength and removed a small amount of hemicellulose, resulting in an increase in the degradation temperature of the fibres. With all characteristics studied, such as the amount of cellulose and lignin, the level of crystallinity and the general order of the fibrous structure indicates that these fibres might be comparable to other well-diffused lignocellulosic fibres used in composites, such as coir, as well as to other bark fibres proposed during last few years. On the other hand, it is likely that treatment would need some further optimisation since its effect, though promising, so far appears to be limited.

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References

- Ahmad, F.; Choi, H.S.; Park, M.K. A review: Natural fiber composites selection in view of mechanical, light weight, and economic properties. *Macromol. Mater. Eng.* **2015**, *300*, 10–24. [[CrossRef](#)]
- Cheung, H.; Ho, M.; Lau, K.; Cardona, F.; Hui, D. Natural fibre-reinforced composites for bioengineering and environmental engineering applications. *Compos. Part. B* **2009**, *40*, 655–663. [[CrossRef](#)]
- Elanchezian, C.; Vijaya Ramnath, B.; Ramakrishnan, G.; Rajendrakumar, M.; Naveenkumar, V.; Saravanakumar, M.K. Review on mechanical properties of natural fiber composites. *Mater. Today Proc.* **2018**, *5*, 1785–1790. [[CrossRef](#)]
- Kathirselvam, M.; Kumaravel, A.; Arthanarieswaran, V.P.; Saravanakumar, S.S. Isolation and characterization of cellulose fibers from *Thespesia populnea* barks: A study on physicochemical and structural properties. *Int. J. Biol. Macromol.* **2019**, *129*, 396–406. [[CrossRef](#)]
- Hemachandra Reddy, K.; Meenakshi Reddy, R.; Ramesh, M.; Mohana Krishnuudu, D.; Madhusudhan Reddy, B.; Raghavendra Rao, H. Impact of alkali treatment on characterization of tapsi (*Sterculia Urens*) natural bark fiber reinforced polymer composites. *J. Nat. Fibers* **2019**. [[CrossRef](#)]
- Amutha, V.; Senthilkumar, B. Physical, chemical, thermal, and surface morphological properties of the bark fiber extracted from *Acacia concinna* plant. *J. Nat. Fibers* **2019**. [[CrossRef](#)]
- Yemele, M.C.N.; Koubaa, A.; Cloutier, A.; Soulounganga, P.; Wolcott, M. Effect of bark fiber content and size on the mechanical properties of bark/HDPE composites. *Compos. Part A* **2010**, *41*, 131–137.
- Chonsakorn, S.; Srivorradatpaisan, S. Effects of different extraction methods on some properties of water hyacinth fiber. *J. Nat. Fibers* **2018**, *16*, 1015–1025. [[CrossRef](#)]
- Baley, C. Influence of kink bands on the tensile strength of flax fibers. *J. Mater. Sci.* **2004**, *39*, 331–334. [[CrossRef](#)]
- Saravanakumaar, A.; Senthilkumar, A.; Saravanakumar, S.; Sanjay, M.R.; Khan, A. Impact of alkali treatment on physico-chemical, thermal, structural and tensile properties of *Carica papaya* bark fibers. *Int. J. Polym. Anal. Character.* **2018**, *23*, 529–536. [[CrossRef](#)]
- Saravanakumar, S.; Kumaravel, A.; Nagarajan, T.; Ganesh, I. Investigation of physico-chemical properties of alkali-treated *Prosopis Juliflora* fibers. *Int. J. Polym. Anal. Character.* **2014**, *19*, 37–41.
- Jayaramudu, J.; Jeevan Prasad Reddy, D.; Guduri, B.R.; Varada Rajulu, A. Properties of Natural Fabric Polyalthia Cerasoides. *Fibers Polym.* **2009**, *10*, 338–342. [[CrossRef](#)]
- Mathew, L.; Joseph, K.U.; Joseph, R. Isora fibres and their composites with natural rubber. *Prog. Rubber Plast. Recycl. Technol.* **2004**, *20*, 337–349. [[CrossRef](#)]
- Rahul, A.B.; Gautham, M.T.; Aswathy, T.R.; Indu, S.; Nair, A.S. Pharmacognostic and antibacterial activity evaluation of *Acacia caesia* (L.) Willd. *J. Pharmacogn. Phytochem.* **2020**, *9*, 48–54.
- Palanisamy, S.; Kalimuthu, M.; Palaniappan, M.; Alavudeen, A.; Rajini, N.; Santulli, C. Morphological Characterization of Soapbark Fibers. *J. Mater. Sci. Res. Rev.* **2020**, *8*, 19–26.
- Palanisamy, S.; Kalimuthu, M.; Palanappian, M.; Alavudeen, A.; Rajini, N.; Santulli, C. Characterization of *Acacia Caesia* bark fibres. In Proceedings of the ICNF2021 Conference, online, 17–19 May 2021; Fangueiro, R., Ed.; Fibrenamics: Guimaraes, Portugal.
- Sathishkumar, T.P.; Navaneethakrishnan, P.; Shankar, S. Tensile and flexural properties of snake grass natural fiber reinforced isophthalic polyester composites. *Compos. Sci. Technol.* **2012**, *72*, 1183–1190. [[CrossRef](#)]
- Van Soest, P.J.; Robertson, J.B.; Lewis, B.A. Methods for dietary fiber, neutral detergent fiber, and nonstarch polysaccharides in relation to animal nutrition. *J. Dairy Sci.* **1991**, *74*, 3583–3597. [[CrossRef](#)]
- Spinacé, M.A.S.; Lambert, C.S.; Femoselli, K.K.G.; De Paoli, M.A. Characterization of lignocellulosic curaua fibres. *Carbohydr. Polym.* **2009**, *77*, 47–53. [[CrossRef](#)]
- Hai, N.M.; Kim, B.S.; Lee, S. Effect of NaOH treatments on jute and coir fiber PP composites. *Adv. Compos. Mater.* **2009**, *18*, 197–208. [[CrossRef](#)]
- Bisanda, E.T.N.; Ansell, M.P. Properties of sisal-CNSL composites. *J. Mater. Sci.* **1992**, *27*, 1690–1700. [[CrossRef](#)]
- Rohen, L.A.; Neves, A.C.; Mantovani, D.D.P.; Mauricio, F.C.; da Silva Vieira, J.; Pontes, L.D.A.; Monteiro, S. Hemp fiber density using the pycnometry technique. In *Characterization of Minerals, Metals, and Materials*; Springer: Cham, Switzerland, 2017; pp. 423–428.
- Mishra, S.; Mohanty, A.K.; Drzal, L.T.; Misra, M.; Hinrichsen, G. A review on pineapple leaf fibers, sisal fibers and their biocomposites. *Macromol. Mater. Eng.* **2004**, *289*, 955–974. [[CrossRef](#)]
- Le Gall, M.; Davies, P.; Martin, N.; Baley, C. Recommended flax fibre density values for composite property predictions. *Ind. Crops Prod.* **2018**, *114*, 52–58. [[CrossRef](#)]
- Delhom, C.D.; Kelly, B.; Martin, V. Physical properties of cotton fiber and their measurement. In *Cotton Fiber: Physics, Chemistry and Biology*; Fang, D.D., Ed.; Springer: Cham, Switzerland, 2018; pp. 41–73.
- Satyanarayana, K.G.; Arizaga, G.G.; Wypych, F. Biodegradable composites based on lignocellulosic fibers—An overview. *Prog. Polym. Sci.* **2009**, *34*, 982–1021. [[CrossRef](#)]
- Bledzki, A.K.; Reihmane, S.; Gassan, J. Properties and modification methods for vegetable fibers for natural fiber composites. *J. Appl. Polym. Sci.* **1996**, *59*, 1329–1336. [[CrossRef](#)]

28. Madhuri, K.S.; Rao, H.R.; Reddy, B.C.M.; Chandrasekhar, V. Characterization of New Cellulosic Fiber from the bark of *Hardwickia Binata* (Narepa). *Int. J. Appl. Eng. Res.* **2018**, *13*, 967–972.
29. Charlet, K.; Eve, S.; Jernot, J.P.; Gomina, M.; Breard, J. Tensile deformation of a flax fiber. *Procedia Eng.* **2009**, *1*, 233–236. [[CrossRef](#)]
30. Ashori, A.; Bahreini, Z. Evaluation of *Calotropis gigantea* as a promising raw material for fiber-reinforced composite. *J. Compos. Mater.* **2009**, *43*, 1297–1304. [[CrossRef](#)]
31. Arthanarieswaran, V.P.; Kumaravel, A.; Saravanakumar, S.S. Physico-chemical properties of alkali-treated *Acacia leucophloea* fibers. *Int. J. Polym. Anal. Charact.* **2015**, *20*, 704–713. [[CrossRef](#)]
32. Reddy, N.; Yang, Y. Natural cellulose fibers from soybean straw. *Bioresource Technol.* **2009**, *100*, 3593–3598. [[CrossRef](#)]
33. Rwawiire, S.; Tomkova, B. Thermal, static, and dynamic mechanical properties of bark cloth (*Ficus brachypoda*) laminar epoxy composites. *Polym. Compos.* **2017**, *38*, 199–204. [[CrossRef](#)]
34. Liu, Z.Q.; Yi, X.-S.; Feng, Y. Effects of glycerin and glycerol monostearate on performance of thermoplastic starch. *J. Mater. Sci.* **2001**, *36*, 1809–1815. [[CrossRef](#)]
35. Nazir, M.S.; Wahjoedi, B.A.; Yussof, A.W.; Abdullah, M.A. Eco-friendly extraction and characterization of cellulose from oil palm empty fruit bunches. *Bioresource* **2013**, *8*, 2161–2172. [[CrossRef](#)]
36. Fatah, I.Y.A.; Abdul Khalil, H.P.S.; Hossain, M.S.; Aziz, A.A.; Davoudpour, Y.; Dungani, R.; Bhat, A. Exploration of a chemo-mechanical technique for the isolation of nanofibrillated cellulosic fiber from oil palm empty fruit bunch as a reinforcing agent in composite materials. *Polymers* **2014**, *6*, 2611–2624. [[CrossRef](#)]
37. Zhang, P.; Dong, S.J.; Ma, H.H.; Zhang, B.-X.; Wang, Y.-F.; Hu, X.-M. Fractionation of corn stover into cellulose, hemicellulose and lignin using a series of ionic liquids. *Ind. Crops Prod.* **2015**, *76*, 688–696. [[CrossRef](#)]
38. Sim, S.F.; Mohamed, M.; Irwan Lu, N.A.L.M.; Sarman, N.S.P.; Samsudin, S.N.S. Computer-assisted analysis of Fourier transform infrared (FTIR) spectra for characterization of various treated and untreated agriculture biomass. *Bioresource* **2012**, *7*, 5367–5380. [[CrossRef](#)]
39. Stanzione, M.; Oliviero, M.; Cocca, M.; Errico, M.E.; Gentile, G.; Avella, M.; Lavorgna, M.; Buonocore, G.G.; Verdolotti, L. Tuning of polyurethane foam mechanical and thermal properties using ball-milled cellulose. *Carbohydr. Polym.* **2020**, *231*, 115772. [[CrossRef](#)]
40. Mansikkamäki, P.; Lahtinen, M.; Rissanen, M. The conversion from cellulose I to cellulose II in NaOH mercerization performed in alcohol–water systems: An X-ray powder diffraction study. *Carbohydr. Polym.* **2007**, *68*, 35–43. [[CrossRef](#)]
41. Nam, S.; French, A.D.; Condon, B.D.; Conch, M. Segal crystallinity index revisited by the simulation of X-ray diffraction patterns of cotton cellulose I β and cellulose II. *Carbohydr. Polym.* **2016**, *135*, 1–9. [[CrossRef](#)]
42. Kathirselvam, M.; Kumaravel, A.; Arthanarieswaran, V.P.; Saravanakumar, S.S. Assessment of cellulose in bark fibers of *Thespesia populnea*: Influence of stem maturity on fiber characterization. *Carbohydr. Polym.* **2019**, *212*, 439–449. [[CrossRef](#)] [[PubMed](#)]
43. Saravanakumar, S.S.; Kumaravel, A.; Nagarajan, T.; Sudhakar, P.; Baskaran, R. Characterization of a novel natural cellulosic fiber from *Prosopis juliflora* bark. *Carbohydr. Polym.* **2013**, *92*, 1928–1933. [[CrossRef](#)]
44. Elanthikkal, S.; Gopalakrishnapanicker, U.; Varghese, S.; Guthrie, J.T. Cellulose microfibrils produced from banana plant wastes: Isolation and characterization. *Carbohydr. Polym.* **2010**, *80*, 852–859. [[CrossRef](#)]
45. Dou, J.; Karakoç, A.; Johansson, L.S.; Hietala, S.; Evtyugin, D.; Vuorinen, T. Mild alkaline separation of fiber bundles from eucalyptus bark and their composites with cellulose acetate butyrate. *Ind. Crops Prod.* **2021**, *165*, 113436. [[CrossRef](#)]
46. Dou, J.; Rissanen, M.; Ilina, P.; Mäkkylä, H.; Tammela, P.; Haslinger, S.; Vuorinen, T. Separation of fiber bundles from willow bark using sodium bicarbonate and their novel use in yarns for superior UV protection and antibacterial performance. *Ind. Crops Prod.* **2021**, *164*, 113387. [[CrossRef](#)]
47. Khalid, M.Y.; Imran, R.; Arif, Z.U.; Akram, N.; Arshad, H.; Rashid, A.A.; García Márquez, F.P. Developments in Chemical Treatments, Manufacturing Techniques and Potential Applications of Natural-Fibers-Based Biodegradable Composites. *Coatings* **2021**, *11*, 293. [[CrossRef](#)]
48. Baskaran, P.G.; Kathiresan, M.; Pandiarajan, P. Effect of alkali-treatment on structural, thermal, tensile properties of *dichrostachys cinerea* bark fiber and its composites. *J. Nat. Fibers* **2020**, *1*–17. [[CrossRef](#)]
49. Peng, Y.; Nair, S.S.; Chen, H.; Farnood, R.; Yan, N.; Cao, J. Application of different bark fractions in polypropylene composites: UV and thermal stability. *Polym. Compos.* **2020**, *41*, 2198–2209. [[CrossRef](#)]
50. Senthamaraikannan, P.; Saravanakumar, S.S.; Sanjay, M.R.; Jawaid, M.; Siengchin, S. Physico-chemical and thermal properties of untreated and treated *Acacia planifrons* bark fibers for composite reinforcement. *Mater. Lett.* **2019**, *240*, 221–224. [[CrossRef](#)]