EasyChair Preprint

№ 7866

# Extraction and Characterization of New Cellulosic Fibers from Moroccan Mallow Stem and Comparison with Other Naturel Fibers

Youssef El Omari, Bouazza Tbib, Mohamed Eddya and Khalil El-Hami



April 30, 2022

# Extraction and characterization of new cellulosic fibers from Moroccan mallow stem and comparison with other naturel fibers

Youssef El Omari, Bouazza Tbib, Mohammed Eddya, Aziz Soufi and Khalil El-Hami*

University of Mohammed V, Rabat, Scientific Institute, BP 703, Av. Ibn Batouta, Agdal, Rabat, Morocco

* Email : elhami_k@yahoo.com

**Abstract.** Natural fibers are one of the good alternative sources for replacing synthetic fiber and reinforcing polymer matrices. Because of their eco-friendly nature. This investigation deals with the extraction and characterization of new natural fibers from the Moroccan mallow plant stem. The physicochemical, thermal, and mechanical properties of Moroccan Mallow Fibers (MMFs) were reported and compared with other natural fibers for the first time. Fourier transform-infrared spectroscopy, X-ray fluorescence (XRF) spectroscopy and X-Ray Diffraction, confirmed that MMFs are rich in cellulose content. Some Moroccan Mallow Fibers were treated with 5% aqueous sodium hydroxide (NaOH) solution. Our object focused in the evolution of crystallinity index.

**Keywords:** Mallow, XRD, XRF, Lead, Rietveld refinement, FTIR. Crystallinity index

## 1 Introduction

In the last few decades, the scientists and industry are given more important to the research and development of composite materials based on organic-materials originally of renewable sources [1]. The natural fibers were replacing synthetic fibers because of their eco-friendly nature, non-toxicity, biodegradability and of their low cost and low density [2]. Cellulose is an

* No academic titles or descriptions of academic positions should be included in the addresses. The affiliations should consist of the author's institution, town/city, and country.

Organic compound with the formula $(C_6H_{10}O_5)_n$ , a polysaccharide consisting of a linear chain of several hundred to many thousands of $\beta(1,4)$ linked D-glucose units. In nature, cellulose chains are grouped together in order to form a compact microfibrils [3], this latter is stable by both inter-molecular and intra-molecular hydrogen bonding. Up to 100 glucon chains are grouped in order to form a long thin crystallites which considered as an elementary fibrils, the cellulose source influence to the width of the crystallites [4]. They are organized in groups to form microfibrils that are between 5 and 80 nm in diameter and a few micro meters for length [5]. The crystal structure of this nanofibers make the plant stem more strength [6]. Chemical and mechanical treatment of the fibers cause a modification on the fiber surface and the cells, which influence the properties of the fibers in composites [7, 8]. The mallow fiber was used in lot of application like making cordage in clothes, carpets, paper money... [9]. but it has not heretofore been of great utility as reinforcing component in industrial composite products [10]. The Mallow fibers can be used as reinforcement in polymer matrix composites as shown by physical and mechanical properties [11]. The properties physical and mechanical of Mallow are presented in the table 1[12].

**Table 1.** Physical and mechanical properties of the mallow fibers [12].

| Natural fiber | Diameter ($\mu$m) | Length (mm) | Specific masse (kg/m3) | Tensile strength (MPa) | Elongation at break (%) | Modulus of elasticity (GPa) |
|---|---|---|---|---|---|---|
| Mallow | 42.6 | 23.8 | 1374 | 160 | 5.2 | 17.4 |

Which prove that there is a possibility to use mallow fibers for polymer matrix composite reinforcement. Therefore, increased crystallinity of mallow fibers can improve mechanical properties of the polymer matrix composites by the mercerization treatment.

## 2 Experimental processes

Moroccan mallow stem fibers (IMFs) were collected from region of khouribga , in the winter weather Figure (1) show Indian mallow plant and its extracted fibers. Table 1 indicates the comparison of various physicochemical constituents and mechanical properties of IMFs with other cellulosic fibers [12].

### 2.1 Fabrication of composites

The products are sampling in winter weather and they are save on roof of the House from winter to summer weather, the colour of plants change and there are transform to fibers.

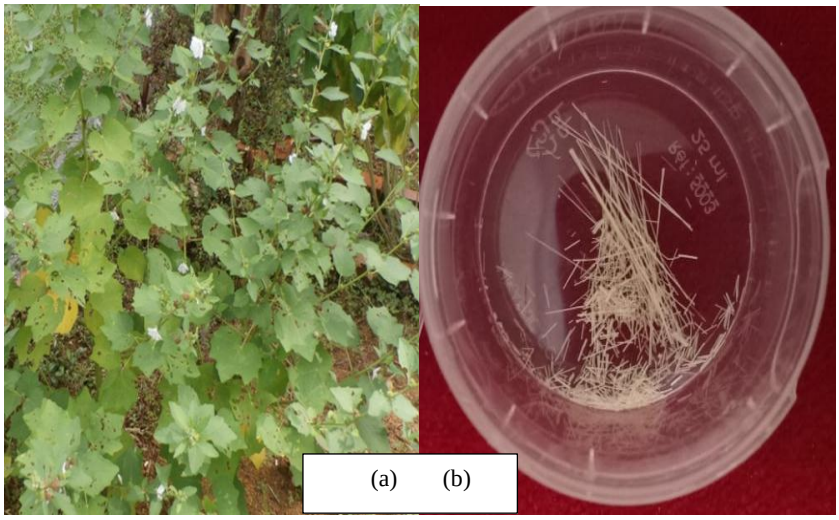


**Fig. 1.** (a) mallow plants (b) Extracted fiber collected from Indian mallow plant.

### 2.1 Extraction of Moroccan corn fibers

The plant corn was first immersed in water to allow microbial degradation for 14 days. The fibers were extracted from the Moroccan corn by retting method after degradation process. The extracted fibers were then dried in sunlight for a week to remove the moisture content. The fibers are cut into small pieces of length 4 mm and 3 mm in width, thus the samples have been prepared.

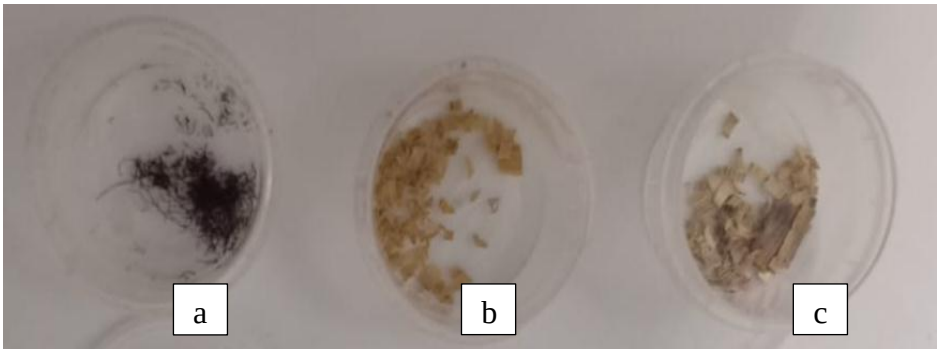


**Fig. 2.** (a) Silks of corn plant (b) tassel internode of plant and d ears of corn plant: Extracted fiber collected from Moroccan corn plant.

## 3 X-ray fluorescence

### 3.1 X-ray fluorescence (XRF) analysis

X-ray fluorescence (XRF) spectroscopy is one of the simplest and most widely used techniques for the non-destructive multielement analysis of materials. This technique has a remarkable progress and proved, its applicability in material science. In conventional XRF, the element detection sensitivities are largely limited to the μg g–1 (ppm) range; mainly because of the large spectral background produced by the Compton scattered X-rays from the specimen. The X-ray fluorescence spectrometer is capable of analysing elements in concentrations ranging from a high percentage up to the ppm level. In our case, the importance of the chemical element analysis is to determine the chemical composition of the cellulose fiber. The results show the existence of oligo elements and major elements. Oligo elements in ppm (parts per million) such as $Cr_2O_3$, $Fe_2O_3$, NiO …, and major elements such as 0.14% in $SiO_2$ ,

0.173% in P2O5 , 0.278% in Cl , 0.489% in CaO , 0.102% in SO3 , the components which are in ppm are the origin of the clay and these components are absorbed by the plant.

**Table.2** Chemical elements concentrations in mallow fiber samples

| | **Samples** | **Mallow** | |
|---|---|---|---|
| | Compound | Conc | Unit |
| **Major elements** | Na2O | 0 | Ppm |
| | MgO | 456,7 | Ppm |
| | Al2O3 | 591,8 | Ppm |
| | SiO2 | 0,14 | % |
| | P2O5 | 0,173 | % |
| | SO3 | 0,102 | % |
| | Cl | 0,278 | % |
| | K2O | 788,3 | Ppm |
| | CaO | 0,489 | % |
| | TiO2 | 290 | Ppm |
| | V2O5 | 10,2 | Ppm |
| **Oligo elements** | Cr2O3 | 8,2 | Ppm |
| | Fe2O3 | 119,7 | Ppm |
| | NiO | 173,8 | Ppm |
| | ZnO | 5,8 | Ppm |
| | Ag2O | 201,9 | Ppm |
| | SnO2 | 0,5 | Ppm |
| | Re | 0,4 | Ppm |

### 3.2 X-ray diffraction (XRD) analysis

The mallow fibre sample was characterised by X-Ray Diffraction analysis (XRD), by this technic we can evaluated the crystallinity of the sample by wide-angle X-ray diffraction analysis using a Bruker D8 ADVANCE Powder XRD apparatus who is the most used. XRD allow the characterization of the mallow fibre microstructure. The use of the diffraction spectrum to go back to the crystallinity index of the analysed substrate can be done using different analytical methods [13]. propose the correlation (equation 1) to estimate the crystal fraction (CrI) from the height of two peaks which corresponds to the crystalline and amorphous fractions baseline [13]..

$$CrI\ (\%) = \frac{I_{002} - I_{\mathrm{am}}}{I_{002}} \times 100 \qquad (1)$$

$I_{002}$: is the maximum intensity of the peak for 2θ=22° which corresponds to the crystalline fraction

$I_{am}$: is the minimum intensity of the peak for 2θ=15° which corresponds to the amorphous fraction as shown in the figure (3).

**Table.3** Crystallinity characteristics

| **Fibers** | **CrI (%)** |
|---|---|
| Untreated corn stem fibers | 58.2 |
| NaOH treated corn stem fibers | 65.7 |
| NaOH-silane treated corn stem fibers | 64.4 |
| Silane treated corn stem fibers | 69.7 |

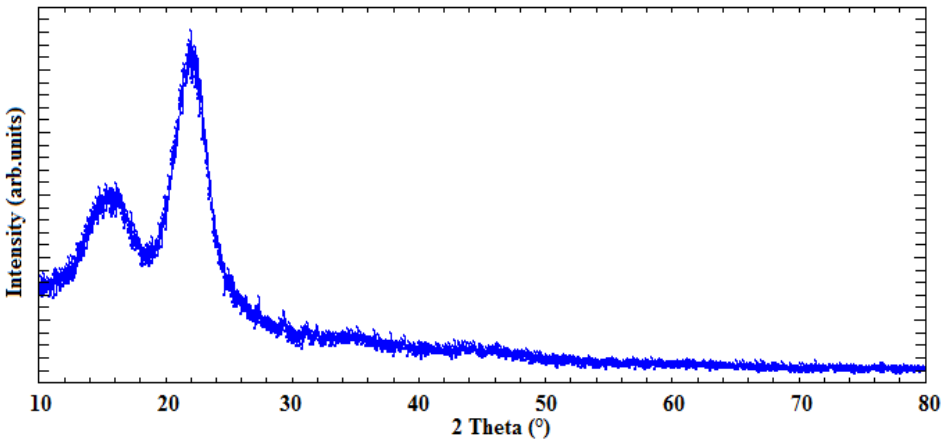


**Fig.3.** Spectrum of X Ray diffraction patterns of Mallow fiber

$$CrI\ (\%) = \frac{7.2 - 4.1}{7.2} \times 100$$

$$CrI = 43\%$$

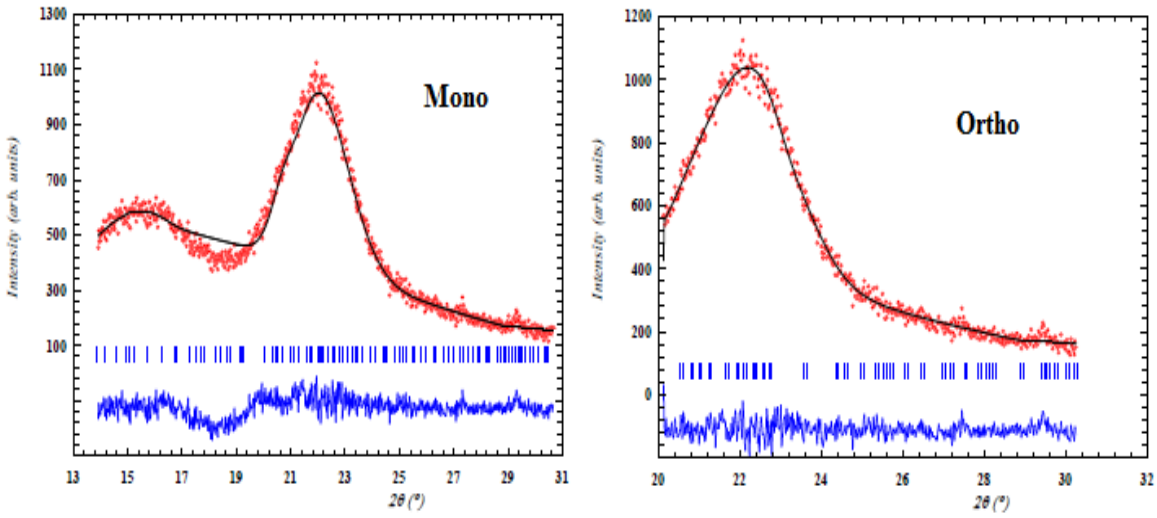


**Fig 4.** Profile matching by fullprof program for mallow plant

**Table. 4** Structural parameters mallow samples.

| Sample | Crystal System | Symmetry Group | Cell Parameters (A) | | | α (°) | β (°) | γ(°) | Cell Volume (A3) |
|---|---|---|---|---|---|---|---|---|---|
| | | | A | B | C | | | | |
| a | Orthorhombic | P m m m | 20.5008 | 12.9599 | 4.2672 | 90 | 90 | 90 | 1133.7345 |
| b | Tetragonal | P m m m | 7.8957 | 7.8957 | 14.7201 | 90 | 90 | 90 | 917.6901 |
| c | Orthorhombic | P m m m | 22.0875 | 6.5661 | 5.1432 | 90 | 90 | 90 | 745.9226 |
| d | Tetragonal | P m m m | 16.6261 | 16.6261 | 8.1613 | 90 | 90 | 90 | 2256.0208 |

### 3.3 Fourier Transformed Infra-Red (FTIR) spectral analysis

FT-IR spectra were used to examine the structure of Moroccan mallow fibers. For obtaining a spectra of each sample we used a Nicolet 560 spectrophotometer. The untreated and treated maize tassel fibers were grounded and mixed with KBr powders and the mixture was compressed into plates for FT-IR analysis. FTIR spectrometer was used to determine the presence of free functional groups on the MMF. Spectral outputs were obtained in the wavelength range of 4000–400 cm−1

using 32 scans and recorded in the transmittance mode as a function of wave number, the figure (5) represent the spectrum of FT-IR of Mallow plant.

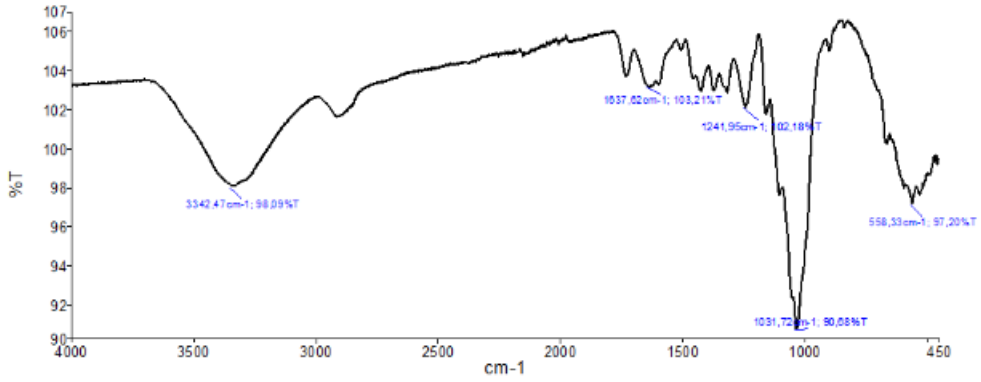


**Fig 5.** Spectrum of FT-IR for mallow plant

**Table. 5** Absorption bands for functional groups of cellulose and hemicellulose and lignin

| Fiber component | Wave number (cm-1) | Functional group | Compounds |
|---|---|---|---|
| Cellulose | 4,000–2,995 | OH | Acid, methanol |
| | 2,890 | H–C–H | Alkyl, aliphatic |
| | 1,640 | Fiber –OH | Adsorbed water |
| | 1,270–1,232 | C–O–C | Aryl-alkyl ether |
| | 1,170–1,082 | C–O– C | Pyranose ring skeletal |
| | 1,108 | OH | C- OH |
| Hemicellulose | 4,000–2,995 | OH | Acid, methanol |
| | 2,890 | H–C–H | Alkyl, aliphatic |
| | 1,765–1,715 | C=O | Ketone and carbonyl |
| | 1,108 | OH | C–OH |
| Lignin | 4,000–2,995 | OH | Acid, methanol |
| | 2,890 | H–C–H | Alkyl, aliphatic |
| | 1,730–1,700 | | Aromatic |
| | 1,632 | C=C | Benzene stretching ring |
| | 1,613 , 1,450 | C=C | Aromtic skeletal mode |
| | 1,430 | O–CH3 | Methoxyl– O–CH3 |
| | 1,270–1,232 | C–O–C | Aryl-alkyl ether |
| | 1,215 | C–O | Phenol |
| | 1,108 | OH | C–OH |
| | 700–900 | C–H | Aromatic hydrogen |

The FTIR spectra for mallow fiber is shown in figure (5). The characteristic peaks at approximately 3342 $cm^{-1}$ correspond to the $O-H$ bond stretching which indicate the presence of α-cellulose , the peak observed at 1730 $cm^{-1}$ for $C-O$ stretching of hemicellulose, the peak observed at 1637 cm-1 correspond to the $C=C$ bond which indicate the presence of lignin, the peak observed at 1241,95 cm-1 correspond to $C-O-C$ bond stretching from ether linkage of lignin, the peak observed at 1031,72 $cm^{-1}$ correspond to the aromatic $C-H$ stretching of lignin, the peak observed at 558,33 $cm^{-1}$ correspond to aliphatic $C-I$ stretching.

**Table.6** Peak positions and assignments of chemical groups in the untreated mallow fiber

| Samples | IR Band cm-1 | Transmittance % | Description |
|---|---|---|---|
| Mallow fiber | 3342,47 | 98,09 | $O-H$ bond stretching |
| | 1730 | 103,5 | $C-O$ stretching |
| | 1637,62 | 103,21 | $C=C$ bond |
| | 1241,95 | 102,18 | $C-O-C$ bond stretching |
| | 1031,72 | 90,68 | aromatic $C-H$ stretching |
| | 558,33 | 97,20 | aliphatic $C-I$ stretching |

In this figure (6), we are observed all peaks are de the same form between 450 $cm^{-1}$ and 4000cm-1; these quantified the character of cellulose fibers of plants in this subject, therefor the absorbance is presented in the figure (7), it can show the different between fibers of mallow and corn plants.

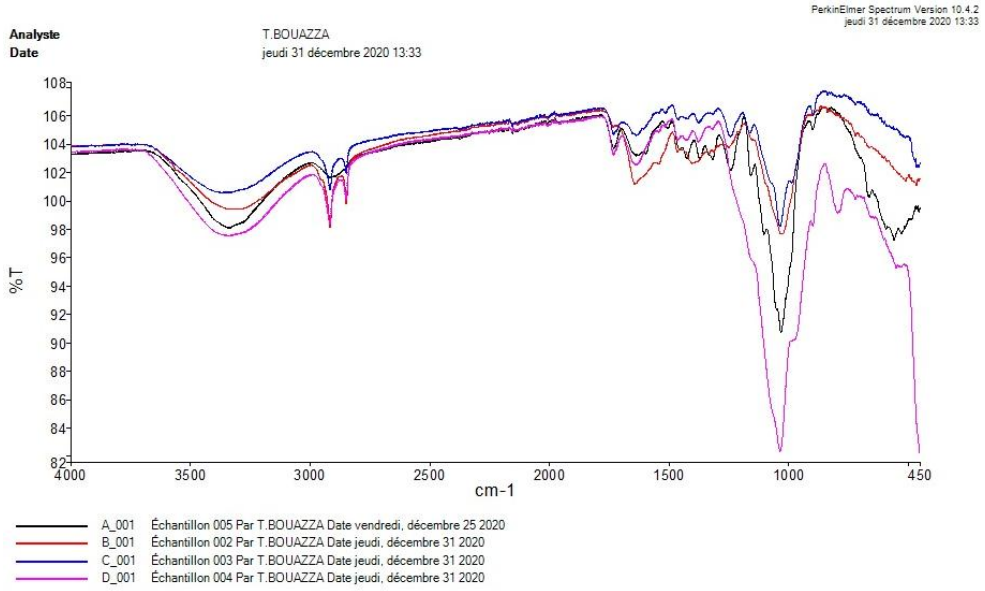


**Fig 6.** Spectra of transmittance of fibers mallow and corn plants

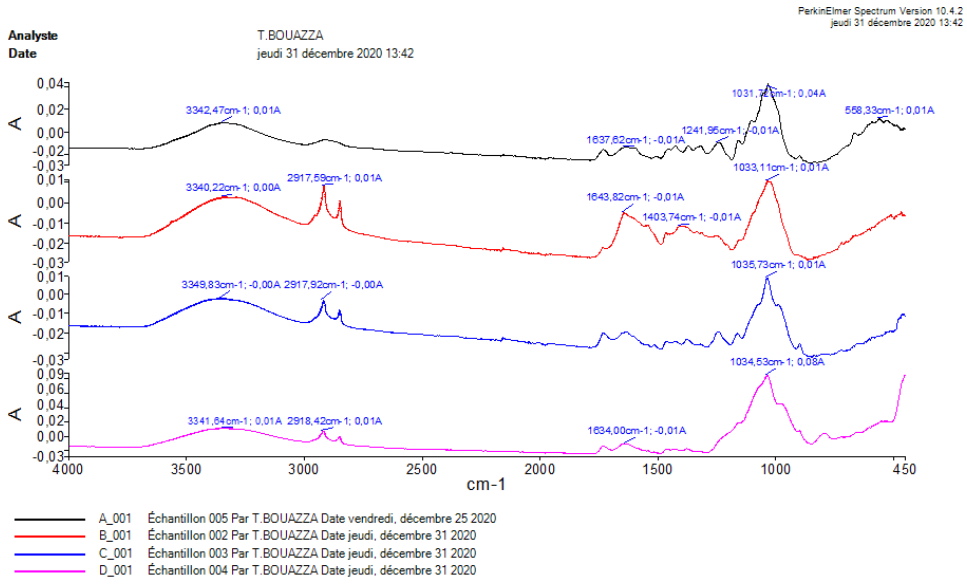


**Fig 7.** Spectra of absobance for fibers of mallow and corn plants

## 4 Conclusion

A residue from agricultural plant, such as the Moroccan Mallow fibers is an attractive substitute as a cellulose resource for a number of applications. This material is renewable and in great abundance in several regions of morocco. It is usually burned off or disposed of for ambient degradation. The result obtained by XRD analysis show that Moroccan mallow fibers had other elements, lignin and hemicellulose which decrease the crystallinity index. The results obtained from FT-IR analyses showed

that Moroccan mallow fibers had a low percentage of cellulose content because the presence of lignin and hemicellulose. This study advances the possibility of utilizing the Moroccan mallow fibers, which is renewable, and biodegradable, as reinforcement in bio composites. In the further works, we will further investigate the effect of alkali-treatment with different percentage of NaOH on the morphological, chemical, and thermal properties of the Moroccan mallow fibers.

## Acknowledgements

Special thanks to dear Abderahim HASRI from Hassan I University of Settat, Morocco for XRD and XRF apparatus.
Special thanks to dear Aziz Essoufi from Sultan Moulay Sliman university of Beni Mellal.

## References

1. Gurunathan, T., Mohanty, S., Nayak, S.K., A Review of the Recent Developments in Biocomposites Based on Natural Fibres and Their Application Perspectives, Composites: Part A (2015), doi: http://dx.doi.org/10.1016/j.compositesa.2015.06.007
2. Chand, Navin (2008). Tribology of Natural Fiber Polymer Composites || Natural fibers and their composites. , (), 1–58.doi:10.1533/9781845695057.1
3. Rongpipi, Sintu; Ye, Dan; Gomez, Enrique D.; Gomez, Esther W. (2019). Progress and Opportunities in the Characterization of Cellulose – An Important Regulator of Cell Wall Growth and Mechanics. Frontiers in Plant Science, 9(), 1894–.doi:10.3389/fpls.2018.01894
4. Harris, D. M., Corbin, K., Wang, T., Gutierrez, R., Bertolo, A. L., Petti, C., et al. (2012). Cellulose microfibril crystallinity is reduced by mutating C-terminal transmembrane region residues CESA1A903V and CESA3T942I of cellulose synthase. Proc. Natl. Acad. Sci. U.S.A. 109, 4098–4103. doi: 10.1073/pnas.1200352109
5. Müller, M., Hori, R., Itoh, T., and Sugiyama, J. (2002). X-ray microbeam and electron diffraction experiments on developing xylem cell walls. Biomacromolecules 3, 182–186. doi: 10.1021/bm015605h
6. Agarwal, U. P. (2014). 1064 nm FT-Raman spectroscopy for investigations of plant cell walls and other biomass materials. Front. Plant Sci. 5:490. doi: 10.3389/fpls.2014.00490
7. Donaldson, L., and Frankland, A. (2004). Ultrastructure of iodine treated wood. Holzforschung 58, 219–225. doi: 10.1515/HF.2004.034
8. Astley, O. M., Chanliaud, E., Donald, A. M., and Gidley, M. J. (2001). Structure of Acetobacter cellulose composites in the hydrated state. Int. J. Biol. Macromol. 29, 193–202. doi: 10.1016/S0141-8130(01)00167-2
9. Nieduszynski, I., and Preston, R. D. (1970). Crystallite size in natural cellulose. Nature 225, 273–274. doi: 10.1038/225273a0
10. Atalla, R. H., and Vanderhart, D. L. (1984). Native cellulose: a composite of two distinct crystalline forms. Science 223, 283–285. doi: 10.1126/science.223.4633.283
11. Ade, H., and Hitchcock, A. P. (2008). NEXAFS microscopy and resonant scattering: composition and orientation probed in real and reciprocal space. Polymer 49, 643–675. doi: 10.1016/j.polymer.2007.10.030
12. V. Vignesh;A.N. Balaji;N. Nagaprasad;Mavinkere Rangappa Sanjay;Anish Khan;Abdullah M. Asiri;Ghulam M. Ashraf;Suchart Siengchin; (2021). Indian mallow fiber reinforced polyester composites: mechanical and thermal properties . Journal of Materials Research and Technology, (), –.doi:10.1016/j.jmrt.2021.01.023
13. Elenga, R. G., Djemia, P., Tingaud, D., Chauveau, T., Maniongui, J. G., and Dirras, G. (2013). "Effects of alkali treatment on the microstructure, composition, and properties of the Raffia textilis fiber," BioRes. 8(2), 2934-2949