Physico-Chemical, Structural, mechanical and thermal behaviour of raw and steamed Borassus flabellifer (Bf) fiber

M. Abisha, S. Anne Kavitha, K.R. Jaya Sheeba, J. Jisha Francy, D.P. Dona, A. Sahaya Soosanna, A.J. Aruthra, A. Hylynn Rowena, S. Prema and *R. Krishna Priya

Department of Physics, Holy Cross college (Autonomous), Nagercoil – 629004.
Affiliated to Manonmaniam Sundaranar University, Tirunelveli - 627012.
*Corresponding Author - Email: phyprinag2017@gmail.com

ABSTRACT

Sustainable environment is an immediate require to safeguard the planet from being exposed to vulnerabilities. Bio composites can minimize synthetic acquisition since they are easily degradable. The reinforcement material deployed is a plant fiber according to which the entire behaviour of composites are decided. Raw and steam treated fibers of Borassus flabellifer (Bf) peels are characterised to see its efficiency as a reinforcement material. Lowered density of 0.91 g/cc is achieved after steam treatment. Chemical analysis computed the cellulose content to be 58.3%. Crystalline parameters are found from XRD analysis. SEM studies gave a clear discrimination between fiber sufaces before and after treatments. Steam has brought about 19% hike in the tensile values from 58 MPa to 70 MPa. FTIR spectroscopic studies found the vibrations associated with different functional groups. TG- DTA and DSC studies graphed the change in mass with rising temperatures as well as the heat flow rate on the fiber surface. Thermal stability upto 240 °C is registered during the degradation of cellulosic components with a maximum degradation peak at 410 °C and 440 °C for the raw and treated Bf. Elemental compositions of Carbon. Hydrogen, Nitrogen and Sulphur are calculated. All the above findings suggest that Bf fiber could serve as an effective reinforcement material for light weight composite applications.

***Keywords**: Sustainability, fiber composites, low density, thermal behaviour*

1. Introduction

Technological development fetches useful products along with waste which are mostly non biodegradable. Composite materials pairing natural fibers as reinforcement material could be the best solution to stabilize the production of synthetic waste. Lowered mass fraction and density are the key things that prioritise the role of natural fibers in reinforcements. Plant fibers contain crystalline and amorphous constituents. Cellulose which add up the crystallinity to the plant is responsible for tensile and thermal stability. Before considering it for any reinforcements, it is essential that the fiber must be properly brought out with lowered amorphous entity [1]. Various chemical treatments like alkalisation, benzoyl, carbonate, silane, permanganate treatments etc., can help on improvising the orderness of fibers [2]. They find applications in automotive parts, insulation equipment, shielding and construction materials. New bio-degradable material is a need of the hour for sustainability and the possibility to develop such material from waste is an added benefit to defend the resources that are scarcely

exist. The present work concerns on Borassus flabellifer (BP) to examine its Physical and chemical details along with characterisation studies such as X-ray diffraction analysis, morphological studies using SEM, Tensile testing, FTIR spectroscopic studies, thermogravimetric analysis and CHNS analysis.

2. Materials and methods

2.1 Steam treatment

Borassus flabellifer (BP) fiber locally called palmyra tuber are collected from Nagercoil, Kanniyakumari. The outer skin of BP tuber is generally treated as a waste and useful fibers can be separated from various layers. Selected fibers are washed in distilled water under room temperature and a part of it are subjected to steam treatment maintained at 110°C for 1 hour. Fibers separated using needle are kept in vacuum descicationg for 2 days. Steam treatment can improve the intimacy between the matrix and reinforced fiber when scrutinizing for composite making [3]. Figure 1. Shows the fibers extracted from palmyra sprout.

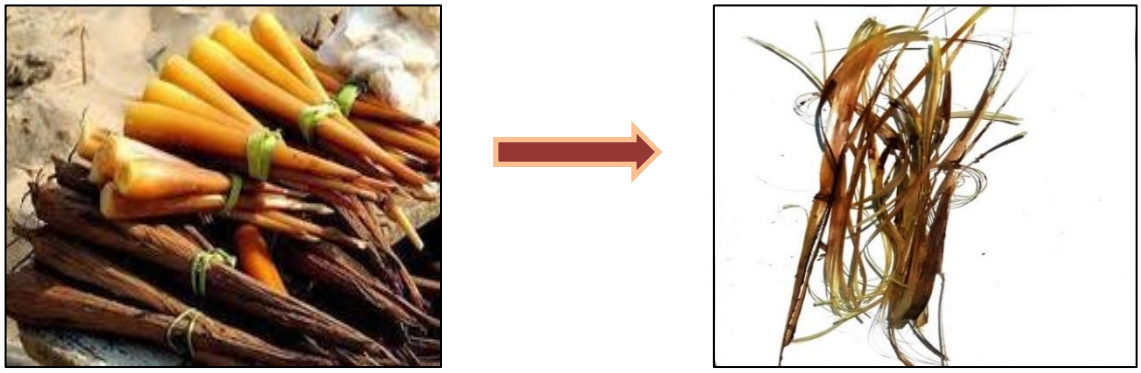

Fig. 1. Fibers from Borassus flabellifer (BF) tuber

2.2 Physical properties of Borassus flabellifer (Bf) fiber

A detailed study on the physical aspects of Bf fiber is essential in knowing and tapping the potentials of fiber to the fullest. Linear density is a measure of fineness of the fibre and density is analyzed with liquid Pycnometer method using the equation [4,5]

$$\rho_f = [(m_2-m_1) / [(m_3-m_1)-(m_4-m_2)]]\times\rho_t \quad (1)$$

Where, m_1 is the mass of pycnometer (kg), m_2 is the mass of the pycnometer filled with fibers (kg), m_3 is the mass of the pycnometer filled with toluene (kg) and m_4 is the mass of pycnometer filled with fibers and toluene (kg). The density of Bf fibers are much lower than Coccinia grandis stem (1.517 g/cc), Acacia leucophloea (1.385 g/cc), Thespesia populnea (1.412 g/cc) [6]. Steam treatment has scaled down the density to 0.9 g/cc, which is lower than synthetic

fibers and it can have a good scope on light weight applications [7, 8]. An average of 30 fibers are chosen to measure its diameter using an air-wedge arrangement and the physical parameters are tabulated in Table.1

Table 1. Physical parameters of Bf fiber

Parameters	Untreated fibers	Steam treated fibers
Length (cm)	15.03	15.26
Diameter (µm)	225	219.5
Aspect Ratio	668	695.22
Linear density (tex)	52.63	50.46
Density (g/cc)	1.31	0.9

3. Characterisation studies

3.1. Determination of Chemical composition

Presence of cellulose, lignin, hemicellulose and wax content in the fiber sample is revealed through chemical analysis. Chemical analysis testing was done from Chemical Testing Laboratory, SITRA, Coimbatore.

3.2. X-Ray Diffraction (XRD) Analysis

Crystalline nature of BP fibres was measured using Powder X-ray diffraction analysis using a Bruker AXS–D8 Advance Model diffractometer. 2θ values are recorded between 3˚and 80˚ under 40 kV and 35 mA. Crystallinity index is calculated using the Segal Emphrical formula [9].

$$CI = \frac{I_{200}-I_{am}}{I_{200}} * 100 \quad (2)$$

Where, I_{200}-maximum intensity of the crystalline diffraction peak, I_{am}-minimum intensity of an amorphous peak

The crystallite size was calculated using the equation Scherrer's equation

$$CS = \frac{K\lambda}{\beta_{200}\cos\theta} \quad (3)$$

Where K is Scherrer's constant, λ is Wavelength of the X ray (0.154 nm), β_{200} is the peak's full width at half maximum, θ is the Bragg angle.

3.3 Single fiber tensile testing

Single fiber tensile testing and elongation was computed using (Zwick/Roell) from Physical testing laboratory, SITRA, Coimbatore. All the tests were carried out at a temperature of 21˚C ± 1˚C with a relative humidity of about 65%.

3.4. Scanning Electron Microscopy (SEM)

Morphological features of fibers are studied using SEM studies. It was done by using a Jeal T220 scanning electron microscope, with the working voltage between of 5-20 kV.

3.5 FTIR Analysis

Vibrational bands of various functional groups of raw and steam treated fibres are found using the FTIR spectrometer (Model FTIR-8400S spectrum, SHIMADZU, Japan) in KBr matrix with a scan rate of 45 scans per minute.

3.6 Thermogravimetric analysis

Response of the fiber to a variety of reactions such as decomposition, degradation, adsorption, vaporization, oxidation, reduction etc., can be studied using thermogravimetric studies. Tg-dta and DSC analysis were taken using HITACHI-STA7300 in nitrogen atmosphere. A heating rate of 20°C /minute was monitored between 40-700°C.

3.7 CHNS Analyzer

Carbon Hydrogen Nitrogen Sulfur (CHNS) elemental analysis do a rapid determination of Carbon, hydrogen, nitrogen and sulphur quantities in the biofiber. The analysis is made using the model Elementer Vario EL III with the Precision >0.1% absorbance.

4. Results and Discussion

4.1 Chemical analysis

Presence of cellulosic and non-cellulosic constituents comprise the fibrillar arrangement. High cellulosic fraction with lower hemicellulose and lignin content enuciate a positive effect. The percent of wax is low around 0.35% which is a good impression since wax debonds fiber from matrix phase while making composites. Higher value of ash quantifies the improved cellulosic content in the fiber surface. Chemical constituents of Bf fiber is compared with other fibers [7, 10, 11] in Table 2.

Table 2. Chemical constituents in Bf fiber

Fibre	Cellulose (wt %)	Hemi cellulose (wt%)	Lignin (wt%)	Wax (wt %)	Pectin (wt%)	Moisture (wt%)	Ash (wt%)
Raw BP	58.35	25.83	19.90	0.35	7.62	9.33	3.90
Acacia leucophloea	68.09	13.6	17.73	0.55	-	8.83	0.08
Prosopis juliflora	61.65	16.14	17.11	0.61	-	9.48	5.2
Arundo donax	35.52	26.81	19.80	-	-	8.40	4.75

4.2 XRD Analysis

Natural fibers itself is considered to be semi-crystalline, due to the presence of cellulose, hemicellulose, pectin etc. These components are composed of various linkages of hydrogen and carbon [6]. A major crystalline peak was seen around 22 degrees in the steam treated Borassus fiber, along with the amorphous peak at around 18 degrees. In the case of untreated fibers, the CI value was found to be 54.48%. Unexpectedly, crystallinity index (CI) for the steam treated fiber is found to be 41%, which is less than the untreated fibre.

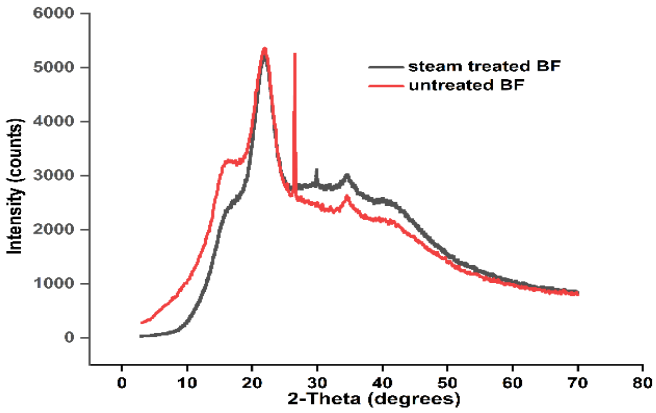

Fig. 2 XRD diffractogram of raw and treated BF fibers

The steam action might be strong enough to abandon the cellulosic components along with the amorphous material which resulted in a drop of cellulose after steam treatment. The values of CI and CS of the experimental fibers are comparable with other natural fibers like *Kigelia africana*, *Areca catechu* L, *Coccinia grandis* L, *Saharan aloevera* cactus leaves, *Furcraea foetida* etc., [12- 14]. Crystallite size of raw and steam bath treated Bf fibre was found to be 0.59 nm and 0.78 nm. XRD diffractogram and crystalline parameters of raw and steamed Bf fibers are shown in Figure 2 and Table 3.

Table 3. Crystalline, Tensile and CHNS values of raw and steam treated Bf fibers.

Sample	Crystallinity Index (%)	Crystallite size (nm)	Tensile strength (MPa)	N%	C%	S%	H%
Raw Bf	54.48	0.59	57.56	0.61	43.95	-	7.01
Steamed Bf	41	0.78	70.78	0.95	42.50	0.13	6.41

4.3 Single fiber Tensile Test

Fiber strength predominantly gears on number of things like maturity of the plant part, habitat, fibers chosen for testing and so on [15]. The tensile values of steam treated fiber show an increment of 70.78 MPa than the raw Bf fiber (57.56 MPa), without subjecting to any other chemical treatments. A better orientation of cellulose micro fibrils along the fiber axis play a role in the fiber strength. An upsurge of 19% was seen in the steamed fibers. Smaller size, enhanced mechanical strength and lowered amorphous impurities influence an advanced adhesion between the matrix and reinforced material [16]

4.4 Scanning Electron Microscopy

Surface morphologies are diligent while considering fibers to be paired with matrix phase. SEM photographs of raw and steam treated fibers are displayed in figure 3: a,b,c,d and figure 4: a,b,c,d. Fibrils are seen to be clouded with impurities, wax, hemicelluloses and lignin on the surface of untreated Bf fiber [17]. Also a number of pores opening into long ridge area is visible in figure 3a. Large vacuoles created by impurities are marked in figure. 3d. However, deformities are largely replaced with more ordered and regular arrangements after steam explosion.

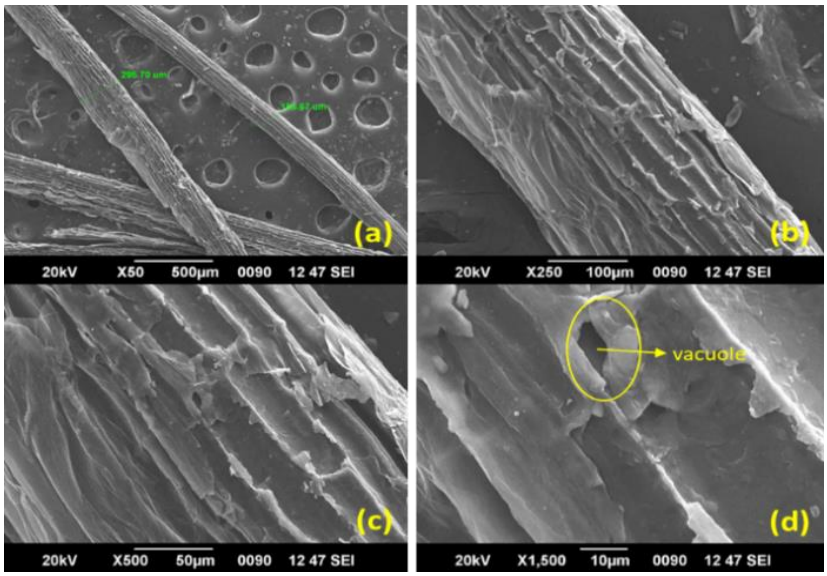

Fig. 3 a,b,c,d: SEM image of untreated BF fibers

It is predicted with the fact that steam treatment has broken the bonds between the cellular constituents of fiber and hence the disorganised hemicellulose might be cleaved off of the fiber surface with magnified, long fissures are visible and it might add up a hold on fiber towards the matrix arrangement [15].

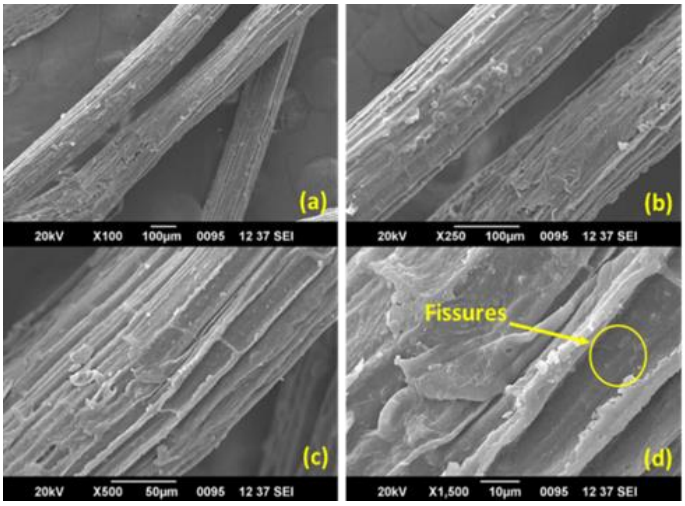

Fig. 4. a, b, c, d: SEM images of steam treated BF fibers

4.5 FTIR Spectroscopic studies

The use of Fourier transform infrared (FTIR) spectroscopy has been considered to be one of the most effective techniques to study the chemical and surface chemistry of biofibers. Wavenumbers at 3428 cm^{-1} and 3435 cm^{-1} in the raw and steam treated fibers indicated the presence of O-H stretching of hydrogen bonded hydroxyl groups in α-cellulose and /or in hemicelluloses. Strong peaks observed at 1643 cm^{-1} in both fibers attribute to C=O stretching vibration of keto carboxylic acid in lignin. Absorption peaks at 2920 cm^{-1} is due to the C-H stretching vibration of α-cellulose and hemicellulose [18]. Vibration of fibers at 1600 cm^{-1} is due to the stretching of C=C groups present in the lignin and medium absorption peaks visible around 1414.34 cm^{-1} is due to CH_2 wagging [19]. Streaming has not inculcated a wide vibrational variations in Bf fibers. FTIR graphs of raw and treated fibers are shown in Fig. 4.

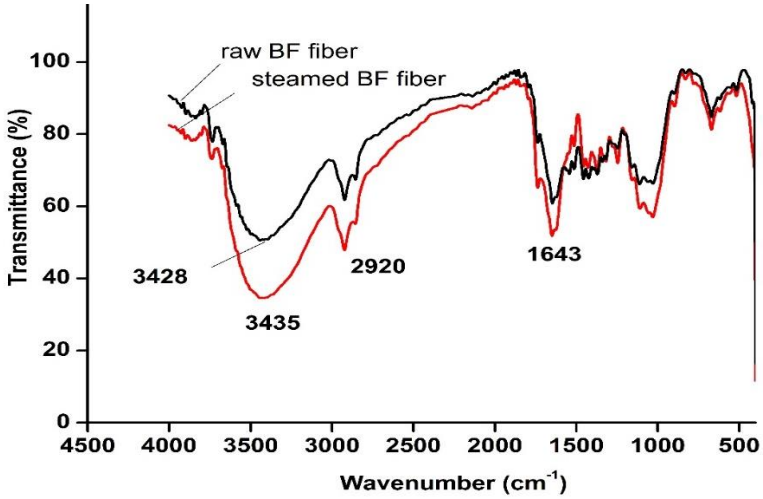

Fig. 4. FTIR spectrum of raw and steam treated Bf fiber

4.6 Thermogravimetric analysis

Heat resistance is very much needed for making composites [20]. TG plot shows that initial mass loss of 12% in the raw fiber at 110°C, is due to the removal of moisture. [21, 22]. Apparent loss of weight between 230°C- 360°C is recorded due to the degradation of cellulose. On rising the temperature to 325℃, huge degradation was initiated due to the decomposition of hemicelluloses and glycosidic linkages of celluloses. Nearly 51% of weight got reduced in this stage. Beyond 400°C, lignin mass has got out of the fiber. Steam treated fibers also follow the same pattern with a slight variation of 42% mass loss between 250°C- 320°C. Clinical degradation of raw fiber was noticed at 230°C and eventually it has manifested to 240°C during the steam treatment. Figure 5. explains the TG-dta and DSC curves for the raw and steam treated Bf fibers.

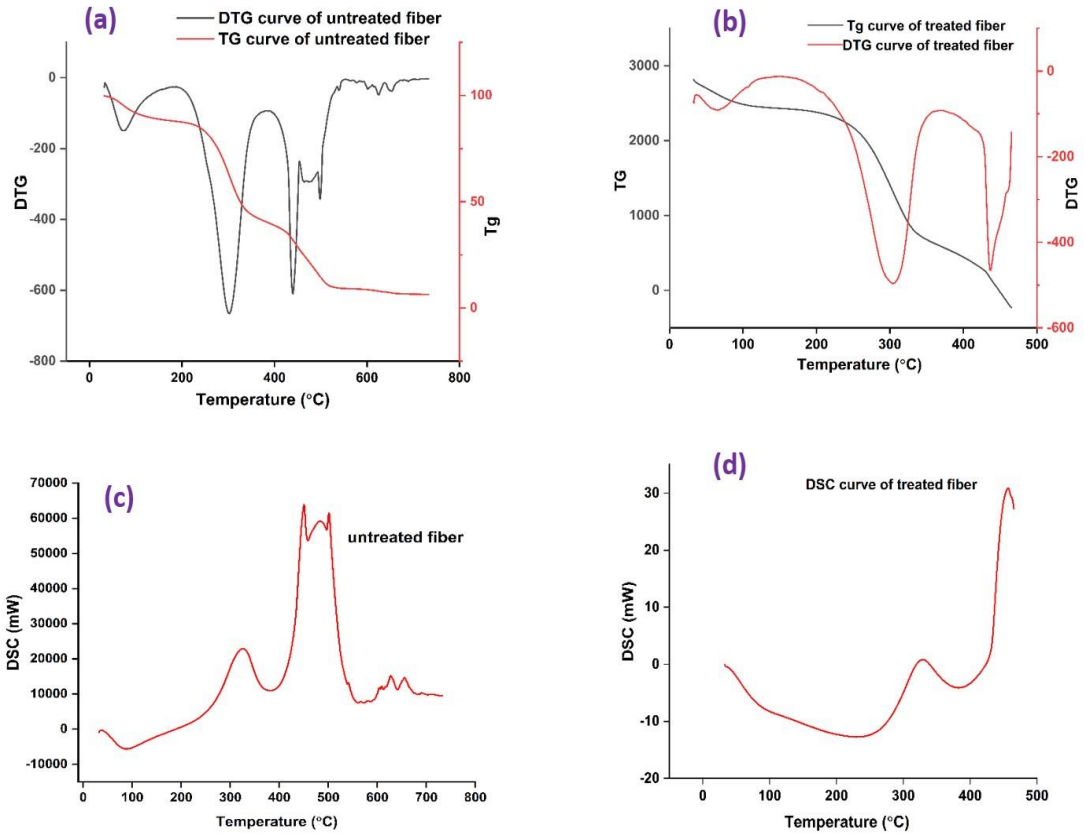

Fig. 5a. Tg-Dtg curve of untreated Bf 5b. Tg-Dtg curve of steam treated Bf
5c. DSC curve of untreated Bf 5d. DSC curve of steam treated Bf

DTG curves marks the maximum degradation temperature of raw and steam treated fibers at 302°C and 304°C, which is very much greater than most cellulosic fibers. Steam treatment has the capacity to bring variation and extend the thermal properties to a considerable level. Another degradation peaks at 420.8°C and 461.3°C in raw and steam treated fibers respectively, are possibly due to the disintegration of lignin and pectin. Similar peaks had

appeared in Coccinia grandis stem fibers [23] and pigeon pea plant fibers [24]. Enthalpy change connected with the fiber is enumerated in DSC curve. An endothermic peak appeared at 410°C in the raw fiber and preceded to 440°C in the treated fiber, clearly indicates a wide constructive discrepancy in the later one. TG data summarises that steam inclusioned Bf fibers can be opted for thermal applications considering the thermal standby temperature not more than 240°C.

4.7 CHNS Analyzer

Raw and steam treatead Bf fiber samples are combusted to generate compound gases of Carbon, Hydrogen, Nitrogen and Sulfur. Purity is found along with this analysis. Presence of sulfur was detected in the steam treated sample. Table 3. shows the CHNS weight percent of fibre sample.

5. Conclusion

The suitability of Borassus flabellifer (Bf) fiber to be consumed for green composites were analyzed and the following conclusions were put forward. Density of steamed fiber show a massive change to 0.9 g/cc from 1.31 g/cc. Chemical composition of fibers witnessed a higher cellulose (58.35%) and ash (3.90%) content adjoined by lowered hemicelluloses (25.83%). Although the Crystallinity Index in the steam treated fiber has been declined, mechanical and thermal behaviour marked greater improvements. This is because the action of steam has cleaved the celluloses along with the amorphous entities. Fibrillar arrangement was more ordered in the steam treated fiber (70.78 MPa) which is 19% more than the raw Bf. FTIR spectroscopic assignments register a slight vibrational variation with the steamed fiber.

The SEM images neatly portray the presence and absence of components on the fiber surface. Tg-dta and DSC plot show the mass loss of cellulosic components and adds that the maximum temperature upto which the fibers can stay active was marked at 240°C, with a maximum degradation peak on the steamed Bf at 461°C. CHNS analysis quantified the present of the elements. The findings give a positive way to introduce the fiber as reinforcement material in composite making. Overall study on Borassus flabellifer (Bf) indicated an augmented results and there is no less probability that different chemical treatments could enhance the properties for optimised composite applications.

References

1. Saravanakumar A., Senthilkumar A., Saravanakumar S.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. Charact. 2018; 23 (6): 529 - 536.

2. Saravanakumar S.S., Kumaravel A., Nagarajan T., Moorthy I.G. Effect of chemical treatments on physicochemical properties of *Prosopis juliflora* fibers. Int. J. Polym. Anal. Charact. 2014; 19 (5): 383 - 390.
3. Challabi A. J. H., Chieng B. W., Ibrahim N. A., Ariffin H., Zainuddin N. Effect of Superheated Steam Treatment on the Mechanical Properties and Dimensional Stability of PALF/PLA Biocomposite. *Polymers (Basel)*. 2019 11(3). doi: 10.3390/polym11030482.
4. Mohanty A. K., Misra M., Hinrichsen G. Biofibres, biodegradable polymers and biocomposites: An overview. Macromol Mater Eng. 2000; 276 (1): 1 - 24. https://doi.org/10.1002/(SICI)1439-2054(20000301).
5. Trunoga M., Zhonga W., Boykob S., Alcock M. A comparative study on natural fibre density measurement. J. Text. Inst. 2009; 100 (6): 525 - 529.
6. 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. Bio. Macromol. 2019; 129: 396 - 406. https://doi.org/10.1016/j.ijbiomac.2019.02.044.
7. 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 (2): 1928 - 1933.
8. Indran S., Raj R.E. Characterization of new natural cellulosic fiber from *Cissus quadrangularis* stem. Carbohydr. Polym. 2015; 117: 392 - 399.
9. Segal L. G., Creely J.J., Martin Jr A.E., Conrad C.M. An empirical method for estimating the degree of crystallinity of native cellulose using the X-ray diffractometer. Text. Res. J. 1959; 29 (10): 786 - 794. https://doi.org/10.1177/004051755902901003.
10. Arthanarieswaran V.P., Kumaravel A., Saravanakumar S.S. Characterization of new natural cellulosic fiber from *Acacia leucophloea* bark. Int. J. Polym. Anal. Charact. 2015; 20 (4): 367 - 376.
11. Wissam Bessa, Djalal Trache, Mehdi Derradji, Houda Ambar, Ahmed Fouzi Tarchoun, Mokhtar Benziane, Bendiba Guedouar, Characterization of raw and treated *Arundo donax* L. cellulosic fibers and their effect on the curing kinetics of bisphenol A-based benzoxazine. International J.Bio.Macromol. 2020; 164: 2931 - 2943. https://doi.org/10.1016/j.ijbiomac.2020.08.179.
12. Siva R.T., Valarmathi N., Palanikumar K., Antony V. Samrot. Study on a novel natural cellulosic fiber from *Kigelia africana* fruit: Characterization and analysis. Carbohydr. Polym. 2020; 244: 116494.

13. Garette Jebadurai S., Edwin Raj R., Sreenivasan V.S., Binoj J.S. Comprehensive Characterisation of natural cellulosic fiber from *Coccinia grandis* stem. Carbohydr. Polym. 2018. https://doi.org/10.1016/j.carbpol.2018.12.027
14. Manimaran P., Senthamaraikannan P., Sanjay M.R., Marichelvam M.K., Jawaid M. Study on Characterization of *Furcrea Foetida* new natural Fiber as Composite Reinforcement for light weight applications. Carbohydr. Polym. 2018; 181: 650 - 658.
15. Seno Darmanto, Heru S. B., Rochardjo, Jamasri. Effects of alkali and steaming on mechanical properties of snake fruit (Salacca) fiber. AIP Conference Proceedings. 2017; 1788, 030060. https://doi.org/10.1063/1.4968313
16. Hou X., Sun F., Yan D., Xu H., Dong Z., Li Q., Yang Y. Preparation of lightweight polypropylene composites reinforced by cotton stalk fibers from combined steam flash-explosion and alkaline treatment**.** J. Clean. Prod. 2014; 83**:** 454 - 462.
17. Amutha K., Sudha A., Saravanan D. Characterization of Natural Fibers Extracted from Banana Inflorescence Bracts. J. Nat. Fibers, 2020; 19: 872 - 881. https://doi.org/10.1080/15440478.2020.1764437.
18. Mabrouk Maache., Abderrezak Bezazi., Salah Amroune., Fabrizio scarpa. Characterization of a novel natural cellulosic fiber from Juncus effuses L. Carbohydr. Polym. 2017; 171: 163 - 172.
19. 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 (2): 1928 - 1933. https://doi.org/10.1016/j.carbpol.2012.11.064.
20. Van de velde K., Kiekens P. Thermal Degradation of Flax: The Determination of Kinetic Parameters with Thermogravimetric Analysis. J. Appl. Polym. Sci. 2002; 83 (12): 2634 - 2643. https://doi.org/10.1002/app.10229.
21. Rajan B. S., Balaji M. A. S., Saravanakumar S. S. Effect of chemical treatment and fiber loading on physico-mechanical properties of *Prosopis juliflora* fiber reinforced hybrid friction composite. Mater. Res. Express. 2018; 6 (3): 035302. https://doi.org/10.1088/2053-1591/aaf3cf.
22. Vijay R., Singaravelu D. L., Vinod A., Sanjay M. R., Siengchin S., Jawaid M., Khan A., Parameswaranpillai J. Characterization of raw and alkali treated new natural cellulosic fibers from *Tridax procumbens*. Int. J. Bio. Macromol. 2019; 125: 99 - 108. https://doi.org/10.1016/j.ijbiomac.2018.12.056.

23. Garette Jebadurai S., Edwin Raj R., Sreenivasan V.S., Binoj J.S. Comprehensive Characterisation of natural cellulosic fiber from *Coccinia grandis* stem. Carbohydr. Polym. 2018; https://doi.org/10.1016/j.carbpol.2018.12.027
24. Nijandhan Kulandaivel R., Muralikannan., Kalyana Sundaram S. Extraction and characterisation of novel natural cellulosic fibers from pigeon pea plant. J. Nat. Fibers. 2020; 17: 769 - 779, https://doi.org/10.1080/15440478.2018.1534184.