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Characterization of New Natural Cellulosic Fibers – A Comprehensive Review

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Abstract. The world is in the need of more eco-friendly materials; therefore researchers around the globe focus on developing new materials that would improve the environmental quality of products. This need for new green materials has led to the utilization of composites made from raw natural fibers as they are low cost and have better mechanical properties and require low production energy consumption. Natural fibers are one of the effective substitutes for switching artificial fiber and concentrating to reinforce polymer matrices due to their decomposable characteristic. This study explains the various tests to be carried out such as mechanical property analysis, chemical composition analysis, surface morphology analysis, thermal stability analysis, etc., in order to characterize a new natural fiber. The impact of treating natural fibers with the suitable chemicals on the desirable properties is elaborately discussed. The testing procedures and the various significant points given by the researchers in this field are presented in detail. This work will surely be helpful for the upcoming researchers those who would like to carry out the research work in natural fiber composites.

1. Introduction

Researchers are doing variety of investigations on different types of application areas of composite structures for various applications. For performing the desirable analysis on the composites structures to ensure their suitability, it is very essential to have the details of the base properties of the constituents of the composites. Hence, characterization study holds most significant role while we are introducing new natural fibers to this field. Standard testing procedures are being followed for carrying out the property analysis of natural fibers. The consolidated details obtained from the extensive literature study are presented in this work. P. G. Baskaran *et al.*, have analysed the mechanical properties, chemical composition, crystalline characteristics and thermal degradation of Dichrostachys Cinerea Fibre (DCFs). The mechanical properties of the DCFs proved that they are the best replacement for manmade synthetic fibres in polymer matrix composites [1]. R. Gopinath *et al.*, have investigated the chemical composition, crystalline characteristics, thermal degradation, and mechanical properties of Sida Rhombifolia Fibre (SRFs). The test results revealed the presence of high cellulose content (75.09 wt. %,) in SRFs, considerably more significant than that of other cellulosic fibres [2]. N. Rajesh Jesudoss Hynes *et al.*, have described the chemical configuration, crystalline characteristic, and thermal stability of the fiber obtained from Heteropogon contortus Plant (HCFs). The reasonably higher percentage of cellulose (64.875%) in HCFs can offer higher strength and the lesser density (602 kg/m³) which make HCFs as fit for low weight composite applications [3]. S. Indran *et al.*,



reported the characterisation details of *Cissus Quadrangularis* Stem Fibre (CQSF). The density of CQSF is significantly lower than that of the popular glass fibre, carbon fibre, etc. and also gives 20% better specific strength. The tensile strength and Young's moduli are 4203 ± 1276 MPa and 131 ± 37 GPa which is significantly higher than any other natural fibres ever investigated and also on par with the widely used synthetic fibres [4]. J. Jayaramudu *et al.*, in their research work stated the properties of the uniaxial natural fabric *G. tilifolia*. The SEM analysis manifested the morphology of the fiber contains uniaxial roughly parallel surfaces present in the fiber [5].

Mabrouk Maache *et al.*, have investigated the properties of a new lignocellulosic fibre extracted from *Juncus Effusus* L (JE). The study of the surface morphology by SEM revealed that the cross-section of the JE fibre bundle has a cellular shape similar to other fibres [6]. C.E. Maepa *et al.*, have described the extraction and characterisation of Tassel Fibre. The authors stated that the residue from maize (tassel fibres) is an attractive substitute as a cellulose resource for some applications [7]. M. V. Maheshwaran *et al.*, have reported the chemical, structural, thermal, and tensile properties of the *Epipremnum Aureum* stem (EAFs). It has been shown that lignin content has little effect on biological attack; and relatively lesser density of the EAFs may enable them for light-weight applications [8]. M. Prithiviraj *et al.*, have studied the mechanical properties of the fibre, chemical composition, crystalline characteristics and thermal degradation of *Perotis Indica* Plant (PIFs). The obtained properties of the PIFs ensured that they are suitable as a good substitute for the conventional materials in the industries [9]. Dhana Lakshmi Sampath Kumar *et al.*, have reported that the Chemical treatment of natural lingo-cellulosic fibres would remove the impurities like pectin, fat and lignin present in the fibres, which may result in rough surfaces in turn the better ability of bonding of fiber with the matrix while preparing composite structures with the help of them. From the literature survey, we found that the natural fibres will be more important in future because of its availability, recyclability and eco-friendly nature. Various natural cellulosic fibres have been investigated which impart different desirable properties.

2. Characterization study

For the characterization study, initially a new natural fiber needs to be identified from a plant. Then, it should be separated from the plant by following the commonly used step by step procedure as described below [11, 12]:

- i. The part of the plant (Ex.: stem, root, etc.) from which the fiber is going to be separated are collected at required level.
- ii. The collected parts of the plant are needed to be immersed in ordinary / distilled water for about two to three weeks for getting microbial degradation.
- iii. Then the fiber is extracted from the stem / root with the help of a metallic comb.
- iv. After that, the fiber is washed in running water in order to remove the unwanted particles present in the fiber surface.
- v. Followed by, the fiber is dried in oven at nominal temperature to remove the moisture content.

2.1. Physical Analysis

Physical analysis is normally performed to predict the density and nominal geometrical details of the chosen fiber. Density is normally measured by using gas pycnometer set up and

diameter is measured by means of micrometer. The procedure to measure the density value is as follows [5]:

- The gas pycnometer operates by detecting the mass difference between the known and unknown density materials.
- Toluene is normally chosen as the known density (867 kg/m^3) liquid for the density measurement of fibers.
- A small amount of the fiber needed to be tested in chopped form and a little amount of toluene are taken for the analysis. The mass of each are measured.
- Distilled water is also sometimes used as the known density liquid [6].
- The mass values are to be noted for the empty pycnometer, chopped fiber only, toluene only and toluene + chopped fiber.
- Then, the density of the fiber can be measured by means of the following formula:

$$\rho_f = \left\{ \frac{m_2 - m_1}{(m_3 - m_1)(m_4 - m_2)} \right\} \rho_t$$

Where, m_1 -Mass of empty pycnometer

m_2 - mass of pycnometer with chopped fibre

m_3 - mass of pycnometer filled with toluene

m_4 - mass of pycnometer filled with chopped fibre and toluene solution

ρ_f – density of fibre

ρ_t – density of toluene

The measured density values of various natural fibers are presented in table 1.

Table 1. Density values of various natural fibers [1, 3, 6, and 7]

S.No.	Name of the fiber	Density (kg/m^3)
1.	Dichrostachys cinerea	1240
2.	Prosopis juliflora	580
3.	Acacia Leucophloea	1385
4.	Nettle	1510
5.	Cyperus pangorei	1102
6.	Lygeum Spartum	1499.7
7.	Sida Rhobifolia stem	1320.7
8.	Jute	1460
9.	Flax	1500
10.	Ramie	1500
11.	Hemp	1480
12.	Kenaf	1400
13.	Sisal	1500
14.	Cotton	1600
15.	Heteropogan Contortus	602
16.	Acacia planiflora	660
17.	Coir	1200
18.	Pineapple leaf	1400
19.	Banana	1350
20.	Palmyrah	1090

21.	Bamboo	910
22.	Bagasee	1200
23.	Sea grass	1500

2.2. Chemical Analysis

Chemical composition analysis is performed to identify the percentage composition of cellulose contents, hemicellulose contents, lignin contents, moisture, wax and ash contents present in the fiber. Since the percentage existence of above said compositions decides the strength and bonding ability of the fiber materials, this study is significant in characterization work [7, 13, 14].

Determination of cellulose content – The amount of cellulose present in the fiber is found out by Kurschner's and Hanack's method. This method is based on the insolubility of cellulose in water and its resistance to the action of dilute acids and bases. The crushed fibre sample was degraded with a mixture of nitric acid and acetic acid and boiled in an apparatus that contained a condenser. The solution is then filtered through a Büchner funnel. Then, the filter paper containing an insoluble residue is dried in an oven and measured which is the direct measure of the cellulose content [6, 15].

Determination of lignin content - Insoluble solution of the lignin content is determined based on Klason lignin of APPITA P11s-78 method. The samples of crushed fibres (300 mg) are hydrolysed with 72% of sulfuric acid in an ultrasonic bath for 1 hour at a controlled temperature of 30 °C. The treated sample is mixed with a solvent solution of methylene chloride (CH₂Cl₂) and placed in an autoclave at 125 °C for one hour and dried. Then, the sample was cooled; the lignin was filtered and measured [5, 16].

Determination of ash content – The ash measurement test is carried out based on ASTM E1755-61 standard. The amount of ash present in the fiber is predicted based on the difference in weight between the burned and unburned fibers [4, 17].

Determination of moisture content – The amount of moisture content is measured by means of the standard weight loss method. Or that, the test sample is heated until there is no weight difference can be noted. At that condition, the weight difference of the fiber is noted which is the amount of moisture content present in the fiber [3, 18].

Determination of hemicellulose content – This test is carried out based on the NFT standard 12-008 [2, 3, 4].

Determination of wax content – This measurement is based on the Conrad method [3, 4].

For achieving better bonding ability and mechanical strength, the percentage of cellulose content should be high, hemicellulose content should be low, lignin content should be nominal, moisture, ash and wax contents should be low [1, 2, 19].

The chemical compositions of various investigated natural fibers are presented in table 2.

Table 2. Chemical composition of few natural fibers [1, 2, 4, 6, 8]

S.No.	Name of the fiber	% of Cellulose	% of Hemicellulose	% of Lignin	% of moisture	% of Ash	% of Wax
1.	Dichrostachys Cinerea	72.4	13.08	16.89	9.82	3.97	0.57
2.	Prosopis Juliflora	61.65	16.14	17.11	9.48	5.2	0.61
3.	Acacia Leucophloea	68.09	13.6	17.73	8.83	0.08	0.55
4.	Sida Rhombifolia	75.09	15.43	7.48	12.02	4.07	0.49

	Stem						
5.	Acacia leucophloea	68.09	13.6	17.73	8.83	0.08	0.55
6.	Jute	72	13	13	12.6	1	0.5
7.	Flax	81	14	3	10	--	1.7
8.	Hemp	74	18	4	10.8	--	2.3
9.	Kenaf	53.14	14.33	8.18	9	3.5	0.8
10.	Sisal	68	13	11.5	17	--	2
11.	Heteropogon Contortus	64.87	19.34	13.56	7.4	--	0.22
12.	Acacia Planifrons	73.1	9.41	12.04	8.21	4.06	0.57
13.	Acacia Arabica	68.10	9.36	16.86	--	--	0.49
14.	Cissus Quadrangularis Stem	82.73	7.96	11.27	6.6	--	0.18
15.	Cissus Quadrangularis root	77.17	11.02	10.45	7.3	--	0.14
16.	Agave	68.42	4.85	4.85	7.69	--	0.26
17.	Epipremnum Aureum	66.34	13.42	14.01	7.41	4.61	0.37
18.	Perotisindica	68.4	15.7	8.35	9.54	4.32	0.32
19.	Red Banana Peduncle	72.90	11.01	15.99	9.36	2.79	0.32
20.	Furcraea Foetida	68.35	11.46	12.32	5.43	6.53	0.24

2.3. Thermogravimetric Analysis

Thermogravimetric analysis is carried out on fibers in order to find out their thermal stability in turn to identify the suitable high temperature applications. The test results will provide the temperature points at which the various constituents such as cellulose, hemicellulose, lignin, moisture etc., present in the fiber getting degraded. The analysis is performed in Jupiter simultaneous thermal analyzer in which an alumina crucible will be there to keep the sample. About 12 mg amount of sample fiber to be tested is taken in the crucible and it is subjected to a varying temperature condition from 15 to 600⁰ Celsius under the atmosphere of Nitrogen gas. The readings are taken for the heating rate of 10⁰C/minute [20, 21, 22].

The thermal stability of various constituents of different natural fibers is illustrated in table 3.

Table 3. Thermal stability values of the constituents of few natural fibers [1, 3, 5, 7]

S.No.	Name of the fiber	Moisture (°C)	Hemicellulose (°C)	Cellulose (°C)	Wax (°C)
1.	Dichrostachys Cinerea	164.4	284.3	359.3	495.3
2.	Lygeum Spartum	110	220 – 307	338.7	450 – 550
3.	Sida Rhombifolia stem	73.5	250 – 375	328.2	433.3
4.	Heteropogon Contortus	90.1	240 – 400	337.7	500 – 650
5.	Cissus Quadrangularis stem	89	294	342	476 – 695
6.	Juncus Effusus	30 – 110	220 – 360	300	400 – 500

7.	<i>Epipremnum Aureum</i>	110	240	240 – 380	380 – 600
8.	<i>Perotis Indica</i>	88.4	277.5	339.1	400 – 500
9.	<i>Areca</i>	241.5	241.5 – 297	297 – 353	499.4
10.	<i>Cissus Quadrangularis</i> root	89	230 – 330	328.9	481 – 696
11.	Red Banana Penduncle	120	200 – 300	353	353 – 500
12.	<i>Areca Palm Leaf Stalk</i>	60 – 110	200 – 350	365	37 – 450

2.4. Fourier Transform Infrared (FTIR) Analysis

FTIR analysis is carried out to discover the chemical functional group present in the natural fiber to ensure the existence of the chemical components. This test is conducted in a FTIR spectroscopy. For the test, 2.5 mg quantum of the fiber sample is mixed with potassium bromide with the ratio of 1:100 ratio and formed as pellets. The test samples will be subjected to infrared radiation and the frequency responses are noted down. The resultant plot obtained out of the analysis has been plotted between wave number and normalized transmittance, which carries a frequency wave with critical peaks. The critical peaks ensures the existence of O-H group in turn cellulose contents, C-H group in turn hemicellulose content, C=C group in turn wax content and C-OH group in turn lignin content [6, 23, 24].

2.5. X-Ray Diffraction (XRD) Analysis

XRD analysis is performed to predict the crystallinity index value of the fiber. Crystalline index represents the degree of crystalline nature of the fiber. Crystallinity index is calculated by the following formula [1, 2, 6]:

$$\text{Crystallinity Index, CI} = \left(1 - \frac{\text{Min. peak intensity representing the amorphous region}}{\text{Max. peak intensity representing the crystalline region}} \right) \times 100\%$$

The crystalline size is predicted with the help of Scherer's formula:

$$\text{Crystallite Size, CS} = \frac{0.89 \times \text{Amplitude of the radiation}}{\beta \cos \theta}$$

Where, 0.89 – Scherer's constant,

β – full width of the peak at half – maximum, and

2θ – Diffraction angle.

The crystallinity index values reported for different natural fibers are provided in table 4.

Table 4. Crystallinity Index values for few natural fibers [2, 4, 6, 7]

S.No.	Name of the fiber	Crystallinity Index (%)
1.	<i>Dichrostachys Cinerea</i>	57.82
2.	<i>Grewia Tilifolia</i>	41.7
3.	<i>Prosopis Juliflora</i>	46
4.	<i>Lygeum Spartum</i>	46.19
5.	Date Palm	38.5
6.	Coconut	41.9
7.	<i>Wrightia Tinctoria</i>	40.6
8.	<i>Sansevieria Ehrenbergii</i>	52.27
9.	<i>Sansevieria Cylindrica</i>	60
10.	<i>Altaea Officinalis</i>	68
11.	<i>Sida Rhombifolia</i>	56.6

12.	Ferula	48
13.	Acacia Leucophloea	51
14.	Heteropogan Contortus	54.1
15.	Cyperus Pangorei	41
16.	Acacia Arabica	51.72
17.	Acacia Planiforns	65.38
18.	Flax	70
19.	Ramie	58
20.	Sisal	75
21.	Coconut	68
22.	Hemp	87.9
23.	Jute	58.9
24.	Pineapple	58.6

2.6. Morphology Analysis

Morphology study is done to know the nature of the surface of the fiber, which is performed with Scanning Electron Microscopy. From the SEM results, it can be understood the distribution of various constituents present over the fiber surface. The presence of hemicellulose contents can be viewed as bright white layers. The existence of lignin content can be noted as nail like parts. The wax and impurity items will be noted by the small discontinuous parts. By observing the nature of the surface, i.e, either rough or smooth, one can come to know the conclusion about the bonding nature of the fiber under analysis with the matrix medium. Chemical treatments will help to make the fiber surface as rough in turn to make the fiber with more bonding ability [4, 9, 25].

2.7. EDX Analysis

The amount of elements present on the fiber's surface (such as Carbon, Oxygen, Nitrogen, etc.,) is tested using Energy Dispersive X-Ray Spectroscopy [2, 5, 6].

2.8. Mechanical Strength Test

In order to find out the tensile strength of the natural fibers, INSTRON 5500 type tensile testing machine is normally used [3, 4 7]. The test is usually conducted at room temperature condition. The tensile strength vales reported for various natural fibers are listed in table 5.

Table 5. Tensile strength values of few natural fibers [2, 6, 8, 9]

S.No.	Name of the fiber	Tensile strength (MPa)
1.	Dichrostachys Cinerea	873 ± 14
2.	Lygeum Spartum	280
3.	Sansevieria	584.64 – 724.12
4.	Banana	529 – 759
5.	Jowar	302
6.	Sisal	274 – 526
7.	Alfa	134 – 220
8.	Kenaf Bast	427 – 519
9.	Petiole Bark	185.52
10.	Spatha	75.66

11.	Arundo Donax	248
12.	Grewia Tilifolia	75.3
13.	Agave	63 – 211
14.	Elephant Grass	292.3
15.	Empty Fruit Bunche	49
16.	Date palm	170 – 275
17.	Sida Rhombifolia stem	673 ± 14
18.	Heteropogan Contortus	476 ± 11.6
19.	Jute	400 – 773
20.	Flax	500 – 1500
21.	Cotton	200 – 400
22.	Coir	95 – 174
23.	Ramie	400 – 938
24.	Hemp	690
25.	Pineapple	362 – 748
26.	Kenaf	930
27.	Sisal	511 – 635
28.	Banana	700 – 800

3. Conclusion

The importance of characterization of natural fibers and the treatment of fibers with suitable chemicals for improving the strength are clearly explained in this work.

- Various tests to be conducted in order to characterize a new natural fiber are detailed with the processing procedure.
- It is also explained that how to interpret the results obtained from each test.
- The mass density, chemical composition, thermal stability, crystallinity index values and tensile strength results of distinct natural fibers are consolidated and presented in tables.
- This work may be an initiative for many future researchers those who are going to work with natural fibers and fiber reinforced polymer composite structures.

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