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Extraction and characterization of various unconventional natural fibres

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In this study, various unconventional fibres have been successfully extracted from crop residue *of Canna indica, Areca catechu* sheath*, Caryota urens* spadix petiole*,* and *Abelmoschus esculentus*, and their physical, chemical, thermal, and mechanical properties are studied. High cellulose contents of the fibres (20.5-67 wt%) provide better tensile strength (525-563MPa) and ensure better bonding with the matrix. Moreover, the low density of the fibre (1.37-1.44 gcm^{-3}) makes it an alternative to hazardous synthetic fibres. The lower crystal size (0.5-12.9nm) structure tends to absorb more water than the higher crystal size structure. The thermogravimetric analysis confirms its stability up to 150-300°C, which is higher than the polymerization temperature. These characteristics show that the fibre may be effortlessly converted into nonwoven fabrics.

Keywords: *Abelmoschus esculentus*, *Areca catechu* sheath*,* Biodegradable textile**,** *Canna indica, Caryota urens* spadix*, Curcuma longa* petiole*,* Eco-friendly textile, Lignocellulosic, Natural fibres, Thermal analysis

1 Introduction

In textiles, natural fibres are mostly used in different areas for various applications, namely wearable textiles, household textiles, and industrial textiles based on the advantages obtained from the particular type of fibres. Large numbers of unconventional natural textile fibres are available, but they are not familiar in the field of textiles. Some of the unconventional fibres like milkweed, pineapple, banana, furcraea, and spider silk, are used in different applications in the field of textiles because of their unique properties. These materials have the advantages of being biodegradable and eco-friendly[1,2]. Natural fibres provide several benefits, such as low price, "green" accessibility, lower densities, recyclable, biodegradable, moderate mechanical properties, etc. Application of natural fibres includes filler or reinforcement materials, insulation or structural parts, a disposable or durable product, like yarns and textiles, ropes, twines, nets, nonwoven materials, tissues, paper and board products, packaging, building and construction materials, fibre boards, geotextiles, composites, and automotive components. In the agricultural sector, there is an increased production of food grains and different agricultural produce leads to a corresponding increase in crop residue. It is predicted that more than 500 million tonnes of crop residues are generated in India per annum. Crop residues embody all agricultural wastes, like straw, stem, stalk, leaves, husk, shell, peel, lint, seed/stones, pulp, stubble, etc. The natural fibres from agricultural residues were extracted by using different methods. Following this, several extraction approaches were used, including the mechanical, chemical, retting, and boiling extraction procedures[3]. *Canna indica*, a plant belonging to the Cannaceae family, is commonly used as a medicinal herb for its antipyretic properties and for relieving gastrointestinal disorders. Additionally, its rhizomes are utilized for medicinal purposes[4]. *Areca catechu* L., on the other hand, belongs to the Arecaceae family. The leaf sheath of this plant completely surrounds the stem, providing a protective covering for the developing inflorescence[5]. The constituents of the leaf sheaths are polyose 43%, crude fibre 33%, and ash 5%. Presently, the Areca sheath is employed for manufacturing eco-friendly plates/cups, fuel, and compost[6]. The *Caryota urens* plant belongs to the family Arecaceae, native to tropical and semi-tropic regions and is generally cultivated as an ornamental. Flowering begins at the highest of the trunk and often continues downward for many years.

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Individual male flowers stay open for 16-20 days, whereas one inflorescence has a flower gap for regarding 6 weeks[7].

Curcuma longa L. is a perennial herb and member of the Zingiberaceae family. It is cultivated mostly in India and China. The leafed plant with terribly large, petiole plant attached to a stem enclosed in a sheath petiole. *Abelmoschus esculentus* is a significant vegetable crop within the Malvaceae family. Its mature fruit and stems contain crude fibre that is employed in the paper industry. A fibre obtained from the stem is used as a substitute for jute. It is also used in making papers and textiles. The leaves are removed and also the stems are water retted till the fibres are stripped off. Vignesh *et al.*[8] reported the extraction of new cellulosic stem fibres from the Indian mallow plant and its characterization has been deeply investigated. To replace synthetic fibres and reinforcing polymer matrices, natural fibres are excellent and eco-friendly alternatives. Indran *et al.*[9] discussed the characterization analysis of new natural cellulosic fibres from the *Cissus quadrangularis* root. Due to the high specific strength and modulus of fibre-reinforced polymer, composites are replacing many metallic structures. Arthanarieswaran *et al.*[10] characterised the cellulosic natural fibres from *Acacia leucophloea* bark. A novel reinforcement is to fabricate lightweight composite structures. The findings of the study show that the cellulose content and the density of the fibre are high. From TG and DTA analyses, it is observed that the fibre starts degrading at 220°C with a kinetic energy of 73.1 kJ/mol. So, it is clear that it can be used as a reinforced material. Santhanam *et al.*[11] reported the characterization of cellulosic natural fibre from the *Ipomoea staphyloma* plant. This natural fibre has superior properties and played a major role as a reinforced material in polymer composite[12].

In this study, various unconventional natural fibres have been extracted from *Caryota urens, Curcuma longa*, *Canna indica*, *Areca catechu* and *Abelmoschus esculentus*. The extracted fibres are then tested accordingly to identify whether these fibres are appropriate for manufacturing nonwoven fabrics by using a needle-punch method.

2 Materials and Methods

The natural fibres were extracted from different parts of *Canna indica*, *Areca catechu, Caryota urens, Curcuma longa* and *Abelmoschus esculentus*. These emerging fibres possess excellent cellulose content and desirable physical properties. Consequently, these fibres were chosen for the study. The selected unconventional waste materials were collected from Tirupur, Coimbatore, and Erode districts, Tamil Nadu, India. The NF1 refers to the fibres extracted from the stem part of the *Canna indica* plant. The *Areca catechu* sheath was used as a source for the extraction of fibre which refers to NF2. The NF2 fibres were extracted from the sheath manually using the needles[13]. The *Caryota urens* fibre (NF3) was extracted from the spadix part of the *Caryota urens* tree. The petiole part of the *Curcuma longa* plant was used as a raw material to extract fibre which refers to as NF4. The raw material sourced from the *Abelmoschus esculentus* stem was used for the extraction of fibre (NF5).

2.1 Selected Agro Wastes

2.1.1 Collection of Agro Wastes

After harvesting, unconventional plant wastes are generally used for ornamental purposes in functions. After that, it becomes biomass residue, which can be used as the source material for making natural fibres (Fig. 1).

2.2 Evaluation of Fibre Extraction Methods

The combination of mechanical and water retting processes was adopted to extract fibres from selected unconventional plant wastes.

2.2.1 Retting

Retting is a process in which fibres in the bark are loosened and separated from the woody stalk by the removal of various cementing tissue components, presumably pectins and gums**.** In this study, only mechanical retting and stagnant water retting were used and chemical retting was not done since it is very expensive and polluting to the environment**.** The colour of the raw fibres varies depending upon the retting method and conditions.'

2.2.2 Mechanical Retting - Combing from Fresh Plant

Fibres found in the inner side are then separated from the outer sheath manually by combing the stems with the serrated needle placed over the wooden board[14]. The separated fibres were then washed thoroughly using fresh water and then dried in the shade at room temperature (27ºC) for 2-4 h to ensure maximum moisture removal. Among the five unconventional fibres plant wastes, *Canna indica*, *Curcuma longa*, and *Abelmoschus esculentus* were adopted for mechanical retting.

2.2.3 Stagnant Water Retting

Both the *Areca catechu* and *Caryota urens* spadix were hammered and tied into bundles and soaked in the tank for degradation of the gum present in the leaves, as suggested by Vastrad *et al.*[15]. Weight was placed over the bundles to keep it submerged in the water. The bundles were left as such until complete microbial degradation of the fleshy pulp of the plants

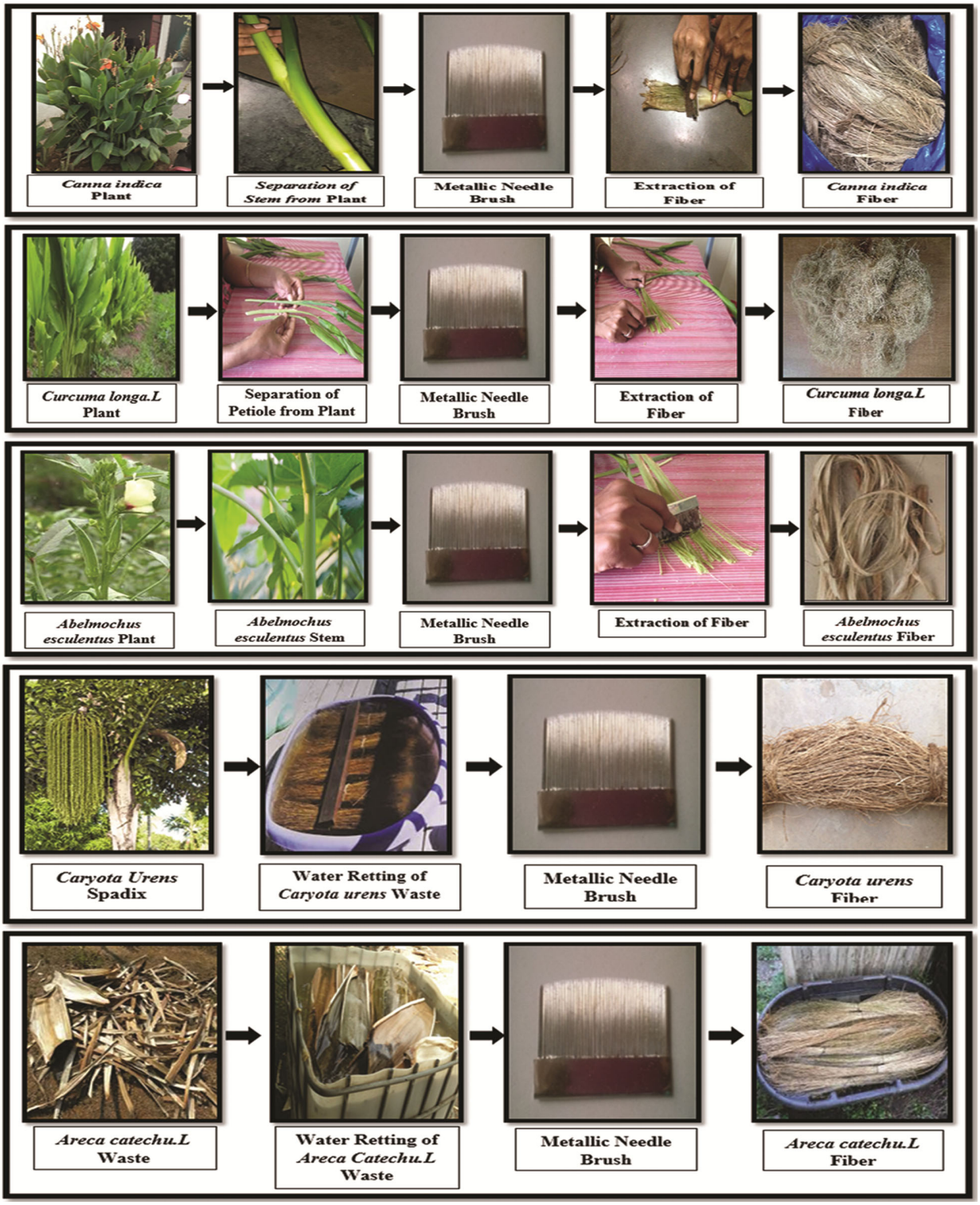

Fig. 1 — Extraction of unconventional fibres

occurs. Water was altered every week to reduce the bad odour coming due to microbial degradation. The loosened fibres were washed thoroughly with plain water and dried in sunlight for about 2 days. Finally, the fibres were conditioned using a hot air oven at 105ºC+ 2ºC until their constant weight was achieved and to remove excess moisture. Without any further treatment, the fibres thus obtained were left in their original state for additional studies. All five selected fibres were extracted by using mechanical and stagnant retting fibre extraction methods. The extraction methods were chosen based on the duration of fibre extraction, quantity, and quality. It is found that both the mechanical and stagnant retting methods are suitable for the extraction of fibres.

2.3 Fibre Characterization

2.3.1 SEM Study

The morphology of agro-waste fibres was examined using a scanning electron microscope (FEI Quanta 200). Sample preparation was done by coating the fibre with 8mm gold/palladium using sputter coater apparatus for 1.30 min to avoid electron beam charging effects during the examination. A high-energy beam of an electron with an accelerating voltage of 25kV was used to scan the samples with a vacuum level of 1.5×10^{-3} pa. Then, the sample surface was observed at different magnifications and the resulting image was captured.

2.3.2 X-Ray Diffraction Study

The amorphous and crystalline phase of the agro waste fibres were analyzed by X-ray diffraction (XRD) spectrometer (Shimadzu) with cu kα (λ=1.5406 ˚A) as a radiation source at a current of 30 mA with an accelerating voltage of 40 kV. All samples were scanned in the 2 range between 10˚C and 90˚C at a rate of 10˚C/ min in order to obtain an acceptable diffraction pattern. The crystallinity index (CI) was calculated using the following equation:

$$CI = \frac{Ic - Iam}{Ic} \times 100 \quad \ldots 1$$

where I_c is the maximum intensity of the peak which corresponds to the crystalline fraction; and

I_{am} represents the minimum intensity of the peak which corresponds to the amorphous fraction.

The crystallographic plane was measured using the following Scherer's equation:

$$CS = \frac{K\lambda}{\beta \cos\theta} \quad \ldots 2$$

where K is the Scherer constant (0.84); λ, the X-ray wavelength (0.154nm); β, the peaks full width at half maximum; and θ, the Bragg angle.

2.3.3 Thermo-gravimetric Analysis

The thermo-gravimetric analysis measures the amount and rate of change in the weight of a material as a function of temperature or time in a controlled atmosphere[16]. Thermal degradation of agro-waste fibres was examined using a thermal analyzer (model NETZSCH STA449F3). The instrument consists of a furnace in which the powdered fibre sample is placed on an alumina crucible supported by a precision balance. The measurement was performed in a high-purity nitrogen atmosphere at a flow rate of 20 mL/min and the weight loss was recorded in a thermogram at a heating rate of 30°C/min in the temperature range from 25°C to 1000°C. The measurement was made using an alumina crucible to maintain good contact between the sample and the thermocouple.

2.3.4 FTIR Analysis

The free functional groups present in the natural fibres and their unique chemical bonds were identified by TENXO 27 using an infrared spectrometer with a scanning rate of 32 scans min^{-1} in a PR Mode and a resolution of $2cm^{-1}$. The sample was analyzed in the wavelength range of 500–4000 cm^{-1} at 25°C.

2.3.5 Mechanical Properties

2.3.5.1 Breaking Strength and Elongation

The tensile strength of the unconventional natural fibres was measured by a single fibre tensile test method as per the specification of ASTM D 3822 standards with a crosshead speed maintained at 0.5 mm/min using a universal tensile testing machine with a load cell of 1kN capacity on randomly selected fibre samples (n=20). The entire test was performed with a relative humidity of 65% and a room temperature of 21°C.

2.3.6 Physical Properties

The fibres were equilibrated under standard conditions (20 C temperature and 65% RH) for 48h (ref. 16). An optical microscope was used to measure the diameter of 100 fibres in three random locations, and longitudinal direction images were taken from different fibre samples. Finally, the mean diameter of the fibres was calculated using the software "Image Pro Plus." The density of the fibres was assessed

using the pycnometer setup where a known toluene liquid density was used to identify fibre density.

3 Results and Discussion

3.1 Results of Extraction Methods

Details about the fibre extraction methods are given in Table 1.

Natural fibres were extracted using two different methods, namely mechanical retting and stagnant water retting. The experimental parameters are given in Table 1. In a typical extraction process, 10 kg of waste collected from different unconventional materials has been used. The extraction process is carried out at different time intervals; 12 h - 10 days for mechanical retting and 10-21 days for stagnant water retting. The processing (retting, extraction, and drying) time depends on the nature of the fibres and their chemical contents. The higher amount of fibres is obtained by the mechanical retting process in the case of NF1, NF4, and NF5 and the stagnant retting process for NF2 and NF3. In the mechanical retting process, a lower quantity of NF2 and NF3 is obtained due to the loss of fibre during washing. Therefore, the mechanical retting method is suitable for the extraction of NF1, NF4, and NF5 and the stagnant retting process is adopted for the extraction of NF2 and NF3.

3.2 Evaluation of Fibres

3.2.1 Chemical Compositions

The chemical composition of samples NF1, NF2, NF3, NF4, and NF5 are given in Table 2. The chemical composition of the fibre is strongly influenced by the region, maturity of the plant, and extraction conditions and methods. The chemical content, present in natural fibres, greatly impacts the fibre properties, such as mechanical, fire resistance, resilience, and biodegradability. In all the fibres, the chemical contents, such as cellulose, hemicellulose, lignin, ash, and other content, are present in the range of 20.05-67.0 wt%, 15.4-53.95 wt%, 6.8-22.3wt%, 2.44-5.2 wt%, 1.3-8.6 wt% respectively. Generally, the increase in the cellulose content in the fibre shows good performance in terms of tensile strength and tensile modulus. The higher hemi-cellulose content leads to the disintegration of cellulose micro-fibrils and decreases the fibre strength. On the other hand, the lower hemi-cellulose content tends to reduce the moisture absorption capacity, which intern increases the thermal stability of the fibre. Lignin content is responsible for the stiffness of the fibre. The increase in ash content in the fibres decreases their fire resistance characteristics. The moisture and other contents found in the natural fibre are favourable for making lightweight nonwoven composite materials.

3.3 Mechanical Properties

3.3.1 Single Fibre Strength

Table 3 shows the mechanical properties of samples NF1, NF2, NF3, NF4, and NF5.

Table 1 — Evaluation of natural fibre extraction methods

Extraction method	Plant name	Quantity of waste, kg	Duration	Quantity of fibres, g
Sample NF1	*Canna indica*			
Mechanical retting		10	12h	2750
Stagnant water retting		10	10days	2300
Sample NF2	*Areca catechu*			
Mechanical retting		10	10days	1500
Stagnant water retting		10	21days	2000
Sample NF3	*Caryota urens*			
Mechanical retting		10	12days	2000
Stagnant water retting		10	21days	3200
Sample NF4	*Curcuma longa*			
Mechanical retting		10	12h	2500
Stagnant water retting		10	10days	2250
Sample NF5	*Abelmoschus esculentus*			
Mechanical retting		10	12h	2100
Stagnant water retting		10	10days	1750

Table 2 — Chemical composition of different natural fibres

Sample	Cellulose content wt%	Hemi-cellulose content wt%	Lignin content wt%	Ash content wt%	Others content wt%
NF1	51.2	23.6	18.7	5.2	1.3
NF2	41.8	31.4	22.3	3.1	1.4
NF3	42.1	30.2	21.1	4.5	2.1
NF4	20.05	53.95	6.8	4.6	14.6
NF5	67	15.4	7.1	2.44	8.6

Table 3 — Mechanical properties of natural fibres

Sample	Fibre strength N	Fibre elongation %	Micro-fibril angle deg
NF1	4.02	2.7	1.02
NF2	3.87	1.8	1.01
NF3	4.37	3.8	1.03
NF4	2.59	1.6	1.01
NF5	2.35	0.62	1.00

A graph, plotted as load vs. strain, for the tested sample fibres also shows the mechanical properties of the natural fibres. Meanwhile, the study on the diameter of the natural fibre samples using an optical microscope with an image analyzer shows a diameter in the range of 525-563 µm. The values of tensile properties such as strength and elongation-at-break of the natural fibres have been calculated as 2.35-4.37 N and 0.62-3.8 % respectively. These fibres exhibit relatively good strength than those of other bast fibres, such as flax (2.7–3.2%), hemp (1.6%), jute (1.5–1.8%), and kenaf (1.6%)[17]. The tensile strength of the natural fibres is mainly influenced by their chemical composition, especially in the presence of cellulose. The higher content of the cellulose and low micro-fibril angle cause an increase in the tensile strength of the fibre. The micro-fibril angle (ε) of the natural fibres is calculated using the following equation:

$$\varepsilon = 1n\left(1 + \frac{\Delta L}{L_0}\right) = 1n(\cos \propto)$$

where ε is the microfibril angle (deg); L_0 the gauge length (mm); and ΔL the elongation at break (mm). The calculated micro-fibril angle of the natural fibres is found in the range of 1-1.03°. However, the micro-fibril angles of the other bast fibres, such as jute, flax, hemp, and banana are 8.1°, 5°, 6.2°, and 11–12° respectively.

3.3.2 Physical Properties

Table 4 shows the physical properties of NF1, NF2, NF3, NF4, and NF5 samples.

The length and diameter of the fibres vary from 30.26 cm to 46.8 cm and from 525 µm to 563 µm respectively. However, there are no substantial changes in the density and moisture content of the fibres. All five fibre samples are cellulosic in nature and have a slight variation in their physical properties. But there is much difference in the fibre strength.

3.4 Characterization Studies

3.4.1 SEM Analysis

The scanning electron microscopic images of NF1, NF2, NF3, NF4, and NF5 samples are shown in Fig. 2. The surface morphology of natural fibres shows the cellular structure and these cells together form fibrils with tissues, connected with each other at several locations [Figs 2 (a), (c), (e), and (g)]. The natural fibres show clean and smooth surfaces and a thick layer of uniformly deposited over the entire length of the fibre that is composed of hemicelluloses and lignin [Figs 2 (a) and (i)]. It consists of several elementary fibres, such as fibrils or fibre cells joined together in the direction of their length by pectin and other non-cellulosic compounds. The traces of waxy materials and a few damaged surfaces are observed in

Table 4 — Physical properties of natural fibres

Samples	Length, cm	Diameter, µm	Fineness, tex	Density, g/cc	Moisture regain %	Moisture content %
NF1	32.4	553.92	3.35	1.37	4.16	4
NF2	30.26	525.30	1.7	1.39	3.09	3
NF3	35.84	536.76	0.75	1.43	5.04	4.8
NF4	46.8	532.64	5.7	1.43	5.5	5
NF5	42.05	563.36	6.1	1.44	4.75	4.1

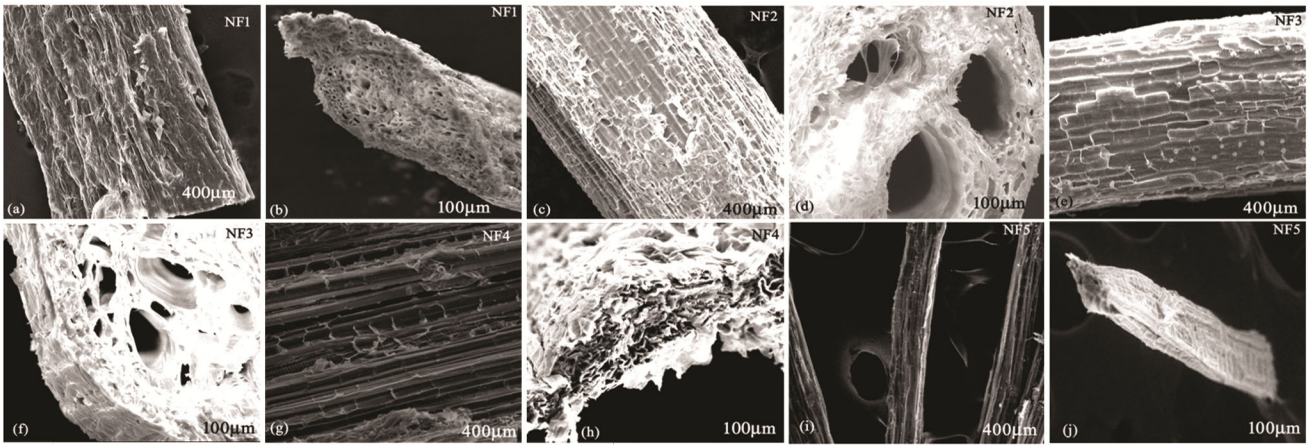

Fig. 2 — Surface morphology SEM image (a, c, e, g, i), and cross-sectional morphology (b, d, f, h, j) of various natural fibres

the fibre due to the manual extraction process. The cross-sectional images [Figs 2 (b), (d), (f), (h) and (j)] show a roughly cylindrical structure and also indicate that the fibres have a multicellular structure. The cells are roughly circular in nature but do not have uniform dimensions. Fibres have small and large voids which can easily hold thermal and sound frequencies.

3.4.2 Evaluation of Functional Groups

Infrared FTIR spectra of NF1, NF2, NF3, NF4, and NF5 samples are shown in Fig. 3.

All the fibres show similar peaks. The broad peak at 3360 cm^{-1} corresponds to the asymmetric vibration of OH groups present in the cellulose. The two bands at around 2840-2930 cm^{-1} are attributed to the asymmetric stretching of the C-H group present in cellulose and hemicellulose. A small band located at around 1730 cm^{-1} is assigned to the C = O stretching of the acetyl and uronic ester groups of hemicellulose or to the ester linkage of carboxylic group in the ferulic and p-coumaric acids of lignin and/or hemicellulose. The smaller peaks may be indicative of a lower percentage of hemicellulose. The absorbance peak at 1640 cm^{-1} corresponds to bending vibrations of the OH group of cellulose and adsorbed water. The intense peak at around 1030 cm^{-1} is due to the C-O-C and/or the C-O stretching and C-H rocking vibrations of the pyranose ring skeletal vibration. The peak at around 900 cm^{-1} is assigned to ß-glycosidic bonds between glucose units of amorphous cellulose. Finally, the peak at around 590 indicates the C-OH out of plane bending, as also reported by many researchers[18-21]. However, depending on the natural fibre, the peaks are slightly shifted, but not substantially.

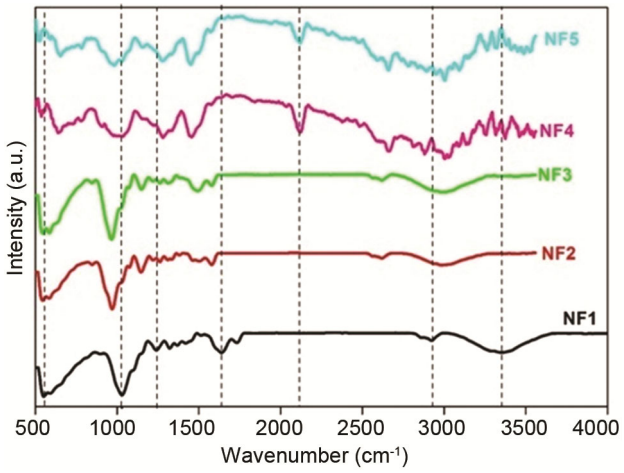

Fig. 3 — FTIR analysis of natural fibres

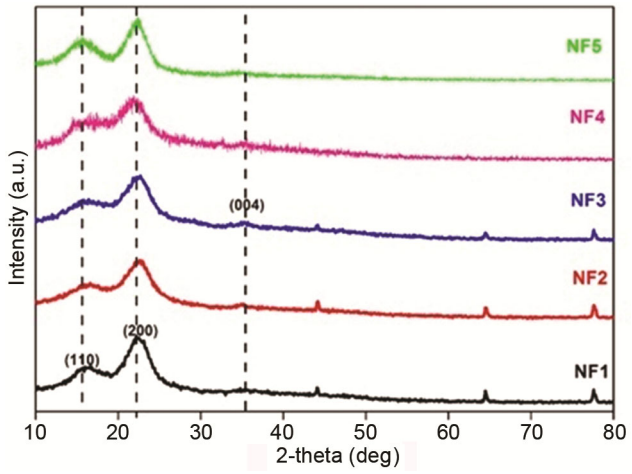

Fig. 4 — XRD analysis of natural fibres

3.4.3 X-Ray Diffraction Analysis

The crystalline index percentage and crystal size of the NF1, NF2, NF3, NF4, and NF5 samples can be determined by X-ray diffraction**.** The results of X-ray diffraction are given in Table 5 and Fig. 4.

Figure 4 shows the XRD patterns obtained for different natural fibres. The patterns of fibres are typically a semi-crystalline material, such as lingo-cellulosic material that yields an amorphous broad hump and a crystalline peak, as described by Guimarães *et al.*[22]. The peak observed at 15° corresponds to the crystallographic (110) planes. The peak at 22° is due to the crystallographic planes (200) and another small peak at 35° is associated with crystallographic planes (004) or (023), which are characteristic of the crystallographic cellulose. The cellulose crystalline properties of crystallographic planes at (110) and (200) of natural fibres are also calculated (Table 5).

Table 5 — Results of X-ray diffraction of natural fibres

Sample	Lattice plane	Degree of crystallinity, %
NF1	(110) (200) (004)	90.98
NF2	(200) (110) (023)	93.34
NF3	(110) (200) (004)	90.13
NF4	(200) (110) (023)	28
NF5	(200) (110) (023)	62

The crystalline size of natural fibres is calculated using the following Debye-Scherrer equation:

$$CS = \frac{K\lambda}{\beta\cos\theta} \quad \ldots 4$$

where k=0.94 is the Scherer's Constant; β, the full-width half maximum of peaks; λ, the wavelength of the radiation (0.154) nm; and θ, the Bragg angle.

The crystallite size of the natural fibres NF1, NF2, NF3, NF4, and NF5 is calculated as 0.5-12.9 nm respectively using the (200) plane, and the lower crystal size structure tends to absorb more water than the higher crystal size structure. Crystallinity index (CI) is performed using the following equation:

$$CI = \frac{Ic-Iam}{Ic} x100 \quad \ldots 5$$

where I_c represents the maximum intensity of the crystalline fraction; and I_{am} represents the minimum intensity of the amorphous fraction. The calculation yields a CI range of 28-90.98%.

3.4.4 Thermo-gravimetric Analysis

The thermal stability of natural fibres has been investigated by the thermo-gravimetric method, and results are shown in Fig. 5. TGA supports the results obtained in the chemical composition analysis since hemicellulose, cellulose, and lignin usually decompose themselves at different temperatures due to its chemical structures. The analyzed fibres present four distinct stages better observed in the DTG curve. The first stage (until 130°C) is due to the vaporization of absorbed water, the release of moisture and low molecular mass compounds such as extractives. The second stage is the decomposition of hemicellulose, pectin and parts of lignin (between 150°C and 320°C). The third stage, which occurs at around 330°C is due to the weight loss promoted by cellulose decomposition, through reactions of depolymerization, dehydration, and decomposition of glycoside units. The last stage at around 400°C is related to the decomposition and dehydration of lignin. The higher weight loss occurring in the third stage of all these fibres is related to the degradation of cellulose because it is the larger component of the fibre structure. The amount of residue remaining at 1000°C is due to the presence of carbonaceous materials. Compared to all other fibres selected for this study, the NF5 fibre is rich in cellulose content. Hence, it's fully degraded at 600°C.

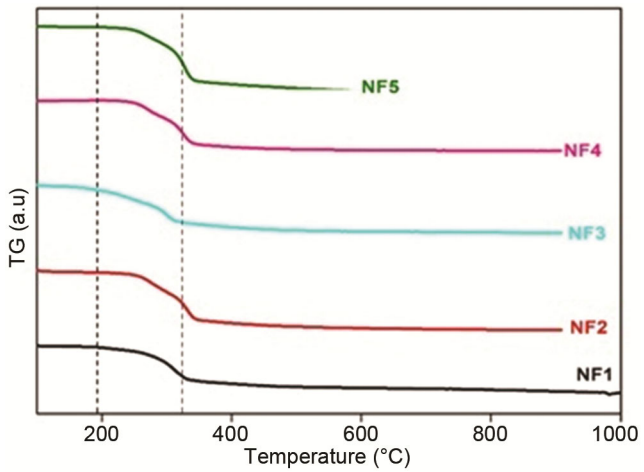

Fig. 5 — TGA curve of natural fibres

4 Conclusion

Based on the fibres characterization evaluated, it can be concluded that the fibre may be easily converted into nonwoven rather than spun into fibres, because of the brittle nature of natural fibres. The density and tensile strength of the natural fibres are found in the range of 1.37-1.44 g/cc and 2.35-4.37 N respectively. Fourier transform infrared spectroscopy, X-ray diffraction, and thermogravimetric analysis confirmed that the unconventional fibres are rich in cellulose content (20.05-67 %), and thermally stable (up to 330°C) with a crystallinity index of 28-93.34%. Hence, all the unconventional waste fibres generated are made nonwoven by using needle punching techniques. The nonwoven thus prepared may be used in final end products, such as acoustic, thermal insulation and sound absorption.

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