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Short Communication

Thermal properties of natural fibres extracted from Palmyra sprouts

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Effect of fibre extraction from Palmyra sprouts by manual method and water retting process has been studied. Mechanical and chemical properties of the fibres have been studied. Cellulose, lignin, ash and wax contents present in both manual and water retted palmyra sprout fibre are found to be 61.72% & 55.63%, 23.25 % & 30.66%, 5.15 % & 3.61 % and 0.66% & 0.84% respectively. The low composition of ash provides light-weight and stiffness to the fibres. Thermogravimetric analysis shows that the residual mass of water retted fibre shows is 26.77% at 498.2ºC and that of manual retted fibre is 21.31% at 498.2ºC. The average single fibre strength values for the water and manual retted samples are 2.34 N and 4 N respectively, with a standard deviation of 0.642 N and 1.81 N respectively. In this study, thermal properties are also analyzed.

Keywords: Chemical properties, Mechanical properties, Palmyra sprout fibre, Thermal properties

Recently, the consumers have been very conscious of their environment to ensure safety to their lives. Thus, ecofriendly products are gaining importance in the market[1]. Natural fibres are obtained from various parts of plants, such as stems, leaves, roots, fruits and seeds. Efforts have been made to use these fibres as reinforcements in polymer composites from ancient days[2]. Natural fibres, being biodegradable, low cost, easily accessible, low density and with good acoustic property, attract the manufacturers from a diversified field to utilize the same as a centric approach[3].

Palmyra palm is referred as a 'miracle' plant due to the wide utilization of most of its parts, such as the trunk, foliage, husk, nut, and flesh[4]. The trunk can be used for furniture and handicrafts. Dried leaves and the flexible sticks in the frond are woven to make some crafts[5,6] *Borassus flabellifer* L., belonging to family Arecaceae commonly known as Palmyra palm or Asian toddy palm, is a native of tropical Africa but cultivated and naturalized throughout India[7]. The Palmyra palm, a multipurpose tree of great utility, occurs extensively in Tamilnadu state, India[8]. The *Borassus flabellifer* is a tall and erect palm, with large, fan-shaped leaves, which are quite unlike the pinnate leaves of other palms. Borassus is derived from a Greek word describing the leathery covering of the fruit and flabellifer means "fan-bearer"[9].

In the present scenario, fabrics and textile products need to be sustainable, and their eco-friendliness is highly essential. It is vitally proved that Palmyra sprouts possess a lot of meritorious physical and chemical properties, which promote its usage for textile applications. Hence, in this research work, an attempt has been made to develop textile product using natural eco friendly fibres. Natural fibres are in great demand today for textile product manufacturing as they have eco-friendly characteristics like bio-degradability and reusability. India offers diverse agro-climatic conditions, providing fibres of various types. Among these, fibres extracted from Palmyra sprouts has been considered as a reliable resource for the future.

Experimental

Materials

Palmyra sprouts were purchased from local merchant farm in and around Kanyakumari and Tuticorin District, Tamil Nadu, India. The collected Palmyra sprouts were authenticated by Botanical Survey of India, Coimbatore, Tamilnadu.

Fibre Extraction

The fibres were extracted from Palmyra sprouts using both manual and water retting methods. The outer surface skin of Palmyra sprouts was removed in the manual method. The sprouts were cleaned initially with water and with the help of knife and a comber. The long fibres were uniformly extracted, and care was taken to avoid fibre damages. This method is economical, though time consuming.

In water retting process, the outer surface skin of sprouts has been removed, followed by cleaning of the foreign matters, dust. dirt, and has been soaked for seven days. Afterwards, they were being removed,

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washed with water and dried under sun light, which resulted in uniform long fibres[7-9].

Fibres from the two processes, viz manual and water retting , evaluated using various analytical process, counting from field-emission scanning electron microscopy (FESEM),differential scanning calorimetry (DSC),thermo-gravimetric analysis (TGA), Wide-angle X-ray diffraction analysis (WAXRD), contact angle and single fibre tensile strength.

Characterization of Palmyra Sprout Fibre

Chemical Analysis

The characterizations of fibres were subjected to chemical composition test at South Indian Textile Research Association (STIRA), Coimbatore, India, laboratory, using the standard test procedures.

Mechanical Properties of Fibre

Field Emission Scanning Electron Microscopic Study

The surface morphology of the fibres was analyzed using Field Emission Scanning Electron Microscope (FESEM). The morphology of the Palmyra sprout fibres was characterized using Field Emission Scanning Electron Microscopy (FESEM) Carl Zeiss microscopy tester.

The fibres were dried at 27 ± 3˚ C, followed by coating with a gold layer for analysis. The FESEM studies were conducted by scanning the samples with a high-energy electron beam at an accelerating voltage of 0.2-30 kV in a vacuum chamber, and the image was captured at different magnifications. It has a much brighter electron source and smaller beam size than a typical SEM, increasing the useful magnification of observation and imaging up to × 500000. The main advantage of using FESEM is the high-resolution imaging, which could be performed with very low accelerating voltages. This enhances the observation of fine surface features, electron beam sensitive materials, and non-conductive materials.

Wide Angle X–Ray Diffraction Analysis

The crystallinity of Palmyra sprout fibre was elucidated using X-ray diffraction method with a powdered sample. An XPERT-3 Diffractometer model Goniometer PW3050/60 was used with Cu as Anode material. X-ray diffraction was designed for obtaining the ultimate quality diffraction data, combined with ease of use and flexibility to switch to different applications. The diffraction intensity of CuKα (wavelength of 0.1542nm) with 2θ range from 10˚ to 90˚ at 45kV and 30 mA powdered sample was analyzed using reflection/Transmission spinner mode[12]. In order to determine Crystalline Index (CI), Segal empirical method is used as follows:

$$\text{C. I \%} = \frac{I_{200} - I_{am}}{I_{200}}$$

where I_{200} is the maximum Intensity diffraction and I_{am}, the intensity diffraction of the amorphous

Thermo Gravimetric Analysis

The thermal changes and the thermal stability of the powdered fibre samples weighting 5 mg, were evaluated using thermo gravimetric analysis, on a NETZCH Germany & STA49F3 Jupiter device in following nitrogen (N2).

The amount and rate of change in the weight of a material as a function of temperature or time in a controlled atmosphere was measured by thermo gravimetric analysis[10]. Measurements were done primarily to determine the composition of materials and to predict their thermal stability at temperatures up to 1200 °C. The technique can characterize materials that exhibit weight loss (or) gain due to decomposition, oxidation (or) dehydration.

Differential Scanning Colorimetry

Five grams (5g) fibres from the two retting methods were analyzed for thermal stability and thermal changes using NETZCH Germany & STA 449 F3 Jupiter device in flowing nitrogen (N_2). Differential scanning colorimetry (DSC) is a technique in which the difference in energy temperature between a sample and a reference is monitored as a temperature function, when both sample and reference are submitted to the same temperature – time programme.

DSC is also a useful method in finding out calorimetric purity and second order transitions. In this technique, aluminum pan is used to accommodate the weighted sample powder. The medium used is air and is supplied at the rate of 100 mL/min. The sample is heated at the rate of 5 ˚C/min up to 600 ºC. Here, change in energy; say in mW, is recorded as a function of temperature, which provide either exothermic or endothermic peaks at the time of dehydration or decomposition reaction[11].

Fourier Transforms Infrared Spectroscopy

The retted fibres were analyzed using Fourier Transform Infrared Spectroscopy. The model of Fourier Transform Infrared spectrometer used was SHIMADZU – FTIR – 8400S. The Fourier Transform Infrared spectrum was obtained at a spectral range of

400 – 4000 cm^{-1} and has a resolution of 0.9 – 1 cm. The Fourier Transform Infrared spectrometer is mostly used for identifying the active chemical constituent present in a compound[12].

Contact Angle Test

A simple way of measuring the interactions between solid and liquid molecules is through the measurement of contact angle. The wettability of the system is characterized by the contact angle. Wetting phenomena are measured by contact angle formed between the liquid drop and solid surface. Contact angle goniometer is an apparatus, which is used for measuring the contact angle between the liquid and the substrate.

Single Fibre Tensile Strength and Elongation

The tensile test was performed on single fibres of Palmyra sprouts in order to find its axial tensile strength and elongation of the fibre. Zwick Asia Private Ltd, Germany (Z010) model was used for the measurement.

Results and Discussion

Chemical Composition of Retted Palmyra Sprout fibre

The chemical composition of manual and water retted Palmyra sprouts fibres has been determined through chemical analysis. It is observed that the percentage of cellulose present in the manual and water retted Palmyra sprouts fibres are 61.72% and 55.63% respectively. The composition of lignin content present in manual and water retted Palmyra sprout fibre is observed to be 23.25 % and 30.66% respectively.

The presence of cellulose and lignin content observed in the fibres provides the strength and stiffness to the fabric. The ash content in manual and water retted fibre is found to be 5.15 % and 3.61 % respectively, which signifies the presence of carbon in the fibre. The low composition of ash provides light-weight and stiffness to the fibre. Similarly, the wax content present in manual and water retted Palmyra sprout fibre is observed to be 0.66% and 0.84% respectively. A very little moisture composition is observed in manual 9.07% and water 9.05 % retted fiber, which can be able to provide excellent dimensional stability and fibre swelling. Fibre density is observed to be 1.4734 g/cc and 1.3638 g/cc respectively for manual and water retted Palmyra sprout fibres.

Field Emission Scanning Electron Microscopic Study

Field Emission Scanning Electron Microscope is used to study the surface morphological characteristics of Palmyra sprout fibres. The surface morphology of the fibre is a very important factor for determining the ability of fibre to act as a good reinforcement and to resist the fibre pull out[5]. The cross-section of both manual and water retted sprout fibres indicates that most of the fibre bundles are relatively intact, but easy to separate from the fibre core after water retting. The water retted fibre results not only in the separation of the fibre bundles from the core, but also the formation of the smaller bundles from larger ones. Hence, it clearly implies that the surface characteristics have been improved significantly in both water and manual retted Palmyra sprout fibres and the presence of longitudinal flutes and smaller pits observed on the surface level provides a smooth surface appearance. It is also observed that the Palmyra sprout consists of several elementary fibres connected in length direction by wax, lignin and other compounds surrounding the fibre surface to protect the cellulose permanently.

Wide Angle X-ray Diffraction Study

WAXRD analysis is used to determine the phase crystallinity of a single crystal (or) a grain of Palmyra sprout fibre obtained using manual and water retting. Fibre properties are influenced mainly by two important quality, namely crystallnity index and degree of fibre orientation[6].

In water retted Palmyra sprout fibre, various peaks have been identified with respect to hkl values at 15.27˚ - (100) , 17.21º- (100) , 21.70˚-(110) , 22.62˚-(110), 28.26˚-(111), 30.90˚-(200), 34.69˚-(210), 52.16˚-(310) and 54.84˚-(311) respectively. The maximum peaks are identified at 21.70˚ and 17.21˚ and the peak intensity of cellulose corresponds to 21.70˚, representing the maximum intensity diffraction and its relative intensity is calculated as 100 %. Similarly, the minimum intensity of amorphous corresponds to 17.21˚ and its relative intensity is calculated as 23.10˚. The observed diffraction peaks are determined using standard JCPDS (File No.09-0432).

The counter reading at peak intensity of 21.70˚ is said to represent the crystalline regions and the peak intensity at 17.21˚ corresponding to the amorphous regions is found to be present in cellulose. In manual retted Palmyra sprout fibre, various peaks have been identified with respect to hkl values at 15.36˚ - (200) , 17.13º- (210) , 17.97˚- (210) , 21.69˚-(220), 23.04˚-(221), 26.67˚-(222), 30.91˚-(400) and 34.81˚-(420). The maximum peaks are identified at 21.69 ˚ and

17.13˚ and the peak intensity of cellulose corresponding to 21.69˚ represents the maximum intensity diffraction and its relative intensity is calculated as 100%. Similarly, the minimum intensity of amorphous corresponds to 17.13˚, and its relative intensity is calculated as 24.17˚. The observed diffraction peaks are determined using standard JCPDS (File No.09-0432).

Thermo gravimetric Analysis

Thermogravimetric Analysis (TGA) measures the amount and rate of change in the weight of a material as a function of temperature or time in a controlled atmosphere. Measurements are done primarily to determine the composition of materials and to predict their thermal stability at temperatures to the maximum of 1200°C. This technique can characterize materials that exhibit weight loss or gain due to decomposition, oxidation, or dehydration.

TGA measures the mass of a sample as it is being heated at a constant temperature in a defined atmosphere. The mass of the sample steadily decreases and leaves the ashes finally. The burning process of a sample can easily be measured using TGA (Fig. 1).

Thermogram of the manual retted Palmyra sprout fibre shows a single-step degradation process. As a first step, at 30º-100 ºC, a significant weight loss is occurred due to moisture evaporation followed by another weight loss which occurred due to the dehydration of light materials like cellulose at 100º-250 ºC. The weight loss occurred at 250º- 350ºC is due to the decomposition of heavy material like lignin of the manual retted Palmyra sprout. The degradation process is started in the cellulose and lignin constituents after the removal of free water. An increase in ash content is noticed at a higher temperature of 350º-500ºC, leading to a residual mass of 21.31% at 498.2ºC respectively.

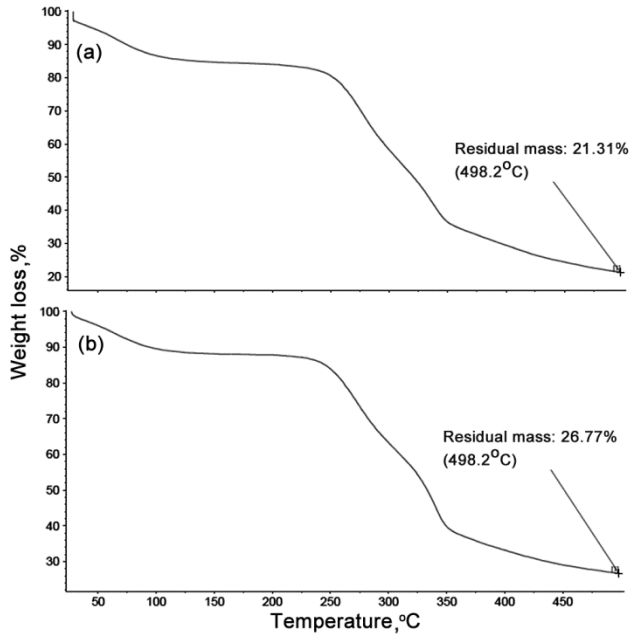

Fig. 1 — TGA analysis of (a) manual and (b) water retted palmyra sprout fibre

The thermogram of the manual retted Palmyra sprout fibre shows a single-step degradation process. As a first step, at 30 ˚C-100 ˚C, a significant weight loss is occurred due to moisture evaporation followed by another weight loss which occurred due to the dehydration of light materials like cellulose at 100º-250 ºC. The weight loss occurred at 250º-350 ºC is due to the decomposition of heavy material like lignin of the manual retted Palmyra sprout. The degradation process is started in the cellulose and lignin constituents after the removal of free water. An increase in ash content is noticed at a higher temperature 350-500 ºC, leading to a residual mass of 26.77% at 498.2 ºC respectively.

Differential Scanning Colorimetry Study

The differential scanning calorimetry (DSC) technique is used to compare the thermal strategies like melting, glass transition and crystallization of manual and water retted fibre. In this process, DSC curves of Palmyra sprout fibres are expressed in terms of heat flow per unit mass.

Water Retted Palmyra Sprout Fibre

DSC curves of water retted Palmyra sprout fibre as represented shows large endothermic peak at 69.2 ˚C, which characterizes that the absorbed moisture in the surface (or) within the fiber is vaporized. At the temperature range 250º-300˚C, a glass transition is formed. Next endothermic peak at 332.9 ºC for water retting fibre is observed due to the cellulose degradation, and the removal of lignin observed at 387˚- 415 ºC.

Manual Retted Palmyra Sprout Fibre

Likewise, the differential scanning calorimetry thermograph of manual retted Palmyra sprout fibre represents the large endothermic curve due to the removal of water molecules at 74.2 ºC. The cellulose degradation peak for manual retted fibre is reduced and converted in to slight exothermic curve at 175 ºC, because of the removal of binding components of fibre, like lignin, which shows the endothermic peak at 325 ˚C and a small shoulder at 250 ºC.

Fourier Transform Infrared Spectroscopic Study

Water Retted Palmyra Sprout Fibre

FTIR spectra of water retted Palmyra sprout fibre is represented in Fig. 2 (a). The absorption bands and the presence of peaks on the spectra of Palmyra sprout samples are listed. The difference in peak noticed at 3988.39-3374.03 cm^{-1} (O-H) stretching & peak observed at 2848.74 cm^{-1} (C-H) symmetrical stretching confirms the presence of protein substances in the fibre. Similarly, the difference in peak at 1732.52 cm^{-1} (C=O) stretching vibration and peak at 1652.03 cm^{-1}, represent (O-H) bending, confirms the presence of absorbed water. The difference in peaks at 1462.80 cm^{-1} (H-C-H) & (O-C-H) bending vibration

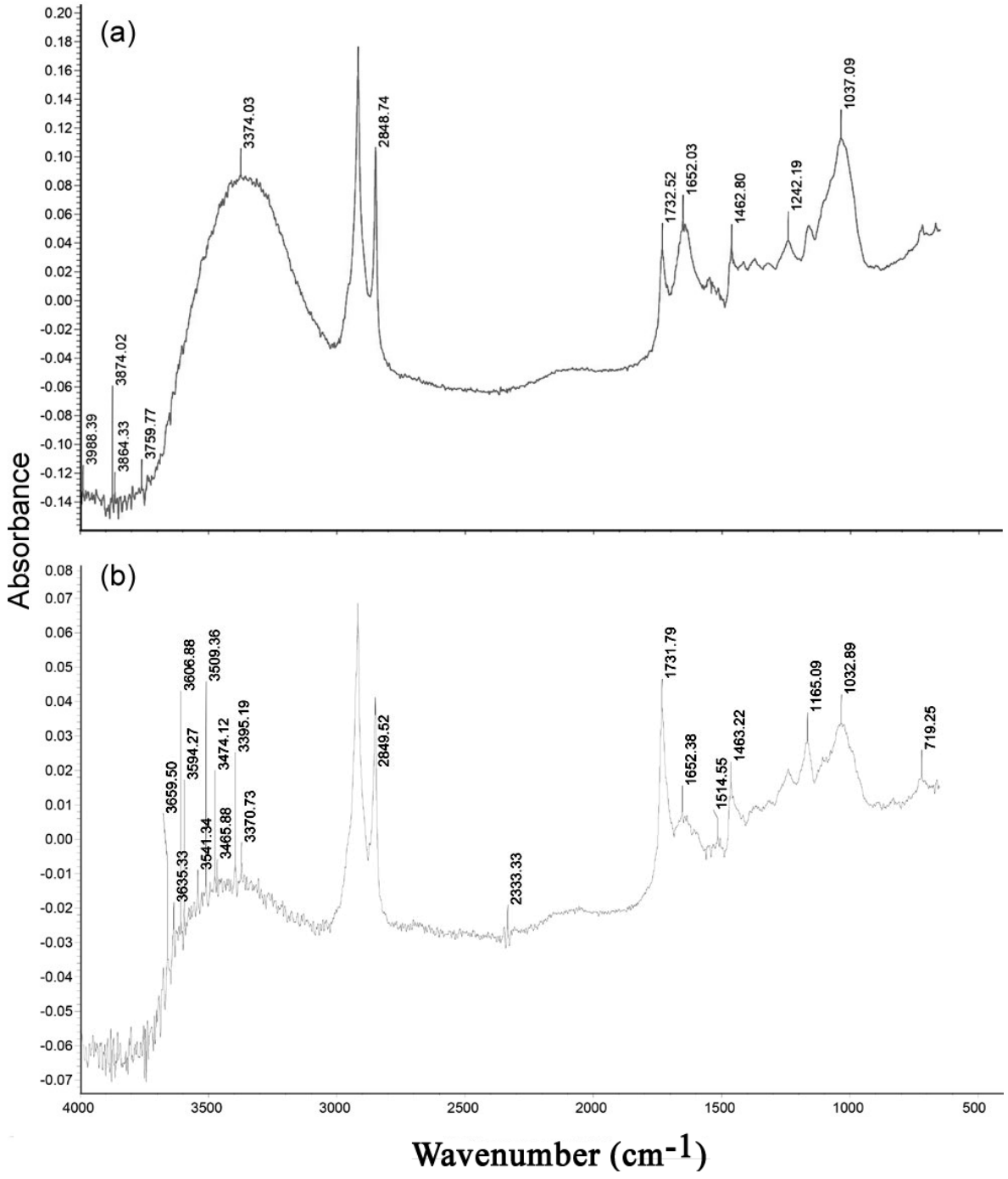

Fig. 2 — FTIR analysis of (a) water and (b) manual retted palmyra sprout fibre

and peaks at 1242.19-1037.09 cm^{-1} (C-O) stretching vibration confirm the presence of phenols, flavonoids content and polysaccharides

Manual Retted Palmyra Sprout Fibre

FTIR spectra of manual retted Palmyra sprout fiber is represented in Fig. 2(b). The absorption bands and the presence of peaks on the spectra of Palmyra sprout samples are listed**.** The difference in peak noticed at 3659.50- 3370.73 cm^{-1} (O-H) stretching, and peak observed at 2849.74 cm^{-1} (C-H) symmetrical stretching confirms the presence of methoxyl groups in the fibre. Similarly, the difference in peak at 2333.33 cm^{-1} (C-H) stretching vibration and peak at 1731.79-1652.38 cm^{-1} represents (C=O) stretching, and peak at 1514.55 cm^{-1} confirms the presence of aromatic chains in the polymer. The difference in peaks at 1463.22, 1165.09-1032.89 cm^{-1} (C-O) stretching and peaks at 719.25 cm^{-1} (C-C) stretching vibration confirms the presence of phenols, flavonoids content and polysaccharides.

Contact Angle Test

The wettability characteristics of the Palmyra sprout fibre is determined using contact angle measurement. It has been observed that the contact angle of Palmyra sprout water retted fibre is found to be 97.6º, whereas manual retted Palmyra sprout fibre is found to be 108.3º. It clearly implies that the surface-free energy of the material has been reduced in both manual and water retted fibre owing to a hydrophobic nature of surfaces in both manual and water retted fibre. But the water retted fibre shows a gradual change in the state of absorption from hydrophilic to hydrophobic surface, whereas manual retted fibre surfaces are strongly hydrophobic in nature due to higher contact angle (> 90º).

Single Fibre Tensile Strength

Besides the fibre length and fineness, the fibre strength is an important fibre property[11]. The experimental results show the tested single fibre strength data. The mean values for two or three replications for force and elongation for approximately 100 fibres tested from each sample in each replication. It shows the values for average single fibre tension at break, which is computed from the peak voltage (Vb) of the force transducer output. The test is performed at a relative humidity of 65% and temperature of 21˚C. The overall average of tensile strength for the water retted and manual retted Palmyra sprout fibre is 27.44 N and 38.76 respectively with young modulus ranging from 1970 MPa to 6330 MPa for the water retted fibre and from 3080-7940MPa for the manual retted fibre respectively. The percentage of elongation at break of water retted palmyra sprout fibre is 35% and 39.56% respectively. The average single fibre strength for the water and manual retted sample is 2.34 N and 4 N, with a standard deviation of 0.64 N and 1.81 N respectively.

Conclusion

The purpose of this study is to investigate the effect of fibre extraction from palmyra sprouts by manual method and water retting process. Mechanical and chemical properties of the fibres have been studied. Fibre shows the best mechanical properties in terms of strength and rigidity. Following inferences are drawn:

It is observed that the cellulose, lignin, ash and wax contents present in both manual and water retted Palmyra sprout fibre are found to be 61.72% and 55.63%, 23.25 % and 30.66%, 5.15 % and 3.61 %, 0.66% and 0.84% respectively. The low composition of ash provides light-weight and stiffness to the fibres. A very little moisture composition is observed in manual and water retted fibre, (9.07 % and 9.05% respectively). This provides excellent dimensional stability and fibre swelling. Fibre density is observed to be 1.4734 g/cc and 1.3638 g/cc respectively for both manual and water retted Palmyra sprout fibers.

The surface morphological study of both manual and water-retted Palmyra sprout fibres infer that most of the fibre bundles are relatively intact, but easy to separate from the fiber core after water retting. The water retted fibre not only results in the separation of the fibre bundles from the core but also avoids the formation of smaller bundles from larger ones. Hence, it clearly implies that the surface characteristics have been improved significantly in both water and manual retted Palmyra sprout fibres, and the presence of longitudinal flutes and smaller pits observed on the surface provide a smooth appearance. It is also observed that the Palmyra sprout consists of several elementary fibres connected to each other in length direction by wax, lignin and other compounds surrounding the fibre surface to protect the cellulose permanently.

In X-ray diffraction (WAXRD) studies, the maximum peaks for the water retted sample is identified at 21.70˚ and 17.21˚, which represent the maximum intensity diffraction of crystalline and amorphous regions respectively and its relative

intensity is calculated as 23.10˚. In manual retted Palmyra sprout, the maximum peaks are identified at 21.69˚ and 17.13˚, which represent the maximum intensity diffraction of crystalline and amorphous regions respectively and its relative intensity is calculated as 24.17˚. Similarly, anorthic and orthorhombic structures has been noted for water retted sample and Ortho-rhombic structure have been noted in manual retted fibre.

TGA studies show that both water and manual retted Palmyra sprout fibres indicate a significant weight loss at different temperature range (30ºC-100 ºC), which is due to moisture evaporation occurred at 100ºC -250ºC, This is due to the dehydration of light materials like cellulose. The weight loss occurred at 250ºC-350ºC is due to the decomposition of heavy material like lignin of the manual retted palmyra sprout. An increase in ash content is also being noticed at a higher temperature of 350 ºC-500 ºC. The residual mass of water retted fiber is 26.77% at 498.2ºC and the residual mass of manual retted fibre is 21.31% at 498.2 ºC.

From DSC studies, It has been inferred that a large endothermic curve occurred at 74.2 ºC due to the removal of water molecules and the cellulose degradation peak is reduced and converted to slight exothermic curve at 175ºC in manual retted fibre. Similarly, a large endothermic peak is occurred at 69.2 ˚C, due to the fact that absorbed moisture in the surface (or) within the fibre is vaporized for water retted sample. It is also inferred that the glass transition temperature of the fibre is noticed at 250º- 300 ˚C.

From FTIR studies, it is inferred that both water and manual retted Palmyra sprout fibres show the difference in peak at various wave numbers. The difference in peak at 3659.50- 3370.73 cm^{-1} (O-H) stretching and peak observed at 2849.74 cm^{-1} (C-H) symmetrical stretching confirms the presence of methoxyl groups in the fibre. Similarly, the difference in peak at 2333.33 cm^{-1} (C-H) stretching vibration and peak at 1731.79-1652.38 cm^{-1} represents (C=O) stretching, and peak at 1514.55 cm^{-1} confirms the presence of aromatic chains in the polymer. The difference in peaks at 1463.22, 1165.09-1032.89 cm^{-1} (C-O) stretching & peaks at 719.25 cm^{-1} (C-C) stretching vibration confirm the presence of phenols, flavonoids content and polysaccharides.

The contact angle of palmyra sprout water retted fibre is found to be 97.6º, whereas that of manual retted Palmyra sprout fibre is 108.3º. It clearly implies that the surface-free energy of the material has been reduced in both manual and water retted fibre owing to a hydrophobic nature of surfaces in both manual and water retted fibre. But the water retted fibre shows a gradual change in the state of absorption from hydrophilic to hydrophobic surface, whereas manual retted fibre surfaces are strongly hydrophobic in nature due to higher contact angle greater than 90º.

The overall mean of single fibre tensile strength for both water retted and manual retted Palmyra sprout fibre is 27.44 N and 38.76 N respectively with Youngs modulus ranging from 1970MPa to 6330 MPa for the water retted fibre and from 3080 MPa to 7940 Mpa for the manual retted fibre and the percentage of elongation at break of water retted Palmyra sprout fibre is 35% and 39.56% respectively. The average single fibre strength for the water and manual retted sample is 2.34 N and 4 N, with a standard deviation of 0.642 and 1.81 N respectively.

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